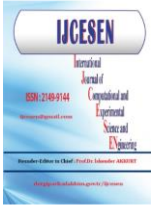
International Journal of Computational and Experimental Science and ENgineering (IJCESEN)
Vol. 6-No.1 (2020) pp. 29-34
http://dergipark.org.tr/en/pub/ijcesen



ISSN:2149-9144

Research Article

Alkaline and Acidic Biodiesel Production from oily Seeds of Terebinth (*Pistacia terebinthus*) plant and Physicochemical Properties of the Biodiesels

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Article Info:

DOI:10.22399/ijcesen.659068
Received : 13 December 2019
Accepted : 13 March 2020

Keywords

Alkaline and acidic biodiesel
Terebinth seed
Pistacia terebinthus
Şırnak.

Abstract:

The objective of the paper is to evaluate the physicochemical properties of the biodiesel produced by alkaline and acidic transesterifications of seed oil of wild Terebinth (*Pistacia terebinthus)* plant from Şırnak province. The terebinth tree is widely distributed in Şırnak province and its oily seeds are consumed mostly as snack by local people. Transesterification is the process of exchanging the alkyl groups of an oil or fat with the methyl groups from short-chain alcohols (methanol) in the presence of an acidic, alkaline or enzymatic catalyst. The results of the alkaline and acidic biodiesels from terebinth oil were as follows: the kinematic viscosity of the alkaline biodiesel was found to be 4.88 mm^2/s (40 °C), density 0.8644 g/cm^3 (15 °C), the flashpoint 134 °C, calorific value 38.250 (kJ/kg), cetane number 41 and a water content <250 mg/kg. The acidic biodiesel had a kinematic viscosity of 5.14 mm^2/s (40 °C), the density of 0.8907 g/cm^3 (15 °C), a flashpoint of 195 °C, the calorific value of 36.300 (kJ/kg), the cetane number of 34, and the water content of <250 mg/kg. In conclusion, the analysis indicated that biodiesel can be produced from terebinth oil by either method and the physicochemical properties of the biodiesels were as good as previously released biodiesel qualities, however, alkaline biodiesel was much qualified than acidic biodiesel.

1. Introduction

With this trend, it is likely that in the future, the depletion of raw resources for energy supply will be exhausted. While petroleum oil prices have risen due to the fear of depletion of limited fossil fuel resources, new research is being conducted to find alternatives to petroleum fuel. The solution focuses mostly on edible and non-edible oils to produce biodiesel fuels. Most of the biodiesel is prepared from edible oils such as soybean, sunflower, canola, palm, animal fats, and cooking oils. The cost of edible oils is much higher than that of petroleum diesel, and the use of edible oils for biodiesel production has received strong criticism [1]. Researchers have begun to focus on developing alternative energy sources like biodiesels produced from non-edible oils such as wild plants oil, algae oil, rendered oils or industrial oils as emissions from fossil fuels harm both the ecosystem and human health. Biodiesel has many advantages, reducing emissions of unburned hydrocarbons, sulfates, carbon monoxide, nitrated polycyclic aromatic hydrocarbons, non-toxic renewable and biodegradable fuel, easy transportation and storing, higher lubrication capacity, better burning (contain O_2), reducing the release of Polycyclic aromatic hydrocarbons (cancer-causing compound) [2,3]. If we want to significantly reduce global warming and the greenhouse effect, we should encourage biodiesel production and use.

Biodiesel is mostly not preferred to be used alone in diesel engines without modification due to its slightly high viscosity and density. In diesel engines, when pure biodiesel is used, many problems such as injector nozzle clogging, plastic tube, and pad corrosion are encountered. Therefore, biodiesel is widely used in vehicles in the form of a mixed fuel mixed with conventional diesel to a certain extent [3-6].

Raw materials that are predominantly used today for biodiesel production, especially for vegetable oils, are seen as problems because they are normally expensive and also have limited availability due to social and economic constraints. Therefore, there is great interest in the use of low-cost raw materials, which do not interfere with the important aspects of human societies and contribute to their sustainability [7]. Terebinth seeds are not widely used as an edible oil in the food industry, as they are difficult to harvest and have low production. This means that the use of terebinth oil in biodiesel production will not turn into any socio-economic problem.

The current study presents the quality and sustainability of the alkaline and acidic biodiesels of terebinth oil as well as comparing the properties of both biodiesels as alternative resources for fuels. Physicochemical properties of the biodiesels show that if the oil can be continuously and efficiently produced from the terebinth plant, it can be a biodiesel raw material for future production.

2. Materials and Methods

Seeds of *Pistacia terebinthus* were purchased from herbalists (collected from the foothills of Mount Cudi and its surroundings during summer by herbalists) in Şırnak and Cizre province. Pure oil of the seeds was extracted with a cold-press machine in the herbalist. Degumming (Separation of phospholipids, proteins, and gums from crude oil), Neutralization (The process of removing the free fatty acids in oil by saponification with sodium hydroxide, the oil is first heated to 75-80 °C utilizing heat exchanger, separated by high-speed centrifugation), Dewaxing (Room temperature insoluble wax molecules precipitate in the oil to cause turbidity, to reduce the turbidity), Bleaching (the process used to remove alpha and beta carotene, xanthophylls, and chlorophyll from the oil). These processes are not applied to the terebinth oil; as such processes are generally applied to edible food oils, and may also cause a quality problem in the biodiesel since their applications may impair the naturalness of the oil. Consequently, the terebinth oil was centrifuged with a high-speed centrifuge to remove contaminating particles. Then, the oil was heated at 110 °C for half an hour to remove any water that might be in the extracted oil. The dehydrated oil was again placed in a centrifuge machine and centrifuged for one hour so that any particles that remained in the oil precipitated.

2.1. Production and purification of alkaline biodiesel

Alkaline biodiesel was produced by alkaline catalyzed transesterification methods using NaOH (Merck KGaA-Pellets pure) as catalyst and methanol (Merck KGaA-Emsure) as a reaction compound, in the laboratory with precision and measuring instruments. In the experiment, 200 ml of oil was weighed into a beaker having a capacity of 500 ml and placed on a magnetic stirrer and heater to warm the oil to reaction temperature (55 °C). Methanol was used in a ratio of approximately 5/1 molar oil/alcohol. About 2g of NaOH catalyst was measured and dissolved in a closed vessel, the mixture was gently heated and stirred. This process took place in a sealed container for about 20 minutes (Approximate time of complete dissolution of methanol). The alcohol-catalyst mixture was then added to the preheated oil and allowed to stand in the shaker for 10 minutes to shake vigorously to increase contact between the reactants. Then the flask was placed on a magnetic stirrer and heater, the reaction was carried out at 55 °C for 2 hours and a stirring speed of 80 rpm. After completion of the reaction time, the sealed beaker was removed from the machine and allowed to cool. The mixture was transferred to a previously weighed beaker to determine its mass and allowed to stand for about 12 hours in a separatory funnel to remove the heavier glycerol phase. The lighter phase biodiesel is placed in the distillation unit at 80 °C to recover excess methanol. It was then placed in a separatory funnel to neutralize with hot water slightly acidified with phosphoric acid. The washing procedure was repeated two or three times using hot water until a neutral pH with a clear phase was formed. A small amount of water may remain in the converted biodiesel, which may adversely affect the quality of the biodiesel. The water washing procedure is performed to completely remove waste materials and glycerol from the biodiesel. The removal of water from the biodiesel can be accomplished by heating the produced biodiesel to 100 °C for dehydration and using molecular sieves as the physical process. The percentages of methyl esters (biodiesel) and glycerine is approximately 86% and 14%, respectively. Glycerol (or glycerine) is a major by-product in biodiesel reactions. Nearly, 206 ml of biodiesel and 34 ml glycerine were produced in the experiments (Figure 1). Subsequently, alkaline biodiesel was stored in dark glass bottles to measure the physicochemical properties of biodiesel.

Figure 1.* 1.** *Wild terebinth seeds,* **2.** *Terebinth oil (dark green) heated at 55 °C,* **3.Terebinth oil + methanol + NaOH solution in magnetic stirrer and heater***,** **4.** *Alkaline biodiesel with two phases: Biodiesel and Glycerol phases,* **5.** *Acidic biodiesel with two phases: Biodiesel and Glycerol phases,* **6.** *Deflagration of alkaline biodiesel*

2.2. Production and purification of acidic biodiesel

The transesterification process was catalyzed by strong acids, preferably by sulphuric acid (Merck KGaA-Emsure) or nitric acid (Merck KGaA-Emsure). These catalysts give very high yields in alkyl esters, but reactions are slow, generally require temperatures above 65 °C and are mixed for more than 1-3 days to achieve complete conversion for the oils. Terebinth oil, in the presence of 1 mol% of H_2SO_4, with an alcohol/oil molar ratio of 1/30 at 65 °C, takes 48 h to reach the complete conversion of the oil. The reaction was slow and for this reason, the formation of the fatty acid methyl ester was slow, too. The properties of the methanol and the laboratory instruments used were the same as those used in the experiment described above, except the acid was used in the experiment instead of NaOH. Besides, purification of acidic biodiesel, separation from glycerol and washing with water were the same methods used in alkaline biodiesel (Figure 1). The sulphuric acid ratio chosen as a catalyst can be increased to 8% or even 10% if the oil is dense (such as animal fat, grease). If the density of the oil is low, as in the case of vegetable oils, the sulphuric acid content can be drawn up to 5%. The alcohol/oil molar ratio is one of the main factors that influence the transesterification. The use of excessive amounts of alcohol can facilitate the formation of biodiesel, but high alcohol makes it difficult to recover glycerol from the fuel, so in transesterification, the ideal alcohol/oil ratio should be determined empirically and previously, by taking into account each process step.

3. Results

Wild terebinth oil is dark green, whereas most oils used for biodiesel are either yellow or reddish yellow and it has a high consistency compared to other vegetable oils. Pure terebinth oil density at 26 °C (g/cm^3) is 0.921, calorific value (kJ/kg) is 39.100, viscosity (mm^2/s) at 26 °C is 44.85, flashpoint is 228 °C and cetane number is 46 [8]. In this experiment, terebinth oil has been converted into biodiesel by both alkaline and acidic transesterification methods. For alkaline biodiesel, the kinematic viscosity was found to be 4.88 mm^2/s (40 °C), the density was 0.8644 g/cm^3 (15 °C), the water content was < 250 mg/kg, the flashpoint was 134 °C, the calorific value was 38.250 kJ/kg, cetane number was 41 and color of the biodiesel was light yellow (golden). For acidic biodiesel, the kinematic viscosity was found to be 5.14 mm^2/s (40 °C), the density was 0.8907 g/cm^3 (15 °C), the water content was < 250 mg/kg, the flashpoint was 195 °C, the calorific value was 36.300 kJ/kg, cetane number was 34 and color of the biodiesel was light brown (Table 1).

4. Discussion

Biodiesel, containing fatty acid methyl esters, is an average of C16-C18 carbon - long organic origin fuel and has some advantages over petroleum-derived diesel fuels. It is safer to transport and store than diesel fuels. Because the flammability point (>110 °C) is higher than diesel fuel, this allows getting a little later ignition. The presence of low sulfur and nitrogen compounds both increases the power of the engine as well as provides an environmentally friendly fuel. The emission rate is less than diesel. The cetane number is higher than conventional diesel fuel; it reduces the impact on the engine. The biodiesel's compound dissolving property leads to deposits dissolving in engines. Biodiesel is dissolved in wastewater at a rate of 95% in a month, while this rate is below 40% in diesel fuel. Providing benefit to the soil as a fertilizer with its nitrogen binding property, it does not also harm the ozone layer like diesel fuel. Polycyclic aromatic hydrocarbon (PAH) emissions, seen as a potential cause of cancer, reduced by up to 85% by the biodiesel [9-11]. Transesterification (alcoholization) refers to the formation of fatty acid esters and glycerol by the reaction of fat or oil with

Table 1.*Physicochemical properties of alkaline and acidic terebinth biodiesels as per ASTM biodiesel standards.*

Characteristics	**The values of alkaline biodiesel**	**The values of acidic biodiesel**	**Procedure of Tests**
Type of source	Pure Terebinth oil	Pure Terebinth oil	-
Chemical formula	CH_3-COO-Rx	CH_3-COO-Rx	-
Kinematic viscosity (mm^2/s, 40 °C)	4.88	5.14	ASTM D 445
Density (g/cm^3, 15 °C)	0.8644	0.8907	ASTM D 1298
Water content (mg/kg)	< 250	< 250	-
Flashpoint (°C)	134	195	ASTM D 93
Calorific value (kJ/kg)	38.250	36.300	ASTM D 2015
Cetane number	41	34	ASTM D 613
Colour of the biodiesels	Light yellow	Light brown	-

short-chain alcohol and catalyst. Methanol or ethanol is generally preferred as alcohol because of its low cost and easy to react [12] and catalyzers are mostly as NaOH, KOH, sulphuric acid, nitric acid or heat resistant lipase [13]. Acid-catalyzed transesterification occurs more slowly than the alkaline reaction. Acidic one is more suitable for oil with high free fatty acid and high water content [12]. Alkaline transesterification is about 4000 times faster than acidic transesterification [3]. Therefore, in biodiesel synthesis, alkaline transesterification is preferred. The lack of water and free fatty acids in the content of the oil has great advantages; these two components reduce the efficiency of the catalyst and cause difficulties in the decomposition of glycerol. Free fatty acids and water cause the formation of soap in biodiesel [12,14]. The characteristics of standard biodiesel and petroleum- diesel fuel are given in Table 2. The specific density of a petroleum-diesel is between 0.82-0.86 g/cm^3, and the specific density of biodiesel is 0.87 - 0.88 g/cm^3 and the kinematic viscosity of the diesel and the biodiesel are shown 1.3 - 4.1 mm^2/s and 4.0 - 6.0 mm^2/s, respectively. The flashpoint of the biodiesel is between 100 -170 and diesel is 60-80 and the water content of the standard biodiesel is <300 mg/kg; whereas, the water content of the diesel is <200 mg/kg.

Table 2*. Fuel characteries of standard biodiesel and petroleum-diesel fuel [15,16].*

Fuel Properties	**Biodiesel** ASTM D6751	**Petroleum-Diesel** ASTM D975
Kinematic Viscosity (mm^2/s,40 °C)	4.0-6.0	1.3-4.1
Density (gravity) (60 °C., g/cm^3)	0.87-0.88	0.82-0.86
Carbon, wt %	77	87
Calorific Value (kJ/kg)	39200	42000
Hydrogen, wt %	123	13
Oxygen, by dif. wt %	11	0
Flash Point, °C	100 to 170	60 to 80
Cetane Number	48-65	40-50
Sulfur, ppm	0	500
Boiling Point, °C	315 to 350	180 to 340
Cloud Point, °C	-3 to 12	-15 to 5
Pour Point, °C	-15 to 10	-35 to -15
Lubricity (HFRR)	<300	300-600
Water content (mg/kg)	<300	<200

The calorific value of biodiesel is about 39.200 kJ/kg, and for the diesel, this is 42.000 kJ/kg. The cetane numbers of standard biodiesel normally range from 48 to 65 while diesel ranges from 40 to 50. The cloud and pour points of biodiesel are higher than those of diesel fuels (Table 2) [15,16].

The aforementioned properties of alkaline and acidic biodiesels of the terebinth oil are given in Table 1. By analysis, the kinematic viscosity of alkaline biodiesel was determined as 4.88 mm^2/s, and the kinematic viscosity of acidic biodiesel was found to be 5.14 mm^2/s. Little high kinematic viscosity of acidic biodiesel is probably since the formation of methyl esters in the transesterification process is not fully achieved. On the other hand, the kinematic viscosity of alkaline biodiesel in this study is within the given standards. The cetane numbers of terebinth biodiesel were found to be 41 for alkaline and 34 for acidic biodiesel. Calorific values of alkaline and acidic biodiesels are 38.250 and 36.300 KJ/kg, respectively. Generally, the values are not far from the standard biodiesel values, particularly alkaline biodiesel. The flashpoint of alkaline was 134 and acidic was 195 °C. Flashpoint for acidic biodiesel is slightly higher than standard biodiesel, however; alkaline biodiesel flash point complied with the standard values. The flashpoint is known as the lowest temperature adapted to normal atmospheric pressure (101.3 kPa) and is the temperature at which the application of the ignition source causes the ignition of a sample's vapors under the specific conditions of the test. Flashpoint is been admitted as a measure of a liquid's hazardousness. Low flashpoint causes the fuel to exacerbate quickly and makes its transportation and storage more dangerous.

Table 3 gives the values of density, kinematic viscosity, cetane number, low heating value, flashpoint, cloud point of some mostly used vegetable oil biodiesels such as peanut, soybean, babassu, palm, sunflower, and rapeseed [17]. Many studies have reported different data on biodiesels from oils and fats. However, these reported data contain substantially approximately similar characteristics. In a study on canola biodiesel, the density, viscosity, and flashpoint were given as 0.880 g/cm^3, 4.29 mm^2/s and 182 °C respectively [18]. In another study about soybean biodiesel, the density has been determined as 0.880 g/cm^3 and flashpoint was >162 °C [19]. The density of safflower biodiesel was 0.890 g/cm^3 (15 °C), kinematic viscosity was 5.34 mm^2/s (40 °C) and flashpoint was 129 °C [20]. Biodiesel from chicken fat was reported as 0.883 g/cm^3 (15 °C), kinematic viscosity was 5.85 mm^2/s, the flashpoint was 171 °C. Beef tallow density was 0.870 g/cm^3, kinematic viscosity was 5.40 mm^2/s, flashpoint 172 °C. Pork lard density was 0.872 g/cm^3, kinematic viscosity was 4.96, flashpoint 147 °C; sardine fish density was 0.886 g/cm^3, kinematic viscosity was 4.33, flash point 160 °C [21]. As seen, chicken fat (5.85 mm^2/s) and beef tallow kinematic viscosity (5.40 mm^2/s) were reported to be higher than normal standard biodiesel. The high value of acidic biodiesel from terebinth seems to be better than some animal fat biodiesels. The viscosity of a fuel refers to how easily the fuel (biodiesel) flows at a specified temperature. Generally, fine oils form low viscosity biodiesels and are more easily poured at lower temperatures than thicker oils with higher viscosity. Fine oil biodiesels reduce friction in engines and help the engines start up quickly in cold weather. Mostly thick fats form biodiesel with higher viscosity. Although the kinematic viscosity values vary due to thick or thin oils, sometimes the chemicals and methods used in the experiment also cause the kinematic viscosity value to be high or low. Almost all of the studies on biodiesel have been reported to produce biodiesel by an alkaline transesterification reaction. The values of the biodiesel mentioned in tables 2 and 3 belong to alkaline biodiesel. There is not much information available on acidic biodiesel. Because the production of acidic biodiesel is not easy and advantageous in terms of time and cost.

In a study about the effect of terebinth oil biodiesel on engine performance and exhaust emissions in a diesel engine, the values of the alkaline biodiesel is given as follows: kinematic viscosity 4.12 mm^2/s, heat value 40.100 kJ/kg, density (15 °C) 0.88 g/cm^3, flashpoint 75 °C and cetane index 55. In the experiment, biodiesel from fabricated terebinth oil has been produced by the alkaline transesterification method. The biodiesel was then mixed with diesel fuel by volume B10 fuel. The experiment was carried out with a mixture of clean diesel fuel and biodiesel (B10 fuel) in a four-stroke compression injection diesel engine. [8]. Although the fuel values of both alkaline and acidic biodiesel obtained from the oil of wild terebinth plant from Şırnak and Cizre province showed similarity in terms of kinematic viscosity, heat value and density, it is seen that there was a difference in flashpoint and cetane index. This suggests that the oil composition of locally collected terebinth seeds may be different or these slight dissimilarities can be originated from other factors such as chemicals, production time, raw material processing and experimental stages.

In conclusion, depending on the geographical conditions, various oil containing plants can be used for biodiesel production. The properties and performance of the produced biodiesels may have some different values due to the characteristics of the geographic region. Generally, most of the parameters are similar to each other in biodiesels. Biodiesels show a slightly lower heating value due to oxygen content (11%). This may increase fuel consumption slightly. As known, the flashpoint of biodiesel is higher than petroleum diesel; this makes it safer for special uses. The cetane number is also high, especially for products containing highly saturated fatty acid esters such as biodiesel from palm oil or tallow. In contrast, the high content of paraffin-like components increases the fuel cloud point and causes clogging of the fuel filter in winter.

As a consequence, the alkaline biodiesel obtained from terebinth oil was found to be better when compared to acidic biodiesel. The fact that the terebinth tree is widespread in the region and its oil is mostly consumed as snack purposes, not for cooking. For this reason, this plant can be a good material for biodiesel production in the future.

Acknowledgements

Some biodiesel values used in this study were taken from the project 2017.03.03.04 - Şırnak University, Coordinatorship of Scientific Research Projects (Project manager: İhsan EKİN) Since there were suspicious differences in some values in the project data, the experiments have been repeated several times to prove and validate the values and then new findings have been obtained and used in the current study.

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