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Physiochemical Assesement Of Biodiesel Blends From *Annona Muricata* Seed Oil From Owerri Municipal Area Of Imo State Nigeria

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Abstract.
This study was designed to study the viablility of producing biodiesels and their attendant blends from Annona muricata seed oil.The oils were obtained from the seed using sohxlet extractor in which case n-hexane was the solvent, the biodiesel was obtained by transeterification method using sodium methoxide and the petrol diesel obtained from the Nigerian national petroleum cooperation. Analysis were carried out according to the American standard for testing material ASTM
Annona muricata seed showed considerable oil content. The biodiesel produced from this oil was regarded in this work as neat biodiesel and was labeled B_{100} *and had comparable pour point, cloud point, flash point, fire point, viscosity and acid number as those produced from other known sources. Interestingly when this biodiesel was blended with petrodiesel and labeled as* B_{10}, B_{20}, B_{30}, B_{40} *and* B_{50} *, they showed marked improvement in fuel characteristics used as yardstick for the determination of the biodiesel quality .Annona muricata seed has considerable extractable oil that could be used for quality biodiesel productions and this make the sample a promising material for the production of biodisel*

Key Words; *Annona muricata, transesterification,biodiesel,,flash point,pour point, cloud point, acid number*

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I. Introduction

The need for fossil fuels and the emissions generated by these fuels are on the increase daily. There is an increasing awareness of global warming as well as climatic changes. Beside, the recent outbreak of corona virus worldwide brought untold hardship to the economy of the nations that depended wholly on crude oil exports and also on countries that consume the crude oils as international trade was put on hold for many months. The attendant consequence is that many countries could not have enough fuel for their machinery, the saving grace was the total lockdown which reduced the energy consumption. Should international trade be put on hold once again, the transportation sector will be worst hit. One attendant problem with the use of fossil fuels is the pollution arising from its exploration and exploitation [1]. Therefore there arise the renewed interest on bio-fuel production. Biodiesel have comparable characteristics compared to petro-diesel, which makes it useful in compression motors engine without any form of modification Biodiesels which are monoalkyl esters of long chain fatty acids are derived from renewable agricultural products such as vegetable oils and animal fats. This fuel is produced by trans-esterification. The process involves the reaction oil or fat and a monohydric alcohol in presence of a catalyst[2]. Some researchers have suggested the methylation of FFA occurred along with methanolysis of the oil.[3] Besides, lipases can be recovered after trans-esterification. Other researchers[4] reported the production of biodiesel from waste vegetable oils through its pretreatment followed by trans-esterification process in presence of methanol using a KM micromixer reactor. Extensive work has been done on the production of biodiesel from waste cooking oil using solid catalyst.[5] Biodiesel are producible from waste sunflower cooking oil by trans-esterification [2], it has been reported that base catalyzed trans-esterification had optimum yield . some other researchers [6] discussed the quality parameters and chemical analysis for biodiesel production in the united states

So many different oils have been used to produce biodiesel; some of which includes Rapeseed oil.[3], palm oil [7] , lipid producing sugar cane, and so many more. Different works has been written or reviewed base on different methods for biodiesel production ; biodiesel production from palm oil and its effluents , a review,[8] biodiesel technologies a review [9], synthesis of biodiesel from vegetable oil[10] , production optimization and quality assessment of biodiesel from waste vegetable oil[11], Review of biodiesel production and emission characteristics from non edible vegetable oil [12] ,Different techniques for the production of biodiesel from waste vegetable oil [13].utilization of three non edible vegetable oil for the production of biodiesel catalyzed by enzymes[14]. One modern method for the analysis of biodiesel is the Analysis of Biodiesel Quality Using Reversed Phase High-Performance Liquid Chromatography[15]

Annona muricata is a small erect evergreen tropical plant belonging to the family Annonaceae. The leaf, bark, fruits and roots are used for medicinal purposes, the fruits are eaten as food but the hard kernel seeds are either planted or thrown away [16]. Recently researches are ongoing on the usefulness of the seed of this plant. The kernel like seed usually has little or no usefulness in this part of the world but it is rich in oil , we therefore felt that this oil could be optimized for the production of biodiesel

II. Materials And Method

2.1COLLECTION AND PREPARATION OF SAMPLE

The fruit pods of Annona muricata was purchased at Afor Ogbe market Ahiazu Mbaise Imo state. The seeds were extracted from the ripped pods and room dried for two months to remove moisture. The dried sample was then milled to fine particles to reduce the surface tension and increase the surface area using a hand grinder. The powdered sample was stored in air tight container to avoid moisture and contamination.

2.2 EXTRACTION OF OIL SAMPLE

The grounded sample was weighed before extraction using a weighing balance and the weight was 1000g. The oil from the sample was obtained by use of soxhlet extractor using n-hexane as the extracting solvent which was recovered through distillation using a rotary evaporator leaving behind pure Annona muricata seed oil

2.3 PRODUCTION OF BIODIESEL FROM SAMPLE OIL

200cm^3 of the extracted oil was measured into a 500cm^3 beaker. The weight of the oil was determined. 1% weight of the oil was the weight of sodium hydroxide used. The base was dissolved in 100 cm^3 of methanol which after dissolution was made up to 500 cm^3 by addition of more methanol ,this solution now contains sodium methoxide. The oil was heated to 60^{o}C and the sodium methoxide was transferred into the oil with continual agitation for 1 hr. The mixture was allowed to stay for 24hr for the reaction to go into completion. This was later transferred into a 500 cm^3 separation funnel and allowed to settle. The mixture separated into two phases, glycerol at the bottom which is denser and biodiesel mixed with soap and methanol at the top. The glycerol was decanted and the top phase which contained methanol, biodiesel and soap was recovered by use of rotary evaporator. The biodiesel was then washed with distilled water until it was free from soap and methanol. After washing, the biodiesel was dried by heating at a temperature of 48^{o}C for 1 hr and stored in an air tight container.

2.4 BLENDING OF BIODIESEL

The pure biodiesel (B_{100}) was blended at different ratios with petroleum diesel to give different blends of B_{10}, B_{20}, B_{30}, B_{40} and B_{50}. This was done by mixing 10cm^3 of biodiesel with 90cm^3 of petroleum diesel to give B_{10} blend, 20cm^3 of biodiesel with 80cm^3 of petroleum diesel to give B_{20} blend, 30cm^3 of biodiesel to give 70cm^3 of petroleum diesel to give B_{30} blend, 40cm^3 of biodiesel to 60cm^3 of petroleum diesel to give B_{40} blend. And 50cm^3 of biodiesel to 50cm^3 of petroleum diesel to give B_{50} blend.

Key;

B_{10} =10% biodiesel and 90% petrodiesel

B_{20}= 20% biodiesel and 80% petrodiesl

B_{30}= 30%biodiesel and 70% petrodiesel

B_{40}= 40% biodiesel and 60% petrodiesel

B_{50}= 50% biodiesel and 50% petrodiesel

2.5 ANALYSIS OF BLENDED AND NEAT BIODIESEL

Color determination ASTM D1500[17]

The samples were placed in a container and their colour was compared with coloured glass disk until a matching colour was found using a standard light source.

2.6 FLASH POINT DETERMINATION (PENSKY MARTENS CLOSED CUP ASTM D93)[18]

The sample was introduced into the cup and heated over sand bath. The sample was stirred at specific rate. The ignition source was directed into the test cup at regular intervals, with simultaneous interruptions of the stirring until a flash is detected and the temperature was recorded at that point using thermometer. This was done with each sample and results were recorded

2.7 FIRE POINT DETERMINATION ASTM D93[18]
The sample was introduced into the cup and heated over sand bath. The sample was stirred at specific rate. The ignition source was directed into the test cup at regular intervals, with simultaneous interruptions of the stirring until the sample catches fire and continue to burn without stopping, the temperature was recorded from the thermometer reading. This was done with each sample and results were recorded

2.8 CLOUD POINT DETERMINATION ASTM D2500[19]
$10cm^3$ of the biodiesel was measured using a measuring cylinder and poured into a $100cm^3$ beaker and the sample was placed into a deep freezer. The sample was inspected at intervals and when the bottom of the container became hazy the temperature was recorded.

2.9 DETERMINATION OF VISCOSITY USING REDWOOD VISCOMETER
The viscometer cup was filled with the sample and electrically heated is filled, Each time a reading is to be taken, the total pressure on the system is made to remains constant. The oil is then heated gradually and the time of flow for $50cm^3$ of the sample at different temperature is recorded. This was repeated with each sample

2.10 DETERMINATION OF ACID VALUE
10g of the biodiesel was dissolved in $30cm^3$ of ethanol and kept in a stoppered bottle so that the alcohol does not evaporate . 10 cm^3 of the biodiesel solution was taken in a conical flask and titrated with KOH solution taken in a burette and phenolphthalein was used as indicator.

2.11 POUR POINT DETERMINATION ASTM D 5853[20]
$10cm^3$ of the biodiesel was measured using a measuring cylinder and poured into a $100cm^3$ beaker and the sample was placed into a deep freezer to allow it to become chilled. The oil was inspected at regular intervals. When the oil appears to have congealed, the beaker was bent, the temperature was taken immediately before the oil started flowing and was recorded

III. Results And Discussion
The results obtained for the production, blending and analysis of biodiesels obtained from Annona muricata seed oil are summarized in tables 1- 6 below. Table 1 gives the physic-chemical properties of neat biodiesel (100%) pure biodiesel from Annona muricata seed oil , tables 2-6 gives the physic-chemical properties of the blends .The parameters used here are cloud point, pour points, flash points, fire point and acid values

3.1 FLASH POINT:
The temperature at which the fuel becomes a mixture that will ignite when exposed to a spark of flame is regarded as the flash point. The flash points of the biodiesels are 158^{o}C for B_{100}, 90^{o}C for B_{10}, 100^{o}C for B_{20} and $B_{30,}$ 103^{o}C and 108^{o}C for B_{40} and B_{50} respectively, tables 1-6 below. This result are within the accepted range of 100-107^{o}C for standard biodiesel . It is higher than that of petrodiesel diesel with values of 68-85 ^{0} C. So, it is safer fuel and can be handled without the risk of fire outbreak. Blending increases the flash point of biodiesel, improves their quality and makes handling safer .these results agree with similar analysis by other researchers [6], [21]. [8] ,[22]

Table 1: PHYSICO-CHEMICAL PROPERTIES B_{100}

Properties	Testing process	Value in ^{o}C
Cloud point	ASTM D2500	10
Pour point	ASTM D6751	4
Flash point	ASTM D93	158
Fire point	ASTM D93	115
Colour	ASTM1500	Amber
Viscosity		6.33 mm^2/S
Acid no		0.23mg

3.2 VISCOSITY:
Viscosity is the measure of a material's resistance to flow. Viscosity is a result of the internal friction of the material's molecules. Materials with a high viscosity do not flow readily; it is a Measure of the fluidity of a substance. Viscosity affects injector lubrication and fuel atomization. Fuels with low viscosity may not provide sufficient lubrication for the precision fit for fuel injection pumps or injector plungers resulting in leakage or increased wear. Fuel atomization is also affected by fuel viscosity. Diesel fuels with high viscosity tend to form larger droplets on injectior which can cause poor combustion and increased exhaust smoke and emissions. The viscosity of biodiesel and blended biodiesels are 6.33mm/s for B_{100}, 6.1, 6.2, 5, 5.3and 5.6mm/s respectively for B_{10} to B_{50} ,this values are within the accepted limits of 1.9 to 6.6mm/s, our results are in agreement with similar analysis by other researchers. [2][3][21][8][22]

Table 2: PHYSICO-CHEMICAL PROPERTIES OF BIODIESEL B_{10}

Properties	Testing process	Value in ^oC
Cloud point	ASTM D2500	8
Pour point	ASTM D6751	1
Flash point	ASTM D93	90
Fire point	ASTM D93	65
Colour	ASTM D1500	Amber
Viscosity		6.1 mm^2/S
Acid no		0.24mg

3.3 POUR POINT:

The Lowest temperature at which an oil will pour or flow under certain prescribed conditions. It is a measure of the ability of a diesel fuel to operate under cold weather conditions. Defined as the temperature at which the amount of wax in a solution is sufficient to gel the fuel when tested under standard conditions .The Pour point is the temperature above which a chilled lubricant shows no movement at the surface for 5 seconds when inclined. For biodiesel it is 6^0C.and for gasoline diesel it is 2-4 ^{0} C. our samples B_{100}, B_{10}-B_{50} have values of 4,1, 3, 6, 8, and 2^oC respectively table 1-6. These results are in agreement with range of results reported by other researchers. [3][21[[8][22]

Table 3: PHYSICO-CHEMICAL PROPERTIES OF BIODIESEL B_{20}

Properties	Testing process	Value in ^oC
Cloud point	ASTM D2500	12
Pour point	ASTM D6751	3
Flash point	ASTM D93	100
Fire point	ASTM D93	66
Colour	ASTM D1500	Amber
Viscosity		6,2 mm^2/S
Acid no		0.33mg

3.4 CLOUD POINT

This is the index of dissolved wax in an oil. it is the temperature at which the cloudiness or haziness appears in a sample of oil due to separation of waxes under standard conditions. We have values of 10^oC for B_{100}, 8, 12, 12 13,and 10^oC for B_{10} –B_{50} respectively table 1-6

Table 4: PHYSICO-CHEMICAL PROPERTIES OF BIODIESEL B_{30}

Properties	Testing process	Value in ^oC
Cloud point	ASTM D2500	12
Pour point	ASTM D6751	6
Flash point	ASTM D93	100
Fire point	ASTM D93	68
Colour	ASTM D1500	Amber
Viscosity		5 mm^2/S
Acid no		0.43mg

3.5 FIRE POINT

The temperature at which the oil vapour will catch fire and continues to burn. Usually the flash point and fire point are indexes of the level of purity of a sample under consideration and also a measure to which the sample under consideration could be handled safely. Values of 115^oC was obtained for B_{100}. Similarly values of 65,66,68, 70 and 70^oC were observed foe B_{10}-B_{50} respectively.table 1-6

Table 5: PHYSICAL PROPERTIES OF BIODIESEL B_{40}

Properties	Testing process	Value in ^oC
Cloud point	ASTM D2500	13
Pour point	ASTM D6751	8
Flash point	ASTM D93	103
Fire point	ASTM D93	70
Colour	ASTM D1500	Amber
Viscosity		5.3 mm^2/S
Acid no		0.44mg

Table 6: PHYSICO-CHEMICAL PROPERTIES OF BIODIESEL B_{50}

Properties	Testing process	Value in ^oC
Cloud point	ASTM D2500	10
Pour point	ASTM D6751	2
Flash point	ASTM D93	108
Fire point	ASTM D93	70

Colour Viscosity Acid no	ASTM D1500	Amber 5.6 mm^2/S 0.46mg

3.7 ACID VALUE

The amount of free acid present in fat as measured by the milligrams of potassium hydroxide needed to neutralize it is the acid value. As the glycerides in fat slowly decompose the acid value increases. The acid value for biodiesel is between 0.2 -1.mg.For general diesel the acid no is 0.02mg. the obtained values range from 0.23 for B_{100} 0.24mg for B_{10}, 0.33, 0.43, 0.44 and 0.46mg for B_{20}-B_{50} respectively table 1-6. Similar results on some classes of biodiesel analysis has been reported [2] [3][22]

IV. Conclusion

The results obtained from the analysis carried out on the biodiesel produced from Annona muricata seed oil has shown that the seeds of this plant is a promising material for future biodiesel production, moreover, blending the biodiesel with petrol diesel improves the flammability of the biodiesel ,we conclude that adding a little petrodiesel to our biodiesel is a very important way of improving it qualities.

AUTHORS CONTRIBUTIONS

[1]Onuh Uchenna Lynda and Iwu Irenus Chinonye wrote the work, she carried out the sampling and extraction of the oils. Ali Bilar and Obiagwu Ifeoma. carried out the trans-esterification of the oil and prouduced and purified the biodiesel and also blended it with petrodiesel . Anozie Augustine, and Ezeokoye Miracle Oluchukwu carried out the physiochemical analysis of the biodiesel and biodiesel blends

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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