Physico-Chemical Properties of Biodiesel from Jatropha and Castor Oils

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Abstract- Biodiesel is becoming prominent among the alternatives to conventional petro-diesel due to economic, environmental and social factors. The quality of biodiesel is influenced by the nature of feedstock and the production processes employed. High amounts of free fatty acids (FFA) in the feedstock are known to be detrimental to the quality of biodiesel. In addition, oils with compounds containing hydroxyl groups possess high viscosity due to hydrogen bonding. American Standards and Testing Materials, (ASTM D 6751) recommends FFA content of not more than 0.5% in biodiesel and a viscosity of less than 6 mm^2/s. The physico-chemical properties of jatropha and castor oils were assessed for their potential in biodiesel. The properties of jatropha and castor oils were compared with those of palm from literature while that of biodiesel were compared with petro-diesel, ASTM and European Standards (EN14214). Results showed that high amounts of FFA in oils produced low quality biodiesel while neutralized oils with low amounts of FFA produced high quality biodiesel. The quality of biodiesel from jatropha and castor oils was improved greatly by neutralising the crude oils

Keywords- Feedstock; biodiesel; physco-chemical properties; transesterification

1. Introduction

Vegetable oils are among the various sources of energy fuels being considered as alternatives to fossil fuels. Rapeseed, soybean, sunflower, coconut and palm oils have been the main raw materials for biodiesel production. However, these oils are required in refined forms to obtain quality biodiesel and, in addition, they are foodstuffs. This makes production of biodiesel from these sources uneconomical [1]. Non-edible plant oils such as found in jatropha curcas and castor beans may provide better alternatives. Plant-derived oils could be used directly in diesel engines or blended with petro-diesel, however, their high viscosity lead to problems in the engine [2,3]. Inefficient oil - air mixing causes poor injector system performance which leads to incomplete combustion hence producing high smoke and causing ring sticking, filter plugging and engine deposits [4].

1.1. Quality Attributes

Kinematic viscosity is one of the parameters specified in biodiesel and petro-diesel standards that require compliance [5,1]. Knothe and Steidley [3], reported that the presence of an OH group in ricinoleic acid increases viscosity significantly oil due to hydrogen bonding. Reducing viscosity is therefore the major reason for processing plant oils to make them suitable for use as biodiesel. Methods of reducing viscosity besides transesterification include dilution, microemulsion, pyrolysis and catalytic cracking [6,7,8,9].

Free fatty acids on the other hand, are undesirable in the fuel tanks because they corrode engines. ASTM D 6751 standard [10] recommends FFA content of not more than 0.5% in biodiesel since higher values lead to corrosion of rubber parts and cause deposits in the engine. Murugesan et al., [4] recommends FFA content of not more than 3% in the feedstock for biodiesel production whereas [11,12] give the value of up to 5%. A high FFA value in the feedstock consumes the catalyst during base catalyzed transesterification reaction to produce biodiesel. It also leads to saponification which lowers the yield and increases formation of emulsions in the product making it difficult to separate biodiesel from glycerine [13]. The presence of FFA is linked to the nature and quality of raw oil. Methods of

reducing FFA in the raw oil include neutralization with an alkaline solution prior to transesterification and two-stage processing; acid esterification in which the FFA is converted into an ester then an alkaline transesterification can follow [14].

Flash point is specified in biodiesel to serves as a restriction of the amount of alcohol in biodiesel for safety measures in transportation and storage. It is also a biodiesel quality related to the fatty acid structure [15]. Flash point can be adjusted through blending biodiesel with petro-diesel in appropriate proportions. Blends of 20% biodiesel to 80% petro-diesel (B20) have been recommended by various researchers [9,16,17].

Cloud point indicates the lowest temperature at which a fuel is usable; this is especially useful in cold countries. Its value is not specified in the ASTM standard but a report is required in some standards, for example, EN14214. Ways of dealing with this problem include blending and winterization [5].

Density is specified in several standards and the purpose is to exclude unrelated materials from being used as biodiesel feedstock [15]. It is also used in the determination of the viscosity of biodiesel.

Calorific value or heat of combustion of plant oils that are commonly used as raw materials for biodiesel production varies from 5443 to14654 kJ/Kg. This is within the range of that of hexadecane, also known as Cetane (10714 kJ/mole), which is used as a reference standard material for the determination of the ignition quality of petro-diesel [5,30].

The composition and physico-chemical properties of oils have been found to vary depending on location of the plant and agricultural practices applied on the raw materials. Aguirrezabal et al., [18] demonstrated that fatty acids composition of sunflower varies with location and sowing season. Knothe [5] showed that kinematic viscosity of biodiesel fuel is influenced by structural arrangement of organic compounds present in the raw material.

A comparative study of vegetable oils by [19] found that biodiesel yield and ester contents were independent of the raw material but both decreased with increase in acid value of the oil. Fatty acid composition of oils affects biodiesel quality [5]. This could exclude some vegetable oils from being used as feedstock for biodiesel production and in any case, the feedstock quality also dictates the type of production process to be undertaken [20].

Jatropha curcus, is a genus that belongs to the Euphorbiaceae family with many species [21]. Castor bean, (Ricinus Cummunis) also belongs to the Euphorbiaceae family. Both plants grow wild in tropical and subtropical countries. Jatropha curcas and castor beans plants were primarily chosen for the study because of their relatively high oil contents and would provide better alternatives to the edible oils mentioned earlier.

The pysico-chemical properties of crude and neutralized jatropha and castor oils grown in Tanzania and of their respective biodiesel were investigated for biodiesel production for use in diesel engines. The physico-chemical properties that were studied included: FFA, kinematic viscosity, flash point, calorific value, cloud point and density. Fatty acids (FA) composition of the oils were also determined and compared with those from literature.

2. Materials and methods

Jatropha and Castor seeds were obtained from Arusha and Dodoma in Tanzania respectively. Seeds were cleaned by hand picking, washed and dried in an oven at 105oC for 1 hour to reduce moisture. These conditions were chosen based on studies by [21]. Dried seeds were crushed and weighed into different thimbles. Crude oil was obtained from the seeds by solvent extraction using n-hexane. Based on literature, n-hexane was considered the best solvent for the extraction [21,22]. Because refined castor and jatropha oils were not available in the market, the crude oils were neutralized (to reduce acid value) using analytical grade NaOH. For jatropha oil, 6.46g of NaOH was dissolved in 40 mls of water and the resultant solution used to treat 100 mls of oil. The quantities were determined based on FFA content of the oil. The oil was heated on a Wagtech (RCT) hot plate while being stirred using Heidolph stirrer (RZR 50L, Germany), set at 600 rpm. NaOH solution was added while heating and stirring continued for 15 minutes up to a temperature of 80^{o}C. The neutralised oil was separated from soap using Martin Christ Osterde centrifuge run at 4,500 rpm for 20 minutes. Soap formed at the bottom of the centrifuge while the oil floated at the top. The oil was removed, washed three times with warm water (50^{o}C) and dried using Heidolph Laborota Evaporator at 700 Pa until no water dripped in the condenser.

In the case of castor oil, five grammes of NaOH were dissolved in 50 ml of water and the solution was used to treat 100 ml of oil. Heating and stirring was done for 10 minutes up to a temperature of 70^{o}C. Separation was done with a Martin Christ Osterde centrifuge set at 3,000 rpm for 20 minutes. Oils were characterized (all measurements done in triplicates) for acid values using AOCS, 1998, Ca 5a - 40; kinematic viscosities at 40oC using ASTM D445 method; flash points using ASTM D 92 (STANHOPE-SETA Semi Automatic Flash Point Tester) Cleveland open cup; calorific values using ASTM D240 (bomb calorimeter, Gallenkamp Auto bomb BS 4791, 1985) and fatty acid composition using AOCS Ce 1-62, 1998.

2.1. Experimental procedure

2.1.1. Fatty acids composition

Four grammes of clear oil were transferred into 100 ml conical flask then 40 ml of methanol and 0.5 ml of methanolic NaOH solution was added. The flask was fitted with a condenser and heated under reflux using a hot plate (Wagtech RCT) for five to ten minutes till the solution became clear. The solution was cooled under running water then transferred to 125 ml separating funnel and the flask rinsed with 20 ml n-hexane and added into the funnel. 40 ml of water was added into the funnel, shaken and allowed to separate. Ester passed into the upper hexane layer and was

removed. The funnel content was again extracted with 20 ml hexane and the two extracts combined and washed with several 20 ml portions of water. Ester was separated and dried over anhydrous sodium sulphate, filtered through cotton wool and evaporated to approximately 10 ml on a water bath to remove excess solvent. This was kept in a refrigerator below -2^{o}C until analysis.

2.1.2. Biodiesel

A two-litre round bottomed-flask with four necks was used as a reactor for transesterification. It was fitted with a refluxed condenser, a thermometer in one port and a Heidolph (RZR) stirrer in another port. One port was plugged and left for sampling and the whole unit was immersed in a Centromat (VR B. Braun Biotech International, Germany) constant temperature water bath.

One litre of oil was weighed and heated separately on a Wagtech (RCT) hot plate to 110^{o}C for 30 minutes to drive off moisture before it was transferred to the reactor. While the oil was heating, NaOH catalyst was weighed (1% wt of oil) and dissolved in methanol corresponding to the molar ratio of 6:1 methanol to oil. The oil was allowed to cool (temperatures of the oil and methanol-sodium hydroxide mixture were equilibrated in the water bath) and the mixture was transferred into the reactor as agitation started immediately. The reaction was allowed to proceed for two hours. The mixture was removed and left for three hours in a separatory funnel to allow glycerol to separate from methyl ester. The methyl ester was washed four times with warm water (50^{o}C) to remove the catalyst, residual methanol and glycerol. It was then dried with anhydrous sodium sulphate and filtered through cotton wool before characterization.

3. Results and discussions

Oil from castor seeds contained high proportion of ricinoleic acid with considerable amounts of lionleic, oleic, stearic and palmitic acids. Oil obtained from jatropha seeds consisted mainly of linoleic, oleic and palmitic acids with minor components of stearic and palmitoleic acids (Table 1). The ability of biodiesel to meet ASTM D 6751 standard criteria is dependent on the fatty acid composition [23,3]. Petroleum diesel is largely made of hydrocarbon with carbon chain length of 8 to 10 carbon atoms compared to jatropha and castor oils that contained fatty acids comprising of 16 to 18 carbon atoms [24]. Cetane number which is a prime indicator of fuel quality for diesel engines is increased with increased carbon number [5]. Branched chains and double bonds improve low temperature flow properties [3].

The physico-chemical properties of castor oil in this study are of significance because of the exceptionally high content of ricinoleic acid. The three functional groups, namely carboxyl, double bond and hydroxyl attached at carbon-12 are of interests for biodiesel production [25]. The low temperature operability of castor oil [26] and its complete solubility in alcohol [27] make it suitable for biodiesel production.

Table 1. Fatty Acid Composition of Castor and Jatropha Oils

Fatty acid	**Structure**	**Castor oil**	**Castor** [26]	**Jatropha oil**	**Jatropha**[23]
Myristic acid	C14:0	Tr	Nd	Nd	Nd
Palmitic acid	C16:0	1.31±0.10	1.0	15.32±0.07	15.3
Palmitoleic acid	C16:1	Tr	Nd	1.33±0.01	1.1
Stearic acid	C18:0	1.22±0.10	0.7	4.06±0.02	5.6
Oleic acid	C18:1	3.98±0.26	3.5	35.38±0.20	37.1
Linoleic acid	C18:2	4.66±0.30	4.4	43.34±0.21	40.4
Linolenic acid	C18:3	0.42±0.04	0.4	Nd	0.3
Ricinoleic acid	C18:1OH	88.55±0.66	89	Nd	Tr

Tr = Trace; Nd = not determined

Heats of combustion of jatropha and castor oils were found to be high. Jatropha oil had a higher heat of combustion (42.47±0.95 MJ/kg) than that of castor (35.50±1.50). Castor oil has more oxygen atoms on its triglyceride structure than jatropha oil. Neutralisation had slight negative effects on heats of combustion. The heat of combustion of jatropha oil was reduced by 0.75% while that of castor by 8.16% (Table 2). However, these are still high compared to the heat of combustion from refined palm oil (38.00 MJ/kg).

Kinematic viscosity, which is an essential property of fuel, is very high in castor and jatropha oils (Table 2). Fuels from these sources may not be used directly in diesel engine. Kinematic viscosity of crude castor oil is almost ten times higher than those of jatropha and palm. This is perhaps attributed to the presence of hydroxyl groups in the castor fatty acid molecule that form hydrogen bonding leading to higher viscosity.

FFA contents of oils from castor and jatropha were found to be high. Oil from jatropha presented higher FFA value than that from castor (Table 2). This is due to stability conferred to castor oil as a result of the hydroxyl group attached to its fatty acid molecule [3]. Acidity is lowered by neutralization of oils because the FFA forms soap with alkali during neutralization and is removed by centrifugation (Table 2).

Cloud points of jatropha and castor oils are generally high making the oils unsuited for making biodiesel, especially for use in cold countries (Table 2). Biodiesel from these oils would not be usable in cold countries without the use of additives to improve their cold filter plugging points (CFPP).

Flash points of crude and neutralised oils were high making them better suited for biodiesel production with respect to safety during storage and transportation (Table 2). Whereas neutralisation increases flash points (7.8% in jatropha and 3.1% in castor oils) (Table 2), transesterification lowers them tremendously (75% and 39% in jatropha and castor oils, respectively (Table 3). These are still within the range specified in biodiesel standards.

Table 2. Physico-chemical properties of crude and neutralised oils

Property	Crude Oils		Neutralized Oils		Refined
	Jatropha	Castor	Jatropha	Castor	Palm [29]
Acid Value [mg KOH/g]	3.38±0.23	2.41±0.03	0.53±0.09	0.51±0.02	nd
FFA [%]	1.70±0.46	1.21±0.02	0.27±0.05	0.26±0.01	nd
Kinematic Viscosity [mm^2/s]	33.86±1.92	222.00±6.93	31.50±1.16	107.50±3.20	36.8
Flash Point [^oC]	292±1.00	294±0.58	315±0.66	303±3.00	280
Density [Kg/m^3]	910±2.64	940±3.00	907±1.00	930±2.00	910
Cloud Point [^oC]	2±0.21	14±1.00	- 4±0.20	14±2.65	32.7
Calorific Value [MJ/Kg]	42.47±0.95	38.65±0.81	42.15±1.30	35.50±1.50	38.00
Oil Yield [% wt]	52±1.13	56±3.13			nd

Heats of combustion of biodiesel from jatropha and castor oils were found to be high. Biodiesel from jatropha oil had a higher heat of combustion than biodiesel from castor oil (Table 2). Rider et al., [29] gives the heat of combustion of petro-diesel as 47 MJ/kg. The heats of combustion (calorific values) of biodiesel are generally similar and are 12% less than that of number two petro-diesel according to [11]. The calculated heat of combustion of biodiesel by this information is 41.36 MJ/kg which is in the same range with the values obtained in this study (Table 3). Based on absolute values, palm biodiesel has the same range of calorific value with jatropha (Table 3). Castor biodiesel has a lower calorific value than jatropha and palm biodiesel. This is attributed to the structural differences in the constituent fatty acids of castor oil [30]. Palm oil is predominantly made of palmitic acid (C16:0), a saturated fatty acid that that is associated with higher energy content than unsaturated acids [30,3]. Neutralisation of oils improves heats of combustion of biodiesel (Table 3).

Kinematic viscosities of biodiesel in the two oil categories were lowered after transesterification (Table 3). Jatropha biodiesel viscosity complied with standard specification while castor biodiesel viscosity did not. Although transesterification lowers viscosity, castor biodiesel viscosity is still out of the range specified in biodiesel standards. Jatropha and palm biodiesel meet the ASTM D 6751 standard specification in their viscosities making them suitable for use in diesel engine while castor biodiesel does not meet the specifications. Biodiesel from crude oils have higher viscosity than that from neutralised oils (Table 3). High viscosity in biodiesel is sometimes associated with unreacted triglycerides in the fuel. Neutralisation therefore improves conversion of oils to biodiesel. Alternative means of reducing the viscosity of castor prior to biodiesel production could be looked into, for example, blending or refining the oil.

Cloud points were generally lowered in biodiesel from jatropha and castor oils. Castor biodiesel had lower cloud point than jatropha biodiesel, making castor biodiesel more suited for use in cold countries than jatropha biodiesel (Tables 3). Jatropha biodiesel may require the use of additives to improve its cold weather property desirable in cold countries.

Flash points of biodiesel from jatropha and castor oils meet the required specification though neutralisation lowers flash points slightly, from 167 to 166^{o}C in jatropha biodiesel and 178 to 160^{o}C in castor biodiesel (Table 3). The impurities responsible for high flash points in crude oils are removed by neutralisation. Acid values of biodiesel from crude oils are still very high (Table 3). Neutralisation improves the quality of the oil and makes it suitable for biodiesel production. Table 4 shows a comparison of biodiesel properties obtained from this study with petro-diesel, ASTM and EN standards.

Biodiesel yield from crude jatropha oil was 76% while the yield from neutralised jatropha oil was 91%. The yield from crude castor oil was 61% and from neutralised oil was 76%. These results indicate that neutralisation of the oil improves biodiesel yield. Castor biodiesel yields were low. This was due to the formation of soap from crude castor oil during transesterification. There was also some oil loss from castor during neutralisation and this was responsible for the low biodiesel yield from neutralised castor oil.

Table 3. Properties of biodiesel from crude and neutralised oils

Oil / Fuel	Type	Kinematic Viscosity [mm^2/s]	Flash Point [^oC]	Acid Value [mg KOH/g]	Cloud Point [^oC]	Density [Kg/m^3]	Calorific Value [MJ/kg]
Jatropha Biodiesel	Crude	5.50±0.91	167±1.00	2.30±0.03	1±0.50	871±1.00	40.83±1.01
	Neutralized	5.25±0.05	166±1.53	0.50±0.03	-6±1.00	871±3.00	42.10±2.1
Castor Biodiesel	Crude	17.10±0.31	178±1.52	2.11±0.10	<-13±2.00	910±3.00	29.60±1.0
	Neutralized	10.75±0.27	160±1.53	0.35±0.02	<-13±1.00	900±9.16	30.40±0.9
Palm	[31]	4.40	178	-	16	855	39.70
Biodiesel	[32]	4.50	174	-	16	855	41.30

Table 4. Comparison of biodiesel properties with petro-diesel and the standards

Property	Jatropha Biodiesel	Castor Biodiesel	Petro-Diesel ASTM D 975-98	Standard ASTM D 6751	Standard EN14214
Kinematic Viscosity [mm^2/s]	5.25±0.05	10.75±0.27	1.9-4.1	1.9 - 6.0	3.5-5.0
Flash Point [^oC]	166±1.53	160±1.53	60-80	130 min	120 min
Acid Value [mgKOH/g]	0.50±0.03	0.35±0.02	-	0.50 max	0.50 max
Cloud Point [^oC]	-6±1.00	<-13±1.00	-15-{-5}	Report	-
Calorific Value [MJ/kg]	42.15±1.30	30.40±0.90	42-46	-	-

4. Conclusion

Biodiesel derived from neutralised jatropha oil is suited for use in diesel engines given that its kinematic viscosity, flash point, cloud point, and calorific value conform to the recommended international standards. This means that neutralisation alone is sufficient to purify jatropha oil for biodiesel production. Savings could be made from the expensive refining processes when jatropha oil is just neutralised and made into biodiesel.

On the other hand, biodiesel from castor oil is not suitable for use in diesel engines due to the high viscosity value. Alternative methods of reducing viscosity like blending castor biodiesel with petro-diesel should be tried. Refining castor oil prior to transesterification should be undertaken in order to get better quality and yield of castor biodiesel. Alternatively improvements on the neutralisation conditions of castor oil could be tried to reduce the oil loss and improve castor biodiesel yield. However, this has to be done with care since castor oil is sensitive to vigorous conditions such as high mixing speed and high temperatures. Based on comparative study, neutralisation of the oils generally improves the quality of biodiesel by lowering amounts of free fatty acids and increasing yield.

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