

Saponification index determines the efficiency in the transesterification process in the production of biodiesel

Índice de saponificación determina la eficiencia en el proceso de transesterificación en la producción de biodiesel

TORRES-RIVERO, Ligia†*, CORONEL-GIRON, Marco, ALCOCER-TORRES, Beatriz L. and BEN-YUSEFF-BRANTS, Sheriff

TECNM/Campus Instituto Tecnológico de Cancún, Mexico.

ID 1st Author: *Ligia, Torres-Rivero* / **ORC ID:** 0000-0002-3303-3437, **CVU CONACYT ID:** 316421

ID 1st Co-author: *Marco, Coronel-Giron*

ID 2nd Co-author: *Beatriz L., Alcocer-Torres*

ID 3rd Co-author: *Sheriff, Ben-Yuseff-Brants* / **ORC ID:** 0000-0003-1468-5365, **CVU CONACYT ID:** IT17B762

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Abstract

For the production of biodiesel from a mixture of used edible oils, the saponification index has played an important role in the elaboration and particularities of biodiesel in its transesterification process by alkaline methanolysis, reaction temperature 35 ° C and molar ratio 6 :1. The objective is to determine the saponification index of used edible oils, from the El Cafecito cafeteria located in the ITCancún, to prevent the free fatty acids from the transesterification process from saponifying, being the causes of saponification the excess of catalyst, or Due to poor treatment in the elimination of the water content in the used edible oil samples, it considerably affects the biodiesel process. The methodology used was based on the analysis of national and international standards in the Determination of the Saponification index by means of the Covenin 323 standard of the NMX-F-174-SCFI-2014, -AOCS Cd 3-25, to the 6 samples of used edible oils. The results of the saponification index test are not acceptable since to saponify said raw material requires a large amount of potassium hydroxide. The contribution is to prevent used edible oils from contaminating the water table, due to its poor disposal.

Saponification index, Biodiesel, Used edible oils

Resumen

Para la producción de biodiesel a partir de una mezcla de aceites comestibles usados, el índice de saponificación ha jugado un papel importante en la elaboración y particularidades del biodiesel en su proceso de transesterificación por metanolisis alcalina, temperatura de reacción 35°C y relación molar 6:1. El objetivo es determinar el índice de saponificación de los aceites comestibles usados, procedentes de la cafetería El Cafecito ubicado en el ITCancún, para evitar que los ácidos grasos libres del proceso de transesterificación saponifique, siendo las causas de la saponificación el exceso de catalizador, o por mal tratamiento en la eliminación del contenido agua en las muestras de aceite comestibles usados, afecta de forma considerable al proceso del biodiésel. La metodología usada se basó en el análisis de las normas nacionales como internacionales en la Determinación del índice de Saponificación mediante la norma Covenin 323, de la NMX-F-174-SCFI-2014, -AOCS Cd 3-25, a las 6 muestras de aceites comestibles usados. Los resultados de la prueba de índice de saponificación no son aceptables ya que para saponificar dicha materia prima requiere de una gran cantidad de hidróxido de potasio. contribución es evitar que los aceites comestibles usados lleguen a contaminar el manto freático, por una mala disposición de este.

Índice de saponificación, Biodiesel, Aceites comestibles usados

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* Correspondence to Author (E-mail: ligia.tr@cancun.tecnm.mx).

† Researcher contributing as first author.

Introduction

Currently in Mexico, the lack of regulation for the proper disposal and management of used edible oils, has made the business of cheap kitchens not a system of good disposal and storage of their residual oils, as in homes, school cafeterias. They regularly dump them down the kitchen sinks and drains or, failing that, because they have been requested to do so, they are stored in containers that are not suitable, they may contain some residue, or after water, they take them to collection centers or to hire a company that goes through such wastes, see figure 1. It is considered that the properties depend on their temperature high caloric power of these used edible oils turn out to be a potential for the production of biofuels. Table 1 indicates the amount of oil collected for the development of this work

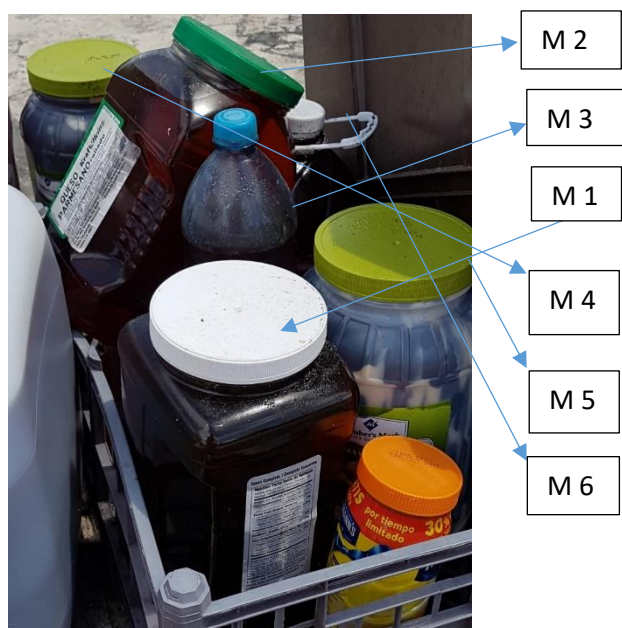


Figure 1 Disposition of used edible oils, in different types of containers, that they have at their disposal
Source; Own source

| Oil collection (Samples) | Quantity | Remarks | Container |
|--------------------------|-----------|--|--|
| M 1 | 8 liters | Waste, black, musty odor, leaked from large amount of food waste | Plastic container, contained candy |
| M 2 | 8 liters | Unpleasant odor, black color, | Plastic container, residue contained candy |
| M 3 | 10 liters | Severely burned | 2.5 l soft drink container |
| M 4 | 8 liters | Honey colored, low residue | Recent dark colored plastic |
| M 5 | 8 liters | Honey color, residues, unpleasant odors | Container contained juices |
| M 6 | 8 liters | Brown color, coarse residue The oil was filtered. | Mayonnaise and juice plastic container |

Table 1 Oil collected in the Cafecito del ITCancún for 4 weeks, 2 liters of oils were collected, one week we collected up to 15 liters of oil distributed in the 6 samples, which was used to determine the IS (index saponification) parameter that we had not considered, to produce Biodiesel

The spills in the kitchen sinks cause the obstruction in the plumbing system, due to the adhesion of the fats, the residues end up in surface water bodies generating contamination of said bodies. Pan American Health Organization PAHO (2019) Others are dumped into bodies of water, wetlands, cenotes since there is no place to dispose of them or any company that collects these oils, this problem generally occurs in areas of irregular settlements, being one of the main pollutants in aquifers concerns oils and fats from urban areas. Taking into account that 1 L of oil contaminates around 1000 L of water. The effects generated by this pollutant are the proliferation of bad odors, infestation of excretions, damage to the ecosystem. Used cooking oil has to undergo a previous treatment in order to be transformed into biodiesel.

The treatment necessary for this conversion begins with the filtration and decantation of the oil, its objective is to eliminate any possible solid food residue in the oil and then it is.

We proceed to eliminate the water content that it may have. Filtering is done with filters for coffee makers. Being of great importance that the oil does not contain water residues since during the alkaline reaction it becomes soap and a complete reaction does not occur to produce biodiesel, figure No.2.

Biodiesel Production is currently one of the best resources available, given that the food industry and the hotel and restaurant sector generate daily



Figure 2 Excess alkali in the transesterification process, soap formation

Source: Own source

High volumes of used vegetable oil, forming a problem from environmental perspectives, it has been estimated that each liter of edible oil that is discharged into the drain contaminates 1'000,000 liters of water (UNAM, 2016).

For this reason, the edible oil used is an excellent raw material in the elaboration of Biofuel with the processes of esterification, acid treatment, transesterification, alkaline treatment, and neutralization to obtain a biodiesel that complies with international parameters and standards.

Biodiesel is a highly biodegradable and water-friendly biofuel, much of it mineralizes in up to 30 days under aerobic or anaerobic conditions, it is also a carbon-free biofuel, since plants and seeds serve as raw material. For their production, they absorb all the carbon from which it is released during the burning of this Esquivel J. (2014), Durán, Torres, Sanhuenza, 2015 mention that the properties of the vegetable oils used depend on the type of oil, it is generally a low-cost oil, duration of cooking, temperature of the oil to which it is subjected during the frying process, time of exposure to air, storage period and conditions in which they are stored and sometimes have content of water and other types of substances such as the type of cooked food.

This does not lead to the transesterification process not being carried out satisfactorily, generating soaps due to water content, or the formation of an excess of glycerin formation, see figure no.3



Figure 3 Formation of soaps by water content when doing the alkaline treatment

Source: Own source

Methodology

Raw material

It is obtained in various places to obtain biodiesel, currently oil-producing algae and sargassum are being handled that in recent years have landed on the coasts of Mexico, the Caribbean Sea, used cooking vegetable oils and fats, it is obtained from school cafeterias, inexpensive kitchens.

Physicochemical properties of the oil

Oils are characterized according to their physical properties: density, viscosity, melting point, refractive index, flash point, moisture index or chemical: acid number, iodine number, peroxide number, saponification number, unsaponifiable material.

Physical properties

Density:

According to the EN-14214 standard, density is defined as the quotient between the mass of a body and the volume it occupies.

Calculations

$$\rho = R + .723 * (T^\circ - 15)$$

Where:

ρ = Density (Kg / m³)

R = Hydrometer liquid level recorded (Kg / m³)

T ° = Sample temperature (° C).

Kinematic viscosity

This method consists of measuring the time that takes a given volume of the liquid to flow through gravity through a calibrated glass capillary type viscometer at a temperature viscometer, C, by means of the following equation:

$$V_{1.2} = C * t_{1.2}$$

Where:

V_{1, 2} = Kinematic viscosity values determined for v₁ and v₂ (mm² / sec)

C = Viscometer calibration constant (mm² / sec)

Melting point

Melting point (in open capillary tubes); slip point: temperature at which a column of fat in an open capillary tube begins to rise under the conditions specified in this Colombian technical standard NTC 213.

The immersion, in water or oil to a specific depth, of the capillary tube containing a column of fat, which has been crystallized under controlled conditions. The temperature is increases to a specific degree. The temperature at which the fat rises in the column.

Chemical Parameters

Saponification

The triglyceride reacts with the basic catalyst, consuming it, in the presence of water, giving rise to the formation of soaps (saponification reaction) when the amount of the catalyst has not been adequate, whether the catalyst is in excess and it has absorbed humidity from the environment, as can be seen in the chemical reaction as indicated stoichiometrically, see figure 4:

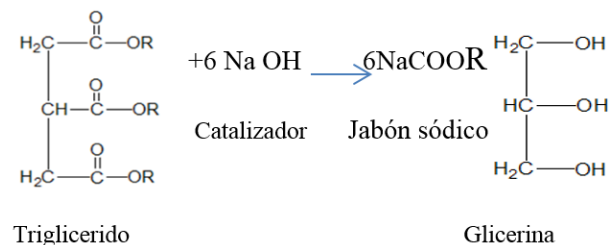


Figure 4 Stoichiometry of the reaction in soap formation
Source: Chang Raymond

Saponification index

The saponification value or index of an oil is the number of milligrams of potassium hydroxide (KOH) needed to completely saponify 1g of oil. Since oils are mainly made up of triglycerides, and each triglyceride needs 3 KOH molecules to saponify, the saponification index can be used to roughly estimate the average molecular weight of the oil used.

Saponification is essentially based on an alkaline hydrolysis of the glyceric esters of fatty acids present in a fatty substance, with the consequent formation of a soap. The above reaction is carried out in practice by the hot action of alcoholic potassium hydroxide, approximately 0.5 N, in sufficient quantity to achieve complete saponification. Uncombined excess alkali is determined by acidimetry in the presence of a suitable indicator, simultaneously a blank is performed for calculation purposes. Timberlake Karen C, 2013

Unsaponifiable material

The unsaponifiable material comprises all the compounds contained in the oil or fat that do not react with KOH to produce soaps, that is, they are not fatty acids or glycerides but are soluble in organic solvents. Among the unsaponifiable materials most found in oils and fats are different compounds that contain phosphorus, such as Phospholipids and phosphates. If the oil has a high phosphorus content, emulsions are formed (see figure no. 3 during the decantation process, separation of glycerin after transesterification and during biodiesel washing, which ultimately leads to losses in process performance.

A control of this parameter must be kept in each batch of oil that is taken to the collection center to produce biodiesel.

Oil pretreatment

There is a large amount of lower quality and lower cost oils and fats generally used in cheap kitchens, school cafeterias, crude vegetable oils, animal fats (lard) that become used oils, used in the production of biodiesel. The problem with processing this type of cheap raw material is that they usually contain free fatty acids, gums, humidity and other impurities that affect the alkaline transesterification process, since they present greater problems of emulsion, the washing process, drying, to eliminate the gums to avoid emulsions in the process, eliminate phosphates to reduce treatment costs and time, eliminate free fatty acids, performing the acid number analysis to facilitate the transesterification process based on the purification of the glycerin obtained.



Figure 5 Formation of emulsions in the process of transesterification of used edible oils, due to excess phosphates, presence of gums that were not eliminated with the washing treatment

Source: Own source

The raw material used edible oil provided by the Cafecito cafeteria, approximately 50 liters were collected which we use in the production of biodiesel in a period of one month as indicated in table No. 1, it was first filtered by gravity using a double coffee machine filter which allows to retain solids contained in the oil of up to 25 μ m, and to facilitate filtering, it was subjected to heating at a temperature of 80 °C for 30 minutes to reduce the viscosity, as well as eliminate the water contained in the oil. Once the oil was filtered, we proceeded to characterize it and for this we proceeded to prepare the assembly of the experiments according to the following Mexican Standards (NMX) and / or international standards, ASTM and AOCS Ca 40, Covenin 323, OACS Cd 3-25 IRAM 5517/88 NMX-F-174-SCFI-2014, to choose the one that we would use before proceeding with the production of Biodiesel, and we do not have soap production when the alkaline catalyst is added. Six samples that were collected in different weeks from the cafecito cafeteria located in the ITCancún were analyzed. From which a 100ml sample was taken from each one and given the filtering treatment to remove food remains, and the presence of any other substance present and washing to remove gums See figure 5



Figure 6 Treatment of oil samples, to determine the saponification and unsaponifiable matter indices
Source: Own source

The figure no. 6 indicates the amount of sample used after the treatment carried out to determine the saponification index.

Where the saponification index is inversely proportional to the value of the molecular weights of the fatty acids of the glycerides present in oils or fats. which is defined as the number of mg of KOH needed to saponify 1g of fat, it should be noted that it is not totally accurate for appreciate the molecular weight, since free fatty acids are included together with glycerides (OACSCd 3-25).

The Saponification index refers to the amount of potassium hydroxide expressed in milligrams, necessary to saponify a gram of oil or fat. To determine this parameter if you referenced the NMX-F-174-2006 standard, the method is based on the chemical reaction of triacylglycerols or triglycerides with an alkali, forming soaps or alkaline salts of fatty acids and glycerin. Saponification is used with potassium hydroxide, since its molecules contain the OH groups responsible for this reaction. In any case, essentially anhydrous oils and alcohols should be used since water favors the formation of soaps by saponification.

For this reason, the water must be removed, by evaporation, from the oils with high moisture contents before carrying out the transesterification. On the other hand, there are two ways to remove the free fatty acids present in the oil.

One by neutralization, since the fatty acids present in the vegetable oil can react with the basic catalyst NaOH in the presence of water, an undesirable reaction also occurring, producing soap as in the previous case, as can be seen in figure 3. Another is to eliminate the free fatty acids is carried out by the esterification reaction with an acid catalyst (H_2SO_4) with which the methyl ester is formed.

For the determination of the saponification index, the sample is heated under reflux with an alcoholic KOH solution for the time specified in the revised standards where the selected conditions depend on the difficulty of saponifying the sample. Once the sample is saponified, it is titrated until the end point of the retro valuation titration, which is clearer than that of the rapid direct titration. The equivalence point of the titration is determined by means of a pachymeter or, failing that, with pH paper strips, it greatly improves the repeatability of the tests.

The alcohol used is ethanol, it allowed us to manipulate the reaction temperature, the increase in the normality of KOH (0.5N) allows us to use saponification conditions, the increase in reaction time, to have quantitative saponification's.

To do this, the reagent is added to the dissolved sample, and it is allowed to react for 10 min and $100^\circ C$ for potassium hydroxide, heated under reflux for saponification for approximately 2 hrs or it can be less than $100^\circ C$. After the reaction is complete, a reagent is added to prepare the sample for titration. With .5M HCl, adding drops of phenolphthalein, a 5 g low-cost virgin sunflower oil blank was prepared with 25 ml of KOH, titrated with HCl at .5 N, with phenolphthalein until color change. See figure No.8.



Figure 7 Reflux system for 2hrs of the sample with KOH

Saponification index: the analysis of this test was evaluated according to the Mexican Standard NMX-F-174-2014, 5 g of the samples were weighed, 25 ml of potassium hydroxide in alcoholic solution were added exactly measured with a volumetric pipette. A reflux condenser was adapted as shown in figure no.7, and it was placed in a boiling water bath for 60 minutes, stirring frequently. Once the 60-minute saponification was finished, 1ml of 1% phenolphthalein indicator solution was added, titrating it cold, with 0.5N hydrochloric acid. The end point of the titration was determined when the solution solidified due to the formation of soap, change of coloration, as indicated in the following figure No. 8 A control test is made using the same amount of reagent, the saponification index is calculated based on the following equation for the case of the three standards

$$I_s = \frac{B - M) \times (N)_1}{P} \times 56.1$$

Where:

B is the volume, mL 0.5 N HCl required to titrate the blank

M is the volume, mL 0.5 N HCl required to titrate the sample

N is the normality of the HCl solution

P is the weight of the sample in grams, and 56.1 is the equivalent of potassium hydroxide

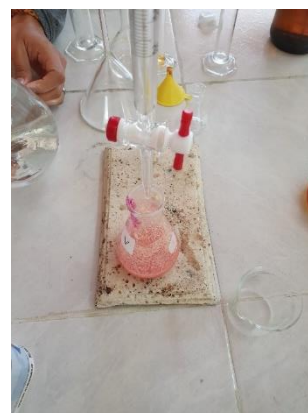


Figure 8 Titration of the sample with HCl with phenolphthalein color change, soap formation, sample no. 5

Source: Own source

The acid number of the reaction oil is determined to evaluate the amount of alkali to be added. In the process, it must be neutralized to a slightly acidic pH, in a range between 5 and 6, to avoid the formation of soaps that can affect the separation of the phases.

Results

The results obtained from the Saponification index parameter of the 6 samples of used edible oil from the cafecito cafeteria of ITCancún, 3 of the samples formed soaps, when subjected to the reflux process for 2 hours at 100 ° C in a Kjeldahl apparatus to that the heat is homogeneous for the sample, see figure 8. They were evaluated using the methods of the Official Association of Analytical Chemists (AOAC, for its acronym in English), Covenin 323, OACS Cd 3-25, NMX-F-174 -SCFI-2014.

The AGL reacted with the alkaline catalyst to produce soap and water, inducing the formation of emulsions. The presence of water can also cause the ester to saponify at alkaline pH. These reactions increase the amount of catalyst required to carry out alkaline transesterification and make biodiesel recovery and purification processes more difficult.



Figure 9 Reflux system in the Kjeldahl apparatus in a time of 2 hrs, T = 100 ° C, samples 1, 5 formed soaps from the heat treatment

Source: Own source

The evaluation of the samples by titration with HCl .5M with phenolphthalein indicator, three replications were made, at the time of adding the phenolphthalein indicator, the titration changed from pink to completely white, as shown in the following figure no. 10 after adding phenolphthalein and titration, a white precipitate formed as shown in the figure, the volume used of 30 ml.

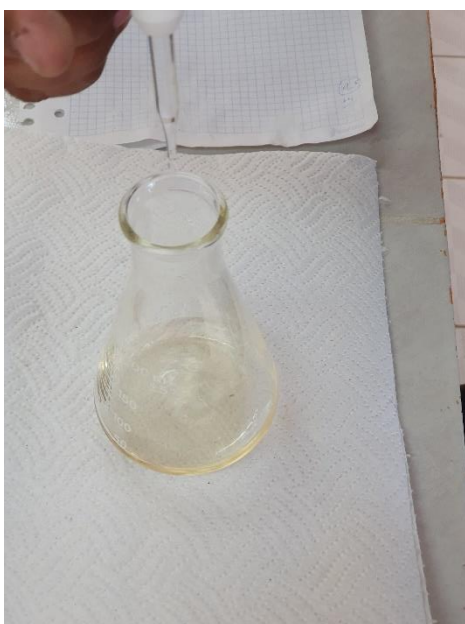


Figure 10 Titration of samples 1.5 with 0.5M HCl with the indicator, to indicate the change of vire, presents solid particles, which now of titration are completely white

Source: Own source.

When carrying out a search on the maximum permissible limits on the saponification index, and the composition of fatty acids, their limits are not set by virtue of being in this case a variable mixture of different oils, in the case of the standard. Different bibliographies manage that for saponification they have been manipulated compounds where the products are known, according to the theoretical value of the saponification index of 345 and 508 mg KOH / g sample respectively. The value of the saponification index is determined by means of the following formula:

$$I_s = \frac{B - M) \times (N)_1}{P} \times 56.1$$

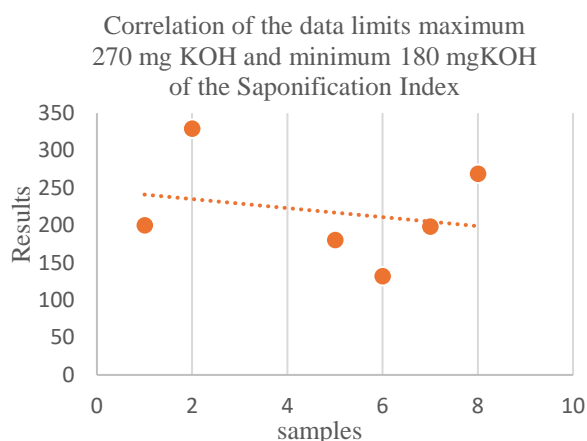
This parameter being of utmost importance since it determines the presence of humidity. It could not be carried out, due to pandemic conditions, since the yield of the reaction decreases, since the water reacts with the catalyst forming soap. Soaps are harmful because they contaminate the final product, and because they form very stable emulsions. For this reason, the least amount of water possible must be ensured during the process, which implies drying of the oil, which is more demanding when using used oil.

| STANDARDS | RESULTS KOH mg/L | | | |
|---------------------|---|-----|-----|--|
| OACS Cd 3-25 | 260 | | | |
| | 360 | | | |
| | 176 | | | |
| | 125 | | | |
| | 90 | | | |
| | 260 | | | |
| Covenin 323 | 210 | | | |
| | 350 | | | |
| | 190 | | | |
| | 126 | | | |
| | 189 | | | |
| | 269 | | | |
| NMX-F-174-SCFI-2014 | 200 | | | |
| | 329 | | | |
| | 180 | | | |
| | 132 | | | |
| | 198 | | | |
| | 278 | | | |
| White | 185 | 180 | 182 | |
| | OACSCd3-25 Covenin 323 NMX-F-174-SCFI-2014 | | | |

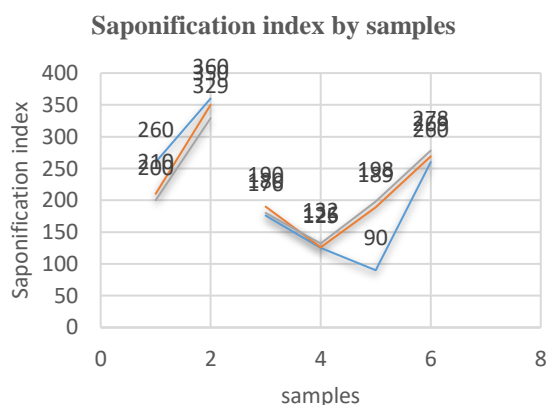
Table 2 Results obtained through the analysis carried out by the different international standards, such as the Mexican one, of the 6 oil samples from the Cafecito cafeteria of ITCancún, which does not show that the results of the 4 standards are very similar between. it is.
Source: Own source

The following table no2 shows the results obtained for each sample of the oil collected from the Cafecito cafeteria of ITCancún by the standards used to choose which one is the best adapted to our experimental development. In the characterization of the physical-chemical parameters and to obtain a better quality of the biodiesel.

The following graphs show us the behavior of each sample with respect to each compared to the behavior of the results of the determination of the saponification index, to obtain a complete transesterification process without the formation of emulsions or soaps.

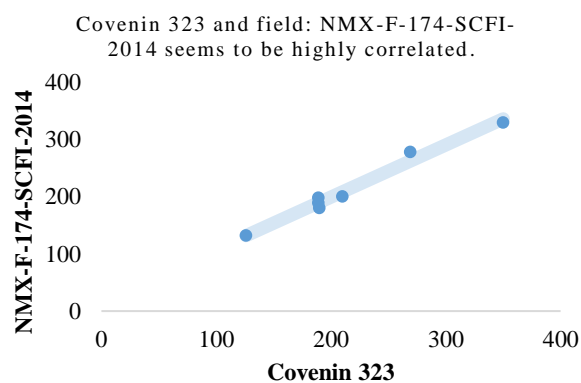


Graphic 1 comparison of the results obtained from the 6 shows with respect to the blank indicating maximum limits of 270 mg KOH and minimum of 180 of minimums. In accordance with the Ecuadorian regulation, of the physicochemical parameters in the production of biodiesel
Source: Own source



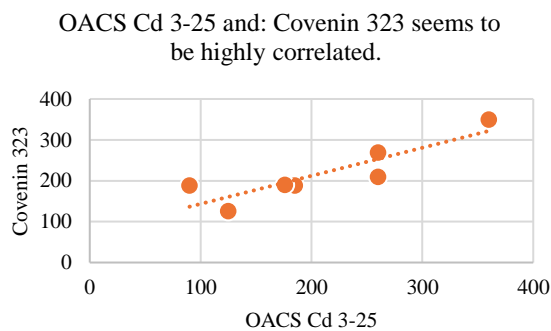
Graphic 2 The behavior of the 6 samples is observed with the data in relation to maximum and minimum limits
Source: Own source

We observe in the graph no. 2 that sample 5 has the lowest saponification index, which presents an excellent transesterification reaction, and therefore a good biofuel was obtained, with a molar ratio of 6: 1, reaction temperature of 40 ° C. In the graph no. 3, we establish a relationship between the Covenin 323 standards and field: NMX-F-174-SCFI-2014. We observe that the use of the methodology proposed in both standards we obtain similar results as well as the behavior in the treatment of each sample.



Graphic 3 According to the results obtained when using the methodologies proposed by each standard, we observe that the results are similar in 350 and 329, it may be the number of reagents used in each treatment. In the 200 and 210KOH mg data the difference is minimal
Source: Own source

When we compare the standards OACS Cd 3-25 and field: Covenin 323 we observe as in the case of the standards Covenin 323 and field: NMX-F-174-SCFI-2014, there are results that coincide 185, 188 KOH mg, although in both they have scattered points of the samples, 90, 125 KOH mg as indicated in graph number 4



Graphic 4 We can see that the standards that we choose to determine the saponification index are adequate, and they give us the same result, with minimal differences, with a similar behavior
Source: Own source

The saponification index refers to the probability that a residual oil turns into soap, therefore, the higher the saponification index, the greater the probability of the presence of soap in the final transesterification product. The result obtained from the saponification index shows a decrease of almost 50% compared to the index of the oil with the highest presence in the raw material used in this project. This result allows us to intuit that our performance in obtaining biodiesel will be high as a consequence of the low soap index. Similar values were also observed with the work of Enweremadu & Mbarawa (2009), which report values of 0.921-0.937 as specific gravity and 193.9-204.3 for saponification index

Gratitude

I wish to express my gratitude TECNAM / Campus ITCancún for the support it has provided for this work, in the use of the Water Laboratory and Chemistry laboratory

Conclusion

Saponification is a chemical reaction; the main product is a salt. constitute a very useful process for the transformation of fats and oils into soaps. The index that is calculated serves to assess the necessary quantities of potassium hydroxide to be used. It contains the edible oil used to produce biofuel and glycerin, we cannot fully deduce about the quality of the oil that has been used in the Cafecito cafeteria since they They take it to the laboratory for the production of biofuel, the used edible oil shows that it presents a higher degree of saturation in samples 2 and 6, since you do not know the conditions in which it was stored.

Regarding the maximum and minimum limits, assuming that the oil in the samples is girasol, la bibliografía consultada reporta que índice de saponificación 188,194 (mg KOH/g de aceite), ya que los aceites comestibles usados se they try a mixture of different types of oils, so each. Saponification index NMX-F-74-2006 90mg of KOH with respect to the author 190 mg of KOH (Bejumbea, 2003)

The soap content constitutes a parameter that evaluates the quality of the oil or fat and the edible oil used is a mixture of many oils. It is said that if an oil does not contain soap it has good quality, in this case the analyzed oil will obtain a better quality of the biofuel. Due to epidemiological conditions, acidity index, unsaponifiable matter could not be determined. Any of the techniques used in the determination of the Saponification index we obtained results that national and international standards with small adaptations can reach the same result in terms of the quality of biodiesel.

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