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OPTIMISATION OF BIODIESEL PRODUCTION FROM SANDBOX (*HURA CREPITANS*) SEED OIL

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ABSTRACT

Transesterification reaction is the most common method of biodiesel production from fats or vegetable oil. In this research, Sandbox (*Hura crepitans*) seed oil is used as feedstock extracted by solvent extraction using N-hexane as solvent. The study evaluates properties of the oil for it suitability in biodiesel production, biodiesel yields as it is affected by the reacting conditions such as methano-oil molar ratio, reaction temperature, catalyst concentration and reaction time, the reaction conditions are optimized using a two-step four factor factorial design. Results indicate that the feedstock is suitable for biodiesel production, catalyst concentration and reaction times interaction are the important factors that has greater effect on the yield of ethyl ester. The optimum yield condition for Sandbox (*Hura crepitans*) were 0.2wt/wt molar ratio, 30^{o}C temperature, 0.4wt% catalyst concentration and 60 min reaction time yielding 93% ethyl ester (biodiesel), and all the measured properties of Sandbox (*Hura crepitans*) of biodiesel met with ASTM6751 standard exception of high polarization rate and slightly high viscosity that can be normalize by appropriate blending. The result showed that only molar ratio showed a negative effect on the reaction. Hence adhering to the standards for high quality biodiesel and economically cheap production process.

Keywords: *Biodiesel; Process Optimization; Renewable Energy; Sandbox*

1 INTRODUCTION

Hura crepitans seed which is highly rich in non-edible oil which falls into group of underutilized species of plants. *Hura crepitans* commonly known as sand box, possum wood, monkey no climb or dynamite tree is about 25m tall with very spiny trunk and branches and it is commonly planted as shade (Okolie *et al,* 2012), it has gained much attention as a feedstoch for biodiesel production (Srivastava et al., 2018).

In the world today, there is ever increasing demand for petroleum resource, although, high speculations concerning its dwindling supplies, unstable price, non-renewable nature and environmental problems has led to an extensive research on the seek for the alternative resource attributes of the petroleum as the most important source of energy (Nakpong and wootthikanokkhan, 2010).

There are four ways in which vegetable oils and fats can be converted into biodiesel namely; transesterification, blending, micro-emulsions and pyrolysis (Verma and Sharma, 2016; Silitonga et al., 2018). The most common method to produce biodiesel is transesterification of vegetable oils and animal fats in the presence of a catalyst such as acid, alkali or enzyme (Gashaw and Lakachew, 2014; Rodionova et al., 2017). This is due to complications faced in biodiesel production of the presence of Free Fatty Acids (FFAs) in non-edible oils. Adoption of homogeneous base catalyst, results in formation of soaps causing strenuous separation thus decreasing ester yield (Marchetti et al., 2007). Therefore, high FFA feedstock, acid catalyst is preferred to produce biodiesel, but it demands more reaction time and alcohol (Meher *et al.,* 2006). For oils or fats having high FFA acid esterification is advantageous, as acid catalyse the FFA esterification to produce fatty acid methyl ester (FAME). (Verma and Sharma, 2016)

Moreover, Type of alcohol, molar ratio and reaction time play significant role in biodiesel yield and its properties. literature are available on optimisation of process variables of biodiesel production from different oils using Response Surface Methodology (RSM) but little work is reported particularly on Sandbox seed oil and comparison of impact of different alcohols on biodiesel production to optimize the reaction parameters.

Saydut et al., (2016), used RSM to optimise biodiesel production from sunflower oil, Ethanol was used as alcohol for transesterification reaction and highest yield of 97.8% was obtained which was close to predicted yield of 99.2%. Galeano et al., (2017) produced biodiesel from palm oil on ethanolysis using 0.2–1 wt.% NaOH at temperature between 60 and 80 C and ethanol to oil molar ratio 6:1. Highest yield of 96% with 100% conversion of fatty acids into methyl esters was obtained on 1 h of reaction. Avramović et al., (2015) produced fatty acid ethyl esters from sunflower oil with yield of 95% on using heterogeneous catalyst Calcium zincate at 78 C reaction temperature. Yatish et al., 2016 optimised sunflower biodiesel production with RSM technique and achieved yield of 98.6% which was well close to predicted value of 98.9%. Optimum reaction conditions found were temperature in range of 50–59 C ethanol-to-oil molar ratio of 12:1, 0.75% catalyst and reaction time of 15 min. Keera et al., (2018) did ethanolysis of castor and soybean oils and found out yield more than 90% for soybean oil biodiesel and around 30% for castor biodiesel. Avramović et al., (2015) applied RSM to ethanolysis of sunflower oil for production of biodiesel and obtained good relation between predicted and actual values of yield obtained with 93.7% accuracy. applied a response surface methodology to determine the optimum condition for the production of biodiesel production from *Raphanus sativus* using two levels three factor experimental design (2^3 experimental design): the ethanol:oil molar ratio (6:1 and 12:1), the catalyst concentration in relation to oil mass (0.4 and 0.8 wt% NaOH) and the alcoholysis temperature (45 and 65 °C), this process yield 95.8% methyl ester. From the above literature, it is noted that, there has been limited study on the use of non-edible feedstock (Hura crepitans) and optimization of reaction parameters on biodiesel production.

Table 1: A summary of applications of transesterification for biodiesel production

s/no	Method	Material	Conditions	References
1	Methanolysis (Complete Factorial design)	Palm oil	0.2-1wt% NaOH, 60oC - 80oC and 0.1 mole ratio	Narvaez et al, Rubio-Caballero et al, Nie et al, 2006, Thliveros et al., 2014
2	Heterogeneous catalysis (RMS)	Sunflower	50–59 oC and 12:1 Mole ratio	Vujicic et al., 2010, Lee et al., 2014, lee and Wilson, 2015
3	Ethanolysis (RMS)++	castor and soybean oils	-	da Costa Barbosa, 2010, Caldas et al., 2016
4	Alcoholysis (RMS)	*Raphanus sativus*	6:1 and 12:1 mole ratio, 0.4 and 0.8 wt% NaOH and 45 and 65 °C temperature.	Domingos and Saad, 2008
5	Ethanolysis (RSM and ANN)	*Sunflower*	50 and 59 °C, 12:1 mole ratio, catalyst loading of 0.75% and reaction time of 15min	Stamenković et al., 2013

2 Materials and Methods

Sandbox seed were collected from Okene in Kogi state, all chemicals such as KOH, alcohols, Sulphoric acid, N-Hexane methanol and ethanol were of AR grade and 99% pure and were purchased from Tunga market in Minna, Niger state.

2.1 Materials Pretreatment

The raw sand box seed collected is removed from husk casing dried in an electric oven for about 24hr at about 65°C to enhance crushing of the seed in order to reduce the size and provide increased contact between the seed the solvent during the extraction process.

Experimental Setup

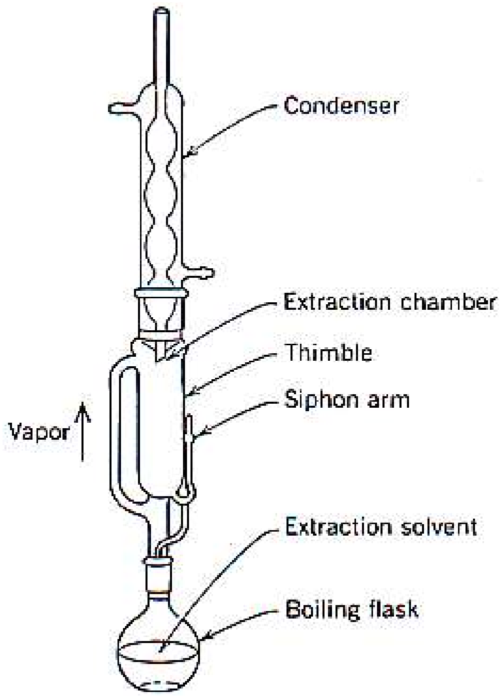


Fig 2.1: Experimental set up for extraction of oil from sandbox seed

The extraction of oil was done using soxhlet extractor, the soxhlet extraction is a set up with the reflux condenser which cools the vapor to convert it to liquid. Weight of the filter paper and steppling pin are taking as (W_1). The samples were then wrapped in filter paper and steppled, the weight of the filter paper, steppling pin and the sample is taking as (W_2). soxhlet extractor uses a method in which the material to be extracted is placed in the extraction chamber which is soaked by the condensed solvent, it dissolves the oil from the sample a for a period of time, the solvent is then removed and replaced with a fresh one, the whole set-up was put on a heating mantle that provides heat which gently heats up the solvent. After each run, the sample was dried for 10 minutes and weighed (W_3). The percentage oil yield is calculated from the formula below;

$$Weight\ of\ extracted\ oil = (w2 - w1) - (w3 - w1) \quad (3.1)$$

Where,

W_1 = Weight of the filter paper and the steppling pin
W_2 = Weight of the sample, filter paper and the steppling pin
W_3 = Weight of sample, filter paper and steppling pin after extraction.

The solvent was recovered from the extract by distillation using the soxhlet extractor. This was done by removing the thimble from the upper of the extractor, then the mixture of the solvent and the extracted oil in the round bottom flask was heated to 30-35°C, at this temperature N-hexane which has the boiling point 40 ^{0}C will evaporate with the aid of water cooling condenser, the solvents were collected in a flask separately from the extracting column, while the oil will be collected in the round bottom flask.

2.2 Analytical Procedure

The oil sample extracted from sandbox seed is analysed for kinetic viscocity, specific gravity, saponification value, free fatty acid content, peroxide value, iodine value and refractive index to ascertain it suitability for biodiesel production. The biodiesel produced is also characterized for it suitability in internal combustion engine, parameters analysed were; viscocity, specific gravity, pour point, flash point, cloud point, acid value cetane number and iodine value.

2.3 Design of Experiment

A factorial design, was employed using four variables to analyse the response patterns and optimise the process variables. The effect of the A (reaction temperature (C)), B (molar ratio of alcohol to oil), C (catalyst concentration (wt.%)) and D (reaction time (min)) at three variable levels in the reaction process is shown in Table 2. A total of 16 experiments were conducted separately for getting the experimental response of yield of sand box oil methyl ester (FAME). The above variables were independent variables selected for optimisation. The experimental matrix is shown in table 3.

Table2: Factorial Design Levels

Variable	Symbl	Levels		
		- α	**0**	**+α**
Mole ratio	A	0.2	0.35	0.5
Temperature	B	30	45	60
Catalyst con.	C	0.25	0.3	0.4
Reaction time	D	30	45	60

Table 3: Design Matrix and Responds

Run No.	Ethanol: oil ratio [wt/wt]	Temperature [^oC]	Catalyst conc [wt%]	Reaction time [s]	Ethyl ester conc [wt%]
1	0.2	30	0.25	30	90.00
2	0.5	30	0.25	30	89.16
3	0.2	45	0.25	30	88.88
4	0.5	45	0.25	30	89.44
5	0.2	30	0.4	30	93.81
6	0.5	30	0.4	30	87.69
7	0.2	45	0.4	30	96.44
8	0.5	45	0.4	30	90.55
9	0.2	30	0.25	60	88.32
10	0.5	30	0.25	60	94.87
11	0.2	45	0.25	60	90.00
12	0.5	45	0.25	60	87.75
13	0.2	30	0.4	60	89.44
14	0.5	30	0.4	60	86.60
15	0.2	45	0.4	60	88.32
16	0.5	45	0.4	60	85.44

The Design Expert 9.0.6.2 software was used for the regression and graphical analysis of the data. The highest value of biodiesel yield was taken as the response of the design experiment for transesterification process. The experimental data obtained by the above procedure were analysed by the factorials design and the general model were developed following the polynomial function equation 1 and 2

$$Y = b_0 + \sum_{i=1}^{n} b_i X_i + \sum_{i=1}^{n} b_{ii} X_{i^2} + \sum_{I=1}^{j-1} \sum_{j=1}^{n} b_{ii}\, X_j X_I \qquad 1$$

$$Y = b_0 + b_1 A + b_2 B + b_3 C + \cdots \qquad 2$$

4.0 Results and Discussion

4.1 Characterization of Sandbox (*Hura crepitans*) seed oil

Sandbox (*Huracrepitans*) seed oil were used as the biodiesel feedstock in this research, the properties such as Kinetic Viscosity, specific gravity, free fatty acid, acid value, peroxide value and saponification of the oil were determined to confirm it suitability for biodiesel production as shown in Table 1. The colour of the oil is golden yellow. The oil yield from the seed is 54.4%, this is although higher than oil yield from coconut seed but less than yield from ground nut seed and palm oil (Kareem et al., 2017) The specific gravity is 0.91 which is slightly less than 0.93 for Jatropher and higher than 0.89 for palm karnel (Musa and Folorusho, 2012; Beccles, 2013). the kinetic viscosity is the measure of resistance of the oil to flow when it is subjected to shear stress (Kareem et al., 2017), the value obtained is 19.69 mm^2/s at 40^oC which is less than 29.8 mm^2/s obtained for coconut and 30.1 mm^2/s for palm karnel (Gillies et al., 2012; Krishnan and Dass, 2012), this indicate that the biodiesel from this oil will have better atomized injection in internal combustion engine. the saponification value of the oil is 207.57 mg KOH/g slightly lower than the value obtained by (Otoikhian et al., 2016) for sesame and Orodu et al., for pineapple peel, while greater than (190 mg KOH/g) for Jatropher, (193.55 mg KOH/g) (Umaru et al., 2016) less than (250 mg KOH/g) for crude palm karnel (David and Julius, 2010) it is determinant of the degree of soap formation by the oil during esterification, this value thus indicate high tendency of soap formation instead of biodiesel formation, this will best be corrected using two step reaction. Free Fatty acid composition of the oil is 12.342 and is higher than value for 1.189 for palm karnel Krishnan and Dass, 2012), which is also in line with other report (Otoikhian et al., 2016). Free Fatty acid are unattached fatty acid in fat and oil unrefined oil contains high percentage of Free Fatty acicd but reduces through refining process, high value of free fatty acid interfere with separation of fatty acid and glycerol during transesterification leading to unsatisfactory conversion to biodiesel. (Beccles, 2013).

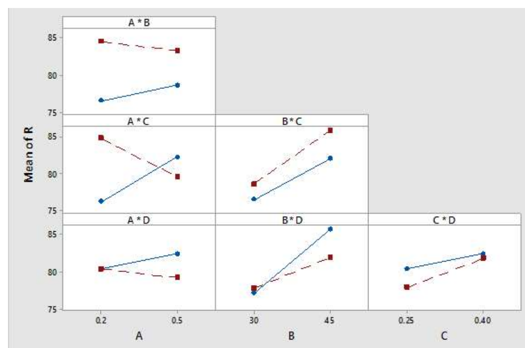
Table 4.: Physico-Chemical Properties of *Hura crepitan* Seed Oil

Properties	Obtained Values
Colour	Golden Yellow
Percentage Yield (%)	54.4
Kinetic Viscosity at 40^{o}C [mm2/s]	19.690
Specific Gravity at 30°C	0.911
Saponification Value (mg KOH/g)	207.57
Free Fatty Acid (as oleic acid; %)	12.342
Peroxide Value (mg Eq/kg)	4.400
Iodine Value (g/100)	163.510
Refractive Index	1.463
Acid Value (mg KOH/g)	24.684

Peroxide value obtained in this research is 4.40 mg Eq/kg, it is slightly greater than (4.073Eq/Kg) and lesser than (6.327±0.006Eq/Kg) for almond nut and frytol respectively (Al-Bachir, 2014). Fresh oils have peroxide values less than 10mEq/kg. The low values of Peroxide value are indicative of low levels of oxidative rancidity of the oils and also suggest high levels of antioxidant, peroxides are possibly not directly responsible for the taste and odour of rancid fats, their concentration is often useful in assessing the extent to which the rancidity has advanced (Atsu Barku *et al.,* 2012), Barku et al., (2012) conclude that a rancid taste often begins to be noticeable when the Peroxide Value is above 20 Eq/kg. Iodine value 163.510 shows high degree of un-saturation of the oil and this categorize it under semi-drying group (iodine value > 100 and <130), which fall within range of iodine value obtained for corn oil (115-130) (Atsu Barku *et al.,* 2012) with sharp difference from (8-10) for coconut and (14-22) for palm krnel (Kareem et al., 2017). The value obtained in this research suggest that the oil is inedible as accumulation of iodine in the body leads to development of goiter, and the oil can be used in production of shoe polish and alkaline resin. (Musa and Folorusho, 2012). Refractive index is 1.463 this value is low compare to other drying oil (1.47) and (1.48) (Abiodun, et al., 2014). It is attributed to the nature of the fatty acids present since refractive index decreases with the molecular weight of the fatty acids. It can also be related to its lower iodine value since refractive index decreases with unsaturation. Acid value defined as the amount (mg) of KOH necessary to neutralize the FFA in 1g of oil or fat sample 24.684 This high value may be due to the moisture contents, refining and deodorization processes. The value obtained is very high compared to palm oil (14.04mgKOH/g), cashew nut oil (0.82mgKOH/g) and rape seed of 7 mg KOH/g stated by Atsu Barku *et al.,* (2012).

4.2 Optimization of Biodiesel Production

Fig 2.11 showing the effect trends and interactions of the factor variables, this plots indicates level of



significance in factor reactions, only factor A*B (mole ratio and reaction temperature) and B*C (reaction temperature and catalyst concentration) show insignificant level of interaction. While other interactions are significant. This however provides basis for model assumption.

Fig2.11: Effect plot of interaction between factor R= yield of the (wt%), A =Methanol oil ratio, B is Temperature (ºC), C is Catalyst Concentration (wt%), and D is Reaction time(Sec)

Table 4.2: analysis of the variance table

S/N	Factor	Factor Effects	Coef	Sum of square	Df	Mean Square	F –Value	P-value (%)
	Constant		80.7					
1	A	-3.1	-1.5	31.9	1	31.9	13.8	10.3
2	B	1.8	0.6	6.8	1	6.8	2.9	2.2
3	C	5.6	2.8	108.16	1	108.1	46.6	34.7
4	D	6.6	3.3	160.0	1	160.0	68.9	51.3

5	AB	1.1	0.5	2.56	1	2.6	1.09	0.80
6	AC	-1.5	-0.8	6.76	1	6.8	2.9	2.2
7	BC	-0.4	-0.2	1.82	1	1.8	0.8	0.6
8	BD	0.7	0.5	1.96	1	1.8	0.8	0.6
9	CD	2.8	1.4	31.3	1	2.0	31.1	10.1
10	ABC	-0.4	-0.2	1.8	1	31.6	0.8	0.6
11	ABD	1.9	0.9	11.6	1	11.6	4.8	3.7
12	ACD	-1.5	-0.8	6.9	1	6.9	2.9	2.2
13	BCD	0.9	0.5	5.5	1	5.5	2.3	1.8
14	ABCD	1.9	0.9	10.6	1	10.6	4.4	3.2

The experimental data in table 4.1 were analyzed with Mini-tab statistic software on all four process variables in which catalyst concentration and reaction time show most significant effect as shown in Figure 4.0 and their interactions. The experimental result was then fit to regression model equation for predicting the yield. Where Y is the respond, A, B, C and D are coded independent variable.

The predicting model equation

Y is the respond, and the coded variables are A = 0.2-0.5 (wt/wt), B=30-45 (oC), C=0.25-0.4 (wt%) and D= 30-60 (min). the regression model for the response (Yield) is govern by the equation:

$$Y = 80.75 - 1.54A + 0.53B + 2.73C + 3.29D + 0.53AB - 0.78AC - 0.46BC + 0.23BD + 1.53CD - 0.21ABC + 0.98ABD - 0.73ACD + 0.46BCD + 0.96ABCD$$

(R^2 = 0.785) (R^2_{adj} = 0.74)

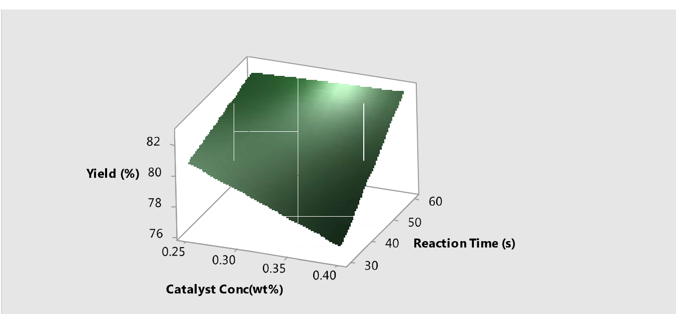


Figure 4.0: Influence of Catalyst concentration and reaction time on ethyl ester yield

This factor exhibits great positive effect on yield of biodiesel, the methyl ester yield increases with increasing time of reaction, this is possibly due to increased mixing of the ethanol in oil with time which is in accordance with (David *et al.*, 2010). Increased molar concentration inhibits the formation of more ethyl ester as indicated in Figure 4.0. But on the other hand, unnecessary prolong of the reaction time leads to the hydrolysis of ester resulting on more formation of free fatty acid.

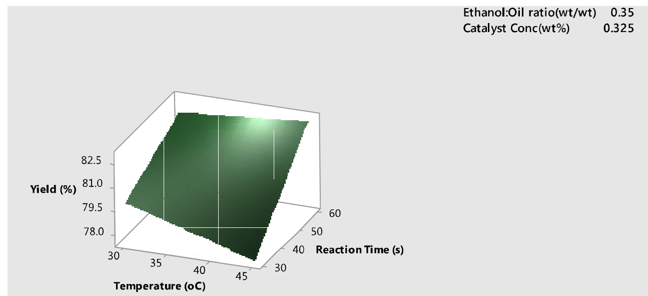


Figure 4.2: Influence of temperature and reaction time on Ethyl ester yield

Temperature has a very slight positive effect or influence on yield of biodiesel, making it possible to operate at low (room) temperature 30^{o}C this is because increasing temperature leads to decrease in ethyl ester yield which is attributed to the formation triglyceride saponification and decomposition of unsaturated methyl esters and unreacted triglycerides in methanol at high temperature.

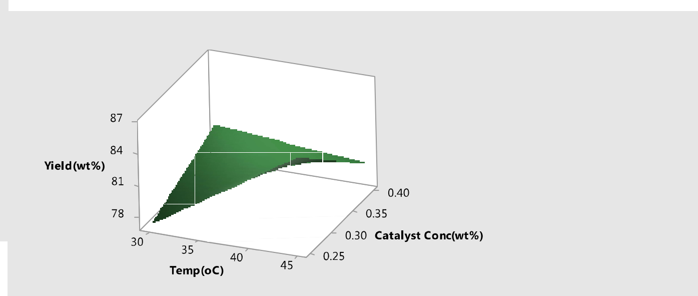


Figure 4.3: Influence of catalyst concentration and reaction time on the ethyl ester yield

The methanol to oil ratios show negative effect, this is obviously an indication of a decrease molar yield with increase molar ratio. This is due to the restriction of the reaction equilibrium and difficulties in separating excessive methanol from methyl esters and glycerol as such

0.2mole with yield 93% is the optimum molar concentration for the production of the biodiesel.

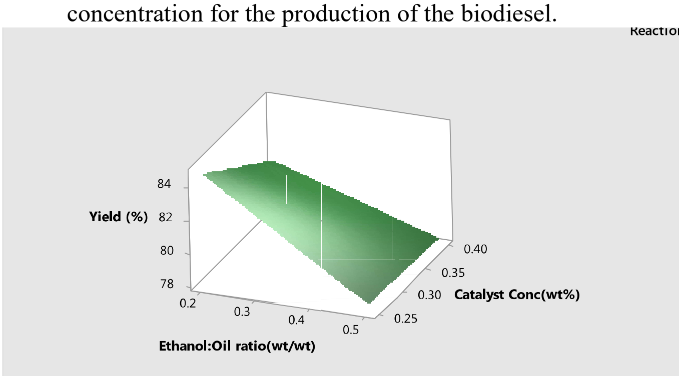


Figure 4.4: Influence of Molar ratio and catalyst concentration on ethyl ester yield

The molar ratio has a significant negative effect on the yield of biodiesel, increase concentration of potassium hydroxide other hand does not significantly affect yield of the biodiesel, though slightly increases the amount of free fatty acid produced through saponification.

4.4 Effect of Interactions

Catalyst and reaction time has significant positive effect on the biodiesel yield responses, this is due to action of catalyst in increasing the polarity of the feedstock with increasing time making the reaction mixture more susceptible to reaction producing more of yield of the biodiesel.

4.5 Biodiesel Characterization

The biodiesel were also characterized for properties such as specific gravity 0.93 which is though less than the ASTM D6751 standard which indicates the degree of impurity in the biodiesel such as water content and fatty acid composition, Viscosity at 40°C is 6.67mm/s^2 which are less than that of the oil by one sixth due to alkaline methanolysis that reduces the viscosity, the viscosity is slightly greater than ASTM D6751 biodiesel standard, which signifies there will formation of deposits in the internal combustion engine, clogging of the injector nozzle and production of dark exhaust gas, an heterogeneous alkaline catalysis will enhance a good viscosity (Nakpong and Wootthikanokkhan, 2010), though the value obtained compares relatively with (4.45mm^2) for palm oil and (4.05mm^2) for ground nut oil (Abiodun, et al., 2014). The flash point is the minimum temperature at which a liquid gives off enough vapour that is sufficient to form an ignitable mixture with air near its surface, the obtained in this research is 185°C which is in the range of ASTM D6751 standard, and is greater than (130°C) for Coconut and (174°C) for Palm oil (Abiodun, et al., 2014), The flash point of pure biodiesels is considerably higher than the prescribed limits, but can decrease rapidly with increasing amount of residual alcohol. As these two aspects are strictly correlated, the flash point can be used as an indicator of the presence of methanol in the biodiesel. (Atsu Barku et al., 2012). Cloud point is particularly important as it relates to the length and degree of saturation of the fatty acid components of the oil, the temperature at which clouds of wax crystals first appear in the liquid when cooled. -5°C is obtained for conformity with the ASTM6751 standard, the parameter estimates the degree of saturation of the biodiesel. Minimum operating temperature in internal combustion engine of the biodiesel is determined by pour point which is -16°C for this research which is in conforming with theASTM6751, insusceptibility to gel or crystallization, the problem of high pour point can be addressed by blending of the biodiesel (Saydut *et al.*, 2016). Acid value is 23.964 mgKOH/g, this value is greater than ASTM6751 standard, and is greater than value reported by (Silitonga *et al.*, 2018) this indicate high polymerization rate with the degree of unsaturation of the fatty acids. Cetane number is 51.251 which is slightly higher than (50 max) for conventional diesel and within the range of the ASTM6751 biodiesel standard, since it is the measure of a fuel's willingness to ignite when it is compressed. It rates the ignition potentials of a diesel fuel, just as octane number determines the quality of gasoline (petrol). The higher the Cetane number, the more efficient is the fuel biodiesel has a higher Cetane number than petroleum diesel because of the presence of oxygen molecules (Opra and Obot, 2009), hence this biodiesel has higher ignition potential than conventional diesel. The iodine value measures the degree of unsaturation and tendency of biodiesel to polymerize, high iodine value means high degree of unsaturation, value obtained is 141.00 g/100 which is slightly greater than (121.19±0.01 g/100) for fryol (Atsu Barku et al., 2012). Biodiesel with high IV tends to polymerize and form deposits on injector nozzles, piston rings and piston ring grooves. The tendency of polymerization increases with the degree of unsaturation of the fatty acids.

5.0 CONCLUSION

Sandbox (Hura crepitans) seed oil have a high value of saponification value, and relatively low acid value, density and viscosity which are characteristics that determine suitability of the oil for biodiesel production.

- Catalyst concentration and reaction time show highest percentage contribution effect on the transeterification process and subsequently on ethyl ester yield
- Increasing reaction time and catalyst concentration increases the ethyl ester yield with decreasing molar ratio and temperature.

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