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RESEARCH ARTICLE

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SYNTHESIS OF BIODEGRADABLE LUBRICATING OILS BY METHYL AND ETHYL EPOXIDATION OF CASTOR OIL ESTERS

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ABSTRACT

This work consisted in the production of biolubricants from castor oil using oil transesterification, followed by methyl and ethyl epoxydation of its esters (biodiesel). The materials involved do not process foram characterized through their chemical and physical-chemical properties. The transesterification, which transforms the oil into biodiesel, provides a yield of 94% for the methyl biodiesel and 92.7% for the ethyl biodiesel. It is epoxydation, a reaction that transforms biodiesel into biolubricant, leading to a yield of 91.3% for methyl biolubricant and 87.6% for ethyl biolubricant. The products obtained have their adequate properties in comparison with the parameters established by the National Petroleum, Natural Gas and Biofuels Agency. In this way, the production of biodegradable lubricants will be able to significantly help in reducing the environmental impact of the use of fossil materials for the production of lubricants.

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INTRODUCTION

Energy sources are essential to human life, as they provide a better quality of life. According to Ramos *et al.* (2017), about 80% of the energy generated in the world comes from fossil fuels, such as coal, oil and natural gas. This fact has triggered a serious environmental problem, caused because the use of fossil fuels releases a high amount of polluting gases, such as carbon monoxide, which contribute, for example, to the intensification of the greenhouse effect, gradually increasing global warming (SANTOS *et al.*, 2019). The rapid decrease in fossil fuel reserves, the extraction, transport and industrial processes of oil transformation are responsible for several environmental damages such as spills, generation of residues and toxic effluents that are difficult to degrade, the contamination of groundwater by some derivatives and its additives, by the accumulation of carbon dioxide in the atmosphere (EREDA, 2004). The biggest aggravating factor in the use of mineral lubricating oils is the low recycling rate, the absence of waste management and disposal control by those responsible, with the largest amount of used lubricants being released into the environment inappropriately (SANTOS *et al.*, 2017). These concerns have led to a growing interest in vegetable oils for use as biofuels, such as biolubricants, as they are considered potential substitutes for petroleum-based mineral oils.

Just as there is a concern about the scarcity of oil and the development of sustainable alternatives for energy production, there is currently another problem that affects everyone: pollution. Population growth and the development of cities require energy needs, however this energy comes from fossil fuels such as oil, and its use has unfavorable consequences for society related to its polluting effect. One way to slow down such damage is to use less polluting fuels, such as biofuels. The production of biofuels and derivatives from castor oil has several advantages, highlighting the low price of the raw material and, consequently, of the lubricant, in addition to the environmental benefits (CRUZ *et al.*, 2020). As disadvantages of the production process, the large amount of free fatty acids found in the raw material need purification steps that increase the expenses during the synthesis and also the seasonality of cultures that offer an unequal supply of raw material at different times of the year. Castor oil can be used in the manufacture of soap, production of biodiesel and other biodegradable products, such as biolubricants. When you hear about castor bean applications, it seems that it is a panacea for everything. In fact, all the uses of the plant are the result of several researches carried out in companies and universities that, little by little, are being transformed into products and quality of life for society. There is also a concern with social inclusion, a decrease in mineral diesel imports and the ambition to provide a new management model for agricultural and energy development (CANGEMI *et al.*, 2010).

Castor bean can be considered the main oilseed for the production of biofuels in the northeast region, as it is easy to grow and has an adaptive property to semi-arid and drought environments. Castor bean is xerophilous and heliophilous, probably originating in Asia. (MELO, 2008). Due to the fact that there are no substitutes in many castor oil applications, as well as its industrial versatility, the need for this oil has increased in Brazil and in other industrialized countries. It is a vegetable oil, known as castor oil and differs from other vegetable oils by the large amount of ricinoleic acid. The presence of this triglyceride in its composition is 90%, on average, containing three highly reactive groups that allow specific qualities for the production of a multitude of industrial products. Castor oil is a viscous liquid, it is the only polyol found in nature and extracted from the seed to be used, being a triglyceride present in levels of 40 to 50% in the seed of the plant scientifically called *Ricinus communis* (PEREZ, 2009). Although unsuitable for human consumption, its importance focuses on its wide industrial application as a raw material used to manufacture a range of products (PRATA, 2007). The hydroxyl group gives this compound stability and high viscosity, which is allowed in wide temperature ranges, explained by the formation of intermolecular hydrogen bonds (MELO, 2008). The oil content of castor beans varies from 35 to 55%, whose commercial standard is 45%. In the extraction process, the oil can be obtained through different methods, solvent extraction or by cold or hot pressing (ALMEIDA, 2004). In addition to their high calorific value, vegetable oils have qualities that differentiate them as sustainable fuels: the absence of sulfur in their chemical composition; their industrial production does not generate harmful substances to the environment, and they are also made from vegetables that consume carbon dioxide from the atmosphere during photosynthesis (MELO, 2020). Despite being favorable from an energy point of view, the direct use of vegetable oils in diesel engines is very problematic, as their direct combustion leads to carbonization of parts, resistance to ejection in the pistons, dilution of the crankcase oil, contamination of the lubricant, among other problems (RINALDI *et al.*, 2007). Hence the need for its use after chemical transformation processes. Research has shown that one of the alternatives to improve the use characteristics of vegetable oils in engines is the modification promoted by chemical reactions, such as transesterification (biodiesel) and epoxidation (biolubricants). (LATHI; MATTIASSON, 2007). This research work was developed with the objective of synthesizing a renewable biolubricant from castor oil, which is less aggressive to the environment, which does not contain synthetic additives, viscosity modifiers, corrosion inhibitors and a high amount of heavy metals, aiming at reduce production costs and minimize impacts on different ecosystems.

MATERIALS AND METHODS

Materials

Castor oil was acquired in the local market and produced by Brazilian industry, and as it is a crude oil, it underwent a purification process and underwent transesterification and epoxidation reactions.

Biodiesel extraction procedure: To obtain the methyl and ethyl esters, initially a calculation of the molar mass of castor oil was made from its saponification index. With the knowledge of this mass, the amounts of alcohol (ethanol and methanol) and catalyst (KOH) necessary to carry out the reaction were calculated. The transesterification reaction was carried out by adopting an oil/alcohol molar ratio 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 point of alcohol can accelerate the saponification of glycerides by the alkaline catalyst before complete alcoholysis (FERRARI *et al.*, 2005). After the transesterification reaction, the reaction mixture was transferred to a separatory funnel allowing the separation of the phases: the upper one containing the ester and the lower one composed of glycerol, soaps, excess base and alcohol. After the waiting time, the lower phase was

removed and stored in a suitable container. Then, the esters were washed with distilled water and hydrochloric acid 0.01 M. Three washes were performed with distilled water (remove glycerol and soap residues from the ester phase) and two washes with HCl 0.01 M solution (neutralize the esters). To verify the efficiency of acid washing, phenolphthalein was used. After the washings, anhydrous magnesium sulfate was added to remove the water that was still present in the esters. Then, in order to remove the alcohol that could still be present in the ester, a rotary evaporator was used.

Biolubricant extraction procedure: In the epoxidation reaction, 100g of ethyl or methyl ester obtained from castor oil were added to a 250 mL round bottom flask, and 140 mL of 15% commercial peracetic acid was added dropwise. The mixture was stirred and heated at 45°C in an ice-water 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 separatory funnel, where the lower phase, corresponding to acetic acid, was removed and the upper phase was washed twice with 50 mL of 10% sodium bicarbonate until the total detachment of the bubbles due to the neutralization reaction. In order to remove residual water, anhydrous magnesium sulfate was added to an erlenmeyer flask containing the epoxide (biolubricant), shaking vigorously for 5 min and then remaining at rest for 30 min (NUNES *et al.*, 2008). To remove magnesium sulfate, vacuum filtration was performed.

Physicochemical characterization: Castor oil was characterized by acid value (AOCS Cd 3d-63), iodine value (AOCS Cd 1-25), saponification index (AOCS Cd 3b-76), soap content (AOCS Cc 17-95), peroxide number (AOCS Cd 8-53), relative density, ash content, moisture content and volatiles (AOCS Da-2a-48), dynamic viscosity. The procedures adopted to characterize the methyl and ethyl esters obtained after transesterification were the same used to characterize castor oil. Epoxides of methyl and ethyl esters from castor oil were characterized by means of iodine indices (AOCS Cd 1-25), peroxide number (AOCS Cd 8-53), hydroxyl (AOCS Cd 13-60) and oxirane oxygen (AOCS D Cd 9-57), relative density, ash content, viscosity, moisture content and volatiles (AOCS Da-2a-48). All characterizations described above were performed according to the techniques described by Wu *et al.* (2000) and were performed in triplicate (DANTAS *et al.*, 2021).

RESULTS AND DISCUSSION

Castor oil after purification process was characterized by its physicochemical properties and the results were compared with the legislation in force (BRASIL, 2006; BRASIL, 2021), according to Table 1. The oil moisture was above that specified by ANVISA, and this fact can become a problem for obtaining a quality biodiesel. Ferrari *et al.* (2005) state that these factors cause problems in the reaction, due to canceling the effect of the catalyst, also causing the formation of soap due to the neutralization of fatty acids and interfering with the final quality of the ester. Inorganic compounds that did not undergo combustion are called ash, the percentage of ash was lower than the sampling carried out by Ramos (2021) which obtained the mark of 0.073% with a difference of only 0.023%. The acidity index is within the acceptable standards established by ANVISA, showing due care for the raw material, which makes it an excellent way to obtain biodiesel. According to Araújo *et al.* (2006) the acidity is influenced due to the treatment that the seed is received, so that the quality of storage and care with it will influence the increase or not of the acidity. The acidity index has as main function the verification of the state that the oil is preserved, as it still has a correlation with purity, quality, processing (SOARES, 2003). The iodine index was lower than the value established by ANVISA, due to the breakdown of unsaturation, especially in fatty acids that have more double bonds, as they are easily degraded (MOGHARBEL; FREITAS, 2003). The iodine index is in the same range as Farias (2012).

Table 1. Physicochemical parameters of castor oil

Parameters	Castor oil	ANVISA Standards
Aspect	Dark yellow	Clean and free of impurities
Moisture and volatiles (%)	0.47	≤ 0.1
Ashes content (%)	0.05	---
Density (g/cm ³)	0.980	0.919 – 0.925
Acidity level (mg KOH/g oil)	0.366	≤ 0.6
Iodine index (g I ₂ /100g oil)	111.5	120 - 139
Soap content (ppm of sodium oleate)	0.364	≤ 10
Saponification index (mg KOH/g oil)	212.0	189 - 195
Peroxide index (meq/Kg)	0.037	≤ 10
Approximate molar mass (g/mol)	796	---
Kinematic viscosity at 40°C (mm ² /s)	27.3	---

Table 2. Physicochemical parameters of castor oil esters

Parameters	methyl esters	ethyl esters	ANP Standards
Aspect	Clear yellow	Clear yellow	Clean and free of impurities
Moisture and volatiles (%)	0.15	0.75	0.02
Ashes content (%)	0.049	0.048	0.02
Density (g/cm ³)	0.964	0.958	0.850-0.900
Acidity level (mg KOH/g oil)	0.666	0.342	≤ 0.5
Iodine index (g I ₂ /100g oil)	118.6	135.2	annotate
Soap content (ppm of sodium oleate)	0.01	0.01	----
Saponification index (mg KOH/g oil)	101.6	97.1	----
Peroxide index (meq/Kg)	0.039	0.043	----
Approximate molar mass (g/mol)	828	870	----
Kinematic viscosity at 40°C (mm ² /s)	4.52	4.71	3.0 – 6.0

Table 3. Physicochemical parameters of castor oil epoxides (biolubricants)

Parameters	Methyl epoxide	Ethyl epoxide
Aspect	Light brown	Yellow
Moisture and volatiles (%)	0.22	0.30
Ashes content (%)	0.032	0.027
Density (g/cm ³)	0.999	0.997
Acidity level (mg KOH/g oil)	1.666	1.221
Saponification index (mg KOH/g oil)	284	270
Iodine index (g I ₂ /100g oil)	7.69	11.11
Peroxide index (meq/Kg)	0.060	0.057
Hydroxyl index (mg KOH/ g oil)	23.4	21.2
Oxygen oxirane (%)	9.8	7.4
Kinematic viscosity at 40°C (mm ² /s)	41.5	40.9

In comparison with the literature, the density was higher than expected. Peres (2008), analyzed a sample of castor oil with density between 0.960 and 0.967. Ribeiro (2006) analyzed the kinematic viscosity of castor oil, with a level of 285.0, a value higher than that analyzed in this oil. The high viscosity of castor oil stands out in relation to oils extracted from other oilseeds, due to the presence of the hydroxyl group of ricinoleic acid. For the saponification index, Léo *et al.* (2004) analyzed three oil samples, one reached the level of 247.9 mg KOH/g oil, a higher value compared to the analyzed one, and in another sample the saponification index was around 180.1 mg KOH/g oil, whose value is below the current legislation. In another analysis, the value was 192.4 mg KOH/g oil, within the specified range. The main reason for the high value of the saponification index is that in the reaction process there was a reaction between the oil and water, this refers to the neutralization reaction of the free fatty acids of the oil demanding more KOH. Saponification consists of the formation of lipid with some alkaline inorganic reagent that produces alcohol, glycerin and salt as a product (OLIVEIRA *et al.*, 2006), an extremely important parameter to verify the quality of the raw material (VIEIRA *et al.*, 2018; FERREIRA, 2006). And as the percentage of humidity was above that allowed by Anvisa, it probably may have influenced the saponification process. A procedure to balance this parameter is distillation to remove water, and use of anhydrous sodium sulfate. The transesterification reaction provided a yield of 94.0% for methyl biodiesel and 92.7% for ethyl biodiesel.

The esters obtained from castor oil by the transesterification process were characterized by their physicochemical properties and the results were compared with the legislation in force (BRASIL, 2014), according to Table 2. One of the parameters used to evaluate the quality of biodiesel is the acidity index. The methyl ester presented in the same range as Canesin (2019) with a value of 0.60 mg KOH/g oil. The ethyl ester is suitable with values of 0.30 and 0.32 respectively. The high iodine content is in particular a good parameter for using the ester for epoxide synthesis. The high value will be reduced from the opening of the double bonds in the reaction process of obtaining the biolubricant. Rockembach *et al.* (2014) analyzed the iodine value with a value of 120 g I₂/100g oil, a value that is similar to that of the methyl ester, but lower than that of the ethyl ester. The kinematic viscosity is of paramount importance for biodiesel to have good functionality in engines. The high viscosity is a result of the molar mass and the entire chemical structure, leading to problems for engines, such as pumping, combustion and in the injection system (LIMA *et al.*, 2012). Charles (2019) synthesized a biodiesel whose viscosity reaches 4.30 mm²/s, a value that is similar to the methyl and ethyl esters obtained, and still fits the ANP legislation. According to Escorsim *et al.* (2015) ash are residual remnants of inorganic origin where there was no combustion, resulting from the presence of metals, where these impurities can cause corrosion and problems such as the formation of deposits, and even endanger human health and environmental risk. Comparing with the ANP, the ash content of the esters obtained was higher. Medeiros (2020) in the synthesis of biodiesel from crude oil in the proportion 1:3 (oil:alcohol) obtained a

value of 0.06% for ash, a higher value compared to the esters obtained. A parameter used to evaluate the process of good yield in the formation of biodiesel by transesterification is the soap content, serving as an investigation of the quality control in the reaction process, because when there is not a reaction with due strict quality criteria, soap can be formed by the characteristic of the oil reacting with KOH and alcohol (methyl and ethyl). ANVISA in the Normative Instruction number 49 says that the soap index is a parameter for verifying the amount of sodium oleate resulting from the neutralization of the alkaline catalyst with the triglyceride fatty acid. In this case, as KOH was used as catalyst, potassium oleate will be quantified. Leite (2016) found a total of 0.0 ppm in the sample, a value that coincides with the analyzed samples of the esters obtained. The analysis of moisture and volatiles serves to define the amount of water in the biodiesel sample, this point is linked to the quality of the biodiesel, if it contains moisture, the acidity can be increased, favoring the corrosion of storage components, still favoring the hydrolysis that will produce fatty acids that causes problems in engines (OLIVEIRA, 2021). The ANP establishes a value of 0.05% for moisture and volatiles. With the moisture content and volatiles of 0.340% for the esters compared to Fernandes (2011) who in their samples exceeded the margin established by the ANP, it is concluded that they performed well in relation to this sample. Methyl and ethyl esters have a greater amount of water, because one of the main ways to increase moisture is due to the ease of absorption of water from the reagents, especially the alcohol and alkali used in the transesterification reaction (ESCORSIM *et al.*, 2015). Compared to the work by Oliveira (2021) the peroxide indices (0.039 and 0.041) for methyl and ethyl biodiesel, the esters obtained vary 0.001 and 0.002 respectively. This is a parameter to observe the quality of the synthesized esters. To avoid the degradability of esters and minimize the appearance of peroxides, an antioxidant can be used (ANTUNES JUNIOR, 2017). The density, according to the ANP, must be between 0.850 and 0.900 g/cm³, whose values the esters obtained do not fit. Density is correlated with the molecular weight of fats, so the lower the weight, the lower the density, and also the double bonds between the carbons will also define the density (COSTA, 2015). The saponification index has the function of characterizing different types of oils by the amount of basic matter that will be needed to saponify all the lipid matter of a given sample (VINEYARD; FREITAS, 2014). Canesin (2019) obtained a value of 178.18 mg KOH/g, the lowest value among all the samples, thus showing that the methyl and ethyl esters synthesized from castor oil were efficient in this regard. The epoxidation reaction of the esters obtained from castor oil provided a yield of 91.3% for the methyl biolubricant and 87.6% for the ethyl biolubricant. The epoxides (biolubricants) obtained from castor oil by the epoxidation process of its methyl and ethyl esters were characterized by their physicochemical properties and the results were compared with the literature, as shown in Table 3. The acid value of the biolubricant had an increase compared to the same index of biodiesel, which would require the use of a higher percentage of sodium bicarbonate for an efficient neutralization in the washing process. Also according to Pereira (2022), more KOH would also be needed for the free acids to undergo alkalization and neutralization to occur. Silva (2019) in one sample obtained the acid value of 0.31 mg KOH/g oil and in another sample 0.38 mg KOH/g oil. The epoxide samples showed high values for the acidity index with an average difference of 75.47%. The acidity index is also a parameter that demonstrates the quality of the product obtained and indicates the care and storage of raw materials and the like. In the work by Starling (2016), the highest value for the saponification index was the sample with 248.3 mg KOH/g oil with a lower difference of 35.7 mg KOH/g oil for the methyl epoxide and a difference of 21.7 mg KOH/g oil for ethyl epoxide. Santos (2014) obtained an epoxy with a degree of unsaturation around 1.3 g I₂/100g oil, a lower value compared to the two epoxides obtained, proving to be more effective in the formation of the biolubricant. The low values, compared to castor oil and biodiesel, reflect the breakage of double bonds. Therefore, the decrease in the iodine index shows that there was the formation of oxirane rings by increasing the oxirane oxygen index, one of the essential characteristics for a parameter as a biolubricant. The decrease in the iodine index of the epoxides compared to the esters

that were used for synthesis shows that there was a breakdown of unsaturations, thus favoring the process of formation of oxirane rings (PEREIRA, 2022). As a reflection of the decrease in the iodine value of the epoxides, there was the formation of oxirane rings, as evidenced by the oxirane oxygen index. The oxirane oxygen index was compared to the work by Trajano (2017) in relation to a sample with a percentage of 6.7%, whose value was lower compared to the index of the synthesized methyl and ethyl biolubricants, while another sample was superior to the epoxides. The methyl epoxide compared to the ethyl epoxide obtained by Pereira (2022) is in the same range, showing the efficiency of the reaction process, and despite the lower percentage of ethyl epoxide, there was also efficiency in the synthesis, since values above 6.3% already demonstrate effectiveness (PEREIRA, 2022). Santos (2014) obtained an epoxide with a density of 0.937 g/cm³, a value lower than the epoxides that were synthesized from the methyl and ethyl ester of castor oil. The presence of inorganic compounds that do not undergo proper combustion can add to the biolubricant causing several problems. This percentage is measured by the ash content (MEDEIROS, 2020). Saboya (2016) in their samples obtained an ash content of 0.005%, a lower value compared to biodegradable lubricants, where methyl peroxide obtained a content of 0.032% and ethyl peroxide a value of 0.027%. However, the values described show that there was no contamination of metals in proportions that reduce the quality of the product. Farias *et al.* (2021) obtained a sample with 0.909 meq/kg for the peroxide index, which compared to the epoxides obtained showed a large reduction in this index, showing efficiency in the epoxide synthesis process and good quality of the raw material. One of the parameters that point to the demonstration of the quality of the synthesized product is the peroxide index, important to demonstrate the care with the epoxide and that its storage occurred correctly. This index is of paramount importance, as it shows the degradation of the product by external agents that favor the formation of by-products and free radicals. The free radical produced causes new oxidation reactions, which generates a multiplication of free radicals and peroxides. Peroxide radicals have poor stability decomposing into intermediate products (ZANELA, 2008; MACEDO *et al.*, 2021). Cavalcanti (2016) says that the measurement of peroxide is empirical, as peroxides are unstable and form transient components during oxidation. According to Pereira (2022) an evaluation method to verify if there was a breakage of molecules by the action of water is the hydroxyl index. Marques *et al.* (2016) obtained in a sample a value for the hydroxyl number of 18.01%, a difference of 5.39% for the methyl epoxide and a difference of 3.19% for the ethyl epoxide. The presence of moisture is an important factor to assess the quality status of the epoxide, and is directly linked to acidity and the formation of peroxides. Silva (2019) obtained samples with a moisture content of 0.41%, which exceeds the percentage of samples analyzed for epoxides. If the epoxides are compared, they will be in the same range, with a variation of 0.04% higher for methyl epoxide and lower for ethyl epoxide. According to Macêdo *et al.* (2021), the values obtained demonstrate the efficiency in the purification process. The viscosity index found by Sánchez (2017) of 30.13 mm²/s is lower than that of the synthesized epoxide, reaching the kinematic viscosity in terms of difference of up to 10.37 mm²/s.

CONCLUSION

The biolubricant was obtained in a viable way, in addition to reflecting an alternative means for replacing the lubricants that are obtained from petroleum refining. This may also reflect an ecological vision, as its use would become a tool capable of reducing the need for fossil material; in addition to being cost-effective that provides savings. The high yield of the reaction allows the possibility of expanding the market and making it attractive to obtain. A reflection of this is the ease of obtaining the raw material, of low economic value and which has enormous qualities for its use. With an eye on society, the entire chain would serve as a social transformer, generating employment and income for farmers who depend on castor bean management.

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