Synthesis and characterization of biolubricants by methyl epoxidation of corn oil

Síntese e caracterização de biolubrificantes pela epoxidação metílica do óleo de milho

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1 INTRODUCTION

As the global population continues to grow, concern is arising over the possibility of future shortages of resources essential to human life, such as fresh water, raw materials, energy and land suitable for food production. In this context, renewable raw materials are destined to gradually replace fossil-based raw materials, ranging from fuels to precursors used in the chemical industry. To meet these challenges, it is crucial to adopt management practices and technologies. This implies the need to develop new methodologies that are based on renewable resources, contributing to the reduction of emissions and waste.

Lubrication is a process of applying a suitable substance, usually oil or grease, between moving solid surfaces. In addition to reducing friction and wear, this process prevents overheating and corrosion of the internal parts of engines and other machines,

facilitating operation, reducing energy consumption and maintaining the functionality of the machines safely (CARRETEIRO, 2006).

Lubricants are generally found in their liquid state, and have three base fluids, these being mineral oils, which are the most common and are derived from petroleum, and vegetable oils produced from the synthesis of chemical components and are designed to meet the demands of high performance. In the historical context, the first lubricants used by humans were vegetable oils, however, in the mid-twentieth century, during World War II, they sought to develop lubricants that would provide equipment with exceptional and lasting lubrication at high temperatures. With this came the production of synthetic lubricants, being composed of synthetic molecules that are able to offer superior performance compared to conventional oils. Currently, the use of synthetic lubricants represents a large part of the lubricating oil market, due to its ability to extend the life of equipment, reduce wear and friction in certain equipment (STARLING, 2016).

Despite being non-biodegradable and environmentally damaging, petroleum-based lubricants are respectively the most widely used in various applications today. In addition, lubricants are generally used in various industrial and automotive sectors, where most of their components are released into the environment. Thus, the excessive use of petroleum-based lubricants can cause environmental damage, such as soil contamination, groundwater contamination, and atmospheric pollution. However, lubricants produced from vegetable oil are becoming, respectively, an alternative to replace petroleum-based derivatives, because they are non-toxic, from renewable sources, and biodegradable (STARLING, 2016).

The search for sustainable and ecologically adequate alternatives for the reality of each region, including oils and fats of vegetable or animal origin, is in constant development and represents a great challenge. These initiatives are very important to meet the growing demand for sustainable products and regional character, thus promoting economic and social development and reducing environmental wear and tear, besides reducing the disorderly use of oil and other non-renewable natural resources (MATOS, 2011). Therefore, among the feasible oilseeds for applications in the area of renewable and sustainable lubricants, corn oil extracted from the germ of corn grain through industrial processes arouses interest due to its easy cultivation and high productivity, as cited by the Ministry of Agriculture, Livestock and Supply.

Corn oil has several application possibilities in the chemical industry, especially when it comes to manufacturing more sustainable and biodegradable products. It can be

processed to produce industrial solvents, such as: methyl and ethyl esters, which can be found in the use of paints, varnishes, adhesives, and cleaning products. In addition, it is an excellent base for the production of industrial lubricants, especially in situations that demand biodegradable lubrication and of renewable origin. In addition, corn oil can be used as a raw material for the manufacture of a variety of chemical products. For example, fatty acids, glycerin, esters, and alcohols. These compounds can be used in various industrial sectors, including pharmaceuticals, cosmetics, food, and agriculture. All these numerous alternatives show the viability of corn oil as a vegetable raw material for the chemical industry.

Corn oil is composed mainly of triglycerides, which are esters formed by the combination of fatty acids and glycerol. It is composed of several fatty acids, of which linoleic acid is the most predominant, representing about 59.6% of the total oil composition. In addition, corn oil contains other important fatty acids, such as oleic acid (25.4%), palmitic (10.9%), stearic (2%) and linolenic (1.2%) (D`ARCE; VIEIRA, 2015). In general, corn oil is widely used in the food, cosmetic and pharmaceutical industries due to its composition rich in essential fatty acids and beneficial to human health. In addition to triglycerides, corn oil may also contain small amounts of other compounds, such as tocopherols (vitamin E) and phytosterols. These compounds have antioxidant properties and may contribute to the stability and nutritional benefits of the oil.

The biolube produced from corn oil plays a key role in promoting sustainability and reducing environmental impact in the industry by offering a less toxic, renewable and biodegradable alternative to conventional petroleum-based lubricants (MATOS, 2011). Furthermore, the use of plant-based can reduce dependence on non-renewable resources, mitigate greenhouse gas emissions and minimize damage to the environment.

While advantageous from an energy standpoint, the direct use of vegetable oils in diesel engines presents a number of significant challenges, regarding thermal and oxidative stability, due to the unsaturations present in the fatty acid molecules. In addition, they tend to have limited performance at low temperatures. However, studies have shown that it is possible to improve these characteristics through chemical reactions, such as transesterification and epoxidation (SANTOS, 2011). These chemical reactions allow the modification of vegetable oils' properties, improving their thermal stability, resistance to oxidation, fluidity at low temperatures and lubricity. Thus, it is possible to overcome the natural limitations of vegetable oils and obtain biolubricants with improved

performance, making them a more viable and effective option for various industrial applications.

Thus, the purpose of this research study was to synthesize a renewable biolubricant from corn oil that was free of synthetic additives, viscosity modifiers, and corrosion inhibitors in order to lower production costs and reduce environmental impacts.

2 OBJECTIVE

Obtain a biolubricant from corn oil by means of transesterification and methyl epoxidation reactions.

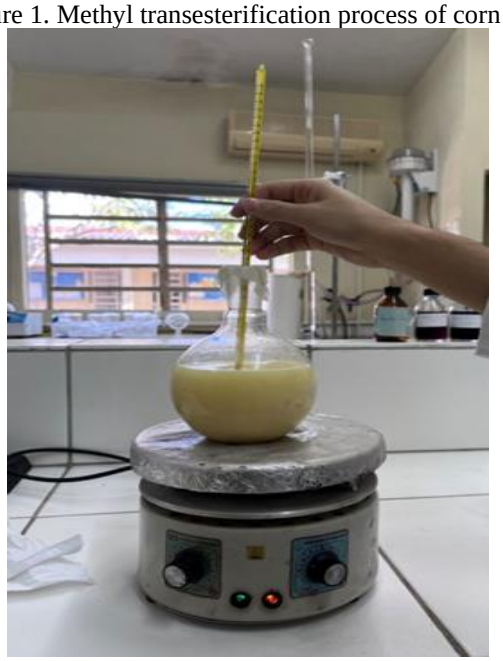
3 METHODOLOGY

The corn oil was purchased in the local trade and produced by Brazilian industry. The refined oil does not need any prior treatment before the reactions to which it was submitted.

3.1 CORN OIL TRANSESTERIFICATION

To obtain the methyl ester, initially a calculation of the molar mass of corn 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°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).

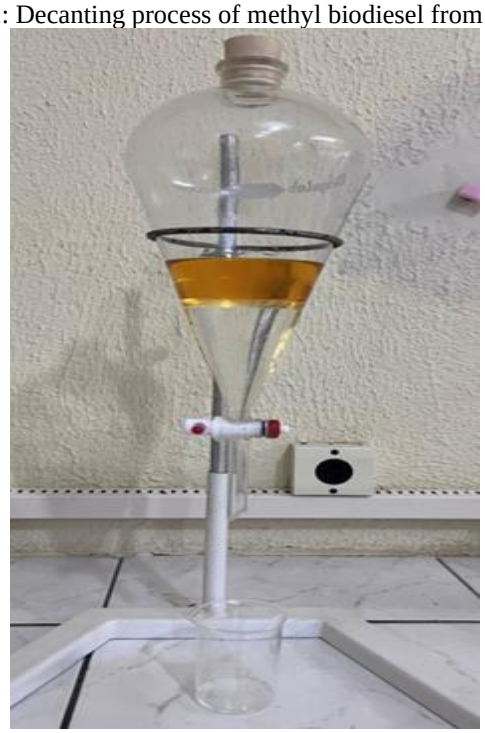
Figure 1. Methyl transesterification process of corn oil.



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 the methyl ester and lower composed of glycerol, soaps, excess base and alcohol.

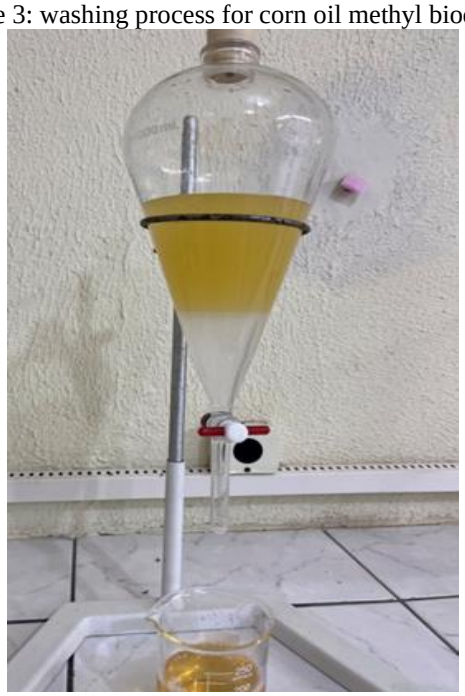
Figure 2: Decanting process of methyl biodiesel from corn oil.



Source: Survey data, 2023.

After the waiting time, the lower phase was removed and stored in a proper container. Then the methyl ester (biodiesel) was washed with distilled water and 0.01M hydrochloric acid solution. Three washes were made with distilled water (removing glycerol and soap residues from the ester phase) and two washes with 0.01M HCl solution (neutralizing the ester). To check the efficiency of the acid wash, phenolphthalein was used.

Figure 3: washing process for corn oil methyl biodiesel.



Source: Survey data, 2023.

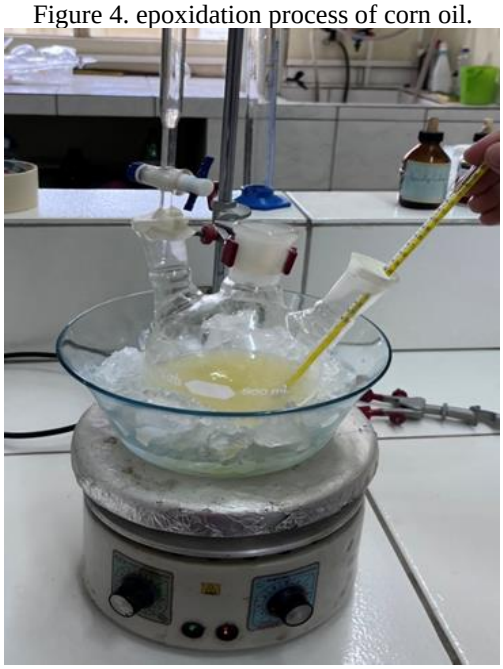
After the washes, anhydrous magnesium sulfate was added to remove any water that might still be present in the ester. Then, in order to remove the ethanol that might still be present in the ester, a rotary evaporator was used.

3.2 EPOXIDATION OF CORN OIL ESTER

In a 250 mL round bottom flask, 100 g of the methyl ester obtained from corn oil, and drop by drop, 140 mL of 15% commercial peracetic acid were added. The mixture was stirred and heated at 45°C in a water and ice bath for 1 hour. The reaction was carried out using a 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 an Erlenmeyer flask containing the epoxide (biolubricant) obtained from cottonseed oil, stirring vigorously for 5 minutes and then kept at rest for 30 minutes (NUNES et al., 2008). To remove the magnesium sulfate, a vacuum filtration was performed.

Figure 4. epoxidation process of corn oil.



Source: Survey data, 2023.

4 PHYSICAL-CHEMICAL CHARACTERIZATION

Corn 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, and moisture content and volatiles (AOCS Da-2a-48). The procedures adopted to characterize methyl ester obtained after transesterification were the same as those used to characterize corn oil. The corn oil methyl ester epoxide was characterized using peroxide indices (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.

4.1 ACIDITY INDEX (AOCS CD3D-63)

In a 250 mL conical flask, a 10 g sample was weighed and 62 mL of the neutralized solvent mixture (31 mL toluene + 31 mL isopropyl alcohol) was added. The sample should be well dissolved in the solvent mixture. To facilitate this process, it could be warmed up a little. Two to three drops of phenolphthalein indicator were added and

titrated with 0.1N KOH until a permanent pink coloration was obtained for 30 seconds. The same procedure was repeated without the presence of sample to determine the blank. The acidity index could be calculated using the following equation:

$$Acidity\ Index = (A - B) .N . 56,1 / W$$

where A: volume of the 0.1N KOH solution used in the sample titration (mL); B: volume of the 0.1N KOH solution used in the blank titration (mL); N: normality of the KOH solution; W: sample mass (g).

4.2 SOAP CONTENT (AOCS CC 17-95)

In a 250 mL conical flask, a 10 g sample was weighed, 0.25 mL of deionized water added, and shaken vigorously. Next, a solution containing 0.1 g of the bromophenol indicator and 50 mL of neutralized acetone was prepared. In the conical flask containing the sample, 50 mL of this newly prepared solution was added, and if soap was present, a phase separation occurred, with the upper layer showing a bluish green coloration. Then the mixture was titrated with a 0.01N standard hydrochloric acid solution until the bluish green coloration changed to yellow. The same procedure was repeated without sample to determine the blank. The soap content could be calculated using the following equation:

$$Soap\ Content = (A - B) .N . 304,4 / W$$

where: A: volume of HCl solution used in the sample titration (mL); B: volume of HCl solution used in the blank titration (mL); N: normality of the HCl solution; W: sample mass (g).

4.3 SAPONIFICATION INDEX (AOCS CD 3B-76)

In a 250 mL conical flask 1.0g of sample was weighed and 25 mL of alcoholic potash was added. This erlenmeyer flask was connected to a ball condenser and the assembly was heated gently for 1 hour so that the sample was completely saponified. Then a few drops of phenolphthalein were added to the Erlenmeyer flask and titrated with 0.5M HCl solution until the pink coloration disappeared. The same procedure was repeated without the presence of sample to determine the blank. The saponification index could be calculated using the following equation:

$$Saponification\ Index = (B - A) .N . 56,1 / W$$

where: A: volume of 0.5M HCl solution used in the sample titration (mL); B: volume of 0.5M HCl solution used in the blank titration (mL); N: normality of HCl solution; W: sample mass (g).

From the saponification index, the molar mass of the oil could be calculated:

$$1\ mol\ triglyceride\ (oil) + 3KOH \rightarrow 3\ soap + 1\ glycerol$$
$$X \text{——————} 3 \cdot (56.1\ g \cdot mol\text{-}1)$$
$$1g \text{————} Saponification\ Index$$

4.4 PEROXIDE INDEX (AOCS CD 8-53)

In a 250 mL Erlenmeyer flask with ground-glass stopper, 3 g of the sample was weighed and 30 mL of 3:2 (v/v) acetic acid:chloroform solution was added and mixed with slight stirring. 0.5 mL of 10% KI solution was added and allowed to stand for 1 minute. Then 30 mL of distilled water and 0.5 mL of 1% starch solution were added. The mixture was titrated with 0.01 N sodium thiosulfate solution with constant stirring until the blue color disappeared. The peroxide value could be calculated using the following equation:

$$Peroxide\ Index = (A - B) . N / W$$

where: A: volume of thiosulfate used in the sample titration (mL); B: volume of thiosulfate used in the blank titration (mL); N: normality of the Na solution $S\ O_{223}$; W: mass of the sample (g).

4.5 RELATIVE DENSITY

In a previously weighed 5 mL pycnometer, approximately 5 mL of distilled water was added and weighed. Next, approximately 5 mL of the sample was added and weighed. The density was calculated according to the expression:

$$d_x = m_x / m_{água}$$

where: d_x : relative density of the sample; m_x : sample mass (g); $m_{água}$: water mass (g).

4.6 ASH CONTENT

Using an oven at 105ºC ± 5ºC, the porcelain capsule was left to dry for 1 hour. After this period, it was placed in the desiccator to cool. Then the empty capsule was weighed and approximately 6g of sample was added. The plate with the sample was placed in a muffle furnace at 550°C ± 5°C for 4 hours. After this period it was placed in the desiccator to cool. Then the capsule with the sample was weighed. The ash content could be calculated using the following equation:

$$\% \, Ash = [(C - B)/A] \, . \, 100$$

where: A = sample mass; B = porcelain capsule + sample after muffle; C = porcelain capsule + sample before muffle.

4.7 MOISTURE AND VOLATILE CONTENT (AOCS DA-2A-48)

Using an oven at 105ºC ± 5ºC, the Petri dish was left to dry for 1 hour. After this period, it was placed in the desiccator to cool. Then the empty plate was weighed and approximately 6g of sample was added. The plate with the sample was placed in an oven at 105°C ± 5°C for 1 hour. After this time it was placed in the desiccator to cool. Then the plate with the sample was weighed. The moisture content and volatiles could be calculated using the following equation:

$$\% \, Humidity \, and \, Volatiles = [(C - B)/A] \, . \, 100$$

where: A = sample mass; B = Plate + sample after oven; C = Plate + sample before oven.

5 DEVELOPMENT

The corn oil was purchased in local commerce and produced by a Brazilian industry. The results obtained in relation to the physicochemical characteristics of refined corn oil are presented in Table 1.

Table 1. Physicochemical parameters of corn oil.

Parameters	Oil	Anvisa Standards[1, 2]
Aspect	Clear yellow	Clear and free of impurities
Humidity and Volatiles (%)	0,034	≤ 0,1
Ash (%)	0,029	---
Density (g/cm)[3]	0,916	0,915 - 0,925
Acid value (mg KOH/g oil)	0,220	≤ 0,6
Soap content (ppm sodium oleate)	0,0	≤ 10
Saponification index (mg KOH/g oil)	190	189 - 195
Peroxide value (meq/Kg)	0,009	≤ 10
Approximate molar mass (g/mol)	886	---

Source: Research Data, 2023;[1] BRASIL, 2021;[2] BRASIL, 2006.

Based on ANVISA (National Health Surveillance Agency) Normative Ruling No. 49 of December 22, 2006. It can thus be seen that, among the data obtained in Table 1, the appearance of the corn oil is within ANVISA standards, obtaining a clear yellow color. The moisture and volatiles are within the allowed levels, that is, for the analyzed corn oil, the moisture content and volatiles are low. ANVISA standards establish a value for moisture and volatiles (%), as being ≤ 0.1. The moisture content indicates the amount of water present in the sample and, therefore, promotes the formation of soap (MARCHETTI, 2005). The ash content, in turn, is high if compared to Araújo et al. (2009). The ash content informs the amount of inorganic residue present after the burning of organic matter in the muffle at high temperatures. The relative density of the sample shows the amount of material contained per unit volume, this will help in characterizing the substances. Given the ANVISA parameters, it can be seen that the relative density of the sample analyzed is within the standard imposed by ANVISA. The acidity index has a characteristic that allows us to show the conservation status of the oil, and is associated with its purity, nature, quality, type of processing and conservation conditions (RIBEIRO; SERAVALLI, 2004; COSTA et. al., 2006 apud PELANDA, 2009). The values expressed in Table 1 show us that the acidity index of the oil analyzed is within the standard, according to ANVISA regulations, which directs the acidity index (mg KOH/g oil) as being ≤ 0.6. The soap content is within the limits expected by ANVISA parameters, which establishes a value ≤ 10, considering that the sample analyzed had a content equal to (0.0), thus highlighting the alkalinity of the sample. The saponification index remains within the predicted by the legislation. The saponification index is an important parameter,

which aims to indicate the amount of alkali needed to saponify a given amount of oil. The peroxide index established in the analyses was 0.009 meq/kg. The established by ANVISA is a maximum of 10 meq/kg. Therefore, this parameter is within the acceptable range. The molar mass obtained was 886 g/mol.

After making the synthesis of methyl biodiesel by transesterification of oil, a yield of 96% methyl ester was obtained. Thus demonstrating its good yield. The table below shows the values for the physicochemical parameters of the corn oil methyl esters (biodiesel).

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

Parameters	**Oil esters**	**ANP Standards[1]**
Aspect	Yellow clear	Clear and free of impurities
Humidity and Volatiles (%)	0,307	0,02
Ash (%)	0,0285	0,02
Density (g/cm)[3]	0,859	0,850-0,900
Acid value (mg KOH/g oil)	0,110	≤ 0,5
Soap content (ppm sodium oleate)	2,55	-----
Saponification Index (mg KOH/g oil)	269,7	-----
Peroxide Index (meq/Kg)	0,021	-----

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

Given Resolution No. 45/2014 of the National Petroleum and Biofuels Agency (ANP), the aspect of the methyl ester analyzed is in accordance with the legislation. On the other hand, if compared to refined corn oil, the moisture content and volatiles of the methyl ester, derived from the same, is high, if related to the ANP standards, this is due to the possible presence of water in the reagents used. The ash content established by the legislation is 0.020%, when compared to the analyses made of the ash content of the sample, it is noted that the corn oil methyl ester is within the acceptable range, obtaining a percentage of 0.0285%. The result obtained by determining the density of the methyl ester analyzed was 0.859 g/cm^3 , showing it is within the ANP parameters. The acidity index, in turn, remains within the standards, reaching a value of 0.110 mg KOH/g oil. The soap content is 2.55 ppm of sodium oleate. And although the legislation has not defined the maximum value of the soap content, this is related to Escorsim et al. (2014) who in their analyses identified a soap content of 0.091 ppm, which is understood as high. The saponification index showed values of 269.7 mg KOH/g oil, the legislation does not have

a maximum index defined in this regard, but if correlated with the saponification index of corn oil, this is high. The same occurs with the peroxide index, which does not have a value defined in the legislation. In the analyses performed, the peroxide index obtained a value of 0.021 meq/kg.

The epoxidation reaction process using corn oil methyl ester in the presence of peracetic acid favored the obtaining of corn oil methyl ester epoxide (biolubricant). The yield of this process was 93%, indicating the efficiency of the procedure. The physicochemical characterizations of the corn oil epoxide are listed in the table below.

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

Parameters	**Epoxide**
Aspect	Clear orange-yellow
Humidity (%)	3,563
Ash (%)	0,028
Density (g/cm)^3	0,882
Acid value (mg KOH/g oil)	1,497
Soap content (ppm sodium oleate)	0,06
Saponification Index (mg KOH/g oil)	294,2
Peroxide Index (meq/Kg)	0,017

Source: Survey Data, 2023.

Although the legislation does not say anything about the maximum values for such parameters of methyl epoxides. The data obtained from the physicochemical parameters of the corn oil methyl epoxides (biolubricant), reveals that the values of moisture and volatiles was 3.563%, compared to the methyl ester, so one can see a significant increase, the moisture can lead to product inefficiency, since it can cause damage to parts. This increase in moisture values contributes to the increase in the saponification index, which showed a value of 294.2 mg KOH/g oil. The data obtained for the ash analysis was 0.028%, relating it to the analysis of the methyl ester, indicates that the presence of inorganic compounds remained the same. The relative density obtained 0.882 g/cm^3 , if compared to the refined corn oil, reached low values. If the molecular weight of triglycerides is lower, the density will tend to be lower, on the other hand, the higher the degree of instauration, the denser the oil will be (CARVALHO, 2016). The acidity index is 1.497 mg KOH/g oil, if we take as a basis the acidity index of the methyl ester, it is possible to see an increase in this index. The soap content is 0.06 ppm of sodium oleate, which is high when compared to the raw material. The peroxide value was 0.017 meq/kg,

relating this value with the methyl ester, it is possible to see that the methyl epoxide value is low, so the methyl epoxide may have less rancid characteristics than the methyl ester.

6 CONCLUDING REMARKS

In conclusion, the synthesis and characterization of biolubricants by methyl epoxidation of corn oil presents a great potential for the production of renewable and sustainable lubricants. In addition, the use of corn oil as a basic raw material for the synthesis of biolubricants contributes to the reduction of dependence on fossil resources and to the reduction of greenhouse gas emissions. The characterization of the obtained biolubricants revealed adequate properties, which meet the requirements for their application in several areas, such as the automotive industry and agriculture. However, further studies are needed to optimize the synthesis processes in order to obtain biolubricants with even better performance and to explore other sources of raw materials, seeking diversification and sustainability in the production of lubricants. In the current context of concern with the environment and the search for cleaner alternatives, the synthesis and characterization of biolubricants by methyl epoxidation of corn oil presents itself as an important contribution to the lubricants industry and to the promotion of more sustainable practices in society.

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