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# Assessment of the Biodiesel Parameters of the Seed Oil of Water Melon (*Citrullus vulgaris*)

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# ABSTRACT

Base-catalyzed transesterification of water melon *Citrullus vulgaris* seed oil (CVSO) was carried out at a methanol/oil ratio of 6:1 at 50°C to produce biodiesel, a fuel comprising mono-alkyl esters of long chain fatty acids derived from vegetable oil or animal fat. The effects of potassium hydroxide (KOH) and sodium hydroxide (NaOH) catalysts were also investigated. The purpose of the transesterification process is to lower the viscosity, density and flash point of the oil. Prior to transesterification, the physico-chemical properties of the CVSO such as specific gravity, viscosity, moisture content, refractive index, density, acid value, saponification value, iodine value, and peroxide value, were determined and the results all fall within the recommended ranges as stated by biodiesel standard quality control organizations. Fuel quality parameters like density, viscosity, cloud point, flash point, pour point, acid value, cetane number, higher heating values and carbon residue were also within the recommended range. The results of the effects of the two catalysts on the yield of the biodiesel showed that KOH catalyst gave a higher yield of biodiesel compared to the NaOH catalyst. The biodiesel yield for KOH catalyzed process was 64.36%, while the yield for NaOH catalyzed process was 41.37%. The fuel properties of CVSO methyl esters were similar to those of rape seed, soya bean and sunflower biodiesel. Hence the refined, chemically processed and degummed CVSO can be used to run compression ignition on engines for longer duration since the major problem of chocking with unmodified vegetable oils has been overcome.

**Keywords:** properties, water melon, biodegradable, engine, renewable energy, substitute, vegetable oil **Abbreviations: ASTM**, American society for testing and material; **DIN**, German Standard; **EN**, European standard; **M**, mole

# INTRODUCTION

Nowadays, fossil fuels mainly petroleum, natural gas and coal have been playing an important role as major energy resources worldwide. However, these energy resources are non-renewable and are projected to be exhausted in the near future. The situation has worsened with the escalating energy consumption worldwide due to rapid population growth and economic development. Global emission of CO<sub>2</sub>, and other harmful gases such as, CO, SO2, NOX and particulate generated by fossil fuel combustion have led to serious green house effects on the environment. These include global warming, depletion of the ozone layer, increase in mean sea level, acidic rain, and the deterioration of human health and the environment (Guan et al. 2009). It has generated a lot of concern and has led to an urgent need for the identification of new energy resources that are environmentally friendly (Shen et al. 2010). Biodiesel is an alternative diesel fuel made from biological sources, primarily vegetable oils, but to a lesser extent, frying oils and animal fats. It is produced by the homogeneous catalytic transesterification or alcoholysis reaction (usually methanolysis), the catalyst being either a base or an acid (Al-zuhair 2007; Papadopoulos et al. 2010). Biodiesel can be used as a basis for a clean substitute for fossil fuel without any modification to diesel engines, boilers or other combustion equipment (Saydut et al. 2008).

Many researchers have reported biodiesel production in several ways depending on the feedstock (Karmee and Chadha 2005; Kaul *et al.* 2007; Chakraborty *et al.* 2009; Papadopoulos *et al.* 2010). However, the critical issues in the production of biodiesel are the availability and cost of the feedstock, cultivation requirement, oil yield and oil composition, the short comings of using edible oils over non-edible oils for biodiesel production and the techniques available for converting non-edible oils to biodiesel (Gui *et*  *al.* 2008). This paper reports the processing and production of biodiesel from the seed oil of *Citrullus vulgaris* (water melon) and the assessment of the oil parameters of the biodiesel produced.

# MATERIALS AND METHODS

# Collection and preparation of seed samples

Water melon fruits were obtained from Farin Gada vegetable market in Jos, Plateau State, of Nigeria. The seeds were removed from the fruits, washed and were subjected to sun drying over a period of time. The dried seeds were de-husked and dry milled into fine powder using mortar and pestle. The milled powder was stored in an air-tight container.

# Reagents

All the reagents used in this work were of analar Grade and purchased from British Drug House (BDH), Poole, England.

# **Oil extraction**

Oil from the powdered seed of water melon *Citrullus vulgaris* was extracted by the method described by AOAC (1980). At the end of the extraction, the solvent was evaporated in a rotary evaporator. Free fatty acids were determined as described in Schliecher *et al.* (2009). The extracted oil which is made up predominantly of 71.90% unsaturated fatty acid and 28.10% saturated fatty acid was then transesterified.

# **Biodiesel production**

The transesterification process proceeded with 6:1 molar ratio of methanol/oil and 0:60 g potassium hydroxide pellets. The metha-

nol was slowly added into weighed 0:60 g KOH in a 150 ml conical flask while stirring until the whole KOH pellets was dissolved to form methoxide solution. The oil was heated at 50°C and the methoxide was slowly added over a period of 30 min, with continuous and slow stirring using a magnetic spin stirrer. The reaction mixture was then allowed to stand overnight. A crude methyl ester phase at the top was formed and a glycerol phase at the bottom of the mixture on settling using a separating funnel. The lower layer is run-off which contains most of the glycerol realized during the reaction. The catalyst is removed by warm water washing and the excess methanol and water in the ester phase is removed by evaporation under atmospheric condition (Eevera *et al.* 2009).

# Tests for fuel quality parameters

#### 1. Density determination

The weight of an empty 100-ml beaker was determined. A specific volume of biodiesel was added to the 100-ml beaker and the weight of the oil and beaker determined. The density is calculated using the formula.

Density ( $\rho$ ) = W2 – W1 / V

where W1 = weight of empty beaker; W2 = weight of beaker and biodiesel; V = volume of biodiesel. The density obtained was compared to the density diesel D<sub>2</sub> (Reece and Peterson 1993).

### 2. Viscosity determination

The method of liquid flow through an aperture in a cup was used for the determination of viscosity of biodiesel. A Prolabo Viscometer was used for the determination of the viscosity. Prolabo cup number 4 Viscometer was washed and rinsed and allowed to dry. The measurement of the viscosity of the biodiesel was carried out by taking the number of seconds it takes for a fixed quantity of biodiesel (60 cm<sup>3</sup>) S to flow through a small orifice of standard length and diameter at a specified temperature. This was done by filling the cup with 60 cm<sup>3</sup> of the biodiesel after closing the aperture with a finger beneath. The finger was then removed from the orifice and the stopwatch started. The stopwatch was stopped after the last drop of the biodiesel and the time taken for the biodiesel to flow out was recorded.

The viscosity was calculated by multiplying the time in seconds by the standard length and diameter of the small orifice at a specified temperature (John and Tyler 1976).

#### 3. Cloud point determination

An improvised method was used for the cloud point determination. 5 ml of biodiesel was poured into a test tube and the clamp carrying the test thermometer held tightly. The position of the thermometer was adjusted to allow the thermometer rest at the bottom of the tube for uniformity. The test tube was placed in an ice bath and the set up inspected at intervals for cloud formation. The temperature at which a distinct cloudiness formed at the bottom of the test tube was recorded (Reece and Peterson 1993).

#### 4. Flash point determination

The Flash Point was determined according to ASTM D93 method for flash point by Pensky-Martens Closed Cup Tester (Anwar *et al.* 2010). The temperature was then compared to the flash point of diesel  $D_2$ .

#### 5. Pour point determination

A modified method using a -4°C cooling bath was used for the poor point determination. 5 ml of the biodiesel was poured into a test tube and clamp with a wooden clamp bearing a thermometer. The sample was allowed to freeze in an ice bath. It was then removed and tilted on the clamp with the thermometer. The setup was observed at intervals and the lowest temperature at which the biodiesel was observed to flow was recorded as the pour point (Purnanand *et al.* 2009).

#### 6. Acid value determination

Acid value determination was carried out according to the method described by Onwuka (2005).

#### 7. Cetane number (CN) determination

CN was determined using the estimated Saponification Value (SV) and Iodine Value (IV) via calculation from the equation below (Meher *et al.* 2006; Eevera *et al.* 2009):

CN = 46.3 - 54.58 / 5V - 0.225IV

#### 8. Higher heating value (HHV) determination

The higher heating values were determined using the estimated Saponification Value (SV) and lodine Value (IV) via calculation from the equation below (Eevera *et al.* 2009):

HHV = 49.43 - 0.041 (SV) + 0.0051 (IV)

#### 9. Carbon Residue (CR) determination

CR determination was carried out by the method described by Demmering *et al.* (1995).

### Sampling and statistical analyses

The results are presented as mean  $\pm$  SD of triplicate readings. Comparison of mean biodiesel quality parameters between the methyl esters produced using either NaOH or KOH were done using the Mann-Whitney U test and p < 0.005 were considered significant.

# **RESULTS AND DISCUSSION**

The results show that the physical and chemical properties of CVSO all were within the recommended limits as specified in the literature.

The conversion of CVSO to methyl esters was achieved by using molar ratio of 6:1 methanol to oil, with catalyst concentration of 0.6 g KOH and 0.75 g NaOH at 50°C for 30 min. Maximum conversion yields of 64.36 and 41.37% were obtained for KOH and NaOH catalyzed processes, respectively. The transesterification is commonly carried out with an excess amount of alcohol in order to shift the equilibrium to the product (methyl ester). But a higher amount of alcohol requires longer time for separation of the ester layer from the water layer. This is due to the fact that methanol with one polar hydroxyl group, can only work as emulsifier which enhances emulsion (Zhang *et al.* 2003). The higher yield of KOH catalyzed process compared to the NaOH catalyzed process might be due to the fact that potassium is more reactive than sodium. This agrees with the findings by others (Dorado et al. 2004; Meher et al. 2006; Rashid and Anwar 2008) who also found that KOH catalyst was the most effective in the methanolysis of Pongamia pinnata and Brassica carinata, a finding they ascribed to the varied chemical nature of the feedstock oils used for the biodiesel production. The fuel quality of the methyl ester produced were evaluated in accordance to American Society for Testing and Materials ASTM), European (EN, and German (DIN) standards. The quality of the methyl esters are of importance for better engine performance characteristics. The results are presented in Table 1.

#### Density

Density is a key fuel property which directly affects the engine performance characteristics such as CN and high heating values which are both related to density (Ertan *et al.* 2008). The density of CVSO methyl ester was 0.87 to 0.86 g/cm<sup>3</sup> for methyl ester using KOH and NaOH respectively. Both values fell within the acceptable limits of 0.86–0.90 g/cm<sup>3</sup> recommended by EN 14214 and 0.87–0.90 g/cm<sup>3</sup> re-

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Table 1	CVSO	Methy	l fuel d	Juality	parameter	analysis.

Fuel Parameters	CVSO Methyl ester using KOH catalyst	CVSO Methyl ester using NaOH catalyst
Density (g/cm <sup>3</sup> )	0.87	0.86
Viscosity (mm <sup>2</sup> /sec)	6.22	6.22
Cloud point (°C)	0	0
Flash Point (°C)	195	202
Pour Point (°C)	2	2
Acid Value (mgKOH/g)	0.28	0.36*
Cetane Value	35.72	35.72
Higher Heating Value (MJ/Kg)	40.98	40.98
Carbon Residue	0.03	0.06*

\*p < 0.05

commended by ASTM D6751–02 (Peng *et al.* 2008), while the density of diesel fuel ranges between 0.075–0.840 g/cm<sup>3</sup> (Demirbas 2009). The density of diesel fuels is generally lower than those of biodiesel. This negative property needs more diesel fuel injected into the combustion chamber in order to gain the same power from the engine. The density of biodiesel also varies depending on the fatty acid composition and their purity (Ertan *et al.* 2008).

A change in the fuel density will influence engine output power due a different mass of fuel been injected, since diesel fuel injection systems measures the fuel by volume. A change in fuel density will also affect the start of injection pressure and spray characteristic, so that they affect engine performance, combustion and exhaust emission (Bahadur *et al.* 1995).

#### Viscosity

Viscosity is an important property of any fuel, as it is an indication of the ability of a material to flow. It was identified that viscosity is one of the most significant properties that affect biodiesel as fuel. It reduces with increase in unsaturation. Viscosities of saturated ethyl or methyl esters are slightly higher than those of unsaturated ethyl or methyl esters. The higher the viscosity the lower the ester content (Candeia *et al.* 2008). The viscosity of CVSO methyl ester is 6.22 mm<sup>2</sup>/sec. It is higher than the recommended value for petrodiesel given by ASTM D445 limits as ranging between 1.9-6.0 mm<sup>2</sup>/sec.

Biodiesel is denser and slightly more viscous than diesel and also presenting a narrower boiling points interval which indicates that the fatty acids have similar boiling points as compared to the wide variety of hydrocarbon composing the petrodiesel. The ASTM D975 limit for viscosity of diesel is 2.60 mm<sup>2</sup>. The viscosity of diesel fuel like density is also generally lower than that of biodiesel.

# **Cloud point**

The cloud point is the temperature at which wax first becomes visible when the fuel is cooled. It is an important parameter for low temperature application of a fuel (Prakash 1998). The cloud point of both CVSO methyl ester, using either KOH or NaOH catalysts are the same,  $0^{\circ}$ C.

Berrios and Skelton (2008) recommended the cloud point of biodiesel ranging from 0 to  $-5^{\circ}$ C and Georgogianni *et al.* (2007) recommended a limit of  $-3^{\circ}$ C. While Sinem *et al.* (2009) gave diesel fuel cloud point as  $-6^{\circ}$ C, and Anh *et al.* (2008) gave cloud point for conventional diesel as  $0^{\circ}$ C. The cloud point ( $0^{\circ}$ C) for CVSO methyl esters indicates that, this biodiesel can be used in cold climate, without flow problems at low temperature.

### Flash point

The flash point of biodiesel is the temperature at which the fuel become a mixture that will ignite when exposed to a spark of flame. Flash point is one of the main properties that are associated with biodiesel composition (Candeia *et* 

*al.* 2008; Anwar *et al.* 2010). Flash point is related to the amount of methanol and is also related to the amount of unconverted triacylglycerides or a low content of mono-alkyl esters. The flash point of both CVSO methyl ester using either KOH or NaOH catalyst are 195 and 202°C, respectively. The values are higher than the ASTM D93 established maximum value of >130°C (Candeia *et al.* 2008). DIN EN 14214 established flash point of biodiesel to be >120°C (Peng *et al.* 2008). ASTM D975 established diesel flash point as ranging between 52-96°C.

Biodiesel generally, has a higher flash point than diesel, hence the high values of both CVSO methyl esters using either KOH or NaOH catalyst is an indication that it is less likely to ignite accidentally and also ensures more safety in handling and storage and thus a safer fuel than fossil diesel.

### Pour point

The pour point is the temperature at which the amount of wax from the solution is sufficient to gel the fuel, thus it is the lowest temperature at which the fuel can flow (Prakash 1998; Purnanand *et al.* 2009). The pour point for both CVSO methyl ester using either KOH or NaOH catalyst are the same, 2°C. The ASTM D6751-02 biodiesel standard established the biodiesel pour point to range from -15 to 10°C likewise DIN 14214 standard, while ASTM D975 established the diesel pour point standard as -35 to  $-15^{\circ}$ C (Peng *et al.* 2008). Anh and Tan (2008) reported that the pour point of conventional diesel as  $-12^{\circ}$ C. The CVSO methyl esters have their point within the range established by both ASTM and EN standards. Higher pour points in fuels affect its use in cold environments, because they have to be kept warm by heating the electric heater insulated tanks (Reece and Peterson 1993).

### Acid value

The acid value indicates the level of free fatty acid in biodiesel. The acid value of CVSO methyl ester using KOH catalyst is 0.28 mg KOH/g while CVSO methyl ester using NaOH catalyst is 0.36 mg/KOH/g, which fell within the acceptable limit recommended by ASTM D6751 as 0.80 max, and EN – 14214 limit of 0.5 max. The acid value of biodiesels generally is lower compared to the acid value of diesel 0.104 mgKOH/g (Maumita *et al.* 2009). The acid value for both KOH and NaOH catalyzed biodiesel are low, showing that the free fatty acid content is low and the biodiesels are not likely to cause operational problems such as corrosion and pump plugging caused by corrosion and deposit formation (Candeia *et al* 2009).

#### Cetane number (CN)

The CN is a measurement of ignition performance of a fuel. It is one of the important parameter which is considered during the selection of methyl esters for use as biodiesels. It is influenced by structural features of fatty acid alkyl esters such as chain length, degree of unsaturation and branching of the chain. Better ignition quality of fuel is always associated with higher CN value (Saydut *et al.* 2008; Anwar *et al.* 2010). CN affects a number of engine performance parameters like combustion, stability, drivability, white smoke, noise and emission of CO and hydrocarbons (Ramadhas *et al.* 2006).

The CN of both methyl ester was at the same value of 35.72, which is a little below the ASTM D6751 limit of 47, Germany (DIN V51606) standard of 49 and European organization standard of 51 (Eevera *et al.* 2009). The CN of biodiesels are generally lower than that of diesel  $D_2$ , which is given as 46. The reason for the low CN is due to the fact that biodiesel fuels from high saturated feedstock (90% and above) would exhibit alkyl chain with higher fatty acids than those from less saturated feed stock, which is the case with CVSO methyl ester (Maumita *et al.* 2009).

### Higher heating value (HHV)

The higher heating value of a substance is defined as the amount of heat released during the combustion of a specified amount of that substance. The higher heating values of biodiesel are relatively lower compared to that of petrodiesel,  $D_2$ , and that of gasoline or petroleum oil but higher than coal (Demirbas 2009). The HHV for CVSO methyl esters was 40.98 MJ/kg, which is within the range recommended for biodiesel ranging between 39 - 41 MJ/kg and 43 MJ/kg for petrodiesel (Demirbas 2009). Low heating value of fuel mainly causes de-rating of engine power. But the use of petrodiesel blend with such methyl esters would lower the viscosity to desired level (Maumita *et al.* 2009).

# Carbon residue (CR)

CR is an important indicator of measures of the tendency to form carbonaceous deposits in engines. The part remaining after the sample has been subjected to thermal decomposition is the CR. It is particularly important in engines because of the possibility of clogging the fuel injectors, corrosion and cracking of components. The most common cause of excess CR in B100 is an excessive level of glycerine (Bradin 1996). The CR of CVSO methyl esters were 0.03wt% for KOH catalyzed methyl ester and 0.06wt% for NaOH catalyzed methyl ester. The ASTM D6751 recommended limit of CR in biodiesel is given as 0.050 max (Ertan and Mustafa 2008). Bayense et al. (1996) also gave acceptance limit of 0.05 max%. The CR for diesel fuel is usually higher than biodiesel. The ASTM D4530 gave diesel standard limit of CR as ranging between 0.1-0.2 wt%, while the EN 14214 gave diesel CR limit as 0.3 max (Maumita et al. 2009). The research result shows that the tendency of CVSO methyl ester to form carbonaceous deposits in engines using potassium catalyst is limited while sodium hydroxide catalyzed biodiesel is likely to form slight CR which could be due to incomplete conversion of the oil to biodiesel due to the strength of NaOH catalyst compared to KOH catalyst.

### CONCLUSION

This research study has shown that CVSO methyl ester has most of the fuel quality parameters meeting the international biodiesel standards. The results also show that KOH catalyst gave a higher yield of biodiesel compared to that of NaOH catalyst, with KOH yielding 64.36% and NaOH yielding 41.37%. Thus, biodiesel from water melon *Citrullus vulgaris* seed oil can be used as a feasible alternative as diesel replacement fuels.

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