BIODIESEL PRODUCTION FROM THORN APPLE (*DATURA METEL)* SEEDS OIL USING NAOH AS CATALYST

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ABSTRACT

The oil used in this work was extracted from the seeds of Datura metel commonly called Thorn apple, Devils weed. 650ml of the oil extract was obtained by using soxhlet extraction and n-hexane as solvent the physico-chemical properties of the oil were evaluated. Two-step Transesterification of the oil in the presence of NaOH as catalyst was done by varying catalyst concentration as 1 %, 2 % of the weight of the oil with 6:1 and 9:1 molar ratio of methanol to oil at constant reaction temperature (60 ^{0}C) and time (120 mins). The biodiesel yields maximally (80 %) at 1 % catalyst (NaOH) concentration with 6:1 molar ratio of the methanol to oil. Acid value, Free Fatty acid, saponification value, iodine value and the moisture content of the biodiesel produced were 4.21 mg KOH/g, 2.10 %, 56.1 mg KOH/g, 22.84 g I_2/100g and 3.0 % respectively. The cetane number, higher heating value and flash point were determined given at 138.451, 49.09 KJ/Kg, and 146 ^{0}C which are in agreement with the ASTM standard for determining biodiesel properties suitable for diesel engines. The 14.3 % yield of the oil from Datura metel seeds makes it a good feedstock for biodiesel production, hence a viable source of renewable energy for future use.

Keywords: Biodiesel, Catalyst, Cetene Number, Transesterification, Renewable Energy

INTRODUCTION

The scarcity of fossil diesel, ever increasing population and the demand for energy, increasing cost of petroleum fuel, strict emission norms, climate change and global environmental problem has renewed interest among scientific community and researchers to search for an alternative source of energy (Rafel *et al.*,2011). Climate change is real and it is happening in an unprecedented rate and decades ahead of what the scientific community projected. The fossil based liquid fuel is majorly used in transportation and agricultural sectors in any developing or developed countries. People and their ever-increasing thirst for energy have brought devastating consequences of global warming, climate change and other environmental problems. Presently energy, environment and sustainability are the hot topics to discuss about across the globe (Ma, and Hanna, 1999). Petroleum based fuels are finite and distributed across certain regions of the world. Transportation and agricultural sector are the two main consumers of finite reserved Petro-diesel (Agarwal, 2007; and Demirbas, 2007). The concept of using vegetable oil in diesel engine is not new. In 1911 Dr. Rudolf Diesel used peanut oil to energize one of his diesel engines due to its similar properties with the fossil diesel. He encouraged using various vegetable oils in diesel engines. Not only Dr. Diesel but Henry Ford has similar kind of vision. In 1925 he hailed biofuel as the “fuel of the future” after he had successfully run his model T ford with bio-ethanol (Mahmud *et al.*, 2007).

Biodiesel is a renewable clean bio-fuel, which is defined chemically as a mixture of methyl esters with long-chain fatty acids. Typically, it is produced from the catalytic Transesterification of vegetable oils and animal fats with alcohol, using either acid or basic catalysts (Da Silva *et al.*, 2011). Usually, methanol is the preferred alcohol for the production of biodiesel due to its low cost and industrial availability (Leung *et al.*, 2008).

Homogeneous catalysis

(Karmakar *et al.* (2010) reported that, homogeneous process leads to higher yields, especially if the content of free fatty acids (FFAs) in the used feedstock is less than 1 %, when compared to the heterogeneous catalytic trans-esterification.

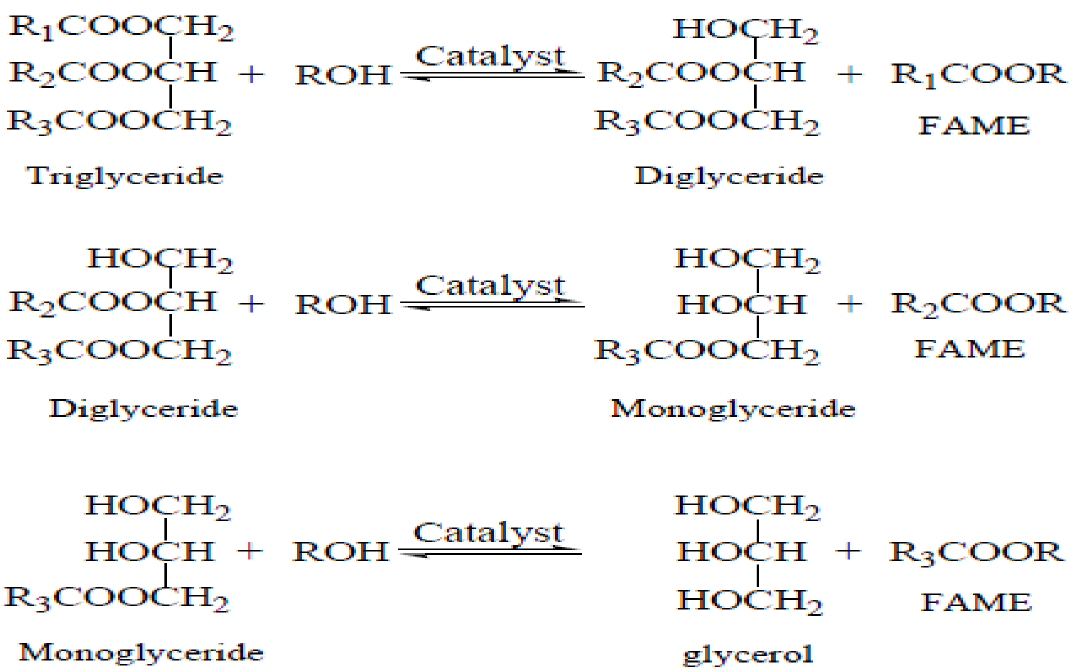
Chemical Reaction in Biodiesel Production

Alcohol reacts with fatty acid to form the mono-alkyl ester (biodiesel) and crude glycerol. The reaction between the biolipid (fat and oil) and the alcohol is a reversible reaction so excess alcohol must be added to ensure complete conversion (Dorado *et al.*, 2003). The overall transesterification reaction is given by three consecutive and reversible equations as shown below.

The Plant

Datura metel L. belongs to the family Solanceae, and is commonly called Thorn Apple in English, in Nigeria, it is locally called: Zakami in Hausa, Apikan/Gegemu in Yoruba and Myaramuo in Igbo (Abdullah *et al*., 2003).

In Nigeria, especially in the northern part, *Datura* is found growing as a weed on abandoned farmlands and on dumpsites. The leaves and seeds of the plant are used for several purposes and in several ways special for its psychoactive activities, thus making the plant parts to be abused by the youths who are more prone to dangers of smoking and drug abuse (Katuma *et al.*, 2010).



The first step is the conversion of triglyceride to diglyceride, followed by conversion of diglyceride to monoglyceride and from monoglyceride to glycerol, yielding one methyl ester molecule per molecule at each step (George *et al.,* 2009).

Figure 1: *Datura metel* plant flower and seed pod

MATERIALS AND METHODS

Sample and Treatment

The sample of Thorn apple seeds (*Datura metel*) was obtained from Gbodoya, Afijio Local Government Area, Awe, Oyo State, Nigeria.

The thorn apple seeds (*Datura metel*) were removed from the pods by mechanical means. The seeds were subjected to sun drying for three days. Then the sample was grinded to powdered form using pestle and mortar and then sieved in order to increase the surface area for optimum oil extraction.

The powdered sample was weighed using electrical weighing balance in the laboratory, all necessary precautions been observed.

Extraction of oil using soxhlet apparatus

A medium sized of the soxhlet apparatus was properly set up with 70 g of the sample in a thimble and 150 cm^3 of the n-hexane solvent was put in a round bottomed flask assembled on a heating mantle. The heating mantle was set at 60 ^{0}C such that the solvent boiled gently. The vapour passing through the vapour tube condensed by the condenser and the condensed hot solvent fell into the thimble in the soxhlet which eventually siphoned over into the round bottomed flask. As the process continued, oil was extracted. The process was repeated until complete extraction was affected. The extract was heated to recover the solvent and pure oil was obtained (Barnwal and Sharma, 2005). The properties the extracted oil is shown in table 2.

Experimental Design

Response surface (Box-Bechnken) statistical experimental design was used to design the optimization. Two independent variables namely reaction catalyst (NaOH) concentration and amount of methanol/oil at constant reaction temperature and time were selected for investigation. Table 3 shows the lower and upper of factors employed based on literature survey (Indhumathi *et al.*, 2014). Each run was completely randomized to obtain a total of two runs. The experiment was design using MINITAB 17 statistical software.

Table 1: The Process Variables and their Levels

Factor	Lower level	Upper level
Temperature (^{0}C)	60	70
Time (min)	120	120
Methanol to Oil ratio (w/w)	6:1	9:1
Catalyst (%)	1	2

Double Step Transesterification Reaction of Thorn Apple (*Datura metel*) Seed Oil

The *Datura metel* seed oil was divided into four portions labeled A, B, C, & D and each portion was esterified using different masses of sulfated zirconia catalyst, saying 1.0 g and 2.0 g. Oil (10 g) each portion was reacted with methanol in the ratio 6:1, and 9:1 of methanol/oil (w/w), 1 %, and 2 % catalyst by weight of the oil. The mixture was then placed on a water bath and heated at a constant temperature of 60 ^{0}C for the reaction time of 120 minutes with continuous stirring. The content was transferred into a separating funnel and allowed to settle for 24 hours. This permits the glycerin to settle down since it is denser than ester (biodiesel). The glycerin was removed from the separating funnel while biodiesel was obtained (Asokan and Vijayan, 2014).

The esterified oil with lowest acid value was transesterified using sodium hydroxide catalyst, the reacting mixture forms two layers or phases: the upper layer containing liquid and the bottom layer. The liquid phase was recovered by decantation and it forms two layers that was separated using separating funnel. The top layer is biodiesel, while the bottom layer is the glycerin. The biodiesel separation was carried out using separation funnel as the glycerin was drained down, which was first collected through the outlet while the biodiesel remained inside the separation funnel (Sokoto *et al.*,2011).

The percentage of biodiesel produced was calculated using the equation below

$$\%\ Biodiesel\ Yield = \frac{Weight\ of\ Biodiesel}{Weight\ of\ Oil} \times 100$$

Physico-Chemical Properties of the Biodiesel Produced

Determination of Acid Value

Exactly 2 g of biodiesel was poured into a beaker and 1 drop of phenolphthalein was added, the beaker was placed into water bath heated at 60 ^{0}C for 1 minute, 0.1 M solution KOH was titrated into the beaker till the color change to pink. (Mukhtar and Dabai 2016). The procedure was repeated three (3) times for both the sample and blank. This was repeated for both samples

Acid value of KOH of the sample can be calculated using the equation.

$$AV = \frac{(A - B) \times N \times 56.1}{W} \ldots \ldots (i)$$

Where A = Volume, ml of standard alkali used in titration.

B = Volume, ml of standard alkali used in titrating the blank.

N = normality of standard alkali

W = mass, grams of sample.

Determination of Density

Measuring cylinder was placed on weighing balance, and weighing balance was calibrated to zero (0), biodiesel was poured inside the measuring cylinder up to 7 cm^3 and mass of the biodiesel was measured. This was done for each biodiesel produced. The procedure was repeated 3 times for both the sample and blank. This was repeated for each sample.

The density of biodiesel can be calculated using the following equation (Almustapha *et al.*,2009).

$$\text{Density (D)} = \frac{M}{V} \ldots \ldots \ldots (ii)$$

Where M = mass of the biodiesel (gram)

V = volume of biodiesel in (cm^3)

Determination of Iodine Value

Exactly 3 g of biodiesel sample was weighed into a conical flask, 5 cm^3 of 5 % HCl was added and the mixture was stirred until the biodiesel sample formed homogeneous mixture. 25 cm^3 of iodine solution was added and the mixture stirred for 5 minutes. The mixture was titrated with 0.1 M $Na_2S_2O_3$ solution until a pale straw colour was obtained. At this point, 1 cm^3 of starch indicator was added to give blue-black colour and titration continues until a colorless end point was reached. The same procedure for blank titration was done. It was repeated one more time for the biodiesel sample to calculate the average titre.

The iodine value was determined using the equation below

$$\text{Iodine value} = \frac{(V_b - V_a) \times M \times 126.9}{W} \ldots \ldots (iii)$$

Where:

126.9 = molecular weight of iodine
M = morality of $Na_2S_2O_3$
V_a = titre value of $Na_2S_2O_3$ (sample)
V_b = titre value Chika M. *et al.*, 2019 k titration)
W = weight of biodiesel sample.

Determination of Saponification Value

Exactly 1 g of phenolphthalein indicator was dissolved in 100 cm^3 of methanol and 4 g of KOH was dissolved in volumetric flask to prepare 0.1 M of KOH. 1.0 g of biodiesel was poured into a beaker and 1 drop of phenolphthalein was added, the beaker was placed into water bath heated at 60 ^{0}C for 60 minutes, 0.1 M solution of NaOH was then titrated into the beaker containing mixture. The colour changed to pink and produced some soap bubbles ((STCAM, 2013). This can be calculated using the equation below:

$$SV = \frac{N(A-B)x56.1}{W} \ldots\ldots\ldots\ldots\ldots\ldots\ldots \; (iv)$$

Where, B = volume of titrate, cm^3 of blank
A = volume of titrate, cm^3 of sample
N = normality of standard alkali
This was repeated for each biodiesel produce.

Determination of Kinematic Viscosity

Exactly 20 cm^3 of oil was taken into the sample container. The spindle to be used was mounted to the machine and the spindle number was selected from the machine. The revolution per minute was also selected. The machine reading was automatically shown on the screen (Ajiwe *et al.*,2006).

$$Viscosity = C \times t \ldots\ldots\ldots\ldots..(v)$$

Flash point

The flash point was determined according to ASTM D-93 method. The sample was placed in the test cup to the prescribed mark in the interior of the cup. The cup was mounted on to its position on the tester. Bunsen burner was used to supply heat to the apparatus at rate of 1 ^{0}C per minute with constant stirring. A small test flame was directed into the cup intermittently. The flash point was taken as the temperature using thermometer when the test flame caused the vapor above the sample to ignite (Indhumathi *et al.,* 2014).

GC-MS Analysis of *Datura metel* Biodiesel Produced

The oil composition and methyl ester content were assayed using a GC-MS machine in Multi-Users Laboratory of Ahmadu Bello University (A.B.U), Zaria, Kaduna State Nigeria.

The GC-MS was equipped with an Econo-cap EC-WAX capillary column (30.0 min length x 250 μm in diameter x 0.25 μm in film thickness). The GC oven was maintained at 50 ^{0}C for 3 minutes, then heated to 210 ^{0}C at a rate of 10 ^{0}C per minute and held at 210 ^{0}C for 9 minutes. The front inlet temperature of the oven was 225 ^{0}C (split less-model). The carrier gas was helium with a flow rate of 12 cm^3/min. The FAME analysis of *Datura metel* biodiesel produced was carried out by injecting 1.0 μL of a sample of biodiesel.

RESULTS

Table 2: Physicochemical parameters of *Datura metel* oil

S/N	Parameter	Unit	Result
1	Oil yield	%	14.3
2	Moisture content	%	2.5
3	Density	g/cm^3	0.80
4	Acid value	mg KOH/g	13.75
5	Free fatty acid	mg KOH/g	4.88
6	Iodine value	mg I_2/100g	88.83
7	Saponification value	mg KOH/g	182.3
8	Viscosity	mm^2/s	256

Table 3: Quality parameters of bodiesel produced from *Datura metel* seed oil

S/N	Parameter	Units	Result	Limits
1	Yield	%	80.0	-
2	Moisture Cont.	(V) %	3.0	0.50max
3	Density	g/cm^3	0.88	0.90max
4	Acid value	mgKOH/g	2.0	0.50max
5	FFA	mgKOH/g	2.15	-
6	Viscosity	mm^2/s	150	-
7	Saponification value	mgKOH/g	56.1	-
8	Iodine value	mgI_2/g	22.84	120max
9	Flash point	^{0}C	146	-
10	Cetane number	-	138.451	47min
11	Higher Heating Value	KJ/Kg	49.09	47min

Table 4. Effect of reaction conditions on the biodiesel of *Datura metel* seed oil.

Sample	Catalyst conc. (%)	MeOH/Oil molar ratio	Temperature (^{0}C)	Time (min)	Biodiesel yield (%)
A	1	9:1	60	120	67.2
B	2	9:1	60	60	57.5
C	1	6:1	60	120	80.0
D	2	6:1	60	60	42.3

Table 5: GC-MS Result of the biodiesel fuel produced from *Datura metel* seeds oil.

S/N	Saturated methyl ester	Quality	Area % composition
1	Methyl tetradecanoate	98	0.11
2	Hexanoic acid, methyl ester	99	50.55
	Unsaturated methyl ester		
3	9,12-Octadecadienoic acid, methyl ester	99	3.18
4	9-Hexadecanoic acid, methyl ester	91	0.28
5	10,13-Octadecanoic acid, methyl ester	99	3.08
6	7-Octadecanoic acid, methyl ester	99	30.21
	Total FAME Content		**87.41**

Table 6: GC-MS Result showing Non-methyl esters

S/N	Non-methyl ester compounds	Quality	Area %
1	n-Hexanoic acid	90	3.26
2	9, 17-Octadecadienal, (Z)-	97	2.19
3	2-Methyl Z, Z-3,13-Octadecadienol	44	4.83
4	9,12-Octadecanoic acid (Z, Z)-	56	2.31
	Total		**12.59**

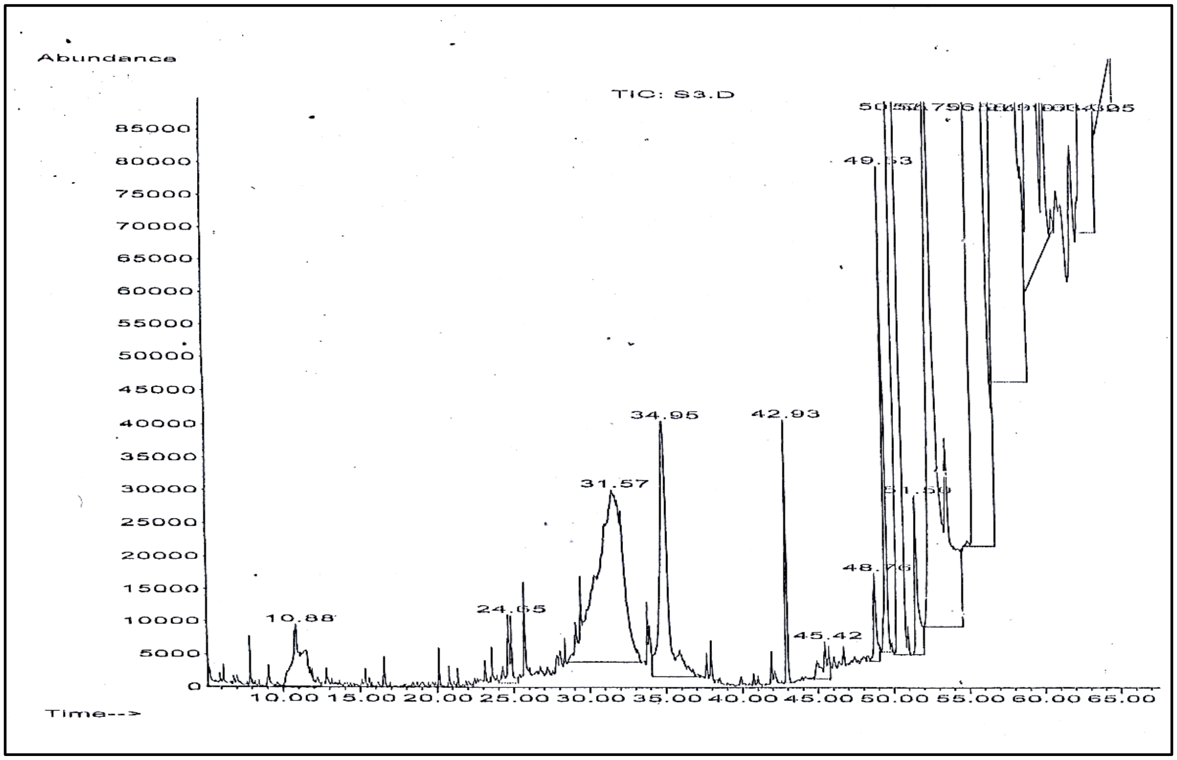


Figure 2: Chromatogram of the GC-MS analysis of the biodiesel produced from *Datura metel* Seed Oil.

DISCUSSION

From Table 2 above, the oil obtained from the extracted *Datura metel* seeds was bright yellow in colour, it was viscous at room temperature. The percentage of oil yield was 14.3 %, and when compared with the percentage yield of oil in *Datura stramonium* (10.3-23.2 %) the result falls within the range as reported by (Babagana *et al.*,2011).

Babagana *et al.*(2011) Reported that the oil yield from seeds of *Balanitea egyptiaca* was 34.52 %, while Jatropha contains 30 to 40 % oil that can be easily expressed for processing and refinement to produce biodiesel (Akpan *et al.,* 2006). The percentage oil content of castor seeds was found to be 33.2 % of the total weight of 155.30 g (Akpan *et al.*,2006). The seed oil showed low oil yield related to Jatropha (30 to 40 %) and Castor seed oil (33.2 %). The moisture content of the oil obtained from *Datura metel* seeds was found to be 2.5 % while that of the *Datura metel* biodiesel was 3.0 %. From a similar work done on *Jatropha*, it was observed that *Jatropha* seed oil contains 0.2 % (Abdulmalik, 2009).

This research work shows that the density of the oil was 0.89 $gcm^{-3}$and that of the biodiesel produced was 0.87gcm^{-3}. The density of biodiesel obtained from NaOH ethanolysis of *Lageneria sincereria* seed oil was 0.80 gcm^{-3}, and the values are within the ASTM limits of biodiesel (Gerpen *et al.,* 2004). The lower density of the methyl biodiesel produced on this research may be due to its degree of unsaturation or chain length (Javidialesaadi and Raeissi (2013); Sexena *etal.*, 2013). The result in Table 3 showed that the oil contains acid value of 13.75 mg KOH/g carried out by titration method.

The free fatty acid is one of the key parameters affecting the yield of biodiesel in transesterification process. The free fatty acid in the *Datura metel* oil sample investigated by titration method was estimated to be 6.88 mgKOH/g as against 14.1 mgKOH/g of *Datura stramonium* oil sample (Koria and Thangaraj, 2010). The biodiesel production process depends on the amount of the free fatty acid in the oil to be processed. Successful alkaline transesterification requires a free fatty acid value lower than 3 % (Freedman and Pryde (1982); Liu (1994); Mittelbach *et al.*(1992)). One step base catalyzed transesterification is applied to feedstock containing free fatty acid level up to 3 %. The oil should not contain more than 1 % FFA for alkaline catalyzed transesterification reactions. If the FFA level exceeds this amount, the formation of soap will inhibit the separation of the ester from the glycerin and also reduces the ester conversion rate. In this study, *Datura metel* oil was subjected to two steps process, a combination of sterification using sulfated zirconia followed by base (sodium hydroxide) transesterification as free fatty acid level is greater than 3 %. The biodiesel from *Datura metel* seed oil in this work has FFA of 2.10 mgKOH/g.

The iodine value of the oil sample was investigated by titration method which was estimated to be 88.83 $mgI_2/100g$. Iodine value of the biodiesel produced from *Datura metel* seed oil was found to be 22.84 $mgI_2/100g$. The saponification value obtained in the oil sample used in this work by titration method was found to be 182.33 mgKOH/g as shown in appendix 1. But the saponification value for biodiesel produced from *Datura metel* seed oil was found to be 56.1mgKOH/g which is lower than that of the Gingerbread plum seed oil biodiesel 84.20 mgKOH/g (Mukhtar and Dabai 2016). Flash point is an important parameter used to determine fuel quality and is the temperature at which fuel will ignite when exposed to flame. Biodiesel usually has high flash point (more than 150 ^{0}C) flash point than petroleum diesel (55-66 ^{0}C) (Atabani *et al.*, 2013). The flash point value (146 ^{0}C) was obtained in this research work and agrees closely with 144 ^{0}C of the *Lageneraria sinceraria* seed oil biodiesel done by (Mukhtar *et al.*, 2014) which is in agreement with ASTM standard (130 ^{0}C min).

Kinematic Viscosity

Kinematic viscosity is the resistance to flow of a liquid under gravity. It is an important property that represents the flow characteristics of the fuel (Bello *et al.*, 2009). Viscosity is one of the properties that determines free flow of fluid (liquid) and is an indicator used in fuel quality. The viscosity value of *Datura metel* seed oil was 256mm^2/s and that of the biodiesel from *datura* oil was 150mm^2/s. The difference in the values may be attributed to the fact that some components were removed during biodiesel production (Akpan *et al.,* 2006).

Higher Heating Value

The result 47.47 MJ/kg obtained as presented in Table 3 is higher than 29.88 MJ/kg reported for *Hevea brasiliensis* (Mukhtar and Dabai, 2016). Fuel having higher heating value gives higher power out and it small quantity will cover long distance drive (Gulum and Bilgin, 2015).

Influence of Concentration of Catalyst and Molar ratio of Methanol to Oil on Biodiesel Production.

The effect of reaction condition was investigated in the production of biodiesel yield from the *Datura metel* seed oil using NaOH as catalyst. Table 4 indicated that varied concentration of catalyst used at 1.0 % to 2.0 % in the MeOH/Oil molar ratio (w/w) of the oil at 60 ^{0}C temperature for reaction time of 120 minutes. The process yielded 67.2 % for sample 'A', 57.5 % for sample 'B' in 9:1 molar ratio of MeOH/Oil at 1.0 % for 'A', and 2.0 % for 'B' respectively. While samples 'C' and 'D' yielded 80.0 % and 42.3 % in 6:1 at 1.0 % and 2.0 %. It was observed that best yield (80.0 %) of the product was obtained at 1.0 % catalyst concentration (w/w) of the oil in the 6:1 molar ratio of MeOH: Oil (w/w). Increase in the catalyst concentration and MeOH/Oil molar ratio more than 1.0 % and 6:1 respectively resulted in the lower product yield.

In a similar study of *Datura stramonium* seed oil (Koria and Thangaraj 2010) reported that best results were obtained at catalyst (NaOH) concentration of 1.0 % in the MeOH/Oil molar ratio of 7:1 of the weight of the oil. It has been reported that alkaline catalyst concentration greater than 1.5% of oil weight leads to the production of large amount of soap (Vincente *et al.,*1998). The soap can prevent separation of the biodiesel from the glycerin fraction (Demirbas, 2003).

GC-MS Analysis

The GC-MS analysis of the *Datura metel* biodiesel shows that saturated methyl ester containing methyl tetradecanoate of 0.11 % and hexanoic acid. The unsaturated methyl esters in the biodiesel sample included 9, 12-Octadecanoic acid, methyl ester; 9-Hexadecanoic acid, methyl ester; 10, 13-Octadecanoic acid, methyl ester and 7-Octadecanoic acid, methyl ester at percentage composition of 3.18 %, 0.28 %, 3.08 % and 30.21 % respectively (Table 7). This brings together the total FAME content of 87.41 % presents in the biodiesel of *Datura metel* seed oil. Other fatty acids present were non-methyl esters having 12.59 % chemical composition.

The composition of saturated methyl ester (50.66 %) can be traced to the amount of acid value of 12.90mg KOH/g in *Datura metel* biodiesel fuel. The unsaturated methyl ester of 36.75 % obtained from the GC-MS indicates that FAME depends on its number of double bonds as well as polyunsaturated fatty acids when related to the iodine value, which permits it to undergo oxidation than the fatty acids (non-methyl ester) having single bonds. It can be deduced that Thorn

Apple (*Datura metel*) seed oil methyl ester will exhibit good shelf life in terms of storage (Dwivedi and Sharma, (2014).

CONCLUSION

The results from the study reveals that appreciable amount of the oil can be extracted from the seeds of *Datura metel*. Physico-chemical properties of the biodiesel obtained conform to ASTM standard. The biodiesel production from a non-edible oil source, *Datura metel* was investigated and optimization of the production process was carried out to determine the required catalyst concentration and methanol to oil molar ratio at constant temperature and time for maximum yield of biodiesel. The two steps alkaline catalyzed transesterification process was followed the production process. 67.2-80.0 % of the product yield from the *Datura metel* oil using 1.0 % NaOH catalyst concentration at a molar ratio of 6:1 with a reaction time of 120 minutes at a reaction temperature of 60 ^{0}C.

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