



Transesterification and epoxidation of coconut oil

Transesterificación y epoxidación del aceite de coco

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Received: 23/07/2023 – Received in revised form: 12/09/2023 – Accepted: 23/12/2023 – Published: 19/03/2024

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Abstract

Due to the advanced demographic increase in the world, pollution and the gradual increase in the price of oil due to the depletion of natural reserves, it causes us to think and act immediately in the face of a solution to replace said fuel with profitable, productive and eco-friendly options. planet; as is biodiesel from animal and vegetable fats through transesterification. Said raw materials are also tested in the epoxidation from which products are obtained such as: intermediate elements for the production of polyurethanes, lubricants, cosmetics or as PVC stabilizers. This article presents a bibliographical review of similar studies and demonstration on a laboratory scale, where it was verified that coconut oil complied with most of the parameters of the NTE INEN 24:1973 standard, except for the percentage of acidity and moisture, making it optimal. for transesterification and epoxidation processes respectively, also complying with most of the parameters of the NTE INEN 2482:2009 standard, with the exception of the iodine value, which is suitable for the use of alternative energy. Regarding epoxidation, the values varied considerably, categorizing it in ISO viscosity grade 5 for lubricant.

Keywords: demographic, transesterification, epoxidation, PVC

Resumen

Debido al avanzado incremento demográfico en el mundo, la contaminación y el aumento gradual del precio del petróleo por el agotamiento de las reservas naturales, provoca pensar y actuar de manera inmediata ante una solución para reemplazar dicho combustible en opciones rentables, productivas y amigables con el planeta; como lo es el biodiesel a partir de grasas animales y vegetales por medio de la transesterificación. También se prueban dichas materias primas en la epoxidación de la cual se obtienen productos como: elementos intermedios para la producción de poliuretanos, lubricantes, cosméticos o bien como estabilizadores de PVC. Este artículo presenta una revisión bibliográfica ante similares estudios y de demostración a escala de laboratorio, donde se comprobó que el aceite de coco cumplió con la mayoría de los parámetros de la norma NTE INEN 24:1973 a excepción del porcentaje de acidez y humedad, haciéndolo óptimo para procesos de transesterificación y epoxidación respectivamente, cumpliendo también con la mayoría de los parámetros de la norma NTE INEN 2482:2009 a excepción del índice de yodo, siendo éste apto para el uso de energía alternativa. En cuanto a la epoxidación los valores variaron considerablemente categorizándolo en el grado 5 de viscosidad ISO para lubricante.

Palabras claves: demográfico, transesterificación, epoxidación, PVC

1. Introduction

The world population growth causes as an adverse effect the obligation to increase the demand for energy, which is produced mostly by traditional non-renewable sources such as methane, coal and oil, contributing to the increase of CO_x emissions where this gas, which is caused by man-made product of the exploitation of these fossil fuels, generates the greenhouse effect, causing several consequences such as climate change [1].

Due to these unacceptable consequences and the rapid industrialization that exploitation entails, it leads to the depletion of the planet's natural reserves and its contamination. Therefore, the demand for all this leads directly to the search for feasible and acceptable alternatives that are favorable, such as obtaining biodiesel from vegetable oils, animal fats, waste oils, sludge from sewage treatment plants, among other sources. [2].

To obtain biodiesel, the most conventional method used was transesterification to reduce the viscosity of the fat or oil, which consisted of combining the triglycerides contained in the oils and fats with a low molecular weight alcohol

(methanol) to produce a mixture of fatty esters and glycerin in the presence of a catalyst (sodium hydroxide) [3].

Another method used to give added value to oils and fats is epoxidation, in which vegetable oils or their corresponding methyl esters are functionalized through the incorporation of an oxygen atom in the establishment of the fatty acid chain. Those obtained from epoxidized FAME (Fatty Acid Methyl Ester) fatty acid methyl ester have proven to have better properties for industrial application. This method allows obtaining a wide range of compounds, since they function as intermediates for the production of polyurethanes, lubricants, cosmetics or as PVC stabilizers. [4].

In the present work, coconut oil rich in medium-chain-length saturated fatty acids was used. Given its low instauration, it is a very chemically stable fat - 0.1% of its total weight [5]; 6% monounsaturated oleic acid, 2% polyunsaturated linoleic acid, 2% saturated stearic acid, 8% saturated palmitic acid, 7% saturated capric acid, 49% saturated lauric acid, 18% saturated myristic acid, 8% saturated caprylic acid [6].

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Therefore, the objective was to transesterify and epoxidize coconut oil at laboratory scale, characterizing the product, referring to the NTE INEN 2482:2009 standard for biodiesel and NTE INEN 24:1973 for coconut fat, for its subsequent evaluation and categorization as an alternative for lubricant or alternative energy.

2. Materials and methods

Oil previously obtained from dried coconut copra was used as raw material. The experimental stages were divided into: (i) characterization (ii) transesterification (iii) epoxidation [7].

2.1. Transesterification

Before transformation to their corresponding epoxidized ester, the vegetable oils were subjected to a transesterification reaction mechanism where the molecule went from tri to di and monoglyceride, respectively, giving as main product three molecules of fatty acid methyl esters (FAME) and one molecule of glycerol [4].

The technique used was the one described by [8]. The transesterification reaction was developed in a molar ratio of alcohol to triglyceride of 3 to 1, reacting in the methanolysis 1 mole of triglyceride with 3 moles of alcohol, using an excess of alcohol. However, this could generate phase separation problems, decreasing the yield and increasing the production cost.

The optimum molar ratio in basic catalysis is 6:1 (yields > 93%) [9].

According to [10] 27 ml of GR methanol was mixed with 1 g of GR sodium hydroxide (99.9% purity) as catalyst and 100 g of coconut oil was added with constant stirring for 1 hour. Finally, the mixture was allowed to stand for 24 hours to achieve the splitting of the two phases: one biodiesel phase and the other glycerol phase.

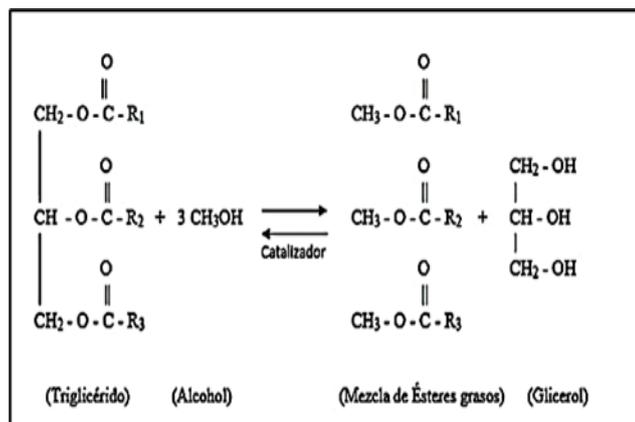


Figure 1. Typical transesterification of a triglyceride with methanol to produce fatty acid alkyl esters and glycerol.

Source: [10]

2.2. Epoxidation

Epoxidation is a method that consists of the functionalization of vegetable oils or their corresponding methyl esters through the incorporation of an oxygen atom in the establishment of the fatty acid chain. [4].

As for the experimentation, 150 g of oil was mixed with 17 ml of formic acid in an Erlenmeyer flask; subsequently, 1 ml of sulfuric acid was added until the mixture reached a yellow to black color. Then, 63 ml of hydrogen peroxide at 30% were added by dripping in a separatory funnel, in a time of 45 minutes.

Finally, the mixture was heated to a temperature of 60 °C and left to react for 3 hours, producing an exothermic reaction; the yellow color gradually disappeared until a white product was obtained.

Once the reaction was neutralized, the water was discarded and consequently the pH was measured, which was 7 (neutral). Finally, the oily phase was washed with distilled water at 60°C until the water was clear.

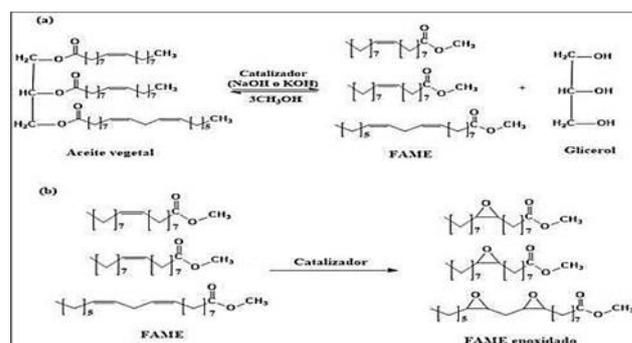


Figure 2. General reaction scheme for the process of obtaining epoxidized FAMES (a) Transesterification of vegetable oils (b) Epoxidation.

Source: [4]

2.3. Physical-chemical tests

The physicochemical properties were determined by laboratory tests based on established methodologies. The specifications and experiments are detailed below, based on the standards set out in Table 1, to carry out the respective quality control of the oil, biodiesel and epoxidized biodiesel oil (lubricant). It is worth mentioning that the tests for each of the physical-chemical parameters were carried out in triplicate.

Table N° 1. Standardized test methods

PROPERTIES	Test Method
IODINE CONTENT	NTE INEN 37
KINEMATIC VISCOSITY 40°C	ASTM D 445-09
RELATIVE DENSITY, 25/25°C	NTE INEN 35
ACIDITY (AS LAURIC ACID)	NTE INEN 38
SAPONIFICATION INDEX	NTE INEN 40
HUMIDITY	NTE INEN 39

Source: [11]; [12]



2.3.1. Iodine Index

Approximately 0.08 g of the samples were placed in an Erlenmeyer flask. Next, 10 ml of chloroform were added to dissolve the fat and 7.5 ml of Wijs' reagent were added, covered and the content was shaken in the dark. Subsequently, the mixture was kept in the absence of light for 1 hour to avoid photo degradation. Then, 10 ml of a potassium iodide solution and 25 ml of water were added. Finally, a titration with the sodium thiosulfate solution was carried out until the total absence of the yellow color produced by the iodine was observed, resulting in the presence of a faint yellow color. Finally, a pinch of starch was added and continued until the exact moment of the color change.

The formula to be used was as follows:

$$\text{Ind } I_2 = \frac{(V - V_1) \times N \times 12.6}{m} \quad (1)$$

Where:

$\text{Ind } I_2$ = iodine value of the sample, in g/g.
 V = arithmetic means of the volumes of sodium thiosulfate solution used in the titration of the test (blank), cm^3
 V_1 = volume of sodium thiosulfate solution used in the titration of the sample, cm^3
 N = normality of the sodium thiosulfate solution.
 m = mass of the sample analyzed, in g.

2.3.2. Density

A 25 ml pycnometer was used to measure the density of the samples.

The relative density will be:

$$d_{25} = \frac{m_2 - m}{m_1 - m} \quad (2)$$

Where:

d_{25} = relative density at 25/25°C.
 m = mass of the empty pycnometer, in g.
 m_1 = mass of the pycnometer with distilled water, in g.
 m_2 = mass of the pycnometer with sample, in g.

2.3.3. Kinematic viscosity

The Ostwald Viscometer was used to measure the viscosity of the samples at 40°C. Its operation was based on the measurement of the time where the fluid traveled a distance between a given space.

The kinematic viscosity is given by the following expression:

$$\eta = \eta_{H_2O} \frac{\rho_m * t'}{\rho_{H_2O} * t} \quad (3)$$

The values of water viscosity for various temperatures are shown below. [13].

η_{H_2O} (millistokes)	1.793	1.522	1.311	1.142	1.006	0.893	0.800
T (°C)	0	5	10	15	20	25	30

Where:

η = kinematic viscosity in mSt
 η_{H_2O} = dynamic viscosity of water in mSt
 ρ_m = sample density
 ρ_{H_2O} = water density
 t' = sample drop time
 t = water fall time

2.3.4. pH

It is important to maintain a regulated pH to neutralize the mixture in the transesterification and epoxidation of the methyl esters. For this, litmus paper strips were used.

2.3.5. Humidity

For moisture determination, the crucibles were washed and placed in the oven for 90 minutes at a temperature of 100_°C. Once dried, the crucibles were weighed on the balance and the sample was added. Next, it was weighed again with the sample and taken to the oven for 2 hours at a temperature of 100_°C. Finally, the crucible with the sample was removed from the oven and placed in the desiccator for 30 minutes. Finally, the crucibles were weighed, and the corresponding data were taken.

La fórmula empleada fue la siguiente:

$$\% \text{ humidity} = [(B - C)/A] * 100 \quad (4)$$

Where:

B = mass of crucible with sample before heating, in g.
 C = mass of crucible with the sample after heating, in g.
 A = mass of empty crucible, in g.

2.3.6. Saponification Index

In an Erlenmeyer flask of 250 ml, 2 g of the sample were weighed, where 40 ml NaOH to 0.081 N were added; later, the sample was heated to 60_°C, and it was shaken at 240 rpm for one hour until saponification. A similar blank assay was performed in all aspects. Once the sample was saponified, it was titrated by adding 4 drops of phenolphthalein indicator.

The formula used was as follows:

$$\text{Saponification index} = \frac{40(V_1 - V_2)(N)}{m} \quad (5)$$

Where:

i = saponification index of the product, in mg/g.
 40 = chemical equivalent of NaOH.



V_2 = volume of hydrochloric or sulfuric acid solution used in the titration of the sample, in cm^3 .

V_1 = volume of hydrochloric or sulfuric acid solution used in the titration of the blank, in cm^3 .

N = standardization of the hydrochloric or sulfuric acid solution.

m = mass of the sample analyzed, in g.

2.3.7. Percentage of Acidity

For the determination of the percentage of acidity, it was made by titration or titration; 2 g of the sample was added in an Erlenmeyer flask with 10 mL of ethanol; later, 3 drops of phenolphthalein were added. The mixture was stirred constantly until homogenized, and by titration 0.081 N NaOH was added dropwise until a pink color was obtained. The formula used was the following:

$$\%A = \frac{M * V * N}{10 * m} \times 100\% \quad (6)$$

Where:

$\%A$ = acidity of the product, in mass percentage.

M = molecular mass of acid used to express the result (200).

V = volume of the sodium or potassium hydroxide solution used in the titration, in

N = normality of the sodium or potassium hydroxide solution.

m = mass of the sample analyzed, in g.

2.3.8. Acidity Index

The formula used was as follows:

$$IA = \frac{56,1 * V * N}{m} \quad (7)$$

Where:

AI = acid number of the product, in mg/g.

56.1 = molar mass of potassium hydroxide.

V = volume of the sodium or potassium hydroxide solution used in the titration, in cm^3 .

N = normality of the sodium or potassium hydroxide solution.

m = mass of the sample analyzed, in g.

3. Results

Table N°2. Characterization of coconut oil.

Chemical physical parameters	Unit	Experimental part	Reference	Specification
Percentage of acidity	%	0.405	0 – 0.2	NTE INEN 38
Acidity index	mg KOH/g of fat	1.14	2 máx	[14]
pH	-	6	-	-
Relative density 25/25°C	-	0.9104	0.907 - 0.919	NTE INEN 35
Kinematic viscosity at 40 °C	mm^2/s	22.69	21.84 - 27.23	[15] [16]
Humidity	%	0.07	0.05	NTE INEN 39
Saponification index	mg NaOH/g of fat	255	250-264	NTE INEN 40
Iodine index	$\text{gI}_2/100\text{g m}$	7.875	7.5 – 10.5	NTE INEN 37

As shown in Table 2, it is observed that most of the parameters are within the permissible range according to the standard established for coconut fat. However, the acidity percentage is not within the range of the standard, but its acidity index to be used as a base material for vegetable oils, as mentioned by [14], which must have an acidity index lower than 2 mg KOH/g of fat, which represents an optimal acidity for the subsequent stages of the process.

As for the percentage of moisture, there was a slight increase in the maximum allowed by the standard, which does not vary much in the transesterification process, since, according to [17], it is very feasible to have values slightly higher than the maximum of the standard, since the probability of saponification decreases, which was verified with the saponification index, which indicates a high purity of the fat.

Table N°3. Characterization of biodiesel.

Chemical physical parameters	Unit	Experimental part	Reference	Specification
Percentage of acidity	%	0.486	0 – 0.5	ASTM D664
pH	-	6	-	-
Density at 15°C	kg/m^3	703.2	880 máx.	ASTM D1298
Kinematic viscosity at 40°C	mm^2/s	1.97	1.9- 6	ASTM D445
Humidity	%	0.048	0-0.05	ASTM D6751
Saponification index	mg NaOH/g of fat	257	370 máx.	ASTM D5558-95
Iodine index	$\text{gI}_2/100\text{g M}$	7.25	120 máx.	EN 14111



According to the values obtained in Table 3, the only relatively low value was the iodine index compared to the maximum acceptable value of 120 mgI₂/g for fuels to be used in internal combustion engines. A low iodine value will result in very low carbon deposits in the internal parts of the engine and the tendency to block the injector orifices, so it is not recommended to be used as an engine fuel, but as a base fuel to be used as alternative energy in subsequent purification and refining processes to increase its autonomy and make it suitable for further use; However, this is also due to the structure of the oil in which 92% are saturated fatty acids, with lauric acid being the predominant one, at 49%, which agrees with the theory that the higher the percentage of unsaturated fatty acids in the sample, the more iodine will react with these double bonds, which ultimately results in a higher iodine content; This is proven by the structure of coconut oil, which has a higher percentage of saturated fatty acids, which is why the iodine value is low, since the remaining 8% is unsaturated fatty acids.

In addition, this characteristic makes it attractive for obtaining biodiesel, since the lower the iodine content in the starting oil, the better the fuel in terms of oxidative stability and increased engine lubrication, which concludes that it is suitable for a lubricant in its subsequent epoxidation process.

Table N°4. Characterization of the epoxidized oil.

Chemical physical parameters	Unit	Experimental part	Reference	Specification
Percentage of acidity	%	0.702	0 – 0.5	ASTM D664
pH	-	3	-	-
Relative density 15 °C	-	0.94	-	NTE INEN 35
Density at 15 °C	kg/m ³	940	880 máx.	ASTM D1298
Relative density 40 °C	-	0.88	-	NTE INEN 35
Density at 40 °C	kg/m ³	880	-	-
Kinematic viscosity at 40 °C	cSt	4.69	1.98-3520	[18]
Iodine index	gI ₂ /100g M	3.15	120 máx.	EN 14111

As shown in Table 4, the percentage of acidity increased compared to the transesterified oil, which in turn exceeds the limit of the standard; one of the causes that provokes this is the oxidation of the oil because in the epoxidation process it incorporates an oxygen atom in the establishment of the

fatty acid, where, this degradation by oxidation increased and in turn also increased the acidity percentage. Due to this addition of oxygen in the epoxidation process, its density at 15°C increased considerably surpassing the limit of the norm; it is worth mentioning that the characterization of the density at 40°C was also carried out to obtain a kinematic viscosity at that temperature, where it is shown that the density reduced by the action of the temperature and that in turn it adjusts to the maximum value of the norm of the density at 15°C. It is also shown that the kinematic viscosity at 40°C was within the reference while the iodine index was below the norm.

3.1. Discussion

The study was conducted to test the theory of epoxidation in which it incorporates an oxygen atom through the unsaturated chain (double bonds) and it was decided to work with coconut oil or fat, which is very rich in lauric acid (saturated) and this theory was affirmed, since the oil or fat did epoxidize because the lowest percentage of this oil (10%) is formed by unsaturated acids, that is to say that 10% of this oil epoxidized correctly and it could be categorized as an ISO grade 5 lubricant.

As for transesterification, according to the results, it serves as a base to be used as biofuel, since due to its low iodine index it cannot be used directly as biodiesel.

4. Conclusions

Biodiesel was obtained from coconut vegetable fat in one stage by using sodium hydroxide as a catalyst, presenting relatively low values required by the standard as: the iodine index, which shows that this biodiesel cannot be used as fuel in combustion engines but it does serve as a raw material for this, and as for the epoxidation it is affirmed with the theory that due to the low percentage of unsaturated fatty acids that the coconut fat has, feasible values were not obtained for this process, but this does serve as raw material for a grade 5 lubricant according to the kinematic viscosity of the ISO.

The oil, biodiesel and epoxy obtained from coconut fat meet most of the parameters established in the standards NTE INEN 2482:2009 for biodiesel and NTE INEN 24:1973 for coconut fat, and its quality depends on these standards.

The optimum time at a temperature of 60 °C for the transesterification was defined that 60 min was the best because it had more combustion time in a test of igniting pieces of paper with the biodiesel and that in the other times of 35 and 45 min it did not have a good combustion stability.

According to its kinematic viscosity of epoxy, it is categorized into ISO viscosity grade 5, which its limits range from 4.14 to 5.06 cSt [18] and where this type of high fluidity and superior quality lubricating oil is specially



formulated to work in high speed and precision systems [19].

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