Epoxidation process of canola oil esters

Processo de epoxidação dos ésteres do óleo de canola

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1 INTRODUCTION

There is growing research for a new way to obtain biodegradable lubricants, the biolubricants are the best choice to replace mineral-based lubricants, aiming to reduce the pollution that mineral-based lubricants cause to the environment, besides its source, oil, being a finite and non-renewable natural resource, Plant-based lubricants are sustainable and, compared to mineral-based lubricants, they cause far less damage to the environment and to living beings all over the planet (MACEDO et al., 2021).

Vegetable oils are the main organic material to replace the convenience lubricants derived from petroleum, they have a high caloric value, besides having specific characteristics that make it possible to be a sustainable product: the low sulfur content in its composition, besides the fact that it comes from vegetable matter that absorbs carbon dioxide from the atmosphere during the photosynthesis process, it is free of heavy metals and its production does not contribute with polluting residues to the environment, (SANTOS, 2011).

The use of biolubricants from vegetable and renewable sources are of total benefit for technological development, where it can contribute beneficially to the country in a way that aggregates to products of ecologically correct perspectives as well as the use of renewable plant materials can be mechanisms for income generation and promoters of growth and regional development, especially for farmers reducing the evasion to urban centers. (MATOS, 2011)

According to Silva and Freitas (2008) of recurrent times, Canola (Brassica napus) or Colza appears as a model for use of cultivation in cold weather and winters. Also, the familiarity of the Brassicaceae species makes it easy to control infestations that are harmful to the plant's health. It can also, concomitantly, be sown with corn or soybeans, also generators of vegetable oils that are sources of raw material for biodiesel. Because sowing takes around 100 days, although it is advantageous for the agricultural system, the use of canola and sunflower oil in the manufacture of biodiesel can be discrepant because they use high quality oils suitable for human consumption, because they have high amounts of polyunsaturated fatty acids, considered beneficial to health and highly valued in food.

However, it should be noted that natural resources are also expended for canola monocropping. Taking this into account, more natural resources are consumed for the production of grain for fuel than for the production of diesel oil from petroleum. Data from the USDA and USDE (1998) cited by Silva and Freitas (2008) show that 1 liter of water is used in the production of diesel oil necessary to generate the energy for a 1 HP engine to run for one hour, however, 18 liters of water are consumed to produce the mixture with 20% biodiesel and more than 85 liters of water to produce pure biodiesel. But, in comparison to burning fossil fuels, which release pollutants in abundance into the atmosphere, such as carbon monoxide (CO) and/or carbon dioxide (CO), carbon dioxide (CO_2), nitrogen oxides (NOx) and sulfur oxides (SOx), especially sulfur dioxide (SO_2).

Carbon dioxide plays a fundamental role in life on Earth, since it is essential for the process of photosynthesis; however, in gigantic quantities it causes environmental imbalance, thus contributing to global warming. The carbon retained for millions of years underground is released into the atmosphere after the burning of fossil fuels, causing the concentration of carbon in the atmosphere to increase considerably, leading to the greenhouse effect. Carbon is also released when biofuels are burned, but in this case the carbon released is only that which has been absorbed by the plants as they grow, establishing a closed carbon cycle. The CO_2 is captured during plant growth and is released when the biofuel is burned (SUAREZ., 2006). In recent years, there has been a significant increase in the concentration of greenhouse gases in the atmosphere, including carbon dioxide (CO_2). This phenomenon is associated with global warming, which results in various climate changes. These climate changes are often attributed to the burning of fossil fuels and their petroleum derivatives.

One of the options that is presented to reduce dependence on petroleum products is the development of products that use vegetable oils as a base, such as biodiesel and biolubricants. According to Silva (2019) There is a limited reserve of input material for fossil fuel production, which increases the demand for alternative fuels from renewable sources. Most of these sources have environmental advantages over conventional fuels, such as in reducing the greenhouse effect and global pollution rates. These advantages are additional points that reinforce the need to replace fossil fuels with renewable energy sources.

As a viable and environmentally friendly alternative to replace petrochemical derivatives is the use and production of esters from vegetable oils (biodiesel) and synthesis of epoxides (biolubricant) from the epoxidation of esters. Biolubricants and biodiesel in Europe have stood out, because the use of these products is already a reality, biodiesel makes if a cheaper product compared to conventional diesel, (GUERRA; FUCHS, 2010), however some fuel of vegetable origin causes damage to engines in the long term, in order to improve their characteristics as lubricants, two widely used and known processes are transesterification and epoxidation, aiming to improve their physical-chemical characteristics. Transesterification is a process in which an ester is obtained from another ester, (FARIAS et al., 2021), while the epoxidation process will improve the poor thermal and oxidative stability of vegetable oils (RIOS, 2015).

Considering this context and its multiple characteristics, such as the possibility of production from vegetable and animal oils and a wide range of residues, biodiesel emerges as a viable alternative to diesel. Besides its high calorific power, vegetable oils present qualities that differentiate them as sustainable fuels: the absence of sulfur in their chemical composition; the fact that their industrial production does not generate harmful substances to the environment and also the fact that they are made from vegetable crops that consume carbon dioxide from the atmosphere during photosynthesis (PIANOVSKI JÚNIOR, 2002, apud SANTOS *et al.*, 2011).

Biolubricating oils originate predominantly from mineral sources, extracted from petroleum, and their classification is based on their physical and chemical properties. The main properties observed in a lubricating oil include viscosity, viscosity index, flash point, fire point, acid index, copper corrosion, specific mass, heat dissipation capacity, and the ability to reduce wear caused by friction. These properties determine which group the lubricant is associated with (CAVALCANTE, 2014).

In terms of chemical reactions, according to Solomons *et. al* (2006) transesterification consists in the reaction between an ester, in this case the triglyceride formed between glycerol and the fatty acids present in canola oil, which with the addition of an alcohol, in this case methanol, forms another ester and another alcohol. And it is customary to use catalysts to accelerate the reaction. There will be the cracking of the triglyceride molecules and the insertion of the methyl substituent of the alcohol, in the carbonyl of the fatty acids, allowing the phase separation between the ester that was formed, and the glycerin (trialcohol). Commonly, homogeneous and heterogeneous catalysts are used, which can have an acid or alkaline character (SUSIN, 2018).

In the epoxidation reaction, there will be the breaking of the unsaturations and the entry of an oxygen at the site where the double bond was broken. The entry of oxygen comes from the use of perfumeric and peracetic acids (SILVA, 2012).

2 OBJECTIVE

The objective of this research is to obtain a biolubricant from canola oil by means of transesterification and epoxidation reactions.

3 METHODOLOGY

To obtain methyl esters, initially a calculation of the molar mass of canola oil was made from its saponification index. With the knowledge of this mass the amounts of alcohol (methanol) and catalyst (KOH) necessary to carry out the reaction were calculated. The transesterification reaction was performed adopting a molar ratio oil/alcohol equal to 1:6 and 0.7% of catalyst (oil/catalyst) (PELANDA, 2009), keeping the temperature at approximately 45^{o} C for 1 h, because temperatures above the boiling temperature of alcohol can accelerate the saponification of glycerides by the alkaline catalyst before complete alcoholysis (FERRARI *et al*., 2005).

Figure 1. Methyl transesterification process of canola oil.

SOURCE: Survey data (2023)

After the transesterification reaction, the reaction mixture was transferred to a separation funnel allowing the separation of the phases: upper containing methyl and lower composed of glycerol, soaps, excess base and alcohol. After the waiting time, the lower phase was removed and stored in a proper container. Next, washing of the esters with distilled water and 0.01M hydrochloric acid was performed. Three washes were made with distilled water (to remove glycerol and soap residues from the ester phase) and two washes with 0.01M HCl solution (to neutralize the esters). To check the efficiency of the acid wash, phenolphthalein was used. After the washes, anhydrous magnesium sulfate was added to remove any water that was still present in the esters. Then, in order to remove the alcohol that might still be present in the ester, a rotary evaporator was used.

Figure 2. process of decanting methyl biodiesel from canola oil.

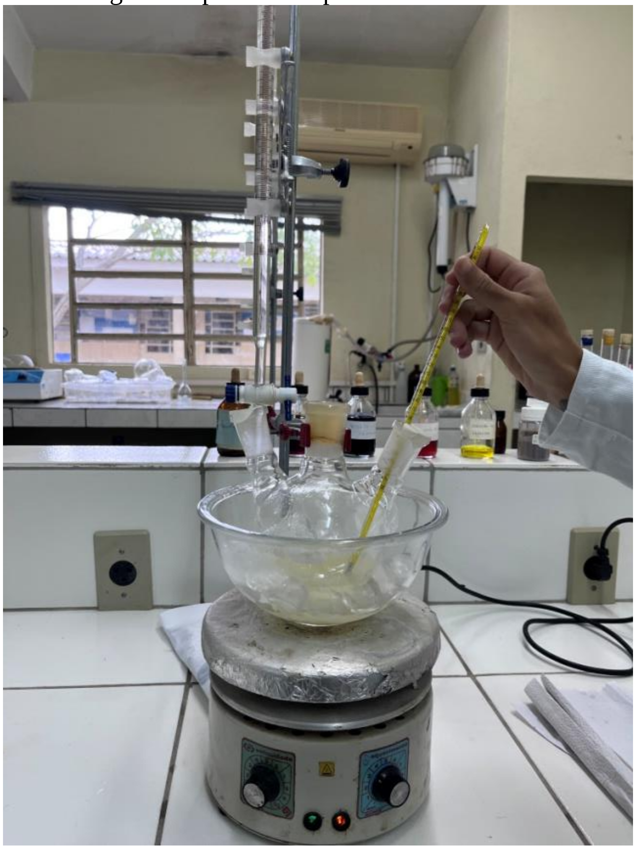
SOURCE: Survey data (2023)

Figure 3. washing process of methyl biodiesel from canola oil.

SOURCE: Survey data (2023)

In the epoxidation reaction, 100 g of the methyl ester obtained from canola oil, and drop by drop, 140 mL of 15% commercial peracetic acid were added to a 500 mL round bottom flask. The mixture was stirred and heated at 45º C in a water and ice bath for 1 h. The reaction was carried out using the molar ratio of 1:1.1 ester/peracetic acid. After the end of the reaction, the mixture was transferred to a separation funnel, where the lower phase, corresponding to the acetic acid, was removed, and the upper phase was washed twice with 50 mL of 10% sodium bicarbonate until the bubbles were completely detached due to the neutralization reaction. In order to remove the residual water, anhydrous magnesium sulfate was added to a conical flask containing the epoxide (biolubricant), stirred vigorously for 5 min and then allowed to stand for 30 min (NUNES *et al*., 2008). To remove the magnesium sulfate, a vacuum filtration was performed.

Figure 4. epoxidation process of canola oil.

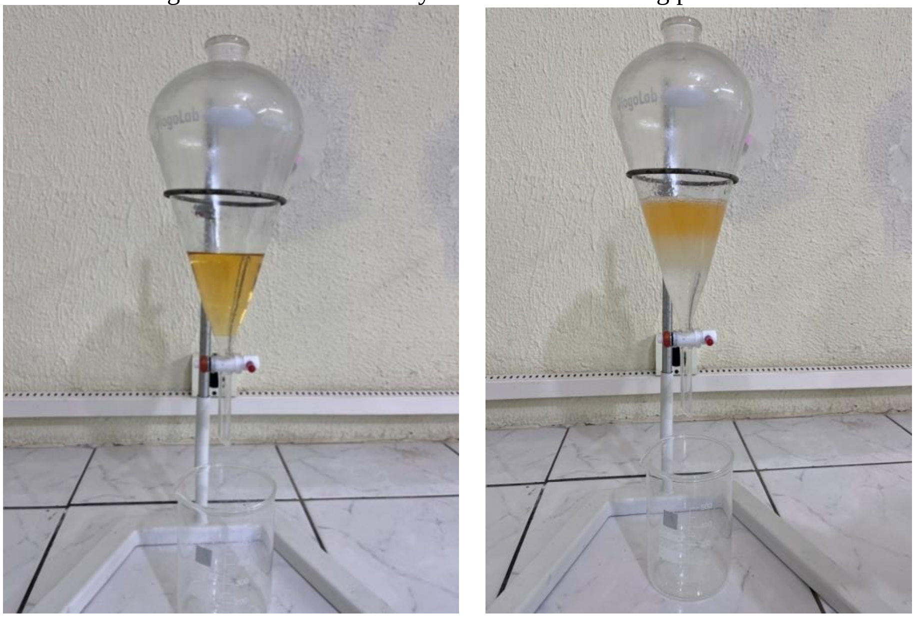


SOURCE: Survey data (2023)

Figure 5. decanting process of canola oil methyl biolubricant.

SOURCE: Survey data (2023)

Figure 6. Canola oil methyl biolubricant washing process.



SOURCE: Survey data (2023)

Canola oil was characterized by acid value (AOCS Cd 3d-63), saponification value (AOCS Cd 3b-76), soap content (AOCS Cc 17-95), peroxide value (AOCS Cd 8-53), relative density, ash content, moisture content, and volatiles (AOCS Da-2a-48).

The procedures adopted to characterize the methyl esters obtained after transesterification were the same as those used to characterize canola oil.

The castor oil methyl and ethyl ester epoxides were characterized using acid value (AOCS Cd 3d-63), peroxide value (AOCS Cd 8-53), relative density, ash content, moisture content, and volatiles (AOCS Da-2a-48).

All the characterizations described above were performed according to the techniques described by Wu *et al*. (2000) and were done in triplicate.

4 DEVELOPMENT

Canola oil can actually be used as a pesticide, industrial lubricant, and biofuel, but any oil can be used for this. Likewise, it can also be used in the manufacture of soap, plastics, cosmetics, and printer ink, and these uses are considered environmentally friendly.

The canola oil presented good quality in such a way that the physical-chemical parameters are in accordance with the norms of the National Health Surveillance Agency (ANVISA).

Table 1. Physicochemical parameters of canola oil.

Parameters	**Oil**	**Anvisa Standards**[1, 2]
Aspect	Clear yellow	Clear and free of impurities
Humidity and Volatiles (%)	0,195	≤ 0,1
Ash (%)	0,021	---
Density (g/cm)^3	0,917	0,915 - 0,925
Acid value (mg KOH/g oil)	0,335	≤ 0,6
Soap content (ppm sodium oleate)	0,060	≤ 10
Saponification index (mg KOH/g oil)	193	189 - 195
Peroxide value (meq/Kg)	0,006	≤ 10
Approximate molar mass (g/mol)	872	---

Source: Survey data, 2023;[1] BRASIL, 2021;[2] BRASIL, 2006.

Density is a measure that helps to characterize substances, indicating the amount of material present in a given volume. In vegetable oils, density is defined by the following aspects: the lower the molar mass of triglycerides, the lower the density of the vegetable oil; also, the higher the degree of unsaturation, the denser the oil is. The canola oil used in this research had a density of 0.917g/cm^3 a value very close in comparison to the sample density that Carvalho(2017) obtained, canola oil density 0.918±1.70x10 g/cm^{33} .

The acidity index is closely linked to the quality of the raw material, and can be influenced by the maturity and storage time of the grain, because the acidity index increases with increasing storage time. In view of this, a high acidity index shows that a breakdown is occurring in the molecular chains of this oil or fat, releasing fatty acids. The acidity index of canola oil showed 0.335 mg KOH/g oil compared to Carvalho(2017)who presented acidity index of canola oil 0.013 ± 3x10^{-4} mg KOH/g oil

The soap content is used to determine the alkalinity present in the sample. the result obtained from canola oil was 0.060 ppm sodium oleate lower index compared to Pereira (2022) in which his index from refined soybean oil is 0.12 ppm sodium oleate.

The saponification index is used to determine the amount of milligrams needed of potassium hydroxide to neutralize the fatty acids present in the sample. The canola oil obtained an index of 193 mg KOH/g oil lower compared to Santos (2011) in which his result was 193.2 mg KOH/g oil, in which his oil used was refined soybean oil.

The peroxide value is a method to measure the oxidation state of fats and oils, the canola oil had a value of 0.006 meq/kg less compared to PEREIRA (2022) in which the refined soybean oil obtained a value of 0.007 meq/kg

The molar mass of canola oil was 872 g/mol, lower compared to refined soybean oil (PEREIRA,2022) which had a molar mass of 837 g/mol.

The yield obtained for methyl biodiesel was 94%. Rosset (2011) synthesized esters with yields of over 95%. Thus, the yield of the sample occurred in a profitable manner. The high yield of esters reflects the transesterification reactions with a small-chain alcohol, which facilitates the reaction, since long-chain alcohol has difficulties in breaking down the triglyceride molecules, making transesterification difficult. The proportion 1:6 of oil to alcohol was used aiming for maximum yield. For, the addition of excess alcohol has as its main function to shift the chemical balance and allow the separation of phases, because the transesterification reaction is an equilibrium reaction, and excess alcohol will favor the formation of products (TAPANES et. al., 2013).

Table 2. Physicochemical parameters of canola oil methyl esters (biodiesel).

Parameters	Oil esters	ANP Standards[1]
Aspect	Clear yellow	Clear and free of impurities
Humidity and Volatiles (%)	0,03	0,02
Ash (%)	0,016	0,02
Density (g/cm)^3	0,859	0,850-0,900
Acid value (mg KOH/g oil)	0,224	≤ 0,5
Soap content (ppm sodium oleate)	4,01	-----
Saponification Index (mg KOH/g oil)	126	-----
Peroxide Index (meq/Kg)	0,007	-----

Source: Survey Data, 2023;[1] BRASIL, 2014.

The content of moisture and volatiles, is a method used to determine the amount of moisture and the presence of any volatile materials in the specific test conditions, in the case of canola oil biodiesel had as a result 0.03%, a lower index compared to the characteristics (PEREIRA, 2022) in which his biodiesel index of refined soybean oil was 0.25%.

The ash content aims to assess the presence of inorganic substances of the methyl esters (biodiesel) of canola oil, by analyzing the sample after burning. The canola oil methyl ester showed an ash content of 0.016%, which was higher than the refined soybean oil methyl ester result of 0.014%.

The density of the oil is determined by the characteristics of the triglycerides present. In general, the lower the molecular weight of the triglycerides, the lower the oil's density. Furthermore, the higher the degree of unsaturation of the triglycerides, the higher the density of the oil. Biodiesel from canola oil had a density of 0.859 g/cm^3 lower compared to Pereira (2022) who synthesized biodiesel from refined soybean oil 0.935 g/cm^3

The acidity index of biodiesel from canola oil showed 0.22 mg KOH/g oil result lower than that of PEREIRA (2022) who presented an acidity index of biodiesel from refined soybean oil of 0.106 mg KOH/g oil. Macedo *et. al.* (2021) found a value of 1.78 ppm of sodium oleate in the soap content of biodiesel. In this synthesized biodiesel, the value was 4.01 ppm of sodium oleate showing that part of the catalyst reacted with the oil forming soap.

The peroxide index of biodiesel characterizes its ability to oxidize the molecules present in oils and fats, the peroxide index of biodiesel determined was 0.007 meq/kg, a lower index than that found in the used oil biodiesel of Pereira (2022) which was 0.0410 meq/kg.

The synthesis of epoxides proved favorable, since their yield was 92%. Silva (2022) synthesized a biolubricant with a percentage of 76.4, a fact that indicates the good residual process due to the complexity of the reactions. And that the lower chain alcohol used during the process may have contributed to the value. This is due to the fact that for the epoxidation reaction to occur, the peracetic acid must react with the esters and the reaction will occur with the opening of the unsaturations of the carbon chain of the esters that were obtained from fatty acids of canola oil (MARQUES, 2015). Canola oil, in its largest composition has unsaturated fatty acids such as oleic acid which varies between 53.0% to 70.0% of the composition of canola oil and has one unsaturation. The linoleic acid has two unsaturations and its presence in canola oil can make up around 15.0% to 30.0% of the oil's composition. It is also possible the presence of linolenic acid around 5.0 to 13.0% in the composition of oil and in its chemical structure has three unsaturations (ALBURQUEQUE, 2006).

Table 3. Physicochemical parameters of canola oil methyl epoxides (biolubricant).

Parameters	**Epoxide**
Aspect	Clear orange-yellow
Humidity (%)	0,3
Ash (%)	0,1
Density (g/cm)^3	0,887
Acid value (mg KOH/g oil)	1,335
Soap content (ppm sodium oleate)	3,40
Saponification Index (mg KOH/g oil)	115
Peroxide Index (meq/Kg)	0,002

Source: Survey Data, 2023

The relative density of the biolubricant was lower compared to Neto (2006). It is noteworthy that density is a property with particularities according to the substance. It is the product of the ratio between the mass and the volume it occupies (GOMES, 2010).

One method to assess the quality and care in the storage and synthesis of the biolubricant is the evaluation according to the peroxide value.

The physicochemical characteristics of biolubricants are what determine their usability, the soap content determines the alkalinity of the sample, the result of the saponification index of the biolubricant was lower compared to the saponification index of Pereira (2022). The presence of soap serves as an evaluative parameter to verify the presence of oils that ended up not reacting in the initial stage to form the ester, and with the passage of the reaction process ended up reacting with the alkaline base leading to soap formation. The low value found, serves as a basis to demonstrate the efficiency of the reaction and that the care to avoid the reaction of alcohol where they can accelerate the saponification of glycerides by the alkaline catalyst before the complete alcoholysis (FERRARI *et al*., 2005).

Peroxide value is a method to measure the oxidation state of fats and oils, the peroxide value of the biolubricant was 0.002 meq/kg, which is lower than the used oil methyl epoxide that was synthesized by Pereira (2022).

The saponification index of a sample determines the amount of milligrams of potassium hydroxide needed to neutralize the fatty acids in the sample, the biolubricant had an index of 115 mg KOH/g oil, a lower index compared to Cruz (2022) who obtained a saponification index of used oil of 178 mg KOH/g oil.

5 CONCLUDING REMARKS

The high yield of the reaction shows that the synthesis of biodiesel is viable, so that its reaction process was efficient, and that it can be employed in large scale aiming at the use as a possible substitute for the usual fuels. Still taking into consideration that biofuels have as a bias a clean, efficient alternative that is able to supply part of the existing demand.

It is also assessed that biolubricants already have literature-based comparisons and that their use is already possible, so that demonstrates the favorable viewpoint for the improvement of compounds and also, in which, it can be used.

It can be seen that the biodegradable lubricant has qualities that are of great benefit, and can also lead to the production chain benefiting a social range that goes from the processing of canola, as well as possibilities in generating employment and income for the chemical industry.

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