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Research Article

ESTIMATION OF PORK LARD BIODIESEL PROPERTIES FROM ITS FATTY ACID METHYL ESTER PROFILE BY GC-MS

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ABSTRACT

The estimation of some fuel properties of pork lard biodiesel from its fatty acid methyl ester (FAME) composition was investigated. Pork lard biodiesel was produced via transesterification of pork lard and methanol using potassium hydroxide catalyst. The biodiesel was characterized to determine the physico-chemical properties according to American Society for Testing and Materials(ASTM) methods. The FAME composition of the produced biodiesel was analyzed with Gas chromatography-Mass spectrometer (GC-MS). The values of the fuel properties from the laboratory test using ASTM methods were correlated with that obtained from their calculation from the fatty acid methyl profile and an average absolute deviation (AAD) of 4.61, 3.96, 3.10, and 0.78% for iodine value, saponification value, cetane number and higher heating value, respectively were recorded. Methyl oleate ($C_{18}H_{34}O_2$) was found to be the dominant ester with percentage composition of 42.6%. The estimated values from FAME profile were found to be very close to the experimental values from ASTM method, hence rigorous laboratory experiments could be mitigated thereby saving cost, time and energy.
Keywords: Pork lard, transesterification, fatty acid methyl ester, ASTM, GC-MS.

1. INTRODUCTION

Due to global population explosion leading to high energy demand, petroleum crises, rapidly increasing prices, uncertainties concerning petroleum fuels availability and environmental concerns on increase in Green House Gas emission and ozone layer depletion, there has been renewed focus worldwide on an alternative fuel.

In spite of the fact that Nigeria is a large producer of oil in the world, it is not left out of the imbroglio in the petroleum sector due to high cost and inadequate supply of the fuel to meet the demands of the increasing population thereby bringing untold hardship to the people. This would have been relegated to the background if there were sustainable alternatives that would compete economically with the petroleum diesel fuel. A viable alternative is biodiesel.

Biodiesel is a renewable fuel which can be obtained from transesterification of vegetable oil, animal fats or waste oil with methanol in the presence of a catalyst. It is biodegradable, non- toxic

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and environmentally friendly (Encinar et al., 2007). Biodiesel quality can be influenced by the quality of the feed stock, the fatty acid composition of the parent vegetable oil or animal fat, the production process and the post production parameters (Ferrari et al., 2010).

The fatty acid profile of biodiesel corresponds to that of the parent oil or fat and is a major factor influencing fuel properties, hence the fuel characteristics of biodiesel depend on the fatty acid composition of the oil used.

A few specifications which define and set the quality standards for biodiesel have been set but the ASTM D6751 and EN14214 standards are the most commonly used standards. Lois, (2007) stated that if the product meets the biodiesel specifications, it may be defined as biodiesel.While many of these specifications are related to fuel quality issues, such as completeness of the transesterification reaction or storage conditions, several parameters directly depend uponthe fatty acid composition of the biodiesel fuel. Among these specifications are cetane number, kinematic viscosity, oxidative stability, and cold-flow properties(cloud point or cold-filter plugging point) (Knothe, 2008; Ramos et al., 2009). Other important issues to consider that are influenced by fatty ester composition but are not contained in biodiesel standards are exhaust emissions, lubricity, and heat of combustion (Ramos et al., 2009).

Cetane number (CN) and higher heating value (HHV) are important properties that affect the utilization of biodiesel fuels because they are involved in the definition of fuel quality. Saponification value (SV) and Iodine value (IV) are paramount in this study for the empirical determination of HHV and CN from its fatty acid methyl ester profile.

The aim of this study is to determine the possibility of estimating these fuel properties as a function of their molecular weight and degree of unsaturation from its fatty acid methyl ester (FAME) composition determined by GC-MS in order to eliminate laboratory testing costs, energy and time as specified by ASTM methods. Effects of process variables on biodiesel yield were studied.

2. MATERIALS AND METHODS

2.1. Materials

Methanol (CH_3OH, 99.8% purity) and potassium hydroxide were bought from Conraws Company Ltd., Enugu and of analytical grade, unless otherwise stated. Mixed pork lard was obtained from New Market in Enugu and was rendered according to the method of Alptekin et al., (2011) and Dias et al., (2008). The pork lard was rendered using dry-rendering method by subjecting it to heating in a pan without the presence of water at 110^oC for 1h (under atmospheric pressure to avoid any degradation) to remove water, the waxy, and other suspended and residual matters. Melted fat was then filtered to remove the insoluble materials (such as meat and bone particles) known as cracklings. The processed pork fat was stored in air tight opaque plastic jars to prevent oxidation.

2.2. Experimental methods

2.2.1. Transesterification Procedure

A batch reactor of 500ml capacity equipped with a reflux condenser and magnetic stirrer was charged with 100ml of oil heated in a water bath with agitation. The catalyst (potassium hydroxide) was then thoroughly mixed in methanol till it dissolved completely to give potassium methoxide. The potassium methoxide was added to the reactor and the reaction timed immediately after the addition of the potassium methoxide. It was transferred into separating funnel and allowed to settle for an hour. Two distinct layers were observed; a thick brownlayer (glycerol) at the bottom and a yellowish colour layer constituting the upper layer (biodiesel)

(Demirabas, 2005). FAME layer (Biodiesel) was then washed and dried. The experiments were carried out by varying different process variables. Temperature, catalyst concentration, reaction time and methanol/oil molar ratio were varied in the range of 50 – 75^{o}C, 0.5 – 1.5%, 10 – 100 minutes and 3:1 – 15:1, respectively. The biodiesel sample produced with the optimum reaction conditions; reaction temperature of 65^{o}, catalyst amount of 1.25%(w/w), methanol to oil ratio of 6:1 and time of 40 minutes was used to determine these fuel properties from fatty acid methyl ester composition (FAME) and ASTM method.

2.2.2. Characterization of FAME.

American Society for Testing and Materials (ASTM) Methods

Standard laboratory procedures as specified by ASTM methods Equations 1 and 2 were used to determine the Physico-chemical properties of the FAME including Saponification value (SV) and Iodine value(IV). Cetane number (CN) and Higher heating value (HHV) were determined using the empirical formulas Equations 3 and 4 suggested by Mohibbe et al., (2005) and Demirbas, (1998) respectively by substituting the experimental results of the Saponification Value (SV) and the iodine value (IV) of the FAME gotten by titration according to ASTM method prescribed.

$$\text{Saponification Value (SV)} = \frac{56.1 * N * (V_a - V_b)}{W} \quad (1)$$

Where

W= weight of oil taken in gram.
N= normality of HCL solution
V_a= volume of HCL solution used in the test in milliliter.
V_b= volume of HCL solution used in blank in milliliter.

$$\text{Iodine value (IV)} = \frac{(b-a) * 1.269}{Weight\ of\ sample\ (g)} \quad (2)$$

$$CN = 46.3 + (5458/SV) - 0.225(IV) \quad (3)$$

$$HHV = 49.43 - [0.041(SV) + 0.015(IV)] \quad (4)$$

Gas Chromatography - Mass Spectrometry (GC- MS)

The fatty acid methyl ester composition of the produced biodiesel was analyzed using an Agilent 6890 gas chromatograph equipped with an on-column automatic injector, flame ionization detector, HP 88 capillary column. 1µL of the biodiesel sample was injected into the gas chromatography with its oven temperature set at 180^{0}C and allowed to warm up with a total analytical time of 15mins at 0^{o}C/min using helium (5 PSI) as the carrier gas in the conduct of the analysis. The components were identified based on software matching with standard mass spectra. Iodine and saponification values of the biodiesel were calculated from the results obtained from the GC-MS of the sample with Equations 5 and 6 below suggested by Mohibbe et al., (2005).

$$IV = \sum \frac{(254\ D_i * A_i)}{MW_i} \quad (5)$$

$$SV = \sum \frac{(560 * A_i)}{MW_i} \quad (6)$$

Where *IV*, is the iodine value, *SV* is thesaponification value, D is the number of double bond, A*i* and MW*i* are the percentage composition and molecular mass of a particular ester, respectively.

Cetane number was calculated by substituting the empirical value of Equations 5 and 6 in Equation 3 as reported by Mohibbe *et al.* (2005) and Demirbas, (1998) while higher heating value of the biodiesel was evaluated using Equation 4 (Demirbas, 1998). Degree of unsaturation (DU)

was determined based on percentages of monounsaturated and polyunsaturated FAMEs of the produced biodiesel using Equation 7 (Mohibbe *et al.*,2005).

DU = (monosaturatedCn :1wt%) + 2 (Polyunsaturated Cn : 2, 3 wt%) (7)

Comparisons between experimental and calculated values of physical properties of FAMEs or biodiesel can be made by means of the average absolute deviation (AAD) defined by

$$AAD = \frac{100}{N}\sum_{i=1}^{NP}\left|\frac{\Phi_{Exp}-\Phi_{CAL}}{\Phi_{Exp}}\right| \quad (8)$$

where NP is the number of experimental points, Φ is the property to be predicted and the subscripts are Exp for experimental and CAL for calculated.

3. RESULTS AND DISCUSSION

3.1. Effect of Process Variables

The effect of the following process variables; reaction temperature, catalyst amount, reaction time and methanol to oil molar ratio on biodiesel yield were considered.

3.1.1. The Effect of Reaction Temperature

Transesterification can occur at different temperatures depending on the type of oil, catalyst and alcohol used. Temperature is necessary in the collision and kinetics of a reaction. Entropy increases with increasing temperature, causing the reacting species to collide more frequently or faster with sufficient energy, thus shifting the equilibrium position towards the favored direction. The influence of temperature on the yield of lard to biodiesel was investigated with five different temperatures;50, 55, 60, 65, and 70^{o}C, while the other parameters such as molar ratio of methanol to oil (6:1), reaction time (40minutes) and amount of catalyst (1.25%) were kept constant. The minimum temperature was selected as 50^{o}C mainly because, if the temperature was too low, the reaction period would be too long that might not be practical for mass production. However, generally higher reaction temperature was noticed to speed up the reaction and shorten the reaction period. Similar observation was recorded by Leung et al., 2010 who stated that higher reaction temperature decreases the viscosity of oils, enhancing the reaction rate. The effect of reaction temperature on the ester yield is shown in Figure 1. Present study shows that ester yield increased as temperature increased, but decreased as the reaction temperature increased above 65°C. Bubbles were formed, lowering the conversion of methyl ester when the temperature was above 65^{o}C. This could be as a result of methanol evaporation and may have probably favored the side reaction; saponification. A reaction temperature of 65^{o}C was observed to be optimal and was maintained to investigate other parameters. Comparable result was recorded by Jeong et al.,(2009).

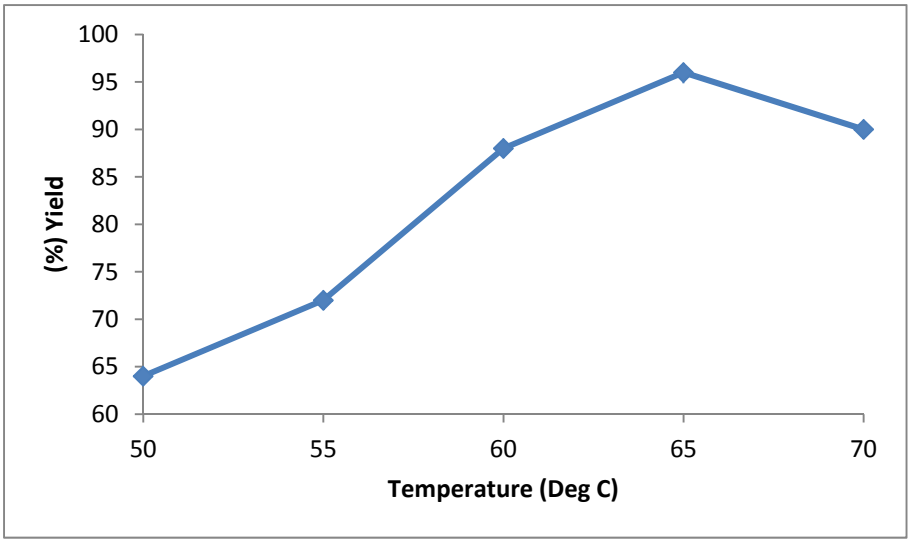

Figure 1. The effect of reaction temperature on % yield of lard methyl ester

Reaction Conditions: Time 40minutes,catalyst amount1.25%, methanol/oil molar ratio 6:1

3.1.2. The Effect of Catalyst Amount.

In a chemical reaction, catalysts provide an alternative reaction pathway for the breaking and remaking of bonds. The activation energy for this new pathway is often less than the activation energy of the normal pathway.

The effect of catalyst was studied by varying the catalyst concentration in the range of 0.5, 0.75, 1.00, 1.25 and 1.5% while keeping other reaction conditions constant at reaction time of 40minutes, temperature of 65^{o}C, oil to methanol molar ratio of 6:1. Figure 2 shows the effect of catalyst concentration on the yield of biodiesel. It was observed that increase in catalyst concentration from 0.5 to 1.25% resulted in an increase in yield. However, the biodiesel yield declined significantly at the catalyst concentration higher than 1.25% probably because the slurry (mixture of catalyst and reactants) became too viscous, making it too difficult for stirring and consumes more energy thereby demanding higher power consumption for adequate stirring (Xie et al.,2006) and also due to the formation of fatty acid salts (soap), saponification.The produced soap prevented a clear separation of biodiesel from glycerol fraction, which increased the viscosity of the biodiesel, thus lowering the yield of biodiesel. The highest yield of methyl ester was obtained at the catalyst concentration of 1.25% weight of oil, hence the catalyst concentration of 1.25%, mass of 0.6g was regarded as optimal value and used for further experimentation to evaluate other process parameters.

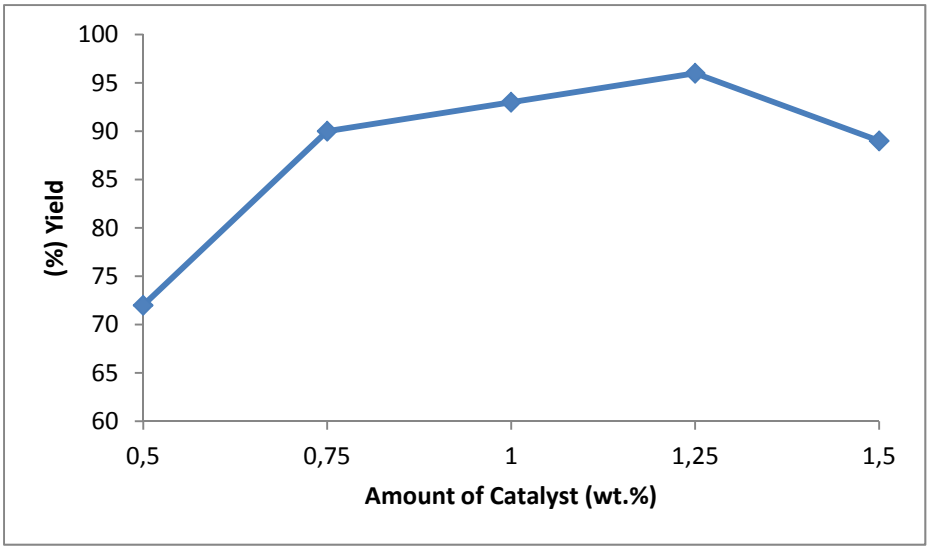

Figure 2. The effect of catalyst amount on %yield of lard methyl ester

Reaction Conditions: Time 40minutes,temperature 65^{o}C, methanol/oil molar ratio 6:1

3.1.3. The Effect of Reaction Time

In this work, the effect of reaction time during the transesterification reaction on the biodiesel yield was investigated by varying the time from 10 – 100 minutes, while other parameters remained constant as depicted in Figure 3 which shows the yield of biodiesel at different reaction time. The reaction was seen to start very fast as within 10 minutes, more than 70% conversion had taken place. This is comparable to the findings of several investigators who found that the reaction starts very fast and almost 80% of the conversion takes place in the first 15mins(Furuta et al.,2006;Ma et al.,1998a). It was noticed that the maximum biodiesel yield was obtained at the reaction period of 40 minutes. It was also observed that the yield increased significantly from 10 to 40 minutes which indicated that most of the transesterification occurred at 40minutes after which the yield declined. The declination in yield after 40 minutes could be due to the reversible nature of the transesterification process.

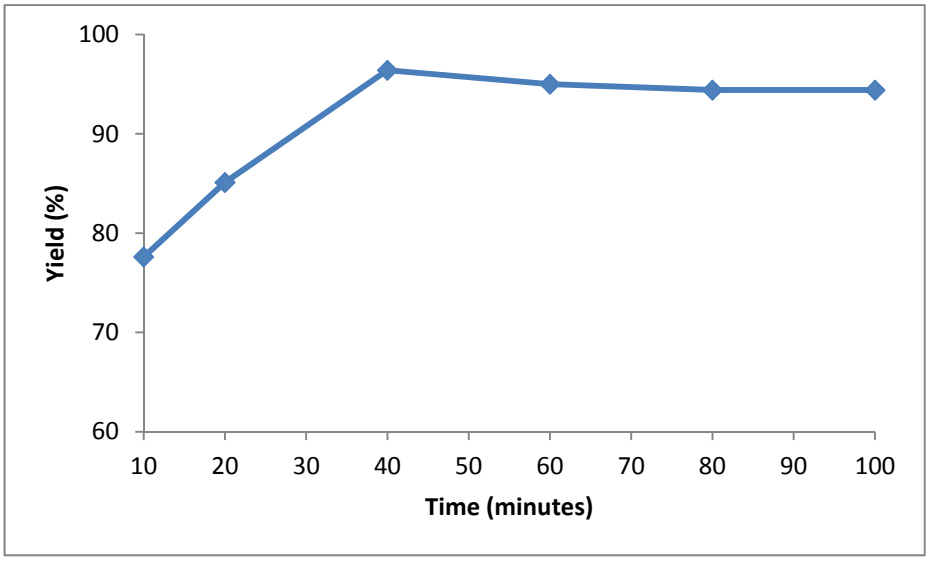

Figure 3. The effect of reaction time on %yield of lard methyl ester

Reaction Conditions: Catalyst amount.1.25%, temperature 65^{o}C, methanol/oil molar ratio 6:1

3.1.4. The Effect of Methanol/Oil Molar Ratio

The methanol/oil molar ratio is considered to be one of the most important factors affecting the yield of biodiesel. The required stoichiometric ratio for transesterification requires 3 moles of methanol for each mole of oil to yield 3 moles of fatty acid methyl ester and 1 mole of glycerol (Kose et al.,2002). Since the transesterification reaction is reversible reaction, excess methanol is required to shift the equilibrium to the expected product, biodiesel side.The molar ratio has no effect on acid, peroxide, saponification and iodine value of esters (Tomaseviset al., 2003). In this study, the effect of molar ratio of methanol to oil on ester yield in the range of 3:1- 15:1, was investigated keeping other parameters constant.

The yield of methyl esters to the different molar ratio of methanol/oil, is shown in Figure 4. The maximum ester yield was obtained at a methanolysis of oil molar ratio of 6:1. The higher molar ratio than the stoichiometric value results in a higher rate of ester formation and could ensure complete reaction. Freedman et al.,(1984) suggested the optimum molar ratio to be in between 4.8:1 and 6.5:1 for the maximum yield of biodiesel from animal fat/used cooking oil which alligned from the result of this present study.

It was observed in Figure 4 that values below 6:1 gave rise to relative poor yield which could be due to incomplete transesterification resulting from insufficient alcohol groups to replace all the acid groups in the triglyceride and is further supported by the report that transesterification is insufficient at the ratios of methanol/oil below 5:1(Board NB,2009). The yield of biodiesel was observed to decline above the molar ratio (methanol/oil) of 6:1 in this study. This could be attributed to the fact that excess methanol deactivated the catalyst thereby reducing its effectiveness (Hogue et al.,2010). Also, the excess methanol could hinder the separation, so that the relative yield of esters decreases because part of the glycerol may remain in the biodiesel phase and also tended to blur the separation border between glycerol and biodiesel, making it difficult to extract the biodiesel. In addition, when glycerin remains in solution, it reverts the

equilibrium to the left, lowering the yield of esters. The observed optimum value was maintained throughout the experimentation to investigate effects of other process variables.

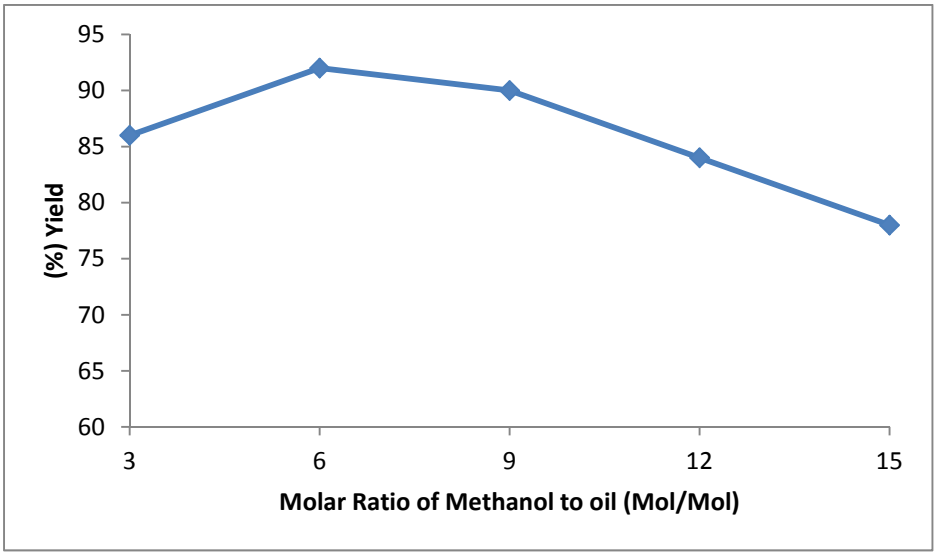

Figure 4. The effect of methanol/oil molar ratio on %yield of lard methyl ester.

Reaction Conditions: Catalyst amount1.25%, temperature 65^{o}C, time of 40 minutes

3.2. Characterization of the FAME

3.2.1. American Society for Testing and Materials(ASTM) Values

Experimental values of various physico-chemical properties of pork lard biodiesel according to American society for testing and materials standards (ASTM) methods using standard apparatus for the measurement are presented in Table 1.

Table 1. Fuel properties of lard methyl ester using ASTM methods

Properties	Units	Lard	Lard biodiesel	ASTM limits
Acid value	mgKOH/g oil	0.84	0.28	0.50 max
Free fatty acid	%	0.42	0.14	-
Specific gravity@30^{o}C	-	0.9088	0.8732	0.86 – 0.90
Viscosity@40^{o}C	mm^2/s	25.26	4.63	1.9 – 6.0
Saponification value	mgKOH/g oil	225.8	190.2	-
Iodine value	gI_2/100g oil	54.57	46.0	3 min
Water content	%	TRACE	-	-
Peroxide value mEq/kg	80.0	-	-	
Cetane number	-	-	65	47 min
Higher heating value	MJ/kg	-	40.92	40 - 42
Flash point	^{o}C	-	135	130 min
Cloud point	^{o}C	-	+9	-3 to 12
Pour point	^{o}C	-	+6	-15 to 10

The experimental results of the physico- chemical properties of the pork lard biodiesel were found to fall within the ASTM limit hence; the produced biodiesel is suitable for use either as a blend or direct replacement of the diesel fuel. Most of these properties could be estimated from the fatty acid methyl ester composition to mitigate the rigorous laboratory procedures and costs.

3.2.2 The Result of GC-MS Analysis of pork lard biodiesel

The fatty acid ester composition of pork lard biodiesel determined by GC-MS showed that different carbon chain compounds are present in the sample as shown in Table 2.

Table 2. Fatty acid ester compositions of pork lard biodiesel.

Fatty acid	Carbon molecule	Formula	Retention time(mins)	Weight(%)
Palmitic	C16:0	$C_{16}H_{32}O_2$	28.123	23.9
Palmitoleic	C16:1	$C_{16}H_{30}O_2$	18.400	2.1
Heptadecenoic	C17:0	$C_{17}H_{34}O_2$	34.193	0.4
Stearic	C18:0	$C_{18}H_{36}O_2$	19.020	12.6
Oleic	C18:1	$C_{18}H_{34}O_2$	18.320	42.6
Linoleic	C18.2	$C_{18}H_{32}O_2$	15.483	15.1
Arachidic	C20:0	$C_{20}H_{40}O_2$	24.066	0.2
Others	-	-	-	3.0

The fatty acid methyl ester profile is one of the key factors that determine the suitability or otherwise of any feedstock for use in biodiesel fuel production (Knothe, 2009). The profile results in Table 2 revealed methyl oleate ($C_{18}H_{34}O_2$) as the predominant compound in the analyzed sample having the highest percentage of 42.6% followed by methyl palmitate with 23.9% which are monounsaturated and saturated fatty acid methyl esters respectively leading to less affinity to oxygen that will lead to quick peroxidation. Hence, makes it a good compound with respect to stability of biodiesel as higher degree of unsaturation in fatty acid methyl esters limits its suitability for use as a fuel due to high polymerization tendency as a result of peroxidation (Gaby

and Peter, 1997). At high temperature, commonly experienced in combustion engines, the peroxidation can be accelerated and engine can quickly become gummed with polymerized fatty methyl esters (Mohibbe *et al.,*2005). Hence, feedstock with high percentage of polyunsaturated acid is not suitable for usage as biodiesel.

3.3. Estimated Values of some Fuel Properties from its Fatty Acid Composition by GC-MS

The fatty acid ester composition of the pork lard biodiesel contained in Table 2 was used to evaluate the fuel properties.

Table 3. Estimated values of the physicochemical properties of lard biodiesel from its fatty acid ester composition by GC-MS.

Parameter	Values
Saponification value (mgKOH)	197.75
Iodine value (mgI_2/100g)	48.12
Cetane number (CN)	63.07
Higher heating value (HHV) MJ/kg	40.60
Saturated FAME (%)	37.1
Monounsaturated FAME (%)	44.7
Polyunsaturated FAME (%)	-
Degree of unsaturation	44.7

Knothe, (2005) stated that the oxidation stability decreases with the increase of polyunsaturated fatty acid methyl esters content. Oils/ fats rich in linoleic and linolenic acids or double bonds impart to fatty acids the susceptibility to reaction with oxygen leading to poor oxidation stability to fuels. Autoxidation of unsaturated fatty compounds proceeds at different rates depending on the number and position of double bonds. It could be inferred from the results in Table 3 that the produced biodiesel will be stable owing to the absence of polyunsaturated fatty acids thus reaffirming the suitability of pork lard for biodiesel production. The estimated values of the physicochemical properties in Table 3 were found to be very close to the experimental values of these properties done with ASTM method in Table 1. This result indicated that from the fatty acid composition, some of the fuel properties could be satisfactorily estimated; hence reducing laboratory cost and saves time.

3.4. Physical properties of Biodiesel.

The various properties of FAME measured in this work such as saponification value, iodine value, cetane number and higher heating value showed an average absolute deviation (AAD) of 3.96, 4.61, 3.10, and 0.78% respectively when the experimental data from ASTM method were compared with the estimated values of these properties from their fatty acid methyl ester which shows a good correlation between them.

Saponification value represents the number of milligrams of potassium hydroxide or sodium hydroxide required to neutralize 1 g of fat under the conditions specified. It is a measure of the average molecular weight (or chain length) of all the fatty acids present. It allows for comparison of the average fatty acid chain length present in oil/fat and is necessary in determining the amount of alkali which is required to convert a given quantity of oil or fat into soap and detecting the adulteration of fat or oil by one of lower or higher saponification value. Gopinath et al., (2009) found a correlation for saponification value based on palmitic, stearic, oleic, linoleic and linolenic acid content in oils. It can be seen from Tables 1 and 3 that the experimental saponification value

using ASTM method is comparable to the estimated result from equation 6 deduced from the fatty acid ester composition of the biodiesel.

Iodine value measures the total unsaturation (double bond) within a mixture of fatty acids. It is the amount of iodine required to iodize all the double bonds in the biodiesel. It is expressed in grams of iodine which react with 100g of the respective sample when iodine is added to the double bonds. Iodine absorption occurs at double bond positions thus a higher IV indicates a higher quantity of double bonds in the sample and greater potential to polymerize in engine and hence lesser stability. A good correlation was found to exist between the experimental and the estimated results from its fatty acid methyl ester composition with equation 5.

Cetane number serves as a measure of ignition quality of a fuel in a diesel engine. It measures how easily ignition occurs and the smoothness of combustion. This is the most pronounced change from animal fat to the transesterified product. Generally, the higher the cetane number, the better the ignition quality and efficiency of the fuel and vice versa (Meher et al., 2006). Fuels with low cetane numbers show an increase in gaseous and particulate exhaust emissions due to incomplete combustion. The values of 65 and 63.07 from the experimental and estimated studies respectively recorded in this study buttress the findings from Encinar et al., (2011) that naturally, animal fat biodiesel has a higher cetane number than plant oil biodiesel because of its high content in saturated compounds. The higher cetane number improves engine performance, cold starting, and warm-up, and helps engines run smoothly. This study takes an approach to determine the experimental cetane number of biodiesel by substituting the experimental values of saponification value and iodine value using ASTM methods in equation 3 and also to estimate the cetane number by using the estimated values of saponification and iodine value from the fatty acid methyl ester composition using GC-MS with equations 5 and 6 respectively in equation 3. It can be seen from Table 3 that the estimated value of cetane number for the biodiesel sample is close to the experimental result in Table 1 as the AAD between experimental and estimated values is 3.10% indicating a minor prediction error. The experimental and estimated cetane numbers obtained in this work are within the ASTM specification that requires a minimum value of 47 (Table 1).

Higher heating value is the amount of heat released by the complete combustion of a unit quantity of fuel. The heat of combustion is important parameter for estimating fuel consumption: the greater the heat of combustion, the lower the fuel consumption. The heat of combustion or heating value is not specified in the biodiesel standards ASTM D6751 and EN14214. However, European standard for using biodiesel as heating oil, EN 14213, specifies a minimum heating value of 35 MJ/kg. The heat of combustion increases with an increasing chain length and decreases with an increasing unsaturation. The higher heating value of a fuel increases with increasing carbon number in fuel molecules and also increases as the ratio of carbon and hydrogen to oxygen and nitrogen increases (Demirbas, 1998). The AAD between experimental and estimated higher heating values is 0.78%, which indicates a very good correlation.

4. CONCLUSION

The physical properties of biodiesel produced from pork lard such as the iodine value, saponification value, cetane number and higher heating value were studied. The experimental values of these properties using ASTM methods and the estimated values from its fatty acid methyl ester (FAME) composition with GC-MS were considered and compared. It was observed that there is a good agreement between experimental and estimated values of the physical properties considered based on the values of the average absolute deviation (AAD) gotten which indicated minor prediction errors hence, the estimated values show sufficient accuracy and as such could be used in engineering applications without rigorous laboratory procedures. The individual process variables were observed to directly influence the biodiesel yield.

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