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Effect of Triacetin on the Quality of Biodiesel Gotten from *Jatropha Curcas L*. and *Thevetia Peruviana S*. Oils

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ABSTRACT

In recent times, there has been a growing need to enhance the quality of biodiesel and consequently increase the acceptability and consumption of biodiesel. The aim of this study was to evaluate the effect of an additive, Triacetin on biodiesel sourced from *Jatropha curcas L*. and *Thevetia peruviana S*. Some basic fuel parameters were tested for biodiesel quality using the American Standard Testing Material (ASTM) such as kinematic viscosity, cloud point, pour point, cetane number and specific gravity and flash point. The results for *Jatropha curcas L*. are as follows: kinematic viscosity, 4.68mm²/s, cloud point, 3°C, pour point, 3°C, cetane number, 57.5, specific gravity, 0.871g/cm³ while for *Thevetia peruviana s*, the following results were gotten: kinematic viscosity, 4.81mm²/s, cetane number, 60.2, cloud point, 2°C, pour point, 1°C and specific gravity, 0.870g/cm³. The flash point of both biodiesels was low when compared to the ASTM standards.

Key Words: ASTM, biodiesel, biodiesel quality, fuel properties, triacetin

INTRODUCTION

There is an intense need for renewable and pollutant-free sources of energy [1]. Increasing concerns about environmental pollutions, the soaring price of petroleum products together with the depletion of fossil fuels have led to considerable research to identify alternative fuel sources [2]. Biodiesel, a biodegradable energy, is defined by Deng *et al* [3] as mono alkyl esters of fatty acids derived from renewable sources such as vegetable oils and animal fats via transesterification processes. Biodiesel has similar and sometimes better physical and chemical properties than petro-diesel, such as flash point, ultra-lower sulphur concentration, better lubricating efficiency, high cetane number and fewer pollutants [3]. Biodiesel has many

advantages which include: its sustainability, safe usage in all conventional diesel engines, offers the same performance and engine durability as petro-diesel fuel, nonflammable and nontoxic reduces tailpipe emissions, visible smoke and noxious fumes and odours [4]. The use of biodiesel from renewable oil sources have the benefits of lower sulphur content, lower aromatic content, higher heat content, biodegradability, readily availability and liquid nature portability [5]. Although in spreading the use of biodiesel in a commercial scale in several countries, it is essential to develop standards to ensure good biofuel quality. Biodiesel is characterized by determining its physical and fuel properties including density, viscosity, iodine value, acid value, cloud point, pour point and volatility according to ASTM standards. In spite of the advantages of biodiesel over diesel fuel, it has some disadvantages. These include injector choking, engine compatibility, and high price, higher Nitrogen oxide (NOx) emissions than diesel [6], relatively poor low temperature flow properties compared to diesel and also the effects of oxidative degradation caused by contact with ambient air (auto oxidation) during long-term storage present a legitimate concern in terms of maintaining the quality of biodiesel fuel.

Due to these disadvantages of biodiesel over diesel fuel, published research has shown that the physical properties of biodiesel can be circumvented by the use of different additives, so that it can solve the problems associated with the properties of biodiesel for their large scale usage in diesel engines. The selection of the additives depends on economic feasibility and toxicity, fuel blending property, additive solubility, flash point of the blend, viscosity of the blend, solubility of water in the resultant blend, and water partitioning of the additive [7].

A number of additives have been tried by different researchers for improving the performance and also reducing emissions from diesel engines. The quality and fuel properties of biodiesel have shown even better improvement with additives. Therefore, the aim of this research is to ascertain the effect of Triacetin on the quality of biodiesel gotten from *Jatropha curcas L*.and *Thevetia peruviana S*. oils. The use of these additives in biodiesel will hopefully help in solving a lot of technical problems which limits the acceptability of biodiesel as an alternative fuel in all conditions.

MATERIALS AND METHODS

Matured *Jatropha curcas L*. seeds and *Thevetia peruviana S*. seeds were collected from National Research Institute for Chemical Technology (NARICT) Zaria, Kaduna State, Nigeria. The dry

pods of both seeds were de-hulled manually by crushing the hard shell with a locally fabricated nut-cracker machine. After which the shells were carefully separated from the kernels by hand. The seeds were crushed to produce fine seed powder so as to expose a larger area from which the oil was extracted.

Oil Extraction: The ground seeds (1.00kg) were subjected to mechanical pressing and thus were extracted manually (without any application of solvent). The expelled oil was filtered using a cloth sieve into a beaker. The difference in weight of seed sample before and after extraction of oil was taken as the weight of extractedoil.

The percentage oil yield was then calculated thus:

% Oil yield = W<u>eight of oil (g) X 100</u> Weight of ground seed (g)

Equipment: Gas Chromatograph-Mass Spectrometer (GCMS-QP2010 Plus Shimadzu, Analytical Equipment, Japan), magnetic stirrer, Fourier transform infrared red spectrophotometer (FTIR 8400S Shimadzu).

Preparation of CaO/Al₂O₃ Catalyst: 15g of hydrated lime was dissolved in 500 ml beaker with distilled water. After thorough mixing, 85g of alumina was added and stirred. The mixture was dried over a hot plate. The dried mixture was loaded into a crucible and was calcined in a muffle furnace at 700 °C for 90 minutes. The calcined catalyst was ground into powder.

Tranesterification procedure: The method of laboratory scale biodiesel production employed by Maher *et al* [8] was adopted. 100 g of the esterified oil was transesterified with 20 g (20% m/m of oil) of methanol, 1.5 g (1.5% m/m of oil) catalyst (Calcium Oxide Alumina). This mixture was poured into a blender which was secured tightly, switched on at full agitation speed and the agitation was maintained for 15 minutes. The product was first filtered to remove catalyst. The filtrate was separated into glycerol and biodiesel with a separating funnel. The percentage yield of biodiesel (fatty acid methyl ester) was calculated as:

Yield= W<u>eight of biodiesel produce x100</u> Weight of oil used

Synthesis of Triacetin additive: The method of batch reaction of Triacetin by Mufrodi et al [9] was adopted. Triacetin was obtained by acetylating a mole of glycerol to nine moles (1:9) of

acetic acid using sulphuric acid as a catalyst. The reaction was carried out in a three-necked flask equipped with a heating mantle, stirrer and a thermometer at a temperature of 90-120°C and 290 rpm stirring speed. The reactants, glycerol and acetic acid, were mixed directly into the three-necked flask along with the sulphuric acid (H₂SO₄) catalyst. The reaction was carried out at a mixing time of 30-60 minutes. The mixture was continuously stirred during the reaction using a magnetic stirrer. The products which contained sulphuric acid were neutralized by the addition of same ratio of sodium hydroxide (NaOH). After neutralization, the contaminated catalyst was removed by using recirculating water respirator equipment. Then, the product containing monoacetin, di-acetin, tri-acetin, glycerol and unreacted acetic acid were poured into a rotary evaporator to remove the solvent from the product.

Biodiesel fuel characterization: Biodiesel properties of *Jatropha curcas L.* and *Thevetia peruviana S.* methyl ester were determined according to ASTM test methods [10]. The following properties were measured experimentally: density (ASTM D5002), kinematic viscosity (ASTM D445), iodine value, pour point (D5949), cloud point (ASTM D2500), flash point (ASTM D93), cetane number (ASTM D613), oxidative stability (EN14112).

Triacetin characterization: The glycerol, mono-, di-, triglcerides, Triacetin were determined according to EN14105 and ASTM D6584 using a QP2010 plus Shimadzu Gas Chromatograph and Fourier Transform Infrared Red Spectrophotometer (FTIR 8400S Shimadzu).

RESULTS AND DISCUSSION

Fatty Acid Profile of Jatropha curcas L. and Thevetia peruviana S. Methyl Eesters

The Gas chromatograms obtained show the fatty acid compositions in both *Jatropha curcas L*. and *Thevetia peruviana S*. methyl esters (Figures 1 and 2), while their respective proposed peaks were identified and tabulated in Tables 1 and 2. The predominant fatty acids are palmitic acid, oleic acid, and stearic acid. It further shows a percentage of saturated, monounsaturated and polyunsaturated fatty acids of both methyl esters.

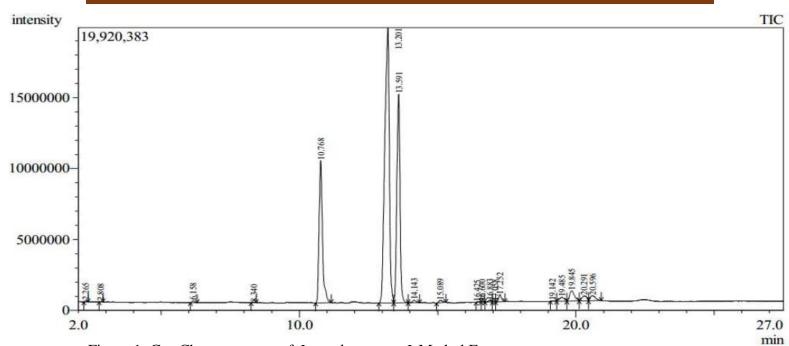


Figure 1: Gas Chromatogram of Jatropha curcas L Methyl Ester

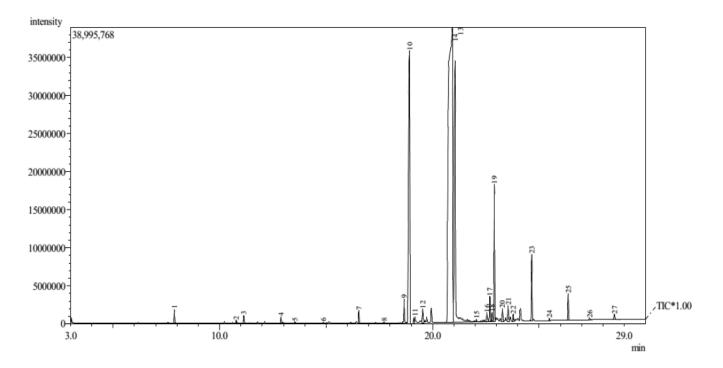


Figure 2: Gas Chromatogram of Thevetia peruviana methyl ester

Peak no	Area %	Compound		
2	2.37	Dodecanoic acid, methyl ester		
		(Lauric acid)		
3	6.44	Myristic acid, methyl ester		
6	2.31	Hexadecanoic acid		
		(Palmitoleic acid)		
7	31.63	Hexadecanoic acid, methyl		
		ester (Palmitic acid)		
8	29.51	9-Octadecenoic acid		
		(Z), methyl ester (Oleic acid)		
10	2.18	9-Octadecenoic acid, methyl		
		ester (Elaidic acid)		
11	3.50	Octadecanoic acid, methyl ester		
		(Stearic acid)		
13	0.69	Eicosanoic acid, methyl ester		
		(Arachidic acid)		
14	2.22	Docosanoic acid, methylester		
		(Behenic acid)		

Table 1: Peak areas of *Jatropha curcas L*. methyl ester gas chromatogram and their suggested compounds

Table 2: Peak areas of <i>Thevetia peruviana S</i> . methyl ester gas chromatogram and their suggested
compounds

Peak No	Area %	Compound
9	0.81	9-Hexadecenoic acid ,methyl ester (Palmitoleic acid)
10	16.69	
		Hexadecanoicacid,methyl ester (Palmitic acid)
13	53.61	7-Octadecanoic acid,methyl ester (Oleic acid)
14	16.15	Ocatdecanoic acid, methyl etser (Stearic acid)
19	4.22	Arachidic acid, methyl ester
23	1.92	Behenic acid, methyl ester
25	0.85	Tetracosanoic acid,methyl ester (Lignoceric acid)

Table 3: Fatty Acid Profile of Jatropha curcas L. and Thevetia peruviana S. Methyl Ester				
Fatty Acid	Molecular Formula	Jatropha curcas methyl ester (JCME) (%)	Thevetia peruviana methyl ester (TPME) (%)	
Myristic Acid (14:0)	C ₁₅ H ₂₀ O ₂	6.44	-	
Palmitic Acid(16:0)	$C_{17}H_{34}O_2$	31.63	16.69	
Palmitoleic Acid (16:1)	C ₁₇ H ₃₂ O ₂	2.31	0.81	
Stearic Acid (18:0)	$C_{19}H_{38}O_2$	3.50	16.15	
Oleic Acid (18:1)	$C_{19}H_{36}O_2$	29.51	53.61	
Arachidic Acid (20:0)	$C_{21}H_{42}O_2$	0.69	4.22	
Behenic Acid (22:0)	$C_{23}H_{46}O_2$	2.22	1.92	
Lignoceric Acid (24:0)	$C_{25}H_{50}O_2$	-	0.85	
Elaidic Acid	$C_{18}H_{34}O_2$	2.18	-	

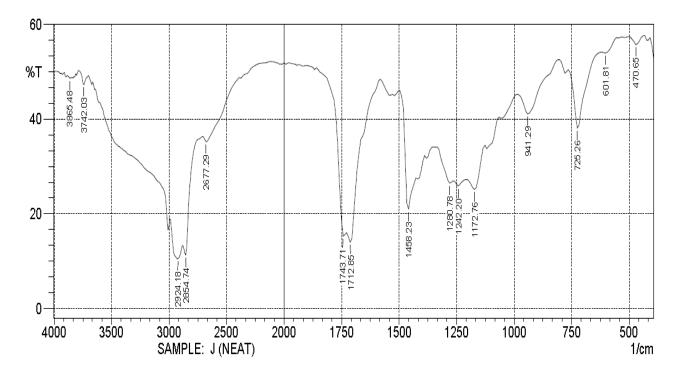


Figure 3: FTIR Spectra of Jatropha curcas L. Methyl Ester

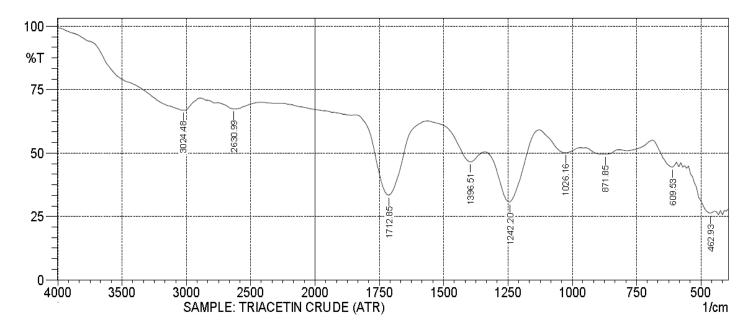


Figure 4: FTIR Spectra of Thevetia peruviana S. Methyl Ester

FTIR Spectra of Jatropha curcas L. and Thevetia peruviana S. methyl esters

The results presented in Figures 3 and 4 are the FTIR spectra of *Jatropha curcas L*. and *Thevetia peruviana S*. methyl esters respectively. Both methyl esters show O-H stretching vibration bands at 2650-3482 cm⁻¹ while the ester groups appeared between 1740-1748 cm⁻¹ although with slight differences in positions for both samples. The C-H bend of alkanes was present within the range of 1450-1462 cm⁻¹ in both samples. Subsequently, C-O stretching appeared at 1160-1170 cm⁻¹ in *Thevetia peruviana S*. methyl ester, while at 722-726 cm⁻¹, a =C-H band was observed for both *Jatropha curcas L*. methyl ester and *Thevetia peruviana S*. methyl ester.

The fatty acid methyl esters of *Jatropha curcas L*. and *Thevetia peruviana S*. (biodiesels) have unique FTIR absorption of carbonyl (C=O) stretching vibration near 1750-1744 cm⁻¹ and C-O stretching vibration in 1160cm⁻¹ region. The band region of 1454.38 cm⁻¹ – 1458.23 cm⁻¹ can be ascribed to the bending vibration of C-H methyl groups in the fuel as well as C=C stretching vibration of the aromatics in the fuels. However the presence of groups with carbon to carbon double bonds (C = C, = C-C) can cause the fuel to remain in liquid state but may be liable to possible oxidation during storage [11]. This spectra separation among the functional groups of vegetable oils, biodiesel and fossil diesel forms the basis of characterization and quantization of FAMEs in biodiesel and in blended biodiesel-diesel fuel through IR spectroscopy [12].

The influence of transesterification was indicated by the formation of a signal at 1458 cm⁻¹ which corresponds to the deformation vibration of methyl ester group (CO)-O-CH₃ present in biodiesel spectrum and absent in the oil spectrum. Similar result was reported by [12-14]. Another visible transformation revealed by the IR spectra of FAMES is a broad signal 1172 cm⁻¹ of C-O group in the ester controlled area.

Triacetin Additive

Additives are utilized for the enhancement of combustion efficiency and ignition, stabilization of fuel mixtures, shielding the motor from abrasions and wax depositions, limitation of pollutant emissions among other vital features. Within the pool of additives, triacetin wasconsidered for this work due to their varying relevant properties such as flash point, viscosity, density, solubility etc. Triacetin as an additive is a colourless, odourless liquid and a low molecular weight glyceride. According to investigation, addition of triacetin in small percentage in the blends of biodiesel increases the oxygen content of fuel which improves the combustion efficiency of biodiesel and reduces emissions [15].

Although it was documented by Saka *et al.* [16], the addition of triacetin decreased the cetane number, increased density and viscosity, slightly improved the cold flow properties and oxidation stability.

Fuel Properties	TPME	JMPE	JCME + TR	TRME + TR	ASTM STD
Specific G. (g/cm ³)	0.867	0.868	0.871	0.870	-
Flash Point (°C)	107 ± 1.8	125	82	65	100 - 170
Cloud Point (°C)	5	5	3	2	-3 to 12
Pour Point (°C)	2	-3	-3	-2	-15 to 10

Table 3: Fuel properties of *Thevetia peruviana S* and *Jatropha curcasL* methyl esters alongside triacetin additive

Total Sulphur Content (% wt)	0.013	0.016	0.015	0.018	15max
Kinematic Viscosity (mm ² /s)	4.4	4.6	4.68	4.81	1.9 - 6.0
Cetane Number	58.9	54	59.5	60.2	48 - 65

TPME: *Thevetia peruviana S* methyl ester; **JMPE:** *Jatrophacurcas L*. methyl ester; **JCME** + **TR**: *Jatrophacurcas L*. methyl ester and triacetin additive; **TRME** + **TR**: *Thevetia peruviana S*. methyl ester and Triacetin additive; **ASTM STD**: American Standard Testing Machine Standards

Comparatively, the fuel properties of biodiesel gotten from *Thevetia peruviana S*. and *Jatropha curcas L*. and the consequent effect of triacetin additive on these methyl esters were investigated and the results are presented in Table 3 alongside the ASTM.

Specific Gravity

The specific gravity of the methyl esters of *Jatropha curcas L*. and *Thevetia peruviana S*. are 0.868 g/cm³ and 0.867 g/cm³ respectively. Similar result of 0.85 was obtained by Dangoggo *et al.* [17]. A specific gravity of 0.871g/cm³ and 0.870g/cm³ were gotten triacetin with *Jatropha curcas L* (JCME+TR), and for *Thevetia peruviana S*, respectively (TPME+TR). The values gotten indicate an increase in specific gravity when blended with additives [18]. These values were obtained at optimum yield and are within the stipulated limits of ASTM (0.90max). The higher the specific gravity of a fuel, the greater the mass of fuel injected into the engine and hence more power output [19].

Kinematic Viscosity

The kinematic viscosity of the fuel samples was determined at 40 °C. The value of kinematic viscosity of *Thevetia peruviana S.* and *Jatropha curcas L.* methyl esters were 4.4 mm²/s and 4.6 mm²/s respectively. A similar work with result 5.25 mm²/s was obtained by Aldo *et al* [19] for *Jatropha curcas L.* methyl ester and 4.33 mm²/s obtained by Dibakar *et al* [20] for *Thevetia peruviana S.* methyl ester. According to ASTM and EN standard for biodiesel, kinematic viscosity must be between $1.9 - 6.0 \text{ mm}^2$ /s and $3.5-5.0 \text{ mm}^2$ /s respectively. The result above falls within the range of both standard limits which indicates the presence of short chain unsaturated methyl fatty esters and is likely to produce less deposits when burnt in combustion engines. The

kinematic viscosity for *Jatropha curcas L* and *Thevetia peruviana S*. methyl esters with triacetin (JCME+TR, TPME+TR) were 4.68 mm²/s and 4.81 mm²/s respectively. From the results obtained, it can be stated that the addition of triacetin to both biodiesel samples resulted in an increase in the viscosity. However, the viscosity only increased slightly. Viscosity increases with an increasing content of saturation, i.e. as the chain length increases the viscosity of the methyl ester increases [21].

Cetane Number

The cetane number is defined by the ability of fuel to ignite quickly after being injected. Consequently, higher cetane numbers help ensure good cold start properties and minimize the formation of white smoke [22]. The results establish that these biodiesels have higher cetane number when compared to petroleum diesel fuel. *Jatropha curcas L*. methyl ester had a cetane number of 58.9 while *Thevetia peruviana S*. methyl ester was 54. The cetane numbers of both biodiesels were found to be within the ASTM. However, the high cetane numbers of the biodiesels obtained indicate its good ignition quality [17]. The cetane numbers for *Jatropha curcas L*, and *Thevetia peruviana S*. methyl esters with triacetin were 59.5 and 60.2 respectively. These cetane numbers reflect an increase on adding triacetin to *Jatropha curcas L*. and *Thevetia peruviana S*. methyl esters with the dilution bythe triacetin additive.

Cloud and Pour Points

The cloud point and pour pointswere 5° C and 2 °C for *Thevetia peruviana S*. methyl ester and 5 °C and -3 °C for *Jatropha curcas L*. methyl ester respectively. While the values of cloud point and pour points obtained on adding triacetin to both biodesel samples were 3 °C and -3°C for *Jatropha curcas L*. methyl ester (JCME+TR) and 2 °C and -2 °C for *Thevetia peruviana S*. methyl ester (TPME+TR) respectively. This marked decrease in the cloud points of both biofuels gives them the ability to burn efficiently in extremely cold regions. In Nigeria, the biodiesel samples will tend to retain their flow properties.

Flash Point

Flash point of a fuel is the temperature at which it will ignite when exposed to a flame or a spark. Flash point varies inversely with the fuel's volatility. The flash point is the lowest temperature at which fuel emits enough vapour to ignite. The flash point of *Jatropha curcas L*. methyl ester and

Thevetia peruviana S. methyl ester as seen in Table 3 are with values 125 °C and 107 °C respectively. These values fall within the ASTM standards of 100-170°C. Similarly, a slightly higher result (140 °C) was obtained by Dagoggo *et al* [17] for *Jatropha curcas L.* methyl ester and a slightly lower result (98 °C) for *Thevetia peruviana S.* methyl ester was obtained by Dallatu [12]. The Flash point of *Jatropha curcas L* and *Thevetia peruviana S.* methyl esters with Traicetin (JCME+TR and TPME+TR) were 82 °C and 65 °C. The decrease in the flash point of the feedstock on the addition of an additive indicates an improvement in the volatile characteristics of the fuel. This is because the additive has a high boiling point which makes the biodiesels become volatile thereby causing a reduction in the flash point and this is in agreement with works found in literature.

Total Sulphur Content

The total sulphur content of *Jatropha curcas L*. methyl ester and *Thevetia peruviana S*. methyl ester as shown in Tables 3 were 0.013% and 0.016%. These values are lower than the ASTM approved limit of 15% max for biodiesels [23]. These results signify the importance of biodiesel in diesel application engines for reduction of sulphuric acid pollutant emissions and protection of exhaust catalytic systems [24]. There was no marked difference in the total sulphur contents of both biodiesels on the addition of triacetin; 0.015 and 0.018 (JCME+TR and TPME+TR) respectively. Invariably, the combustion of *Jatropha curcas L*. methyl ester and *Thevetia peruviana S* .methyl ester will therefore yield products with low oxides of sulphur making it more environmentally friendly than fossil diesel.

CONCLUSION

The addition of triacetin to *Jatropha curcas L*. and *Thevetia peruviana S*. methyl esters had enhanced their fuel properties. Some of these fuel properties (specific gravity, kinematic viscosity and cetane number) of the *Jatropha curcas L*. and *Thevetia peruviana S*. methyl esters increased on the introduction of triacetin; while cloud point, pour point and flash point all tend to decrease.

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