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OPTIMIZATION PROCESS FOR THE PRODUCTION AND CHARATERIZATION OF BIODIESEL FROM VEGETABLE OILS

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ABSTRACT

Energy plays a crucial role in the development of modern society. However, fossil fuels, which remain the world's primary energy source, are limited and their use significantly contributes to climate change. In this context, renewable energies, which are sustainable and environmentally friendly, have attracted growing attention. Biodiesel, produced from vegetable oils or animal fats, represents a promising alternative to fossil diesel due to its similar physicochemical properties, allowing its use in existing engines without modification.

In this study, two types of biodiesel were synthesized from different vegetable oils: new sunflower oil and waste cooking oil. The produced biodiesels were characterized by determining key physicochemical properties, including viscosity, acidity, and cetane index. In addition, FT-IR and FT-Raman spectroscopic analyses were performed to identify the characteristic functional groups and confirm the conversion of triglycerides into fatty acid esters. The experimental results were compared with European and American biodiesel standards in order to evaluate their conformity and potential as substitutes for petroleum diesel.

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1.INTRODUCTION

Diesel is the most widely used fuel for heavy-duty vehicles in the world. Its demand is expected to

rise in the coming years. However, the extraction of petroleum, the source of diesel, is becoming increasingly difficult. Furthermore, the combustion of diesel contributes to air pollution. It is therefore urgent to consider alternative fuels. Biodiesel is a promising solution. It is produced from vegetable oils, such as sunflower oil, olive oil, and palm oil, or from animal fats. Biodiesel has several advantages over diesel. It is less polluting, as it emits fewer nitrogen oxides (NOx) and fine particles. It is also renewable, meaning that it can be produced without depleting natural resources. There are several methods for producing biodiesel. The most common method is transesterification. This method involves reacting the triglycerides contained in vegetable oils or animal fats with alcohol, typically methanol. The reaction produces fatty acid methyl esters (FAME), which are the main component of biodiesel. Other methods of producing biodiesel exist, such as emulsification, pyrolysis, or blending with diesel. The scientific literature has documented numerous studies on the production of biodiesel from waste cooking oils using various methods. Y. Zhang and co-workers [1] investigated the technical feasibility of four continuous processes for the production of biodiesel from virgin vegetable oil or waste cooking oil. The results showed that the alkaline-catalyzed process using virgin vegetable oil was the simplest, but it had the highest raw material cost. The acid-catalyzed process using waste cooking oil was more complex than process I, but it had a lower raw material cost. Osmano Souza Valente and Al [2] olleagues produced a biodiesel blend of waste cooking oil and castor oil. They mixed this blend with diesel fuel and tested the resulting fuel for sulfur content. They found that the sulfur content of the blended fuel was significantly lower than that of the diesel fuel alone. Esther O. Babatundea and colleagues [3] investigated the feasibility of biodiesel synthesis from restaurant waste oil (RWO) using two different heterogeneous catalysts, R-SC1.5 and C-SC1.5. They found that the C-SC1.5 catalyst was the most efficient for the transesterification of RWO.

In addition to studies devoted to biodiesel production processes from vegetable or waste oils, a growing body of research has focused on the characterization of this biofuel using spectroscopic techniques such as FT-IR and FT-Raman, which enable the identification of functional groups and the monitoring of chemical transformations during transesterification.

For instance, M. N. Mohammed and colleagues [4] conducted a spectroscopic characterization of various blends of hemp (Cannabis sativa L.) biodiesel with Euro diesel, butanol, and diethyl ether using FT-IR spectroscopy. Their study highlighted the main functional groups present and provided valuable insights into the molecular interactions within these blends.

Similarly, Alisson M. Miranda and co-workers [5] investigated pure biofuels and their blends based on ester compounds through the analysis of Raman spectral line shapes. Their findings emphasized the relevance of Raman spectroscopy for distinguishing between different components, owing to the sensitivity of this technique to specific molecular vibrations, particularly those associated with C–H and C=O bonds.

This article presents a comparative study of biodiesel production from used cooking oil (UCO) and fresh sunflower oil. The physicochemical properties of the two oils are first described, then the biodiesel production process is described in detail, these properties of the produced biodiesel are compared to those of diesel. Finnaly the FT-IR and FT-RAMAN for these samples are presented.

2. MATERIALS AND METHODS

2.1 Materials and reagents required

Two types of biodiesel were produced from pure sunflower oil and used cooking oil collected from various restaurants in Castelo Branco. Used cooking oil was treated before transesterification. First, it was dried in a fan-assisted oven at 105°C for 24 hours to remove any water. Water may be present due to use or storage conditions. The next day, the oil was cooled by natural convection, i.e., by allowing it to sit on a flat surface for a period of time. After dehydration, the used cooking oil was filtered to remove impurities. The filtration was performed using a number 41 filter paper, as shown in Figure 1.

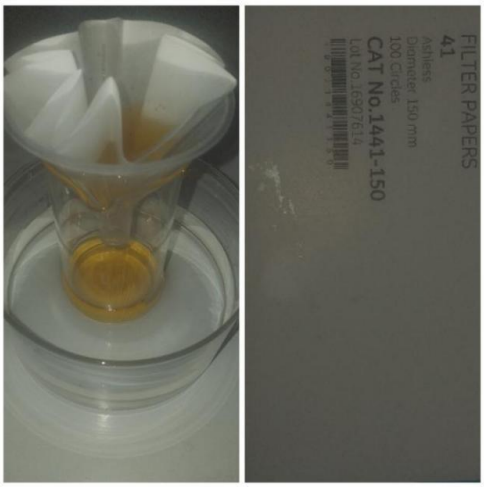


Fig. 1 Filtration of used cooking oil with number 41 filter paper

In addition to sunflower oil and used cooking oil, the following reagents were also used: NaOH (a basic catalyst to accelerate the transesterification reaction), H_2SO_4 (an acidic catalyst for the esterification reaction), and methanol (a solvent). All reagents were provided by the laboratory of the Instituto Politécnico de Castelo Branco IPCB.

2.2 Characterization of the oils used

The characterization of the oils used is a critical step in biodiesel production. This step is essential for selecting the optimal production method. Table 1 below summarizes the key properties of an oil and the methods used to obtain them.

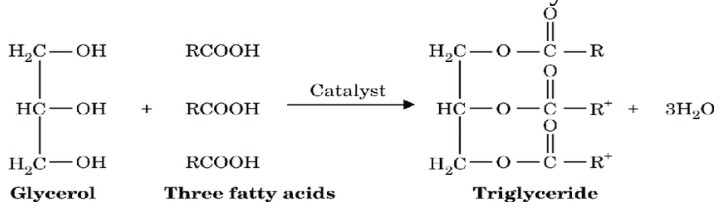
Table. 1 Characteristics of oils used for biodiesel production

Properties	Acid number	Density	Viscosity
Sunflower oil	0.199 mg_{NaOH}/g_{Huile} (0.0995%)	À 40C° → 0.8549 g/cm^3	35 mPa.s
Used cooking oil	4.003 mg_{NaOH}/g_{Huile} (2.0016%)	À 40C° → 0.8655 g/cm^3	/

It is observed that waste cooking oil has a much higher acidity than sunflower oil, its acidity is 2.002%, or 4.003 mgNaOH/goil. Sunflower oil, on the other hand, has an acidity of 0.0995%, or 0.199 mgNaOH/goil. This difference in acidity is due to the fact that waste cooking oil has undergone significant hydrolysis and degradation reactions during its use [6]. The EN14214 standard limits the acidity of biofuels to 0.5 %. Waste cooking oil therefore exceeds this limit. Consequently, waste cooking oil must be treated before being used as a biofuel. This treatment must reduce its acidity. For this purpose, a pretreatment by esterification with an acidic catalyst, sulfuric acid (H2SO4), has been proposed.

2.3 Pretreatment and transesterification of waste cooking oil

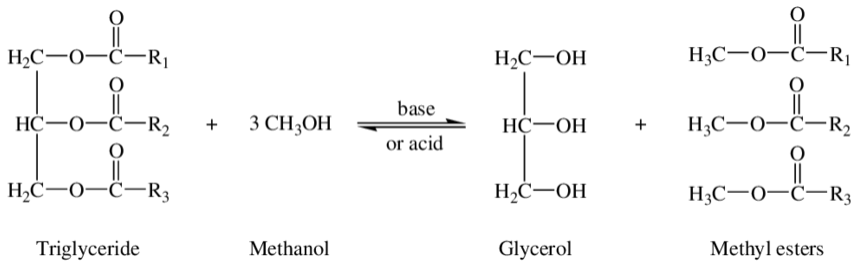
Esterification of free fatty acids is a recommended step prior to basic transesterification. This reaction reduces the acidity of the fat by forming methyl esters. Sulfuric acid is the most effective catalyst for this reaction [4]. Esterification of FFAs with an acidic catalyst occurs according to Equation (1) [7].



(1)

Acid-catalyzed esterification was performed by mixing 1.5 g of sulfuric acid, 34 g of methanol, and 50 g of waste cooking oil at 60 °C for 1 hour. The methyl ester was then recovered by decanting. The acidity of the methyl ester was then measured by titration, which gave a value of 2.05 mgNaOH/goil, or 1.02%. This value was above the limit set by the standard, which is 0.5%. Therefore, a second esterification was performed. The second measurement of the acidity of the methyl ester gave a value of 1.06 mgNaOH/goil, or 0.53%, a value that can therefore be considered acceptable.

After obtaining an ester with an acidity level that meets the standard limits, the transesterification step for the production of biodiesel is carried out according to Equation (2) by mixing 137 g of ester (produced from 3 samples) with 29.74 g of methanol (a methanol:oil molar ratio of 3:1) and 1.7125 g of NaOH (1% + (%FFA / 2)). The reaction is carried out at a temperature of 60 °C to prevent methanol evaporation. After 1 hour of reaction, the mixture is poured into a separatory funnel and allowed to stand for 12 hours to allow the separation of glycerol and ester. The biodiesel produced is collected, purified (by repeated water washing and drying), and the volumetric fuel yield is calculated using Equation (3).



(2)

$$\text{Biodiesel Yield (\%)} = \frac{\text{Weight of Biodiesel from RWO}}{\text{Weight of RWO used}} \times 100\ \% \qquad (3)$$

2.4 Transesterification of Sunflower oil

Sunflower oil, with an acidity of about 0.0995%, has a sufficiently low acidity that transesterification can be carried out directly, without the need for a prior esterification step. To do this, 150 g of oil is heated to 60°C, and 32.57 g of methanol (a methanol:oil molar ratio of 3:1) and 1.5 g of NaOH (1% of the mass of the oil) are added. After 1 hour of reaction, the mixture is poured into a separatory funnel and allowed to stand for 12 hours to allow the glycerol and ester to separate. The biodiesel obtained is collected, purified (by repeated water washing and drying), and the volumetric fuel yield is calculated using equation (3).



Fig. 2 The transesterification reaction for sunflower oil and used cooking oil



Fig. 3 Sedimentation of biodiesel samples

3.RESULTS AND DISCUSSION

3.1 Transesterification Reaction Yield

The transesterification reaction is a chemical reaction that converts triglycerides into methyl esters, which is biodiesel. The yield of this reaction is defined as the ratio of the amount of biodiesel produced to the amount of vegetable oil used. It is expressed as a percentage. The table below summarizes the results obtained at the end of the transesterification reaction for two oils used.

Table. 2 Biodiesel Yields from Sunflower Oil and Used Cooking Oil

Oils	*Used Oil (g)*	*Produced Biodiesel (g)*	*Yield (%)*
Sunflower oil	150	141.95	94.63
Used cooking oil	137	119.48	87.21

3.2 Biodiesel Characteristics

The biodiesel developed was subjected to a series of tests to verify its compliance with the European EN14214 standard.

- ***Density***

The Density is measured using a precision densimeter with a resolution of 0.0001.

Table. 3 Density of biodiesels

Temprature (°C)	*Density of Biodiesel from Sunflower oil*	*Density of Biodiesel from Used cooking oil*
20	0.8801	0.8814
25	0.8767	0.8767
30	0.8743	0.8759
42	0.8642	0.8662
51	0.8583	0.8580
60	08521	0.8521
65	0.8472	0.8491

The density of biodiesel produced from sunflower oil is 0.8743 at 30°C, which is close to the European (0.86 to 0.9) and American (0.85 to 0.9) standards. Similarly, the density of biodiesel produced from used oil is around 0.8759 at 30°C. These values are lower than those of crude oil, which is explained by the transesterification reaction that produces biodiesel.

- ***Viscosity***

In a rheometer, oil fills the gap between two coaxial parts, the rotor and the stator. The ratio of the torque transmitted by the sheared substance from one part to the other to the rotor's rotational speed gives the viscosity, up to a geometric constant.



Fig. 4 Viscosity measurement of biodiesels using a rheometer

- ***For Sunflower oil-based biodiesel***

After introducing sunflower oil methyl ester into the rheometer, the dynamic viscosity was measured. The results are summarized in the table below, as well as the corresponding graph.

Table. 4 Sunflower oil-based biodiesel viscosity as a function of temperature

Temprature (°C)	Viscosity (mPa.S)
20.04	6.4484
25.54	5.809
30.47	5.0726
35.48	4.4958
40.51	3.9477
45.54	3.5364
50.56	3.1412
55.59	2.8767
60.62	2.5840
69.84	2.1108

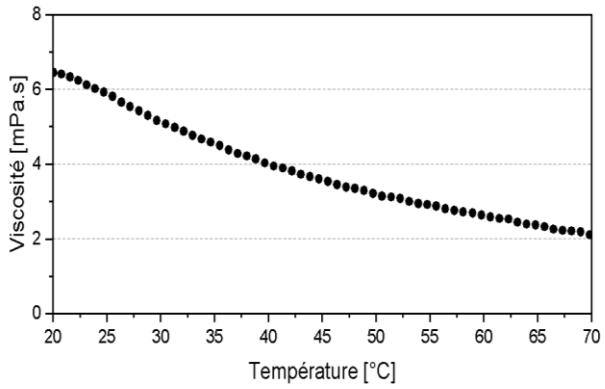


Fig 1. Sunflower oil-based biodiesel viscosity as a function of temperature

- ***For Biodiesel from Used cooking oil***

After introducing cooking oil oil methyl ester into the rheometer, the dynamic viscosity was measured. The results are summarized in the table below, as well as the corresponding graph.

Table. 5 Used cooking oil based biodiesel viscosity as a function of temperature

Température (°C)	Viscosité (mPa.S)
20.11	7.2177
25.60	6.3912
30.53	5.5344
35.52	4.8227
40.54	4.1677

45.56	3.6402
50.58	3.2073
55.60	2.8093
60.63	2.4932
69.01	2.0188

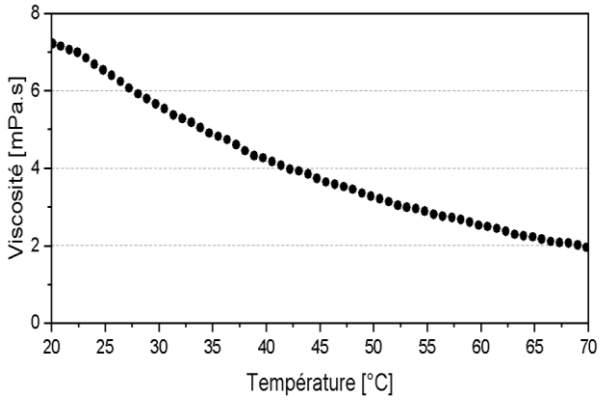


Fig 2. Used cooking oil -based biodiesel viscosity as a function of temperature

The viscosity of biodiesel is a key property that affects its performance in engines. High viscosity can make it difficult for the fuel to flow through the engine's fuel system, which can lead to poor fuel economy and increased emissions. The viscosity of biodiesel produced from sunflower oil is typically around 5.1 mPa.s at 30°C, which is significantly lower than the viscosity of raw sunflower oil (around 35 mPa.s). Similarly, the viscosity of biodiesel produced from used oil is typically around 5.53 mPa.s at 30°C. The lower viscosity of biodiesel is due to the transesterification reaction, which breaks down the long hydrocarbon chains in the oil into shorter chains. Shorter chains have lower viscosity, making them easier to flow through the engine's fuel system and burn more completely.

➢ ***Acidity Number***

Titration was used to measure the acidity of the different biodiesel samples prepared, The results are summarized in the tables below.

- ***For Sunflower oil-based biodiesel***

Table 1. Sunflower oil-based biodiesel Acidity Number

Biodiesel sample	**1**	**2**	**3**
Masse (g)	2.0051	1.9955	2.0084
NaOH (ml)	0.1	0.1	0.1
Acidity Number (mgNaOH/gEster)	0.0997	0.1	0.0995
(FFA%)	(0,04985 %)	(0.5 %)	(0,04975 %)

- ***For Biodiesel from Used cooking oil***

Table .2 Used cooking oil -based biodiesel; Acidity Number

Biodiesel sample	1	2	3
Masse (g)	2.0186	2.0026	2.0210
NaOH (ml)	0.1	0.1	0.1
Acidity Number (mgNaOH/gEster)	0.198	0.199	0.197
(FFA%)	(0,04985 %)	(0.5 %)	(0,04975 %)

Experimental results show that there is a decrease in the acid value of oils compared to their biodiesels. This confirms that the esterification and transesterification reactions refine the oils of these free fatty acids. They thus reduce their acidity rate, which explains this variation. When compared to American standards, we find that our biodiesels comply with the standards.

Table. 6 Physicochemical properties of WCO-based biodiesel compared to ASTM D6751 standards

	Biodiesel de tournesol vierge	Biodiesel d'huile de caisson usagée	ASTM D6751
Densité (a 20°C)	0.8801	0.8814	≤0.90
Viscosité (a 40°C) mPa.s	3.9477	4.1677	1.4 a 6.0
Indice d'acidité (mgNaOH/gEster)	0.0997	0.198	0.50 max

3.3 Fourier Transform Infrared Spectroscopy (FT-IR)

The FT-IR spectra highlight significant differences between the raw waste cooking oils and the produced biodiesel. Figure 7 presents the FT-IR analysis of waste cooking oil and biodiesel. The appearance of an intense peak centered around 1740 cm^{-1}, corresponding to the stretching vibration of the ester carbonyl group (C=O), represents a major indicator of the transesterification reaction. The intensification of this band confirms the formation of methyl esters. In addition, the enhancement of the bands within the 1240–1160 cm^{-1} region, characteristic of C–O and C–O–C vibrations, further supports the effective transformation of triglycerides into biodiesel. These findings therefore demonstrate the successful conversion and the production of a fuel exhibiting the typical spectral signatures of methyl esters.

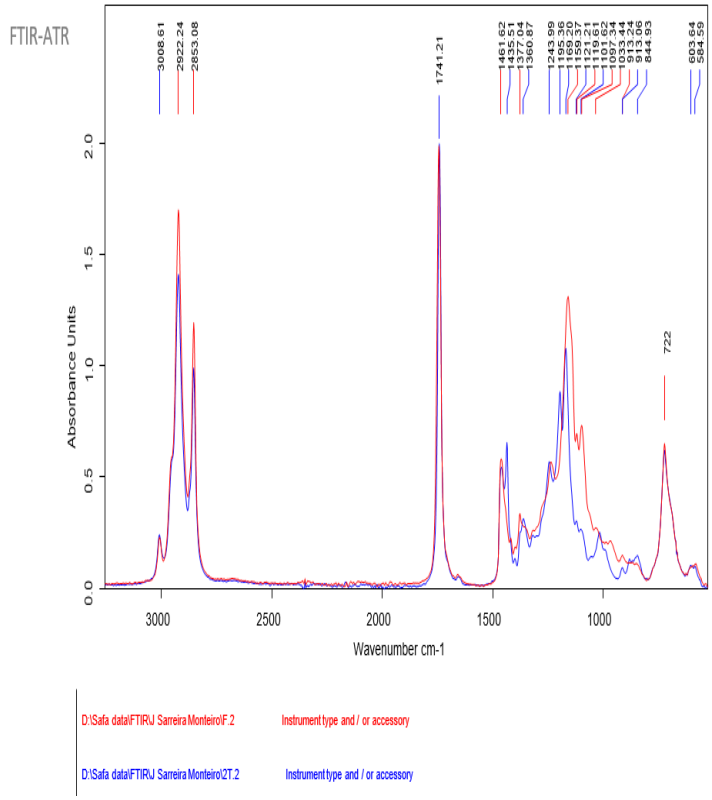


Fig 3. FTIR-ATR analysis of waste cooking oil and the corresponding biodiesel

3.4 FT-RAMAN

FT-Raman analysis revealed the main vibrational modes characteristic of fatty acids and fatty acid esters. Figure 8 presents the FT-Raman spectrum of biodiesel produced from waste cooking oils. The band observed at 1743 cm^{-1}, corresponding to the stretching vibration of the carbonyl group (C=O) of esters, is a reliable indicator of biodiesel formation following the transesterification reaction. The bands in the 2850–2950 cm^{-1} region are attributed to the stretching vibrations of C–H bonds in methylene and methyl groups, typical of long aliphatic chains. In addition, the band at 3004 cm^{-1}, assigned to the =C–H stretching of cis double bonds, confirms the persistence of unsaturations in the biodiesel.

These results demonstrate that FT-Raman spectroscopy effectively complements FT-IR analysis by highlighting the vibrations associated with non-polar bonds and providing additional structural information on fatty acids and the esters formed.

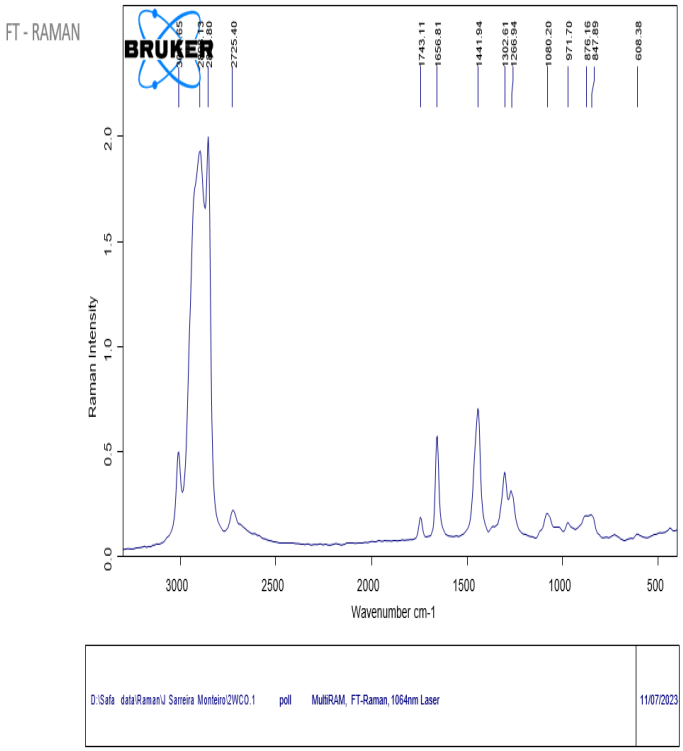


Fig 4. FT-Raman spectral profile of biodiesel derived from waste cooking oils

4.CONCLUSION

In this article, the valorization of two types of oils, sunflower oil and used cooking oil, was carried out and production processes were developed at the laboratory scale. Basic-catalyzed transesterification proved effective for converting sunflower oil into biodiesel. The physical properties of the biodiesel produced from this oil complied with both European (EN 14214) and American (ASTM) standards.

For used cooking oil, the analysis revealed clear signs of severe degradation. The free fatty acid content was estimated at 2%. This high level makes it unsuitable for direct use in engines. Among the various methods available for reducing acidity, acid-catalyzed pre-esterification was found to be the most suitable. The two-step reaction yielded a product with an acidity of 0.53%. This product was then subjected to basic-catalyzed transesterification, resulting in methyl esters that met the European (EN 14214) and American (ASTM) standards.

Furthermore, FT-IR and FT-Raman spectroscopic analyses confirmed the formation of methyl esters. The FT-IR spectrum showed a characteristic peak at 1740 cm^{-1} corresponding to the stretching vibration of the ester carbonyl group (C=O). The FT-Raman spectra exhibited bands between 2850 and 2950 cm^{-1}, assigned to C–H stretching of methylene and methyl groups, along with a band at 3004 cm^{-1} indicating the persistence of cis-configured double bonds. These results confirm the conversion of triglycerides into esters and provide additional structural information on the biodiesel produced

5. ACKNOWLEDGEMENTS

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