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Oil Extraction, Biodiesel Synthesis, Blending and Characterization of Biodiesel from Papaya Seed Oil

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ABSTRACT

The increased demand for energy worldwide, climate change due to emission of greenhouse gases from fossil fuel, increase in price of crude oil are the major key factors leading to need for alternative sources of energy. Biodiesel can serve as alternative to fossil fuel because it is environmentally friendly and easily renewable. This research was aimed at synthesis of biodiesel from Papaya seeds and blending it with petro-diesel to determine their potential as alternative to fossil fuel. The sample were collected, sundried, ground prior to oil extraction using Soxhlet apparatus and nhexane as solvent. The oil extracted were characterized of acid value, viscosity, SG, %FFA, %Moisture content e.t.c The oil was converted to biodiesel by acid-catalyzed esterification followed by base transesterification and properties like SG, Iodine value, Pour and Cloud point, Cetane number, Flash and Fire point were analyzed in the biodiesel. Furthermore, the biodiesel (B100) were blended with petro diesel on two different ratio i.e B20 (20% biodiesel and 80% Petrodiesel) and B50 (50% biodiesel and 50% petro diesel) and their properties were also analyzed. The % oil yield of Papaya seed was 25.40%, its Acid value was 12.20 mgKOH/g, Iodine value was 58.979mg/g. The % yield of biodiesel was 90%. The %FFA of B100, B50, B20 were 0.63%, 0.47% and 0.41% respectively. Their specific gravity were 0.902, 0.890 and 0.850 respectively; their Cetane number were 52, 50, 49 respectively, their Flash point were 115^{0}C, 88^{0}C and 75^{0}C respectively. Most of the fuel properties of B100, B20, B50 were within the standard of ASTM D6751 and EN 14214. These showed that Papaya seed oil can serve as alternative to petro diesel

Keywords: *Biodiesel, Papaya Seed Oil, Petrodiesel, Transesterification, Esterification, Alternative Fuel, Fuel Properties, Renewable Energy.*

INTRODUCTION

Energy is one of the most vital commodity for mankind and its sustainable development. The rate of increase in global population and rising needs of humans have led to increase in world energy demand[1]. Fossil fuels such as petroleum, coal and natural gas are the major energy sources that are consumed globally for decades [2]. However, fossil fuel has its' own draw backs. It is not considered to be renewable energy which means that the supply will run out in near future. Thus, it is not a sustainable energy source. Also the volatility of price of fossil fuels, crude oil market monopoly, environmental degradation, energy crises due to advances in technology are major problems associated with the use of fossil fuels [3]. Fossil fuel combustion leads to emission of greenhouse gases which in turn leads to global warming and charges in weather conditions. Fossil fuels associated pollution are major challenges that humans and scientists have faced globally [4].

Fossil fuel pollution also have serious effects on human health for instance, exhaust emission that contain nitrogen oxides (NOx), particulate matters (PM), carbon monoxide (CO) and unburned hydrocarbons can cause lung cancer, morbidity promotion, detrimental effects on fetuses and oedema [5.6]. These problems can be resolved by the use of energy resources that are renewable and less toxic to the environment and human health [7,8].

There are global interest in searching for clean-burning, eco-friendly, biodegradable, non-toxic and renewable energy resources like energy from wind, sun, hydroelectricity, biofuels, geothermal etc. [9,3].

Biodiesel is an example of a biofuel. Biodiesel are made up of variety of ester-based oxygenated fuels derived from vegetable oil, animal fats, seed oil etc. by a process known as "Transesterification" [2]. Biodiesel contains mono-alkyl ester of a long chain saturated and unsaturated fatty acids. Its constituents are made up of 75% carbon, 13% hydrogen, and 12% oxygen by weight [10, 11, 12].

Biodiesel has many advantages over petroleum diesel which includes non-toxic emission [13], lower risk [14], biodegradable [15], ease of production [16], high cetane value [17] higher Flash point (100^{0}C minimum), [18,19] and also have the capacity to be blended with petro-diesel [20]. More so, absence of sulfur and aromatics compounds and presence of oxygen in the chemical structure of biodiesel makes it more environmentally friendly fuel compared to petroleum diesel. [21,22].

The process of converting vegetable oil, fats and seed oil into biodiesel is known as Transesterification. The process involves reaction of seed oil, animal fats or vegetable oils with a short-chain aliphatic alcohol [23]. Transesterification, also known as Alcoholysis involves substituting of alcohol from ester [18]. This is explained further by [24,25] that synthesis of biodiesel by transesterification involves the use of alcohols, often the first C1 and C2 chain alcohol (Methanol and Ethanol), in a chemical reaction with lipids in presence of alkaline catalyst to form long chain of fatty acid of methyl ester.

Caricapapaya is a main fruit crops and has a yearly production of over 10 million tons [26]. Moreover, the seed of papaya have been studied and reported to contain appreciably amount of oil which are needed for industrial applications, biofuels, and medicinal purposes. [27]. When dried, papaya seeds contain up to 30%oil [1] and thus the oil content of papa seed is relatively high [1]. Researchers like [2, 28, 29] have all researched on oil content and characterization of *papaya* seeds in the synthesis of biodiesel but there are no much report of blending the *Carica Papaya* seed oil biodiesel with petroleum diesel.

This research is aimed at extracting oil from *Carica Papaya*seed and converting the oil to biodiesel via transesterification and then blending the biodiesel with petroleum diesel and characterizing the properties of both the biodiesel and its blends.

MATERIALS AND METHOD

SAMPLE COLLECTIONS AND PREPARATION

The samples used for this research was *papaya* seed that was purchased from a local market in Awka, Anambra state. The *papaya* fruits were washed to remove dust, the skin of the *papaya* fruit were peeled-off and the edible part of the *papaya* fruits were taken off. The *papaya* seeds were cut into pieces and sundried for two weeks in order to remove the water content of the seeds. The sun dried seeds were ground mechanically to a powder prior to oil extraction.

EXTRACTION OF PAPAYA SEED OIL

300g of the ground *Papaya* seeds were weighed into a thimble and loaded into soxhlet extractor with 250ml of n-hexane solvent in the extraction flask. Condenser was placed into the soxhlet extractor. The apparatus was placed on a heater and was left to reflux for about 2 hours.

The method was repeated until the oil was extracted from the *Papaya* seeds. The solvent was recovered using a rotary evaporator. This procedure was clearly shown by [30].

PRODUCTION OF BIODIESEL

The biodiesel was produced by 2-step process. Firstly, acid-catalyzed technique followed by base catalyzed transesterification. This was because of high acid value of the *papaya* seed oil (12.20 MKOH/g). Direct based catalyzed transesterification will produce soap and difficulty in separation thereby decreasing the yield of biodiesel produced from the papaya oil.

Acid pretreatment(acid catalyzed esterification)

300ml of *papaya* seed oil was heated to about 55^{0}C using a heating mantle. 20ml of alcohol (methanol) was mixed with 6ml of conc. H_2SO_4 and was added in a round-bottom flask that contained that papaya seed oil. The mixture was sealed, placed on a thermostated magnetic stirrer at 400rpm and was allowed to reflux for 2 hours.

The mixture was then poured into a separating funnel and was allowed to settle and stand for 4hrs after which the aqueous phase which was on the lower layer was tapped off.

Base catalyzed transesterification

200ml of the pretreated *papaya* seed oil was measured out into a round bottom flask and was placed in a water bath set at 55^{0}C. 1g of potassium hydroxide was weighed into a conical flask containing 20ml of methanol and was swirled gently to attain proper solubility of the KOH. The solution (methoxide) was added to the heated oil, and the mixture was heated for 1hour at 55^{0}C.The mixture was then poured into a separating funnel and was allowed to stand for 4 hours. After the separation, the glycerol phase and the biodiesel (methyl esters) which was in upper position was obtained. The glycerol layer was drained off the separating funnel and the subsequent crude *papaya* oil biodiesel was washed with warm distilled water six times to eliminate residual impurities, catalysts and soaps. After the washing, the *papaya* seed oil biodiesel was subsequently dried in a hot oven at around 100^{0}C in other to remove any moisture from the biodiesel.

CHARACTERIZATION OF OIL AND BIODIESEL

The oil and biodiesel were characterized of the physical and chemical properties. The physical properties characterized were: Kinematic viscosity, Relative density (Specific Gravity), Pour point, Cloud point, Fire and flash point, refractive index, Cetane number and the chemical properties were: Acid value, Iodine value, Saponification value, Free fatty acid (%) and Peroxide value.

DETERMINATION OF RELATIVE DENSITY

An empty clean specific gravity bottle was weighed using an electronic weighing balance, and the weight (W_1) was noted. It was filled with distilled water and the weight (W_2) was noted. After weighing, the specific gravity [bottle] was emptied, dried and cooled and was then filled with the sample (oil/biodiesel) and the weight was noted. The formula used to determinate relative density was:

$$RD = \frac{W3 - W1}{W2 - W1} \quad \dots 1$$

Where,

W_1 = weight of empty bottle

$W_{2=}$ weight of empty bottle + weight of water

$W_{3=}$ weight of empty bottle + weight of oil.

KINEMATIC VISCOSITY

The sample was poured into a viscometer tube and the tube was then inserted into a viscometer bath and was maintained at 40°C. The oil inside the tube was sucked up to the upper limit mark with a suction pump and was allowed to drop under gravity. A stopwatch was started and the set up was monitored till the sample got to the lower-limit of the tube and the stopwatch was stopped. The time was recorded and the procedure was repeated twice. The viscosity of the sample was calculated using the method of [31] using equation 2 below;

$$KV = t \times K \quad \dots 2$$

Where:

KV = Kinematic viscosity

t = time in seconds

K = Viscometer Constant = 0.00768

FLASH POINT AND FIRE POINT DETERMINATION: The sample was poured into a clean crucible and a mercury thermometer was inserted in the centre of the sample. Heat was applied to the sample. Flame was subsequently passed over the sample at an interval until a point when a flash was noticed on the sample. The temperature at which it happened was the flash point and the subsequent temperature at which the sample ignited fire for at least 10 seconds was the fire point.

POUR POINT AND CLOUD POINT: These were analyzed using the pour point and cloud point apparatus. The sample was inserted into the glass tube, which was enclosed in an air jacket filled with a freezing mixture of crushed ice and sodium hydroxide pellets. The glass tube that contained the sample was taken out and inspected at every 5°C. The point at which a haze was noticed at the bottom of the sample was noted as the cloud point, and the temperature at which no motion of the sample was observed for at least five seconds was taken to be the pour point.

CETANE NUMBER: The Cetane number was calculated according to the [32] which is:

$$CN = 46.3 + \frac{5458}{\mathrm{SV}} - 0.225 \times IV \quad \dots 3$$

Where, CN = Cetane number;
IV = Iodine value;
SV = Saponification value.

PEROXIDE VALUE: 10g of the sample was weighed into a conical flask and 30ml of acetic acid chloroform was added. The conical flask was shake very well in order to dissolve the sample after which 0.5ml of KI solution was added. The conical flask was allowed to stand for 1 minute and swirled at intervals. After a minute 30ml of distilled water was added and was titrated with standard thiosulphate solution using starch as indicator. Also, a blank was carried out in the same way without the sample. The PV was calculated using equation 4.

$$\mathrm{PV} = \frac{(\mathrm{T} - \mathrm{B}) \times N \times 1000}{\mathrm{g}} \quad \dots 4$$

Where: T = titration volume for sample;
B = Titration volume for the blank;
N = Normality of thiosulphate used; g = weight of the sample.

SAPONIFICATION VALUE: 2g of oil was weighed into a 250ml of alcoholic KOH and the oil was dissolved completely. An air condenser was connected to the flask and was boiled for about 30 mins in a water bath. It was then cooled to room temperature, after which 2 drops of phenolphthalein indicator were added. It was then titrated against standard 0.5 N HCl until the pink colour disappeared. A blank was carried out in a similar way in the absence of oil.

$$\text{Saponification value} = \frac{(\text{Blank} + \text{Titre}) \times 100}{\text{weight of oil}} \quad \dots 5$$

IODINE VALUE: 0.2g of the sample was weighed into 500ml conical flask. 20 ml of chloroform was added and dissolved the sample completely. It was kept in dark for 1 hour. 20ml of KI solution was added to the sample and was mixed properly. It was titrated against 0.1 N $Na_2S_2O_3$ solution using starch as an indicator with vigorous shaking. A blank was carried out similarly in the absence of the sample.

$$\text{Iodine number} = \frac{\mathrm{A} \times \mathrm{N} \times 0.1269 \times 100}{\text{weight of oil}} \quad \dots 6$$

Where, A = ml of $Na_2S_2O_3$;
N = Normality of $Na_2S_2O_3$.

FREE FATTY ACID ANALYSIS: 2g of the sample was dissolved in 20ml of ethanol inside a conical flask. 3 drops of phenolphthalein indicator was added to the solution and was titrated against 0.1N NaOH. It is calculated as follows:

$$\mathrm{FFA} = \frac{\mathrm{V\ NaOH} \times \mathrm{N\ NaOH} \times \mathrm{MW} \times 100}{\mathrm{Sample(g)} \times 1000} \quad \dots 7$$

Where: V NaOH = Volume of sodium hydroxide used;
N = Normality of sodium hydroxide;
MW = Molecular weight of predominant fatty acid.

RESULTS AND DISCUSSION

Table 1: Physico-chemical Properties of Papaya Seed Oil (Aso)

Property	Unit	Value
Colour	—	Orange
Oil content	%	25.40
Acid value	m KOH/g	12.20
FFA	%	6.10
Specific gravity	—	0.920
Viscosity	Mm^2/s	11.80
Appearance	—	liquid

Table 3: Physico-chemical properties of papaya biodiesel (B100 and its blends)

Fuel Parameters	ASO Biodiesel (B100)	B50	B20	Biodiesel (ASTM D6751)	Biodiesel (EN 14214)
Biodiesel yield (%)	90	—	—	—	—
Colour	Orange	Orange	Orange	—	—
Specific Gravity	0.902	0.890	0.850	0.86 - 0.90	0.86 - 0.90
Viscosity (mm^2/s)	6.30	4.80	5.80	1.9 - 6.0	3.5 - 5.0
Moisture content (%)	0.28	0.12	0.18	0.05 Max.	0.05 Max
pH	6.92	6.88	6.12	—	—
Flash Point (°C)	115	75	88	93 Min.	≤100
Fire Point (°C)	123	83	98	≤100	≤100
Cloud Point (°C)	2	-1	1	-15 to 10	
Pour Point (°C)	-3	-6	-5	-10 to -15	
Cetane no.	52	49	50	≤47	≤51
Acid Value (mgKOH/g)	1.28	0.86	0.98	0.50max	0.50 Max
FFA (%)	0.63	0.41	0.47	—	—
Iodine value (g/100g)	68.20	48.10	55.28	—	120 Max
Peroxide value (Meq/kg)	6.38	4.20	5.24	—	—
Saponification value (mgKOH/g)	158	110	138	—	200 Max

DISCUSSION

The physical/chemical properties of the *papaya* oil were presented in Table 1 above. These physico-chemical analysis were done to ascertain the potential of *papaya* seed oil in biodiesel production.

The colour of the oil was orange and this was probably because of the pigments that was present in the *papaya* seed. The oil content of the *papaya* seed was 25.40% and this was considerably high owing to the fact that *papaya* seed oil is not considered an edible oil and therefore not contribute to food versus fuel debate. However, the yield was lower compared to yield of *papaya* seed (31.0%) as reported by [3] and 27.98% oil yield from *papaya* seed as reported by [2]. The % oil content was higher than what [31] reported for oil content of Avocado seed (13.50%).

The acid value and % FFA were 12.20MgKOH/g and 6.10%, respectively. This indicated that the *papaya* seed oil needed an acid pretreatment (acid esterification) before the alkali transesterification because the acid value was very high. Direct base-catalyzed transesterification will have the tendency of producing soap and difficulty in separation thereby reducing the % yield of the biodiesel.

The specific gravity, (0.92) and viscosity (11.80mm^2/s) were also high and this indicated that the *papaya* oil cannot be used directly in a diesel engine without subsequent pretreatment, hence Acid transesterification and base trans-esterification were used to produce the biodiesel from *papaya* seed oil.

The transesterification reaction was done with oil to alcohol (methanol) ratio of 1:6 and NaOH as the catalyst.

After the production of *papaya* seed oil (PSO) biodiesel, it was blended with petroleum diesel in the biodiesel to petro-diesel ratio of 20:80 (B20) and 50:50 (B50) and their properties were characterized.

The results of the physicochemical properties of both the papaya oil biodiesel (B100) and its blends (B50 and B20) were presented in Table 2.

The specific gravity (SG) of the *papaya* oil biodiesel (B100) and its blends (B50 and B20) were 0.902, 0.890 and 0.850 respectively and they are all within the standard limit of both the ASTM D6751 and EN 14214. Fuel biodiesel with SG over 1 indicates that the fuel maybe relatively heavy which may lead to incomplete combustion of the fuel. [32] reported the specific gravity of *Papaya* seed oil (ASO) biodiesel and its blends of B10 and B20 to be 0.90, 0.85 and 0.88 respectively.

The kinematic viscosity is one of the most important properties of a fuel because it indicates the flowability capacity of the fuel. It is used to measure the atomization and fluidity of a biodiesel. The kinematic viscosity of the *papaya* seed oil biodiesel and its blends at (B50 and B20) were 6.30mm^2/s, 4.80mm^2/s and 5.80mm^2/s respectively and they were within the standards of both ASTM (D6751) and EN 14214 except for B100 which was slightly higher than the standard of both the ASTM and EN. High viscosity can affect the rate of flow of the fuel and pumping of the fuel to the injector most especially at very low temperatures.

Moisture content (%) is used to ascertain the quantity of water in a fuel. High moisture content in biodiesel lowers its heating value, reduces combustion capacity and increases the release of harmful gases like carbon monoxide (CO). It can also lead to incomplete burning and delayed ignition of the diesel fuel. The *papaya* biodiesel (B100) and its blends (B50 and B20) had the moisture content (%) of 0.28%, 0.18% and 0.12% respectively and they were above the standard limit of both ASTM D6751 and EN 14214 which was 0.05% Max.

The Flash Point and Fire Point of a diesel fuel are used to ascertain its flammability and combustion behaviour. The Flash point is used to denote the minimum temperature at which fuel vaporizes enough to get ignited whereas the fire point is the least temperature at which the vapour of fuel continues to burn for at least 10secs after ignition. Fuels with low flash point releases vapours easily and can catch fire easily whereas fuels with high flash point and fire point does not easily form explosive vapours and this means that are safer both in storage and transportation. The *papaya* biodiesel (B100) and its blends (B20 and B50) flash point were 115^{0}C, 88^{0}C and 75^{0}C respectively and their fire points were 123^{0}C, 98^{0}C and 83^{0}C respectively and they were all within the limit set by both ASTM D6751 and EN 14214.

Cloud point and Pour Point are essential parameters of a fuel's performance in cold weather. The cloud point is the temperature at which tiny solid (wax like) particles can be observed as the fuel cools. The pour point on the other hand is the temperature at which the fuel losses its flow properties [32]. The Cloud points of the papaya biodiesel (B100) and its blends(B20 and B50) were 2^{0}C, 1^{0}C and – 1^{0}C respectively while their Pour points were – 3^{0}C, - 5^{0}C and – 6^{0}C respectively and they were all within the standard limit of ASTM D6751 as shown in table 2 above.

Cetane number is used to measure diesel fuel ignition quality. Biodiesel with high Cetane number indicates that the fuel ignites faster when injected into the cylinder, and this helps the engine start easier and prevents sudden pressure spikes thereby reducing the sharp sound known as diesel knock. A high Cetane value also promotes more complete combustion and lowers emissions of carbon monoxide and hydrocarbons emissions. Low Cetane number causes loud knocking, vibrations to the engine. It can cause hard starting especially in cold weather, long ignition delay and can also increase the amount of unburned hydrocarbons. Etc.

The *papaya* biodiesel (B100) and its blends (B20 and B50 had Cetane Value of 52, 50, 49 respectively and are all within the standard limit of both ASTM D6751 and EN 14214.

Acid value and free fatty acids are very important variables used to determine the shelf life of a diesel fuel and how corrosive the fuel can be in fuel tanks and injection systems. High acid value and % FFA in a fuel indicates that the fuel is already degrading or oxidizing in storage. Also free fatty acids attack metal fuel lines causing corrosion in the engine and injection systems. The acid value of the papaya biodiesel (B100) and its blends (B20 and B50) were 1.28MgKOH/g, 0.98MgKOH/g and 0.86MgKOH/g respectively and their free fatty acid values were 0.63%, 0.47% and 0.41% respectively and the acid values were slightly above the standard limit (0.50MgKOH/g) of both ASTM D6751 and EN 14214.

Iodine value are used to indicate the degree of unsaturation of a fuel and it affects fuel properties like oxidation stability, engine deposits etc. fuel with high iodine values indicates high double bonds and unsaturation which may improve low – temperature fluidity of a fuel but also lowers the cetane number of the fuel leading to delay in ignition and rougher combustion. High iodine value also indicate low shelf life stability of the fuel, easy oxidation which causes rapid degradation and acid formation during storage of the fuel. The papaya biodiesel (B100) and its blends (B20 and B50) had values of 68.20, 55.28 and 48.10 g/100g respectively, and they are within the standard limit of both ASTM D6751 and EN 14214.

Peroxide value is another important properties of a fuel used to determine the shelf life of the fuel.

It is used to measure primary oxidation in fuels. A high Peroxide value in fuels indicates high oxidative degradation which usually leads to sediment and gum formation, fuel system corrosion etc. High Peroxide value indicates shortened shelf life of the fuel because of poor oxidation stability and this means the fuel degrades or becomes rancid quickly during storage.

High Peroxide value also increases the total acidity of the fuel, leading to the aging and swelling of plastic engine parts. The Peroxide values of the papaya oil biodiesel and its blends (B20 and B50) were 6.38, 5.24 and 4.20 Meq/kg respectively, and they were within the limit of both the ASTM D6751 and EN 14214 standards.

Saponification value is another good property of a fuel because it measures the average molecular weight of fatty acids in an oil or fuel. High saponification values indicate lower average molecular weight which affects the density and fluidity of the fuel. High saponification value also lead to deposit formation because fuels with high soap formation tendencies introduces impurities that lead to fuel filter clogging, combustor chamber deposits etc. The saponification value of the *papaya* oil biodiesel and its blends (B20 and B50) were 158, 138 and 110 MgKOH/g respectively and they are within standard limit of EN 14214.

CONCLUSION

Papaya oil biodiesel was successfully synthesized from non-edible *carica papaya* seed. The *carica papaya* seed oil contained appreciable amount of oil (25.40%) and this indicates that it can be used as a commercial feedstock for biodiesel production. The oil was converted to biodiesel by transesterification with a methanol to-oil ratio of 6: 1 and NaOH was used as catalyst. Most of the properties of both *carica papaya* seed oil and its blends were within the American standard limit (ASTMD6751) and the European standard (EN14214) and this indicated that *carica papaya* oil can serve as an alternative to petroleum diesel.

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