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Research Article

Evaluation and Characterization of Biodiesels Obtained Through Ethylic or Methylic Transesterification of Tryacylglicerides in Corn Oil

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Abstract: This work was devoted to the transesterification of corn oil either with methyl or ethyl alcohol and to the characterization of the biodiesels (composed by FAME—fatty acid methyl esters—or FAEE—fatty acid ethyl esters, respectively) produced. As an initial hypothesis, it was argued whether or not the two alcohols, both with short molecular chains, would impart significant differences to the chemical characteristics of the two biodiesels from corn oil. The most common properties of the biodiesels were evaluated by determining corresponding parameters for acid value, peroxide value, water content, oxidative stability, free and total glycerin, kinematic viscosity at 40 °C and density at 20 ºC, for both chemical routes, FAME and FAEE. In general, values were found to be well within the recommended limits for commercial biodiesel, in accordance with the Brazilian, European and American standard recommendations, except only for the oxidative stability. The methyl biodiesel presented acidity of 0.08 mg KOH/g; peroxide index, 23.77 meq/kg; oxidation stability, 3.10 h; water content, 297.1 mg/kg; total glycerin, 0.092 %; free glycerin, 0.009 %; viscosity, 4.05 mm^2/s and density, 878.7 kg/m. The methyl biodiesel presented acidity of 0.11 mg/ KOH; peroxide index, 22.39 meq/kg; oxidation stability, 2.13 h; water content, 264.8 mg/kg; total glycerin, 0.25 %; free glycerin, 0.02 %; viscosity, 4.37 mm^2/s and density, 874.0 kg/m. From a direct inspection of chemical data for the two products prepared via the two chemical routes, it can be drawn that values of the physical and chemical parameters for both, methyl and ethyl biodiesels, are essentially similar, except for the oxidative stability. However, the oxidative stability can be suitably adjusted by adding an anti-oxidizing agent to the ethyl biodiesel medium. The two biodiesels are thus

promising alternatives to fully replace or to be admixed to the mineral diesel. Relatively to the pure petrol diesel, the resulting admixture would clearly gain towards preserving the natural environment by reducing the emission of harmful gases that would also direct and significantly affect the human health.

Keywords: Biofuel processing; transesterification; physico-chemical properties; biodiesel-diesel mixture

1. Introduction

The extraction, production and final use of petroleum—based fuels are known to have negative effects on the natural environment and the global climate [1]. The environmental consequences of its use have been progressively affecting the mankind welfare and lifestyle. Smokes released to the atmosphere and the plethora of hardly degrading materials being industrially and widely disposed to the environment have been continuously imparting local chemical and physical changes to the Earth natural equilibrium. The most frequent anomalous meteorological events such as strong winds, extreme temperatures, variations of the sunlight spectrum on the Earth surface, big marine waves and currents are assumed to be to a significant extension caused by such an imbalanced ecological system [2, 3].

Biofuels are thought to be interesting alternatives in the sense that they are renewable sources of energy and that they could help mitigate the hazardous effects to the environment coming from gas emissions on their burning, relatively to petroleum fuels. Regarding fuels from biomass, the released carbon dioxide to the atmosphere, as from the combustion of biofuels in mechanical engines, is re-trapped by the cropped plants and by other photosynthetic organisms, as green algae, to produce the precursor biomass and to cyclically produce biofuels. However, arguments based on benefits to the environment, relatively to the use of mineral fuels, have not been enough to completely prevent criticisms and many warns, particularly those related to the production of biomass destined to fuels in competition to those directly destined to foods for the human needs [4].

Biofuels can be broadly understood as being renewable fuels derived from biomass. Biodiesel is a biofuel that can be prepared from vegetable oils from seeds or pulps of many fruits, including corn, sunflower, soybean, castor and babassu, or oils from animal fats [5, 6]. Its use replacing the mineral diesel is thought not only to reduce gas emissions to the atmosphere by about 78 % of carbon dioxide (CO_2) and 50 % carbon monoxide (CO) but also 50 % of solid particles [7].

In Brazil, soybean oil responds for as much as 73.9 % of the precursor biomass for the industrial production of biodiesel [5, 8, 9], but seeds or fruits of many other cropped or native plants are being considered to be economically valuable as oil source. Corn is one of them. The grain may contain largely variable contents of proteins (lets say, in average, ~10 %), oil (~5 %) and carbohydrate (~70 %), depending on the maize variety, post-harvest management, climate and cropping conditions. It may be advantageous to consider the starch or the oil as starting materials for the industrial production either of bioethanol, by hydrolyzing the starch and fermenting the resulting oligosaccharides, and of biodiesel, by transesterificating the triacylglycerides of its oil. The protein-rich by-product is often destined to feed domestic animals. In average, about 172 liters of oil may be obtained for each hectare of a typical field of cropped maize, which represents from the

economical viewpoint a real potential for biodiesel production [10-13].

This article reports on a work aimed at synthesizing biodiesel in the laboratory through alkaline transesterification of tryacylglicerides in corn oil and at characterizing the corresponding mixture of esters formed through either chemical reaction methyl or ethyl alcohol.

2. Materials and Method

2.1. Production of the biodiesel via transesterification

The conversion of triglycerides of commercially refined corn oil to esters was made via the transesterification reaction either with methyl or ethyl alcohol.

Initially, the potassium methoxide was obtained by mixing 30 g methanol with 1 g potassium hydroxide (KOH). For a better homogenization, the mixture was placed in an ultrasonic bath for about 10 min.

Then, the mixture of potassium methoxide was mixed to 100 g of corn oil; the mixture was then placed on a plate with a magnetic stirring rod, in order to allow the transesterification reaction to proceed for 60 min, at the room temperature.

The same procedure was followed for the ethylic transesterification reaction, except for using 40 g of ethyl alcohol in place of the methyl alcohol.

2.2. Acidity Index

The acidity analysis was performed according to ASTM D-664 [14], which consists of titrating 3 g of sample in 30 mL of a solution of 1:1 (v:v) toluene:ethanol with 0.1 mol L^{-1} KOH. The end point was monitored with the aid of the titrator software, equipment model Auto Titrino Plus of the 848-Metrhom Pensalab instrument.

2.3. Peroxide index

5 g of sample was put into a 100 mL-beaker. 30 mL of a 3:2 (v:v) acetic acid:chloroform was added and stirred until complete dissolution of the solid sample. 0.5 mL of saturated KI solution was then added and the whole system was put protected from light for 1 min. 30 mL of water was added and the solution was put in the automatic titrator under constant stirring. The value of the sample mass was entered as a required datum to the instrument software. A blank sample was prepared the same way as described except for the step of adding the solid sample.

Readings were done with an automatic titration apparatus model Metrohm 848 Titrino Plus – Pensalab, by using a 0.0992 mol/L thiosulfate solution, which was standardized with potassium dichromate. The end-point was indicated by a potentiometric platinum electrode.

So far, there is no recommended value for the peroxide index for biodiesel. Consequently, the ANVISA recommended index for oils and fats [15] was used in the present work.

2.4. Determination of Oxidative Stability

This analysis was performed according to the standard EN 14112 [16]. The measure of stability

to oxidation was performed with a Metrohm 873 Rancimat equipment coupled to a 873 Rancimat Control software.

2.5. Water Content

The water content of the biodiesel was determined according to the ASTM D 6304 Karl Fischer method [14], with Metrohm AG equipment, based on the following equation:

Ek, F = (5.6 × VKF × 100)/m

For which:

Ek, F = Equivalent water Karl Fischer reagent, in mg/mL

5.6 = standardized mass water for every 1 mL of Karl Fischer reagent (mg)

VKF = volume (mL) of the Karl Fischer used in titration

M = mass (g) of the sample

2.6. Free and Total Glycerin

Free and total glycerin fractions were determined according to the methodology described by Pisarello and co-workers [17]. The glycerin produced from the transesterification reaction under reflux followed by alkali neutralization was extracted with hot water. Contents of glycerin resulting from the transesterification reaction were determined via neutralization titration with a standard solution of sodium hydroxide. The free glycerin was determined the same way as above except for the transesterification step at reflux.

2.7. Viscosity at 40 °C

Viscosities for both methyl and ethyl corn biodiesels were determined according to the ASTM D-445 method [14]: the sample was stir-homogenized in the original bottle and filtered through a 75-micron (200 mesh) sieve. Subsequently, the sample was analyzed with an ISL Instruments viscometer.

3. Results

Results of physicochemical characteristics for both, methyl and ethyl biodiesels, are shown in Table 1. The recommended standard limiting values for each of these characteristics, according to ANP [14], ASTM [15] and EN [16] are correspondently presented in Table 2.

Table 1. Physico-chemical characteristics for the methyl- and ethyl-biodiesels from corn oil.

Characteristic	Unit	Method	FAME	FAEE
Peroxide index	meq/kg	ASTM D-1563	23.77	22.39
Oxidative stability	h	EN 14112	3.10	2.13
Viscosity	mm^2/s	ASTM D-445	4.05	4.37

Density	kg/m	EN 14214	878.7	874.0
Acidity Index	mg KOH/g	ASTM D-664	0.08	0.11
Free Glycerin	mass%	ASTM D-6584	0.01	0.02
Total Glycerin	mass%	ASTM D-6584	0.09	0.25
Water Content	mg/kg	ASTM D-6304	297.1	264.8

Table 2. Recommended limiting values for these characteristics according to ANP [14], ASTM [15] and EN [16].

Characteristic	**Unit**	**Limiting values**		
		ANP 14/2012	EN 14214	ASTM D6751
Aspect		Clear and free of impurity	---	---
Density	kg/m	850-900 (20 °C)	860-900 (15 °C)	---
Viscosity (40°C)	mm^2/s	3.0-6.0	3.5-5.0	1.9-6.0
Acidity Index, max.	mg KOH/g	0.50	0.50	0.50
Free Glycerin, max.	mass%	0.02	0.02	0.02
Total Glycerin, max.	mass%	0.25	0.25	0.24
Oxidative stability at 110 °C	h	6	6	3
Water Content, maximum	mg/kg	350	500	---

4. Discussion

4.1. Apparent aspect

The formed biodiesels were visually clear and apparently free of suspended impurities or any solid precipitate. This characteristic is only considered by the Brazilian standard recommendation [18].

4.2. Acidity Index

The value of acidy index is taken as the mass (mg) of potassium hydroxide used to neutralize the free acids in one gram of the oil sample [19]. The free acidity may be not a constant or a single intrinsic characteristic of oils and fats as it may also be originated from the partial hydrolysis of triacylglycerides. In a wider understanding it usually reflects the nature and the quality of the raw precursor bio-material, or the quality and purity of the oil itself, by also the way it was processed and, very often, the conditions it was stored.

High acidity indexes mean a very negative evaluation of the quality of biodiesel, in limiting cases, making it even unsuitable to be used as fuel. Continuous monitoring the value of acidity in stored biodiesels is thus of real importance. Significant changes of values may mean the occurrence of water, which would promote corrosive effects on metallic inner parts of the engine [18, 19]. The

values found for both these methyl and ethyl biodiesels from corn are well below the maximum recommend value, which is 0.5 mg g^{-1} KOH, according to Resolution 14 by ANP 2012 [20], and standards ASTM D6751 [14] and EN 14214 [16].

4.3. Peroxide index

The peroxide value is a measure of the reactive molecular oxygen content, expressed in terms of milli-equivalents of oxygen per 1000 g of fat. The analytical method is actually directed to determine all substances that are able to oxidize potassium iodide. The oxidized products from the reaction are taken as peroxides or any similar chemical forms rendered from the oxidation of fats. This index is then directly related to the oxidation stability [18]. Even though the chemical test is not used to provide a control parameter for biodiesel according to the Brazilian, European and American standards, it clearly represents an inverse correspondence with oxidative stability. High peroxide indexes (Table 1) of 23.77 meq/kg and 22.39 meq/kg found in this work are indeed consistent with reported oxidative stabilities [21] of 3 h 10 min and 2 h 13 min for these methyl and ethyl biodiesels from corn, respectively.

4.4. Oxidative stability

The oxidative stability, or the induction time, of oil is of critical importance especially to control its quality and storing conditions. It is expressed as the time in hours required reaching the point at which the degree of oxidation abruptly increases [19, 22, 23].

The oxidative induction time is used to estimate the relationship of the relative stability of various species exposed to the passage of an oxidizing gas isothermally (dry atmospheric air) and at high temperatures.

The recommended method EN 14112 by the ANP [20] and the standard EN 14214 [16] consider a limit of 6 h minimum. According to the ASTM D6751 [14], the lower limit is 3 h if the analytical method EN 15751 [16] is used. According to data in Table 1, the value found for this methyl biodiesel from corn oil is within the specification limits by ASTM. The lower oxidative stability for the ethyl biodiesel is accompanied by higher acidity and total glycerin, relatively to the methyl biodiesel [24-26]. Thus, to comply with the Brazilian and European standards it would be necessary to, for instance, add some anti-oxidant.

4.5. Water Content

The water content was determined in order to check about the condition by which the oil would favorably or not react to form the methyl and ethyl esters. The water content was also measured for the obtained esters mixture. In case of high water contents the corresponding fuel would tend to more easily deteriorate, due to accelerating oxidation process, and also to decrease the combustion rate, to undesirably generate free fatty acids, corrode metals and allow more easily microorganisms growing [19].

Only ASTM D6751 recommends this characteristic, obtained through the ASTM D2709 analytical method, as a control parameter for the quality of biodiesel [14]. ANP [20] recommends the coulometric European (Karl Fischer) EN ISO 12937 [16] and the Brazilian standard ASTM D6304

analytical methods [14]. Comparing methods, the coloumetric appears to be of higher sensitivity, repeatability and lower response time, if compared to the volumetric method ASTM D2709 [14].

Results obtained for methyl and ethyl corn biodiesels are well satisfactory as they presented 297.1 mg/kg and 264.8 mg/kg, respectively; the ANP recommended upper limit is 350 mg/kg [20].

4.6. Free and Total Glycerin

Contents of free and total glycerin also reflect the quality of biodiesel. A high content of glycerin may cause problems comprising formation of crystals, crusts on the internal wall of the storing tank of fuel, contributing to waste deposits on pistons, injectors, valves, thread, filter rings, and plugging in nozzles thus reducing the engine life. The amount of free glycerin (as a by-product) depends on the efficiency of the process efficiency to separate the ester mixture and the glycerin itself [19].

According to ANP legislation, 0.02 mass% free glycerol is the maximum amount allowed. The combined glycerin (total glycerin) including mono-, di- and triacylglycerides are derived from an incomplete reaction, thus being an important parameter for evaluation of conversion of fats and oils into biodiesel. The total glycerin will be the sum of the concentration of free and combined glycerin. According to EN and ANP standards the maximum the upper limit are 0.25 mass% and 0.24 mass%, respectively, following the ASTM analytical method [18]. Results for these methyl and ethyl corn biodiesels are found to be within limits recommended by the Brazilian, European and American standards.

4.7. Viscosity

Viscosity is the measure of resistance to flow under the gravity action of a body in the fluid relatively to its volume, i.e., the ratio dynamic viscosity: density of the fluid [19].

Higher viscosity of biodiesel appears due to higher carbon chain length and to higher degree of instauration of the fat acid, which directly affects the combustion rate and efficiency in the engine combustion camera [18, 27]. The viscosity turns higher as higher is the polymerization processes and the thermal or oxidative degradation.

EN 14214 recommends as acceptable viscosity values ranging from 3.5 to 5.0 mm^2/s (EN ISO 3104 method) [16], whereas ASTM D6751 establishes the wider range from 1.9 to 6.0 mm^2/s (ASTM method D445) [14]. The Brazilian standard adopts some more wider range, relatively to the already mentioned recommendations: for values of viscosity obtained through the ABNT NBR 10441 method, limits are 3.0 to 6.0 mm^2/s [28].

The upper limit value for viscosity is critically different depending on the available standard for biodiesels: from ANP, 6.0 mm^2/s; from EN 14214, 5.0 mm^2/s. These differences may restrict the use of certain raw bio-materials, as, for instance, oil from castor bean [29]. Results obtained for both of these methyl- and ethyl-biodiesels from corn oil are 5.4 mm^2/s and 4.37 mm^2/s, respectively.

4.8. Density

The fuel density is also a property that affects the engine performance. The quantity of fuel taken into the piston cavity is measured according to the volume passing through the injection pump

of the engine, not by the fuel mass. Thus any greater or smaller mass of fuel may be injected depending on their density. The air and fuel energy contents inside the combustion chamber, and, consequently, the engine performance, are influenced by the density of the fuel.

The density is related to the molecular structure, i.e., the longer the carbon chain of the alkyl ester is the greater is the density. However, the unsaturated chemical bonds along the molecular chain tend to reduce the density.

Biodiesel has higher density than diesel, but values vary according to the precursor oil, excess of residual alcohol, and many other transesterification condition. Very high values can indicate some saponification or residual oil; the excess of alcohol causes a decrease of the specific mass [19].

The European standard EN ISO 3675 recommends upper limit values of 860–900 kg m^{-3}, for data obtained through the analytical method of the glass hydrometer or through the method EN ISO 12185, based on automatic digital hydrometers. This latter has better repeatability [16]. The ASTM D6751 standard does not take into account the specific mass to evaluate the quality of biodiesels [14]. ANP provides a range comprising values between 850–900 kg m^{-3} [20], basing on analytical methods of the European standard, ASTM D1298 (manual handling) or ASTM D4052 (automatic instrumental procedure). Those methods are recommended by the NBR 7148 and NBR 14065 standards, respectively [14, 16, 29, 30].

In any case, the obtained values of density for both for both methyl and ethyl biodiesels from corn oil, i.e., 878.7 kg m^{-3} and 874.0 kg m^{-3}, respectively, were well within the recommended limits for EN, ASTM and ANP standards.

5. Conclusion

From these data, it is possible to obtain high standard biodiesels from refined corn oil, both via the methyl and ethyl chemical routes and alkaline homogeneous catalysis. The only characteristic non-complying with the recommendation by ANP 14/2012, ASTM D6751 and EN 14214 standards is that related to the oxidation stability, for which the obtained value of 6 h is below the recommended upper time limit. This characteristic may however be eventually corrected through addition of an antioxidant.

In general, these methyl and ethyl biodiesels from corn oil present physicochemical features with values lying well inside the recommend ranges by official Brazilian (ANP), European (EN) and American (ASTM) standards, being thus suitable, for instance, to be directly used or to be admixed to the mineral diesel. In any case, the resulting fuel will favor preventing the hazardousness of smokes released from internal combustion engines to the Earth atmosphere.

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