OPTIMIZATION OF BIODIESEL PRODUCTION FROM *DANIELLA OLIVERI* OIL SEED USING WASTE SNAIL SHELL AS HETEROGENEOUS CATALYST

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Abstract: *Biodiesel has gained support and recognition as a fuel to replace fossil fuel which has cause a lot of damage to the environment. In search of locally cheap raw materials that could be used for biodiesel production at a cheaper rate. An investigation was carried out with Daniela oliveri oil seed and waste snail shells as raw materials. One step alkaline transesterification was conducted to produce the biodiesel. Snail shell was used a source of CaO heterogeneous catalysts by calcination process at 9000C for 5 h. Four process parameters was optimized; methanol to oil ratio 7:1, reaction temperature 500C, catalyst concentration 2.0 wt% and reaction time 60 min to obtained high yield of biodiesel 77% from the oil. The fuel properties of the produced biodiesel from D.oliveri oil were compared with ASTM standard and found within the requirements.*

Keywords: *Biodiesel, Daniella oliveri seed, Fuel properties, Optimization, Snail shell, Transesterification*

1. INTRODUCTION

Biodiesel is a bio-based diesel fuel that has received worldwide acceptance as a substitute and also a blending agent to fossil fuel. It is a mono-alky ester that is mainly produced through transesterification of oil obtained from vegetable or animal fat (Ani and Bakheit, 2014).

There are two major problems that must be considered when selecting biodiesel as a substitute to fossil fuel; production process and cost of feed stock. These two issues have made the researchers shifted to underutilized oil seeds prevalent in North Central Nigeria for their potential biodiesel feedstock (Ndana *et al.,* 2011).

Over time edible oil such as sesame, melon, and groundnut were used but this has resulted to food crisis causing the research on biodiesel production to shift to underutilized oil seed.

These seeds are produced in large quantities annually from this region of the country and it is aim to use it to produced biodiesel (Rashid *et al.,* 2011). The oil of this plant was reported to contain both saturated and unsaturated fatty acid in appreciable quantity (Ikwuagwu *et al.,* 2013), which makes it better for the production of biodiesel. The oil contain low fatty acid which is transesterified by single step alkaline transesterification.

The use of nonconventional heterogeneous catalysts for transesterification from waste snail shells, sources has become imperative and popular as one of the alternative to reduce the production cost (Nakano *et al.,* 2010). The snail shells are wastes generated from household, restaurant, eatery and kitchen. The major advantages of using heterogeneous catalyst over homogeneous catalyst (KOH and NaOH) is it reusability that can reach 18 times, renewable, reduce the amount of water used, cost effective and resistance to corrosion of the metallic part of the engine (Chen *et al.,* 2012).

Heterogeneous catalysts prepared from mollusk shell such as periwinkle, crab, oyster and mussel are found to contain high percentage of CaO (Buasri, 2013).

The aim of this study is to optimize biodiesel production from oil derived from *Daniella oliveri* seed using waste snail shell as heterogeneous catalyst. The physicochemical properties and process parameters that influenced the yield of the transesterification of *D. oliveri* oil seed was also investigated.

2. MATERIALS AND METHODS

2.1 Collection of sample

Feed stock which are non-conventional such like *D. oliveri* was used in this research, This plant was sample was source from Gaba in Niger state, North Central Nigeria which are yet to exploit in other to know there benefit as material used in the production of biodiesel. This plant seeds were collected from different farm in Gaba. The description of this plant is given below:

Table 1: Botanical Description of *D. oliveri*

Common name	Local name		Botanical name	Family
Ilorin Balsam	English:	Ilorin balsam	*Danielia oliveri*	Fabales
	Yoruba:	Emi ya		
	Hausa:	Maje		
	Igbo:	Ofo		
	Nupe:	Danchi		

2.2 Pretreatment of *D. oliveri* Seeds

The seeds was unshelled, clean and dried at room temperature for a period of five days. The dried seeds were then pulverized into uniform powdered, sieved, weighed, bottle, labeled which was stored using plastic container (air-tight), this was used in order to ensure that prepared sample is free from moisture content that will interact with it.

2.3 Extraction of *D. oliveri* oil

50 g of powdered sample was used for extraction using n-hexane through soxhlet apparatus on a water bath for 2 h and this was repeated three times in order to obtain sufficient amount of oil to be used for this purpose. The solvent was dried using rotary evaporator, where percentage oil yield was calculated by means of this formula:

$$\text{Oil content} = \frac{\text{weight of oil extracted}}{\text{weight of seed}} \text{ x} \frac{100}{1}$$

2.4 Refining of Extracted *D. oliveri* Oil

Degumming

Degumming was done to remove phospholipids from the oil. This was done by the addition of 50 cm^3 boiled water to 100 cm^3 of the extracted oil. The mixture was stirred for 5 min and allow to stand in the separating funnel.

Thereafter, the aqueous layer was then removed. The procedure was repeated three times to ensure complete removal of most gums (AOAC, 2006).

Neutralization

This was done to remove the free fatty acid. 100 cm^3 of the degummed oil was poured into a beaker and heated to 1000 ^{0}C, after which 130 cm^3 oil of 0.1 M NaOH was added and stirred to obtain a uniform solution. 20 g of sodium chloride was added to salt out the soap formation. This was further transferred into a separating funnel and allowed to stand for 1 h. The soap formed was separated from the oil. 50 cm^3 of hot water was added and used to wash the oil solution several times until the soap remaining in the solution is removed. The neutralized oil was then drawn off into a beaker and heated in a heating mantle at 1000 ^{0}C for 30 min to remove the water completely from oil (Oderinde, 2009). The neutralized oil was characterized for its physicochemical properties.

2.5 Physicochemical Properties of *D. oliveri* Oil

All the test done for physicochemical chemical properties of *D. oliveri* were according (AOAC, 2006). The tests performed were iodine value, pH, free fatty acid, saponification value, peroxide value, acid value, odour, specific gravity, ash content, moisture content and colour. The test was tabulated in Table 2.0.

Table 2: Physicochemical Properties of *D. oliveri* Oil

Properties	Values
Yield (%)	28.57±0.31
Specific gravity	0.821±0.01
Moisture content (%)	1.67±0.05
Refractive index (40^{o}C)	1.34±0.03
Ph	4.10±0.10
Colour	Yellow
Viscosity (mm^2/s) at 25^{o}C	24.73±0.15
Ash content	1.25±0.02
Iodine value (g/100g)	134.5±0.21
Peroxide value (meq/kg)	8.57±0.15
Acid value (mgKOH/g)	4.8±0.03
Saponification (mgKOH/1g)	90.23±0.2
FFA	2.4

Values are mean±SD of duplicate determinations

2.6 Preparation and Characterization of Catalyst

The waste snail shells were collected from different restaurants in Bida. The collected shells were washed and dried in a hot air oven at 105 ^{0}C for 24 h. The dried shells was pulverized in a mortar until they become powered.

The powdered snail shells was clalcined in an electric furnace at different temperature 500, 600, 700, 800, 900 and 1000 ^{0}C for 6 h. The weight change after calcinations process was noted. The percentage weight loss was calculated according to the equation;

$$\%\ \text{Weight Loss} = \frac{W_1 - W_f}{W_1} \text{ x } 100\%$$

Where W_1 = initial weight

W_f = final weight

% yield = 100 - %weight loss

The powdered calcined snail shells was then sieved with a mesh size of 107 – 102 µm and kept in a desiccator to prevent moisture. The powdered calcined shell containing calcium oxide (CaO) was characterized for thermo gravimetric analysis (TGA) to determine the calcinations temperature and time. The elemental composition of the calcined powdered shells was determine using x-ray fluorescence (XRF), the chemical composition was analyzed using x-ray diffraction (XRD). The morphology was analyzed using scanning electron microscopy (SEM), while the crystallinity is determined using Brunauer Emnet Teller (BET) analysis.

2.7 Transesterification by Heterogeneous Catalysts from Snail Shells

The oil extracted from *Daniella oliveri* seed was transesterified by one step alkaline transesterification process because the oil has low fatty acid content less than 3%. 100 g of the *Daniella* oil seeds was heated to 55 ^{0}C and 150 cm^3 of methanol followed by addition of 3 w% of CaO prepared from snail shells. The reaction of the sample oil was carried out in a stirring speed of 300 – 400 rpm under reflux condition. The mixture was stirred at all-time throughout the transesterification process.

The reaction temperature was carried out through the range of 50 – 65 ^{0}C using reaction time 40 – 60 min. After the reaction time, the catalyst was filtered using separation funnel and filtrate was kept overnight for proper separation of methyl ester as well as triglyceride to occur.

After separation the lower layer which is triglyceride (glycerol) is drawn out of separating funnel and the methyl ester was the product obtained which is biodiesel. The biodiesel yield is calculated as;

$$\text{Yield (\%)} = \frac{\text{Raw } D.oliveri \text{ oil seed (g)}}{\text{Biodiesel (ester) weight (g)}} \text{ x} \frac{100}{1}$$

3. RESULT AND DISCUSSION

The result of the physicochemical properties of the oil seeds of *D. oliveri* is presented in Table 2.0. The percent oil yield is 31.5% through soxhlet extractors using n-hexane. The range of (25 – 55%) as reported by Ajayi and Oderinde (2009) for Piloswillgma thonmingi.

This results indicate that the oil seed of *D. oliveri* contain significant amount of oil which will be used in production of biodiesel for commercial purposes. The FFA value of the *D. oliveri* oil is (2.4 mgKOH/g). The oil has low fatty acid which is less than 3%. The low fatty acid content is as a result of refining process (Okullo *et al.,* 2011). The oil with high free fatty acid 3% need to be esterified before transesterificaiton. One step alkaline trans-esterification is used in this study because of low fatty acid content (Akintayo, 2014).

The acid value is a value in milligrams of potassium hydroxide per gram (mg KOH/g) required to neutralize the free fatty acids (Bamgboye and Hausan, 2008). The acid value of *D. oliveri* oil is (4.8 mgKOH/g). This value is within the standard for biodiesel of (2.5 – 5.0 mgKOH/g) (Belewu *et al.,* 2014).

The moisture content obtained for *D. oliveri* oil is (1.67 $\pm$ 0.05). This value is significantly higher than that of cotton seed oil (1.2) but lower than those reported by Belewu *et al.,* (2014) for neem oil (4%), castor oil (8%), rubber seed oil (8.6%) and shea butter oil (10%) and also comparable to 9.4% low pea and 7.8% green pea reported by (Wang *et al.,* 2011). High moisture content in a seed reduces it quality and the oil yields.

The peroxide value of the oil is 8.57 $\pm$ me_q/g. This value is lower than neem oil peroxide value of 9.5 meq/g reported by (Anigo *et al.,* 2013). Low level of peroxide value is an indication of oxidative stability and indicator of lipid oxidation (Kyari, 2015).

The refractive index of *D. oliveri* oil is 1.34 $\pm$ 0.03 from this result the value are in consistent with that of *Jatropha* oil is 2.45$\pm$ 0.05 reported by (Olagunju and Olajumoke, 2013) for the biodiesel production. The refractive index of oil increases with increases in unsaturation of its component fatty acids (Alamu *et al.,* 2008).

The pH value of the oil as presented in Table 2.0 is 4.10 $\pm$ 0.10. This value is within the range according to Codex, (2011) for biodiesel production. The pH for this oil is less than seven is as a result of fatty acid content.

The colour of the extracted *D. oliveri* oil is yellow. This colour is acceptable for biodiesel production according to Codex, (2011). Singh and Singh, (2010) reported brown colour for Jatropha oil for the product of biodiesel, Sanjay (2013) reported brown yellow colour for neem oil for the production of biodiesel which are all accepted by Codex Standard for biodiesel production.

The ash content in *D. oliveri* oil is presented in Table 2, which shows that oil has low ash content of 1.25 $\pm$ 0.02. The low value is in an indication that the mineral contents are low (Eze, 2012).

The iodine value for *D. oliveri* oil is (134 $\pm$ 0.2). Oils with iodine values less than (100I_2/100g) of oil are non-drying oils. Aremu *et al.,* (2015) reported that oil with low iodine value would have low number of unsaturated bond.

Specific gravity, the density of oil is usually express as specific gravity and measured at specific temperature 20^{0}C (Muthu *et al.*, 2010). From this study the specific gravity of *D. oliveri* oil is reported as (0.82 $\pm$ 0.02). This values is in agreement with values of (0.83g/cm^3) cotton reported by (Yahaya *et al.*, 2016).

Characterization of the Snail Shells

The amount of weight loss during the calcination of the snail shells is presented in Table 3.0. The shells is calcined at different temperature of (500^{0}C to 900^{0}C for 6 h). The carbon and oxygen content in the shells were lost after calcination under high temperature and time. More than 30% weight loss were recorded after these temperature and time. The yield at these temperature and time was (60.0%) (Llgen, 2011).

The crystalline structure of the shell was changed after calcination. The change is as a result of decomposition of the calcium carbonate (CaCO3) present in the shell to give carbon IV oxide (CO2) and calcium oxide (CaO) (Viriya et al., 2010). As the temperature increases over the time, the yield also increase. The determination of chemical composition of the catalyst from the snail shells was performed using X-ray fluorescence (XRF) at temperature different of ranging from (500 – 9000C) and results are shown in Table 4.0.

Table 3: Snail Shell Weight Loss after Calcinations

Time(hr)	Temp. (^{o}C)	Wt loss (%)	Yield (%)
1	500	45.25	54.75
2	600	42.78	57.22
3	700	40.25	59.75
4	800	40.15	59.85
5	900	40.00	60.00
6	1000	40.30	60.00

CaO was derived from calcined snail shell. The CaO was derived from the calcined snail shell on a dry basis by XRF. It has shown that, the content of ash from snail shell mainly CaO (96.72%), considered a favorable base catalyst in biodiesel production with high basic strength, minor toxicity and easy reactions with water (Zhang *et al.*, 2010). The remaining (3.28%) was composed of different metal oxides (MgO, SiO, P_2O_5, Na_2O, Al_2O_3, K_2O, FeO, Cr_2O_3, ZnO and SrO) in trace amounts. The acidic components (P_2O_5, SiO_2 and SO_3) potential to mediate esterification of the oil free fatty acid content.

Table 4: Chemical Composition of Snail Shells with XRF

Compounds	CaO	MgO	SiO	P_2O_5	Na_2O	Al_2O_3	Cr_2O_3	TiO_2	SrO
%n concentration	96.72	0.038	1.42	0.123	0.012	0.32	0.004	0.001	0.41

The rebuilt of XRD of CaO derived from snail shell calcinated at 900^0C for 5h is shown in Figure 1.0. It is seen from the figure that CaO was formed on calcnation at 700, 800 and 900^0C.

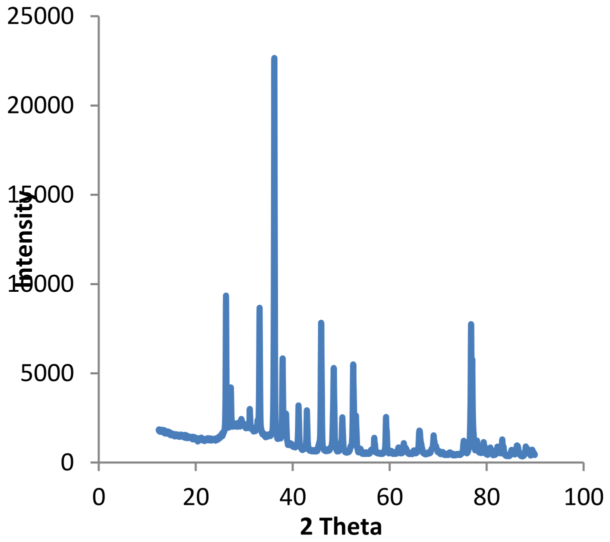

Figure 1: The XRD of Calcined Snail Shell

This is an indication of the presence of diffraction peaks 2θ at 280, 350, 380, 450, 500 and 750. The CaO diffraction pattern is in accordance with standard of JCPDS. Catalyst calcined at 6000C to 8000C for 5h contained lime (CaCO3) at 2θ of 380 and 450. This shows that the decomposition of CaCO3 to CaO on calcination of snail shell at 7000C is not completed (Utami and Wurhajati, 2013). It is clearly observed by the highest peak that the major component of the calcined shell at 9000C at 5h was CaO compounds. The SEM image of a catalyst (CaO) has reported in Figure 2.0. The morphology of CaO with irregular shapes, size as well as pores, differs from one surface to another (3 – 6 µm of width). The profile of the calcined shell was formed through tiny crystals entrenched on the large particles, which may be as a result of heterogeneous distribution in the mechanical properties of the snail shell used that can be regarded as an attribute of high catalytic activity (Tang *et al.*, 2013).

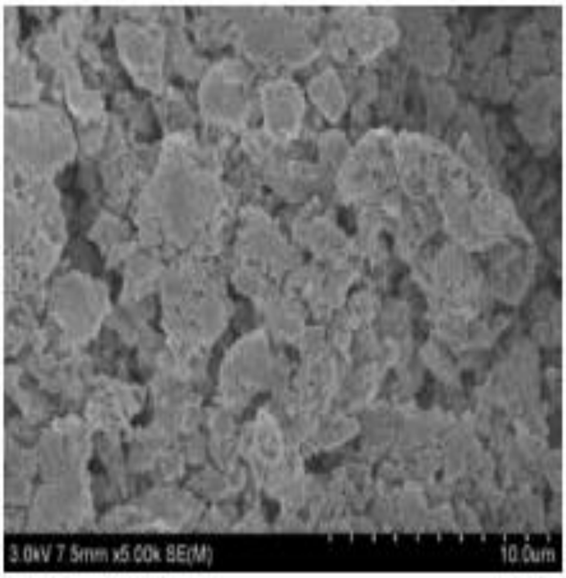

Figure 2: SEM Studies of Calcined Snail Shell

The prepared catalyst was characterized for (BET) analysis; the result is presented in Table 5.0. The pore size for the calcined shell is (98.05 A) with high surface area of (21.15 m^2/g) which allow reactants to diffuse easily into the interior of the catalyst. (Siddharth and Sharma, 2011) reported that a high pore size is desirable for better diffusion of reactant and product molecules. The observation is in agreement with that of (Birla *et al.*, 2012) with a slightly higher value of (99.18 A) pore size of CaO catalyst.

Table 5: Brunauer Emmett and Teller Analysis of Calcined SSC

Properties	Calcined
Pore size (A)	98.50
Pore volume (cm^3/g)	17.50
Specific surface area (m^2/g)	25.15

Optimization of process parameter for transesterification reaction

In this study, four process parameters that influence transesterification reaction namely methanol to oil ratio, temperature, catalyst and reaction time were determine for optional biodiesel production.

Effect of methanol to oil molar ratio Figure 3 Presented the effect of methanol to oil molar ratio. Optimum biodiesel was observed at methanol to oil ratio of 7:1 and yield of 75%. An increase in mole ratio beyond the optimum mole ratio of 7:1 and yield of 75%. An increase in mole ratio of 7:1 had a negative effect on the yield. The yield decreased above this optimum point. Khemthong *et al.* (2012) showed that the most suitable molar ratio was found to be within the range of 6:1 to 10:1, in the production of biodiesel using egg shell ash (CaO) as heterogeneous catalyst.

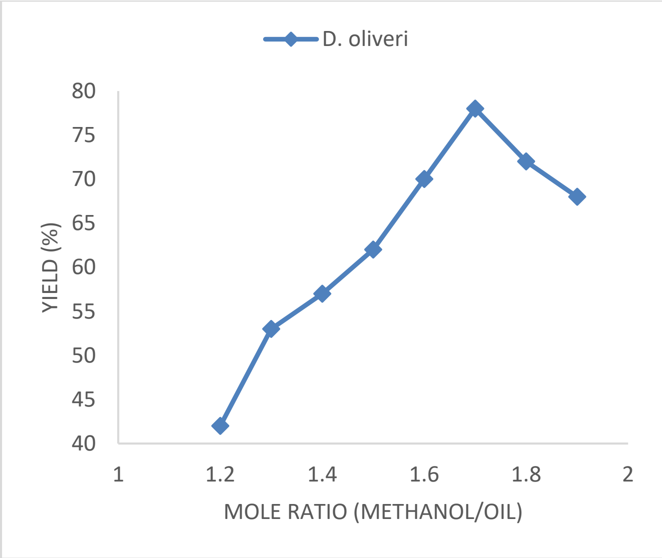

Figure 3: Effect of methanol to oil mole ratio on the biodiesel yield. Reaction condition: catalyst concentration 2.0 wt%, reaction time 60 min, reaction temperature 50^{0}C

Effect of Temperature

The effect of temperature on the biodiesel conversion yield are illustrated in Figure 4. The optimum temperature was observe to be 50^{0}C with a yield of 77%.

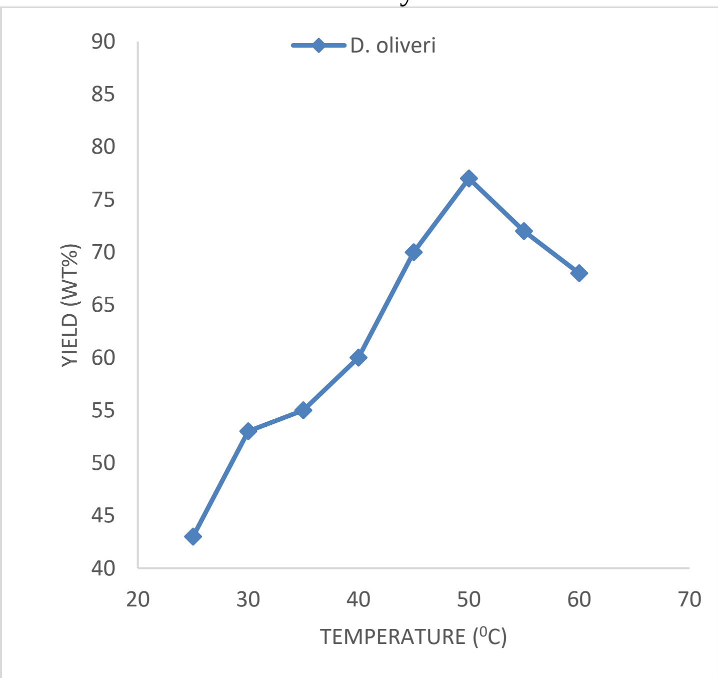

Figure 4: Effect of reaction temperature on the biodiesel yield. Reaction condition: catalyst concentration 2.0 wt%, reaction time 60 min, mole ration (7:1)

In this study when the reaction time temperature increases above the optimum value, there was also an increase in the reaction rate due to high energy input and reduced mass transfer (Tariq et al., 2014).If the temperature is above 600C resulted in decreased in the yield due to the fact that reaction occurring beyond the methanol boiling point 650C and this will result to vaporization (Birla et al., 2012).

Effect of catalyst concentration.

As presented in figure 5, 2.0 wt% of catalyst produced 75% of biodiesel yield of *D. oliveri* oil. This result is in line with Lee *et al.* (2009) reported, that heterogeneous catalysts for the production of biodiesel should range from 0.4 – 2.3 wt% for high yield. Beyond this range a decrease in biodiesel yield was observed for *D. oliveri.*

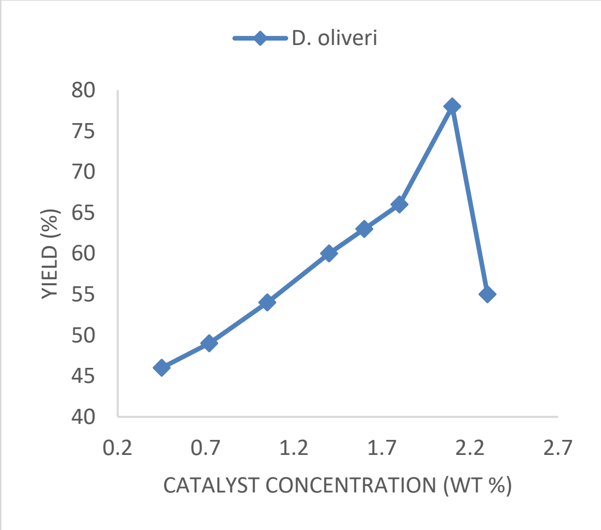

Figure 5: Effect of catalyst concentration on the biodiesel yield. Reaction condition: temperature 50^{0}C, reaction time 60 min, mole ration (7:1)

Effect of Reaction Time

Figure 6 reflects optimum transesterification tome for *D.oliveri* oil. The maximum biodiesel yield was obtained at 60 min. the yield was found to be 76%. At first few minutes the reaction time was slow due to dispersion of interaction between molecules is still in progress (Birla *et al.*, 2012). As the reaction time increases, the yield also increased due to high effective collision occurring beyond the reaction time of 60 min. There is decreased in the yield of the feedstock.

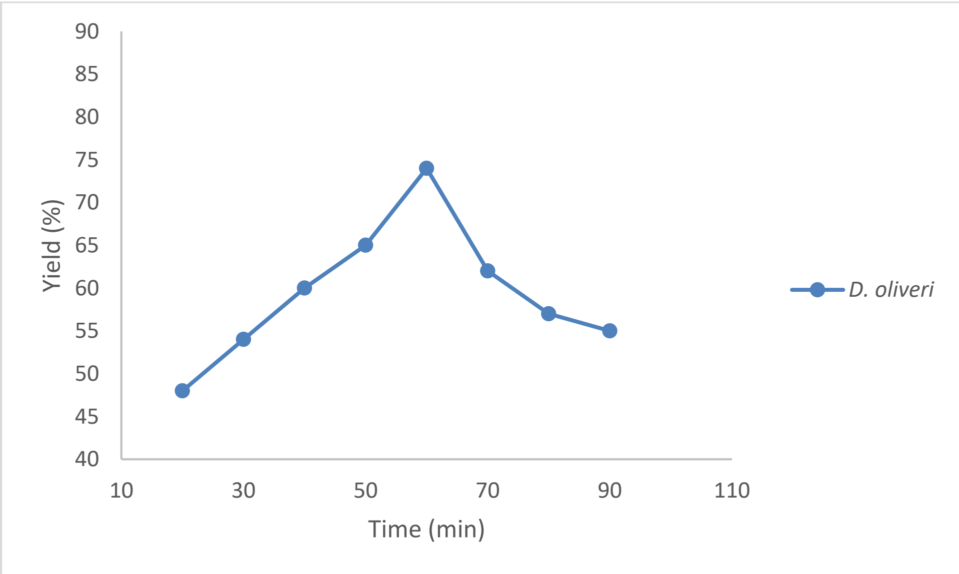

Figure 6: Effect of reaction time on the biodiesel yield. Reaction condition: temperature 50^{0}C, catalyst concentration 2.0 wt%, mole ration (7:1)

Fuel properties of the produce biodiesel

Some of the important fuel properties of the biodiesel produced from *D. oliveri* oil seed are compared with the biodiesel standard (ASTM 6751) and are presented in table 6.

Table 6: Comparison of biodiesel properties with ASTM standard

Property	Biodiesel (DOO)	ASTM 6751	Diesel
Ash content (wt %)	0.03	0.05	-
Flash point (^{0}C)	162	100 - 170	70 – 100
Acid value (mgKOH/g)	0.04	0.05	0.02 – 0.05
Pour point (^{0}C)	-7.0	-10	-
Viscosity (mm^2/s)	4.10	1.9 – 6.0	5.0

4. CONCLUSION

Biodiesel production from one step alkaline transesterification of *D. oliveri* oil seed using waste snail shells is investigated in this study. The waste shells contains $MgCO_3$, $CaCO_3$, Al_2O_3, K_2O and Na_2O in different proportion. The most predominant is $CaCO_3$ which is calcined at 900^{0}C for 5 h and changed into CaO. The CaO was characterize in order to determine it catalytic properties, if suitable for biodiesel production.

The extracted oil from *D. oliveri* seed was transesterified with CaO prepared from the snail shell as heterogeneous catalyst. The optimum biodiesel yield was found as high as 77% with four optimize parameters playing active roles; methanol to oil ratio (7:1), temperature 50^{0}C, catalyst concentration (2.0 wt %) and reaction time 60 min to achieved optimum biodiesel yield. The produced biodiesel properties was compared with the standard and some of the properties are in line with ASTM standard for biodiesel. The waste snail shell had high potential as cost effective heterogeneous catalysts than the convectional (KOH and NaOH) for biodiesel production. *D oliveri* oil seed is a promising feedstock for biodiesel production.

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