

SYNTHESIS AND CHARACTERIZATION OF BIODIESEL FROM AVACADO PEAR OIL

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Abstract

The world energy demand is increasing by day as a result of increase in population and high spate of industrialization. This has therefore resulted in the search of alternative energy sources to replace the fast depleting fossil fuels. This research work is therefore focused on the synthesis and characterization of avocado pear oil fatty acid methyl ester (APOFAME) as a renewable energy. The oil content of avocado pear was extracted using solvent extraction method. Avacado pear oil was characterized based on American Society for Testing and Materials (ASTM) method. The fatty acid profile was determined using gas chromatography mass spectrometry while the functional group of the oil was analysed using Fourier transform infrared spectroscopy. The effect of process parameter on the yield of APOFAME was investigated using one factor at a time method. The APO was pretreated to reduce the free fatty acid below 1% and then traesterified using ethanol in the presence of potassium hydroxide (KOH) catalyst. The fuel properties of the APOFAME produced was determined based on ASTM standards. The physiochemical properties of APO , free fatty acid, saponification value, iodine value , kinematic viscosity, fire point, flash point, cloud point, pour point, density , moisture content, gave the values 7.15%, 201.4mgKOH/g, 74.8gI₂/100g, 38.5mm²s⁻¹ @ 40⁰C, 167⁰C, 120⁰C, 13⁰C, 3⁰C 919Kg/m³, 6% respectively. The fatty acid profile of APO shows the constituents to be lauric acid 12.30%, palmitic acid 24.20%, stearic acid 18.20%, myristic acid 14.54%, while the unsaturated fatty acid constituents are linolenic acid 10.61%, sapentaenoic acid 12.57%, and linoleic acid 7.55%. The process parameters , catalyst concentration, reaction temperature, ,methanol to oil molar ratio and reaction time greatly affected the biodiesel yield as their increase resulted in the increase of biodiesel yield until the optimum parameter was reached when the yield started decreasing. The experimentally determined properties of the APOFAME; acid value, density, kinematic viscosity, fire point, flash point, cetane number, refractive index, calorific value, iodine value, cloud point and pour point gave the values, 0.29, 873Kg/m³, 4.95mm²s⁻¹, 160⁰C, 154⁰C, 62.69, 1.5464, 34.683MJ/Kg, 34.2gI₂/100g, 7⁰C, 4⁰C respectively.

Key words: Avacado pear oil, characterization, synthesis, transesterification

1.0 INTRODUCTION

The Global energy demand is sky-rocketing due to increasing world population and industrialization. The world has been heavily dependent on coal, petroleum and natural gas for energy and feedstock for industrial production. These energy sources are commonly termed as fossil or nonrenewable resources (William, 2006). These resources are extracted from the earth's crust, processed and burnt as fuel or used as feedstock in the chemical industries. The burning of fossil fuels cause environmental concerns such as greenhouse gas emission, which is the major substance responsible for climate change. Other harmful substances released during fossil fuel production and utilization includes sulphur oxides (SO_x), nitrogen oxides (NO_x) and methane (Lee and Shah, 2013). The world economy depends on energy generation, hence the consequences of inadequate energy could be severe. These have prompted organizations, educational and research institutions to look for other sources of energy that are sustainable, renewable, with less negative effect on the environment. Among various options investigated for diesel fuel, biodiesel obtained from vegetable oil and other sources has been universally recognized as one of the contenders for reduction of exhaust emission (Fukuda et al 2001). However the bulk of biodiesel produced all over the world now has edible oil as its feedstock. This has therefore raised the fear of many researchers that the continuous use of edible oil for biodiesel production might stress the food uses, prize, production and availability of these oils. Consequently this has ignited research into the use of non-edible oil for biodiesel synthesis. Biodiesel, a mono-alkyl ester of long chain fatty acid has properties that

approximate that of diesel with added advantages of high lubricity, high cetane number and being highly biodegradable. It is a promising nontoxic alternative fuel used in the transport sector. Biodiesel is produced by the reaction of fat with monohydric alcohol. Various processes have been adopted for biodiesel production from vegetable oil and animal fat, namely; microemulsion with alcohol, catalytic cracking, pyrolysis and transesterification (Demirbes, 2009; Leung, et al 2010; Lu, et al 2009; Aderemi and Hamid, 2010). Among these methods, transesterification is the key and the most important process for production of a cleaner and environmentally safe biodiesel (Younis et al 2009; Athanatho et al 2004). Transesterification means conversion of one type of ester to another. During transesterification a basic catalyst breaks the fatty acid from the glycerine one by one. If an alcohol typically methanol contacts a fatty acid, it will bond and form biodiesel (Vendkata et al., 2012).

The global trend towards increased use of renewable energies has led to investigation of non-traditional oil producing crops. Some crops have been discovered in the tropical Sub-saharan regions of Africa that have potential for use as bio-fuel feed stocks. Oil seeds and feedstock such as Jatropha, cotton, palm kernel, soy beans and rice bran has been proposed as Potential sources of oil for biodiesel production .In the course of this research, the use of avocado pear oil as a possible biodiesel feedstock will be considered. Avocado (*Persea Americana* Mill) of the plant family Lauraceae produce fruit with high oil content (Mooz et al., 2012). The pulp or mesocarp (fleshy part of the fruit) makes up 60 to 75% of the total weight of avocado fruit (Costagli and Betti, 2015). Mesocarp is composed of parenchyma cells that surround uniformly distributed specialized oil containing idioblast cells (Reddy et al., 2012). The endocarp (stony part of the fruit) makes up 13% of the total weight of the fruit. Avocado is a tropical fruit that stands out for its high nutritional value (Mooz et al., 2012). The oil consist mainly of saturated fatty acid and low amount of polyunsaturated fatty acid (Ogunwusi and Ibrahim, 2016). Avocado oil has been used for cooking, cosmetics, and treating diseases, but has not been widely studied as a good source of oil for renewable energy (Knothe, 2013).

2.0 MATERIALS AND METHODS

2.1 Materials

Mashed avocado pear (plate 2.1), reagents, glass wares, equipments including gas chromatography mass spectrometer (GC-MS), Fourier transform infrared spectroscopy (FTIR), viscometer, magnetic hot plate, soxhlet extractor. Design expert software version 12.0 etc.

2.2 Experimental Methods

2.2.1 Sample preparation

The waste or mashed avocado pear fruits used were collected from a local market in Uga Aguata L.G.A, Anambra state. The seed, seed coat and skin were removed and the pulp was sundried for 7 days followed by oven drying till it was sufficiently dry for application of solvent extraction.



Plate 2.1 Avacado pear fruit

2.2.2 Extraction of oil from avocado pulp

Solvent extraction was used for extraction of oil from the dried avocado pulp. Ethanol was employed in the extraction of oil from the dried avocado pulp. The solvent choice of ethanol for extraction of oil from avocado pulp was based on the study by (Genevieve, et al. 2019 ;Anawe & Folayan, 2018). Both researchers reported higher yield of oil from ethanol extraction when compared to extraction with n-hexane.

3kg of the dried, ground pulp was introduced into a plastic container containing 3 liters of ethanol. The mixed content of the container were vigorously shaken after covering the container. The container was made air tight to prevent evaporation of the ethanol and then kept to macerate for a day. Then the dissolved oil in ethanol was decanted and the slurry filtered. The filtrate was then distilled to recover the ethanol at 65⁰C (AOAC 1990). The percentage oilyield was calculated as:

$$\% \text{ oil yield} = \text{weight of oil obtained} \div \text{weight of seed sample} \times 100 \quad (2.1)$$

2.2.3 Characterization of avocado pear oil

The physiochemical properties of the oil extracted from avocado pear was characterized based on American Society for Testing Materials, ASTM 6751 (1973) method. Analytical equipments, GC MS (QP2010 plus Shimadzu, Japan) and FTIR (M530 Bulk scientific FTIR) were used to determine the fatty acid profile and the functional groups of the oil respectively.

2.2.4 Effect of process parameters on biodiesel yield

The effects of process parameter on biodiesel yield were investigated using one factor at a time method involving keeping a factor constant at a time and varying the others in turn. The four factors investigate are, molar ratio of methanol to oil, catalyst concentration, reaction time and reaction temperature.

2.2.5 Pretreatment of the oil extracted.

The avocado pear oil was first heated on a heating mantle at 110⁰C for 10 minutes for any available moisture to be driven off. The sample was cooled to 60⁰C in a water bath, and then weighed into 500ml three necked round bottomed flask. Then methanol of 60% w/w of oil mixed with concentrated sulphuric acid of 7% w/w of oil was added. A reflux condenser was fitted into the middle arm of the flask and water circulated at the outer jacket of the condenser. A thermometer was inserted into the sample in the flask from one of the side arms. The whole setup was placed on a magnetic heating mantle and heated at 60⁰C for 120 minutes at an agitation speed of 450rpm. The mixture was then transferred into a 250 ml separating funnels where it later separated into three layers comprising of water at the bottom, pre-treated oil in the middle and methanol at the upper layer. The mixture of components were carefully separated by removal the water first, followed by the oil and finally the methanol. Hot distilled water was poured into the oil, shaken and allowed to stand. This was done to wash the esterified oils. After a while, 2 layers were observed; water (below) and oil (above). The water was tapped off

from the separating funnel. The pre-treated oil was poured into beakers and dried carefully in an oven regulated at a temperature of 105⁰C until the residual water evaporated completely. After this process, the pre-treated oil was ready for transesterification (Ogunsuyi et al., 2015)

2.2.6 Production of Biodiesel

2.2.6.1 Transesterification reaction

A 500ml three-necked round bottomed flask fitted with a condenser on the middle arm, a thermometer and sample outlet on the side arms respectively served as the reactor. The heating system consists of an electromagnetic hot plate which heats the reactor and rotates the metal knob in the reactor through an electromagnetic field. Specified quantity of the oil sample was introduced into the flask and the flask content heated to the temperature established for the reaction. Then methanol and the catalyst mixture (KOH) was added in the amount established for the reaction, and the stirrer switched on at a specified speed, taking this moment as zero time of the reaction. The reaction mixture was vigorously stirred and refluxed for the required reaction time. At the end of methanolysis, the transesterified product was made to stand for a day in a separating funnel where it separates into the upper biodiesel layer and the lower glycerol layer. The lower glycerol layer was tapped off first followed by the upper biodiesel layer.

2.2.6.2 Biodiesel purification

After transesterification, the upper ester layer may contain traces of methanol and glycerol. The remaining unreacted methanol has safety risk and might corrode engine components and glycerin within the biodiesel will lessen the fuel lubricity and cause injector coking and other deposits (Hanumanth et al., 2012). Such traces of methanol is soluble in water and therefore is removed by wet washing. A drop of 1M sulphuric acid was added to the biodiesel in a separating funnel. Hot distilled water was as well added and the mixture vigorously shaken. The mixture was allowed to settle when it separates into two, the upper layer consisting of the biodiesel and the lower layer consisting of water and water soluble impurities. The water was discarded and tested with three drops of phenolphthalein indicator. Washing was continued until the waste water was bright and does not turn pink when tested with phenolphthalein. The washed sample was dried by heating at 105⁰C on a laboratory hot plate until all residual water molecules is evaporated. The percentage biodiesel yield is given by the expression

$$\% \text{ biodiesel yield} = \text{volume of biodiesel produced} \div \text{volume of oil used} \times 100 \quad (2.2)$$

2.2.7 Determination of the fuel properties of the avocado pear oil biodiesel

The properties of the biodiesel fuel were characterized based on ASTM standards. The properties characterized for include density, viscosity, iodine value, saponification value, cetane number acid value, free fatty acid, calorific value and flash point.

3.0 RESULTS AND DISCUSSION

3.1 Characteristics of avocado pear oil

3.2.1 Physiochemical properties of APO

The summary of characteristics of avocado per oil are as shown in table 3.1. From the table, it could be seen that the acid value and the free fatty acid of APO, 14.30mg and 7.15% respectively are high. The free fatty acid and the moisture content of the oil are each greater than 1% which is the maximum required of the oil for high yield of biodiesel from alkali transesterification. Oil of high moisture content are prone to hydrolytic oxidation. Again oils of high free fatty acid and moisture content has the tendency for soap formation which inhibits glycerol separation from biodiesel and therefore retards biodiesel production. The oil is therefore pretreated or esterified before being transesterified. The kinematic viscosity and the density of the oil are higher than that of the biodiesel produced from it and much higher than that of diesel. High density and viscosity makes atomization of the oil in internal combustion engine difficult and has been associated with increase in engine deposits, hence they cannot be used directly as biodiesel (Angeilo et al 2005).

Iodine value, a measure of degree of unsaturation of the oil obtained is below 100gI₂/100g oil, indicative of the oil being nondrying and therefore suitable for biodiesel production. High iodine value of oil corresponds to high degree of un-saturation of the fatty acid in the triglyceride, and if heated, such an oil is prone to thermal oxidation and polymerization of the triglyceride causing formation of deposits. The cloud and pour point of 13°C and 3°C respectively determined for the oil are high and therefore unsuitable for cold weather. Peroxide value, an index of rancidity obtained as 16meq/Kg was high and indicative of poor resistance of the oil to peroxidation during storage and handling.

Figure 3.1: Physiochemical properties of APO

Properties	Unit	APO
Acid value	mgKOH/g	14.30
Free fatty acid	%	7.15
Saponification value	mgKOH/g	201.4
Iodine value	(gI ₂ /100g oil)	74.8
Peroxide value	meq/kg	16
Kinematic viscosity	mm ² s ⁻¹ @ 40°C	38.5
Fire point	°C	169
Flash point	°C	120
Cloud point	°C	13
Pour point	°C	3
Refractive index		1.4614
Specific gravity		0.919
Moisture content	%	6
Density	Kg/m ³	919

3.2.2 Fatty acid profile of avocado pear oil

The Fatty acid profile of APO was determined using GC-MS analysis. The individual peaks of the gas chromatogram were identified as shown in figure 3.1. The relative percentage of fatty acids were calculated from total ion chromatography by computerized integrator and results are presented in table 3.2. As shown in the table the saturated fatty acid constituents of the oil were identified as palmitic acid (C16:0) 24.20%, lauric acid (C12:0) 12.30%, myristic acid (C14:0) 14.54% and stearic acid (C18:0) 12.57%. The di-, tri-, and polyunsaturated fatty acid constituents of the oil are linoleic acid (C18:2) 7.55%, linolenic acid (C18:3) 10.61% and sapaentaenoic acid 12.57% respectively.

3.2.3 Fourier transform infrared (FTIR) spectra analysis of vacado pear oil

The fourier transform infrared spectra of APO was analysed using fourier transform infrared spectroscopy (M530 Buck scientific FTIR). This analysis was carried out in order to detect the various functional groups contained by the oil. The FTIR spectrum of APO oil is shown in figure 3.2. The different group assignments of the FTIR spectra of APO are summarized in table 3.3 which shows the presence of mostly alkane, alkynes and hydroxyl groups. The presence of hydroxyl groups are detected at 3853.171, 3539.869, 3149.224, 1390.83 cm⁻¹ with O-H stretching. The vibration of C-H bending of alkanes were evident at 775.3272 and 878.8254 cm⁻¹. The C=C stretching at 2042.03 and 2201.238 cm⁻¹ shows the presence of alkynes. While the C-H stretch at 2805.394 and 1852.449 cm⁻¹ depicts the presence of aldehydes and aromatic compounds respectively. The presence of conjugated alkane was also detected by the C=C stretch at 1622.394 cm⁻¹.

Table 3.2: Summary of fatty Acid Profile of APO

Components Common Name	Systematic Name	Structural Formula	Concentration (%)
Lauric C12	Dodecanoic Acid	$\text{CH}_3(\text{CH}_2)_{10}\text{COOH}$	12.30
Palmitic Acid C16	Hexadecanoic Acid	$\text{CH}_3(\text{CH}_2)_{14}\text{COOH}$	24.20
α Linolenic Acid C18:3	Octadeca-9, 12,15 Trienoic Acid	$\text{C}_{17}\text{H}_{29}\text{C}_2\text{O}_2\text{H}$	10.61
Sapentaenoic Acid	Icosa-5,8,11,14,17- Pentaenoic Acid	$\text{C}_{19}\text{H}_{29}\text{C}_2\text{O}_2\text{H}$	12.57
Stearic Acid C18	Octadecanoic	$\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$	18.20
Linoleic Acid C18:2	Octadeca-9, 12- Dienoic Acid	$\text{C}_{17}\text{H}_{31}\text{C}_2\text{O}_2\text{H}$	7.55
Myristic Acid C14	Tetradecanoic Acid	$\text{CH}_3(\text{CH}_2)_{12}\text{COOH}$	14.54

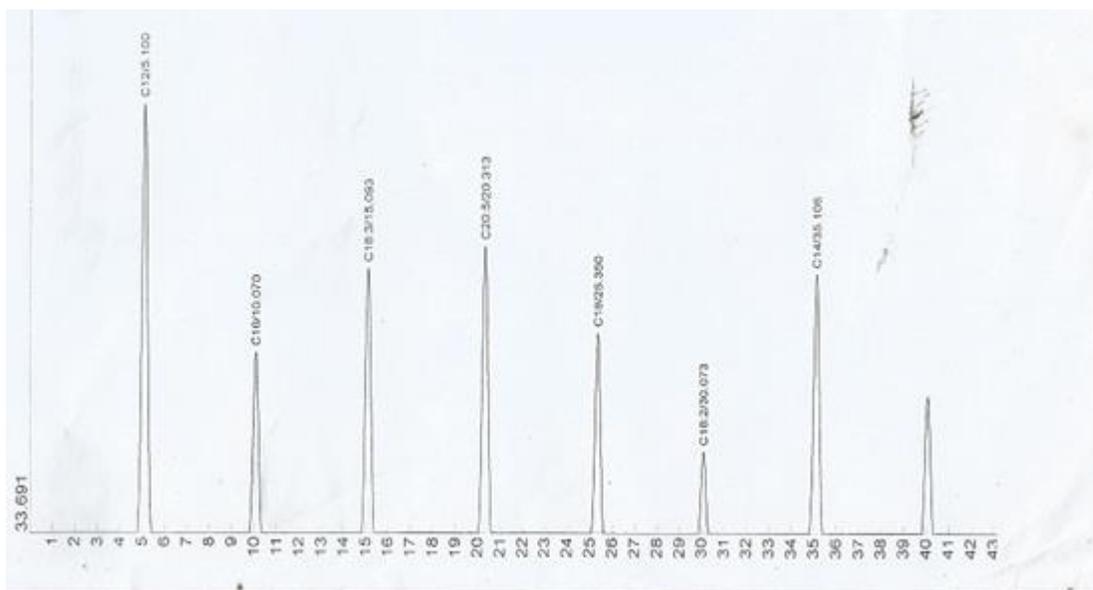


Figure 3.1 GC-MS plot of avocado pear oil

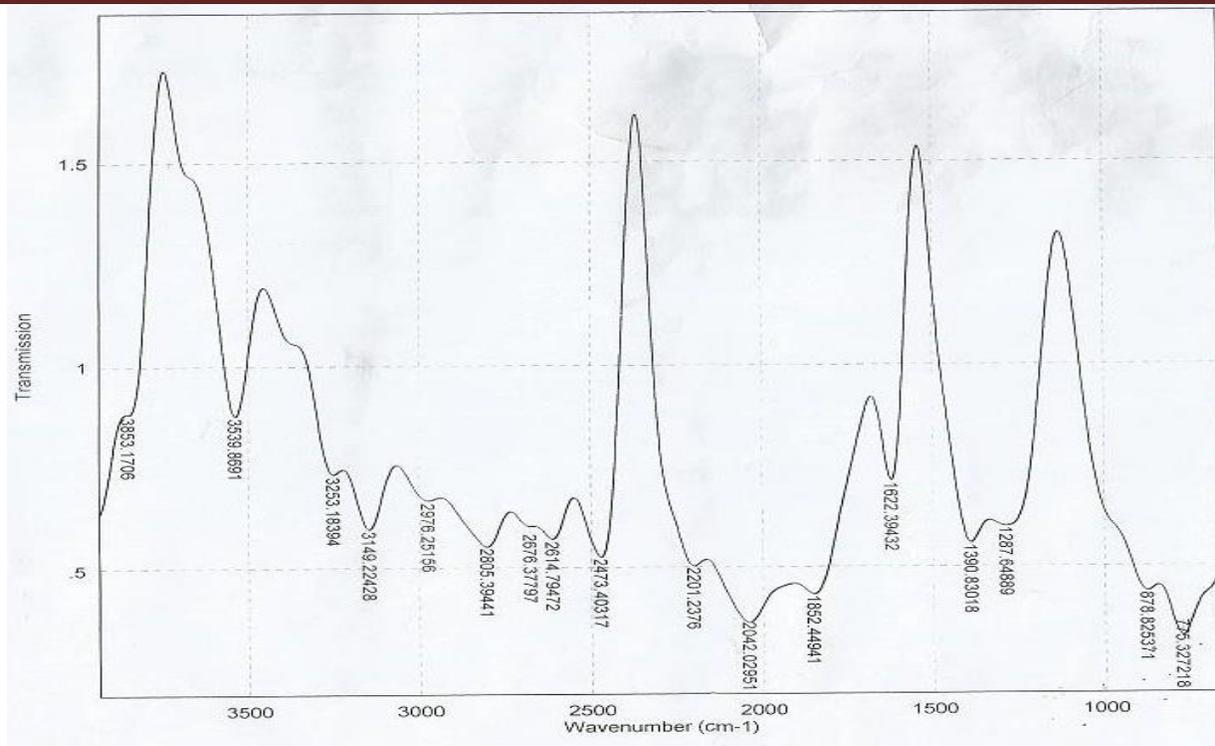


Figure 3.2: FTIR spectra of avocado pear oil

Table 3.3: FTIR functional group frequencies of APO

Frequency wave number (cm ⁻¹)	Types of Vibration	Functional Group
775.3272	Bending	C-H (Alkane)
878.8254	Bending	C-H (Alkane)
1287.649	Stretch	C-O (Aromatic)
1390.83	Bending	O-H (Phenol)
1622.394	Stretch	C=C (conjugated alkene)
1852.449	Stretch	C-H (Aromatic Compound)
2042.03	Stretch	C≡C (Alkyne)
2201.238	Stretch	C≡C (Alkyne)
2805.394	Stretch	C-H (aldehyde)
2976.252	Stretch	C-H (alkane)
3149.224	Stretch	O-H (alcohol)
3253.184	Stretch	Normal polymeric O-H
3539.869	Stretch	O-H
3853.171	Stretch	O-H

3.3 Effect of Process Parameters on avocado pear oil biodiesel yield

3.3.1 Effect of methanol to oil molar ratio on APOFAME yield

The effect of methanol to oil molar ratio on avocado pear oil biodiesel yield is shown in figure 3.3, which reveals that APOFAME yield increased with increase of methanol to oil molar ratio until a maximum yield was attained at 9:1 methanol to oil molar ratio when the yield started decreasing. The decrease in yield beyond the

optimal methanol to oil molar ratio of 9:1 is attributed to the fact that while the increase in methanol to oil molar ratio favours transesterification reaction, very high ratio of methanol to oil decreases the catalytic activity of the catalyst, resulting in the reduction of biodiesel produced. This is in agreement with the findings of Zhang et al, (2003) and Freedman et al (2003). Again Rashid and Anwar (2008) reported that when too much alcohol is used in transesterification reaction, the polarity of the reaction mixture is increased, thus increasing the solubility of glycerol which promote the reverse reaction between glycerol and biodiesel, thereby decreasing the biodiesel yield.

3.3.2 Effect of catalyst Concentration on APOFAME yield

The effect of catalyst concentration on the biodiesel yield is depicted in figure 3.4. The biodiesel yield increased with increase in catalyst concentration and reached a maximum value at catalyst concentration of 1% when the yield started to decrease. The decrease in yield beyond the optimal catalyst concentration results as the excess catalyst react with the triglyceride to form soap which hinders effective dispersion and mixing of the reaction mixture and retards the separation of

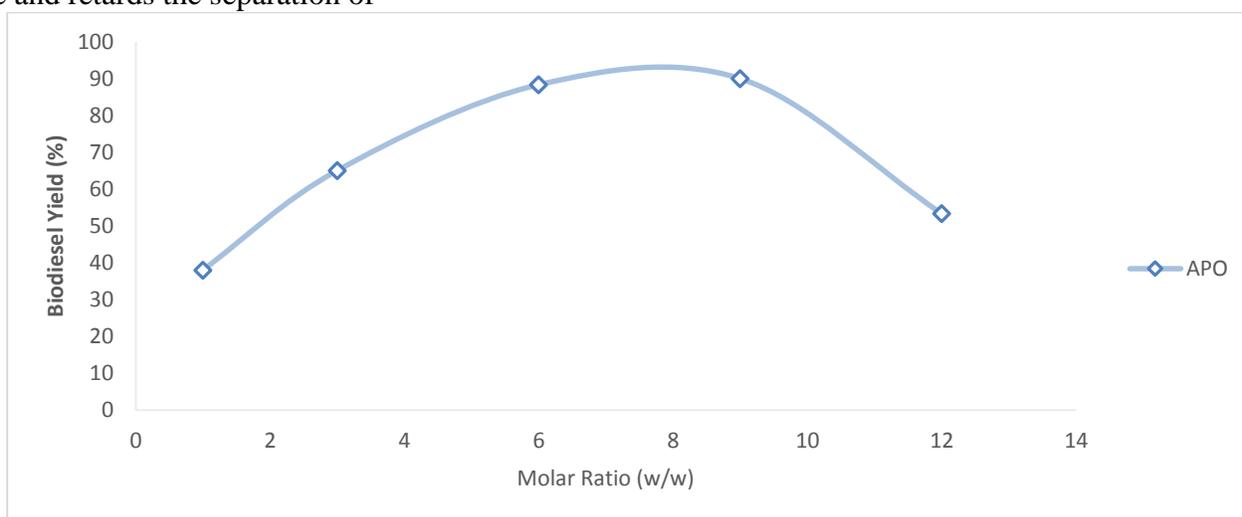


Fig 3.3: Effect of methanol to oil molar ratio on APO biodiesel yield

glycerol from biodiesel and thus give rise to reduction of biodiesel production. This is in conformity with the findings of Jagadele and Jugulkar (2012).

3.3.3 Effect of reaction temperature on APOFAME yield

Avacado pear oil biodiesel yield increased with increase in temperature until the maximum value was attained at 65°C when the yield starts to decrease. This is shown in figure 3.5. The decrease in biodiesel yield beyond the optimum temperature of 65°C results because below the boiling point of methanol (65°C) production of biodiesel is favored but beyond the optimum temperature of 65°C most of the methanol is lost by evaporation, leaving a reaction mixture with higher concentration of alkali catalyst that favour soap formation. The formation of soap retards proper dispersion and mixing of the reaction mixture and hinders separation of glycerol from biodiesel and thus reduced the ester yield. This trend conforms with the findings of Lu et al, (2009).

3.3.4 Effect of reaction time on APOFAME yield

The effect of reaction time on the yield of avocado pear oil biodiesel is shown on figure 3.6. The yield of the biodiesel increased with increase in reaction time and attained a maximum value at the reaction time of 60minutes when it started decreasing. The decrease of biodiesel yield beyond the optimum reaction time of 60minutes may be attributed to the reversal nature of transesterification reaction. For the process conditions

used, the forward reaction leading to ester formation is favoured below 60minutes while the reverse condition leading to ester depletion is favoured at reaction time beyond 60minutes

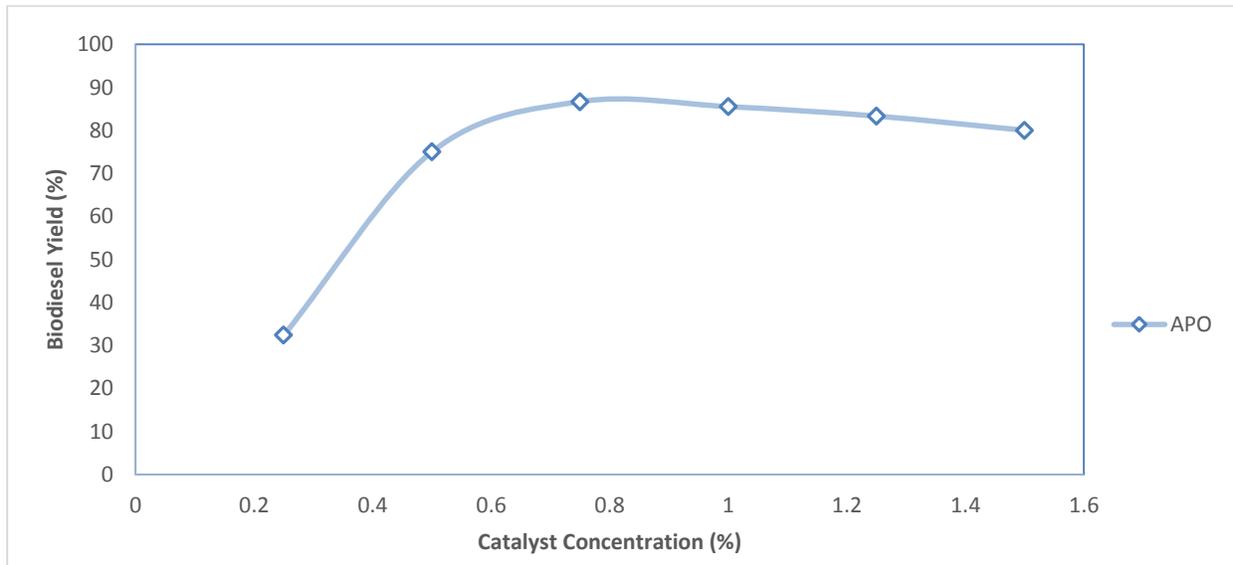


Fig3.4: Effect of Catalyst Concentration on APO biodiesel yield

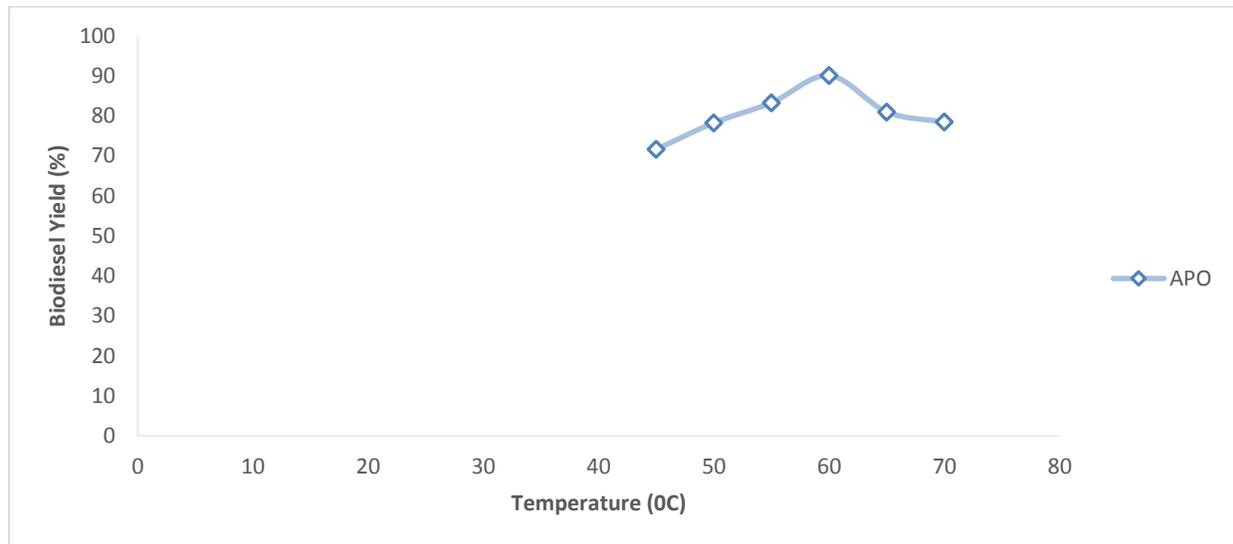


Fig 3.5: Effect of reaction temperature on APO biodiesel yield

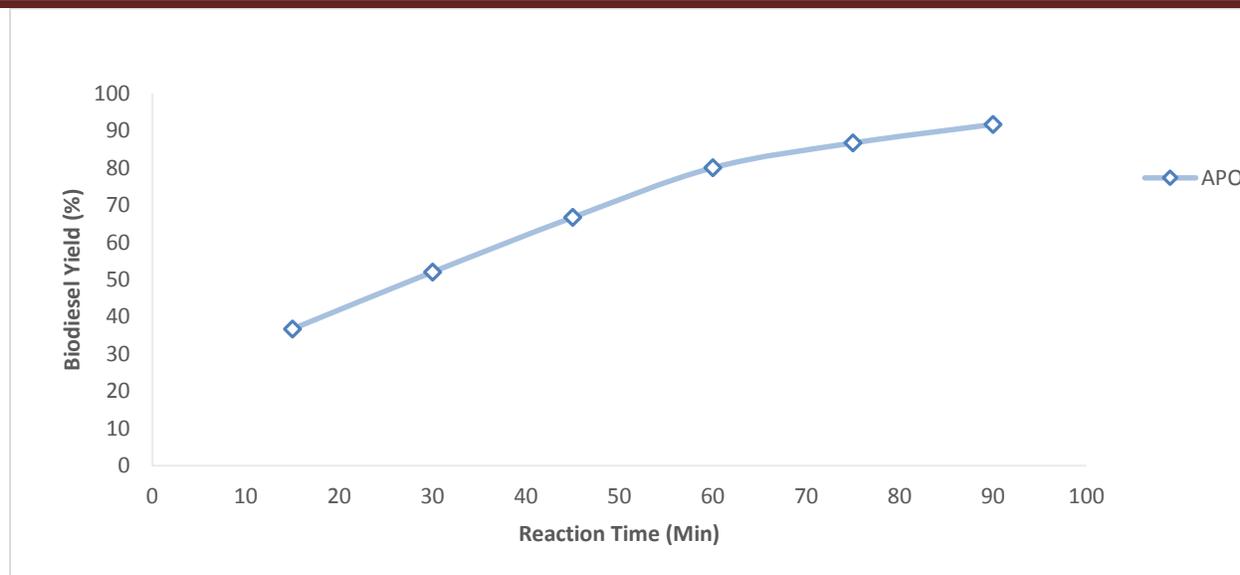


Fig 3.6: Effect of reaction time on APO biodiesel yield

3.4 Fuel properties of avocado pear oil biodiesel

The fuel properties of APOFAME produced are given in table 3.4. Biodiesel generally has a higher density than petro-diesel. This has a significant impact on fuel consumption as the fuel introduced into the combustion chamber is determined volumetrically. The density of the biodiesel was evaluated to be 873kg/m^3 which is within the ASTM limit for biodiesel. The biodiesel density is however lower than that of the oil from which it was derived. This underscores the essence of transesterification in reducing the density of oil to a level where it could be properly atomized in the engine in order to exhibit good combustion characteristics.

The kinematic viscosity of the biodiesel produced was evaluated as $4.95\text{mm}^2/\text{s}$ and is therefore within the ASTM limit. High kinematic viscosity of biodiesel result in poor atomization and incomplete combustion which give rise to cocking of injector tips and hence engine power loss. This conforms to the findings of (Tat and van Garpen (1999). The viscosity of biodiesel is typically higher than that of diesel (Hoekmana et al , 2012). On the other hand very low viscosity fuel produces very subtle spray which cannot properly get into the combustion cylinder, thus forming a fuel rich zone that give rise to sooth formation (Endah et al, 2012; Ezekwe and Ajiwe, 2014).

Flash point measures the degree of flammability of the fuel. The ASTM standard for flash point is $\geq 130^{\circ}\text{C}$. However during biodiesel production and purification, some traces of methanol may remain in the fuel making the flash point to be less than 130°C and thus making it flammable and dangerous to handle or store. The flash point of the GSOFAME is 154°C and thus is withing the ASTM standards which make it safe for handling and storage. Cetane number serves as a measure of ignition quality of the fuel. Fuels with low cetane number shows an increase in emission due to incomplete combustion. The higher the cetane number the better the fuel burns in the combustion chamber of the engine. Since biodieel is composed of long chain hydrocarbon.

Table 3.4: Fuel properties of APOFAME

Properties	Unit	APOFAME	ASTM Standards
Acid vlue	mgKOH/g	0.209	o.50
Density	Kg/m ³	873	860-900
Kinematic viscosity@ 40 ⁰ C	mm ² /s	4.95	1.9-6.0
Flire point	⁰ C	160	197
Flash point	⁰ C	154	100-170
Cloud point	⁰ C	7	-3-15
Cetane number		62.69	48-65
Refractive index		1.5464	1.38
Specific gravity	Kg/m ³	0.873	0.860-0.900
Calorific value	MJ/Kg	34.683	42.06
Pour point	⁰ C	4	0.5
Iodine vlue	gI ₂ /100g	34.2	42.46

groups with virtually no branching or aromatic structure, it typically has higher cetane number than petro-diesel (Hoakama et al 2012). The ASTM lower limit for cetane number is 47. The cetane number of the GSOFAME is 62.69. Thus it is within the ASTM standards and therefore of good ignition quality. Calorific value which is an important property for measuring the energy content of the fuel suggest the suitability of GSOFAME as an alternative to petro-diesel as its determined calorific value of 38.2MJ/Kg approximate that of diesel of 44.34MJ/Kg The low calorific value of methyl ester is usually attributed to the presence of oxygen in the ester.

Cloud point and pour point are cold flow properties which indicate the ease of handling and storage during cold weather. Cloud point which is the temperature of first appearance of wax like material on cooling the biodiesel was determined as 7⁰C. Pour point which is the lowest temperature at which the fuel will stil pour was obtained as 4⁰C. The cloud point and the pour point of the biodiesel are not sufficiently low and therefore might give rise to handling and storage problems during cold weather especially in the temperate and cold regions. However this problem could be overcome by the use of cloud point and pour point depressants or by blending with diesel (Prافليا, et al 2012)

CONCLUSION

The properties of avocado pear oil biodiesel are within the ASTM limit and the biodiesel is therefore suitable as a compression ignition engine fuel. The process parameters , molar ratio of methanol to oil, catalyst concentration, reaction temperature and reaction time significantly affected APOFAME yield as their increase resulted to higher FAME yield until the optimum parameter value was reached when the yield started declining.

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