IN-SITU TRANSESTERIFICATION OF *Datura metel* SEED OIL USING CaO DERIVED FROM SNAIL SHELL AS A CATALYST FOR BIODIESEL PRODUCTION

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Abstract: Biodiesel is considered as one of the alternative environmental benign fuel. It production encountered many problems such as low energy balance and food verses fuels competition. To resolve some of these problems, in-situ transesterification of *Datura metel* seed oil was carried out with 1:1 methanol to n-hexane co-solvent and calcium oxide derived from snail shell as catalyst at 65 ℃ temperature for 3 hours in soxhlet extraction apparatus. This research work produce biodiesel yield of 91% with higher heating value close to petro-diesel and cetane number higher than the minimum standard set by ASTM. The iodine value which is lower than the upper limit set by EN 14214 specification. The density of *Datura metel* seed oil biodiesel is within ASTM Specification but it record high acid value. GC-MS results revealed that methy-11-octadecenoate, methyl hexadecanoate and methyl octadecanoate are major fatty acid esters of *Datura metel* seed oil biodiesel. This research revealed that *Datura metel* seed oil are potential non edible feedstock for biodiesel production.

Keywords: *Datura metel* seed oils, Biodiesel, X-ray fluorescence, GC–MS

Introduction

Increase in world fuel consumption, rising in fuel price (Luu *et al.*, 2014), depletion of fossil fuels(Ahmed *et al.*, 2014) and global warming problems encouraged the search for alternative renewable sources of energy (Marinković *et al.*, 2016). Fuels production will be considered as environmentally benign and economically sustainable, if obtained from renewable resources with higher energy balance. Biodiesel considered as a green fuel (Li *et al.*, 2014), due to its properties such as renewability, sustainability and environmental friendly. It is use in diesel engine without modification and could considerably overcome the forecasted future and current energy crisis (Ahmed *et al.*, 2014).Therefore, development of biodiesel as an alternative fuel have received considerable attention (Rabiah-Nizah *et al.*, 2014; Andréia *et al., 2015*).More than 80% cost of biodiesel production are due to high cost of feedstock (Sani *et al.*, 2014, Šánek *et al.*, 2016). Large proportion of biodiesels was obtained from edible vegetable oils (Shajaratun *et al.*, 2014, Firdaus *et al.*, 2016). Hence, the use of edible vegetable oil as biodiesel feedstock might immeasurably contributed to the high biodiesel price (Lee *et al.*, 2013; Teo *et al.*, 2014). Moreover it might led to some negative impact such as starvation and food inflation in developing countries (Yunus khan *et al.*, 2014). Many researchers agreed that non-edible oils are the most appropriate alternative feedstock for biodiesel production (Sahoo and Das, 2009; Abdul Khalil *et al.*, 2013 Krishnakumar *et al.*, 2013; Tarabet *et al.*, 2014; Yunus khan *et al.*, 2014; Ahmad *et al.*, 2014; Khiari *et al.*, 2016; Jung *et al., 2016;*Onoji *et al.*, 2016). Therefore, search for alternative feedstock like *Datura metel* seed oil is very important in order to reduce the cost of biodiesel production. Some of the important factors to be considered in the selection of biodiesel feedstock are its chemical composition; cost, availability and possible pretreatment required (Chien-Chih and Tsair-Wang, 2013).

The name Datura was derived from Hindi words Dhatura (thorn-apple). Datura species are herbaceous plant, perennials which is up to 2 m in height. The leaves are alternate, 10-20 cm long and 5-18 cm broad, with a lobed or toothed margin. It flowers are erect or spreading, trumpet shaped, 5-20 cm long and 4-12 cm broad at mouth; colors vary from white to yellow, pink, and pale purple. Most parts of the plants are toxic (Murch *et al.*, 2009; Wannang *et al.*, 2009). Datura have oil content ranging from 27% to 37% of oil, it is feasible for biodiesel production and it is possible to get very high fatty acid methyl ester (FAME) conversion (Rabiah-Nizah *et al.*, 2014; Mathiarasi and Partha, 2016).

Biodiesel is a mono-alkyl ester of vegetable oils or animal fats which have similar fuel properties to petroleum based diesel fuel (Feyzi *et al.*, 2013), with typical characteristics such as biodegradability, non-toxicity, essentially free sulphur, low emission of carbon monoxide, particulate matter and unburned hydrocarbon (Rabiah-Nizah *et al.*, 2014). Biodiesel is produces through transesterification reaction of triglycerides from vegetable oils or animal fats with methanol or any lower molecular mass alcohols in the presence of acid catalysts (such as H_2SO_4) or alkaline catalysts (such as NaOH, KOH and $NaOCH_3$) to form a mixture of fatty acid alkyl esters and glycerol (Pingbo *et al.*, 2014). However using liquid acid catalyst will rust the equipment and relatively slow down the reaction rate while homogeneous alkaline catalysts are generally liable to water, and it react with free fatty acid of oils which resulting in saponification (Pingbo *et al.*, 2014). Furthermore, these homogeneous catalysts have other problems such as difficulty in removing catalysts after the reaction, production of large amount of wastewater and emulsification (Chakraborty *et al.*, 2010). To solve these problems, heterogeneous catalyst is seen as good alternative due to its several advantages such as simple catalyst recovery, catalyst reusability and the less energy and water utilization (Niju *et al.*, 2014). Pingbo *et al.* (2014) reported that the alkaline earth metal oxides were used as heterogeneous catalysts for biodiesel production and CaO shows an active catalytic performance in transesterification reaction among the reported ones. From economic point of view, CaO shows potential in biodiesel production because it can be obtained from the waste consisting of $CaCO_3$ such as waste egg-shell, oyster shell, shrimp shell, mud crab shell, mollusk shell and fish scale which is a kind of innovation in economy of resources, making use of waste and improving economic efficiency (Pingbo *et al.*, 2014). Kawashima *et al.* (2009) reported that CaO catalyst is an economical and highly active for biodiesel production. The aim of this research was to produce biodiesel from *Datura metel* seed using in-situ transesterification method with CaO derived from snail shell as heterogeneous alkaline catalyst

Materials and Methods

Sampling

The mature and sun-dried capsules of *Datura metel* were harvested from plant, at new bus stop along the Usmanu Danfodiyo University, Sokoto, Nigeria and it was identified by Herbarium officer of botany unit Usmanu Danfodiyo University, Sokoto, Nigeria. The capsules were pilled to obtain the seeds. Undesired impurities were removed by hand-picking and the seeds were prepared for extraction by grinding, using the laboratory mortar and pestle and sieved with 2.00 mm siever. The snail shells were collected from Niger State, Nigeria and were also washed, dried and grinded into powdered form.

Experimental Procedure

Catalyst Preparation

The snail shells were washed thoroughly and oven dried at 130℃ for 1 h. It was ground and sieved with 2.00mm sieve mesh. It was calcined at 500°C for 3 h which later stored in desiccator for more use.

X-ray fluorescence analysis of the catalyst

The calcined snail shell (5 g) was measured in a platinum crucible and heated in the furnace at 1000℃ for 30 min to determine the lost on ignition which was found to be 41.90%. Hence, 4-general method was used which involve mixing of 8.4 g of the melting agent (which contain Lithium tetraborate, Lithium metaborate and Lithium bromide) and 1.2 g of the powdered sample in a platinum crucible and placed on a claisse machine which converts the mixture into glass in 13 minute. The glass bead formed was placed in an x-ray fluorescence analyzer to determine the percentage of oxides present in the sample.

Transesterification of Datura metel seed oil

Datura metel seed powder (50 g) was weighed and added to the thimble, 100 ml of n-hexane and methanol was measured and transferred to 250 ml round bottom flask and 3 g of CaO derived from snail shell was added to the solvent mixture. The round bottom flask was fitted in a soxhlet extractor {n-hexane to methanol ratio [(v/v) of 1:1]}. The transesterification reaction was carried out at 65℃ temperature for 3 h with stirring. At the end of the reaction period, the round bottom flask was removed from soxhlet extractor apparatus allowing cooling to room temperature. The mixture was filtered and the remaining solvent in filtrate was then evaporated using a rotary evaporator. The mixture was then transferred to separating funnel and allowed to settle overnight. Two layers were formed, the lower layer was dark brown in color contained glycerol and the upper layer was amber yellow in colour contained fatty acids methyl esters. Gas Chromatography- Mass Spectroscopy (GC-MS) was used to determine the fatty acid methyl esters and free fatty acid composition of the produce biodiesel.

All fuel properties analysis were conducted using ASTM standard methods reported by Gerpen *et al.* (2004), while cetane index and HHV were calculated using equations reported by Demirbas, (2008) and Mohibbe *et al.* (2005), respectively.

GC-MS (gas chromatography- mass spectrometry) analysis

GC-MS analysis was carried out using Agilent Technologies 5973 Network GC/MS System with 122-5533 capacity column (DB-5ms, 0.25 mm × 30 m × 1. 00 µm). 1µl of the sample was injected onto the column. The helium gas was used as the carrier gas at 1.2 ml/min flow rate. The inlet temperature was maintained at 230ºC and the oven temperature was programmed initially at 50ºC for 5 min, then programmed to increase to 300ºC at a rate of 10ºC ending with 25 min, this temperature is held for 15 minutes, total run time was 45 min. The MS transfer line was maintained at a temperature of 250ºC. The source temperature was maintained at 230ºC and the MS quad at 150ºC. The ionization mode used was an electron ionization mode at 70eV. Total ion count (TIC) was used to evaluate for compound identification and quantization. The spectrum of the separated compound was compared with the database of the spectrum of a known compound saved in the NIST02 reference spectra library. Data analysis and peak area measurement was carried out using Agilent Chemstation Software (Mukhtar *et al.,* 2017).

Results and Discussion

The X-ray fluorescence analysis results in Table 1, revealed that CaO (95.01%) and SiO_2 (3.47%) are principal constituents of prepared catalyst which indicates that snail shell are very good sources of high grade CaO at 500℃ calcined temperature.

Table 1: X-ray fluorescence analysis of calcined snail shell

Oxides	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	P_2O_5	SiO_2	SO_3	TiO_2
$wt.\%$	0.45	95.01	0.43	0.13	0.17	0.25	3.47	0.04	0.04

Table 2, Physico-chemical properties of *Datura metel* seed oil biodiesel

	Datura metel seed oil FAME	ASTM/ EN14214 Limit
Biodiesel Yield (%)	91.00	-
Iodine Value (mg I_2/100g)	65.00	115
Density (g/cm^3)	0.83	0.90
Kinematic viscosity @ 40℃ (mm^2/s)	3.41	3.5-5.0max
Saponification Value (mg/KOH)	193.50	-
Cloud Point (℃)	4.85	-
Pour Point (℃)	8.61	-
Cetane Number	70.20	47
HHV (MJ/kg)	38.05	35
Water and Sediment (%)	0.04	0.05
Acid Value (mg/KOH)	2.24	0.50

Biodiesel (91% yields) was produced. This yield is high but slightly lower than 95.50% of *Datura metel* biodiesel reported by Mathiarasi and Partha (2016) produce from *Datura metel* Linn oil using SO_2Cl_2 catalyst. But disadvantage of SO_2Cl_2 catalyst in biodiesel production from Datura metel oil is it increase biodiesel acidity (Mathiarasi and Partha, 2016).

Iodine value measure the degree of unsaturation of biodiesel. It is used to estimate the oxidation stability of biodiesel. The iodine value 65 mg I_2/100g presented in Table 1, indicate that *Datura metel* oil biodiesel has moderate degree of saturation. The value is within the ASTM biodiesel standard. Hence, it may have high oxidation stability (Mukhtar *et al.,* 2017).

Density is the mass per unit volume. It is an important fuel quality parameter used to determine its purity (Barabás and Todoruţ, 2011). The *Datura metel* oil biodiesel density 0.83 g/cm^3 presented in Table 1, revealed that *Datura metel* oil biodiesel has high density but it is within the ASTM biodiesel standard limits which means that volumetrically-operating fuel pumps will inject greater mass of *Datura metel* oil biodiesel into compression ignition engine if use as transport fuel and it is cleaned (Evangelos, 2013).This may influence engine output power due to a different mass of fuel injected which directly affects engine performance (Jahirul *et al.*, 2015). It may also causes decrease in NOx and particulate matter emission in old diesel engines technology (Wan Ghazali *et al.*, 2015)

Kinematic viscosity is an important fuel property as it plays main role in fuel spray and combustion process (Wan Ghazali *et al.*, 2015). The use of high kinematic viscosity biodiesel in diesel engine may cause poor atomization during fuel spraying, deposits and wear in the fuel system (Ali *et al.*, 2016). The kinematic viscosity result of *Datura metel* oil biodiesel (3.41 mm^2/s) revealed that *Datura metel* seed biodiesel may have good fuel/air during atomization and may not lead to wear in fuel operating system.

Cloud and Pour Points are the most important diesel fuel cold flow properties (Verma *et al.*, 2016). It is use to assess the suitability of using fuel in low temperature region, thus biodiesel with low pour point and cloud point is the most appropriate fuel in cold weather condition (Jahirul *et al.*, 2015). Generally, convectional diesel has low cloud and pour points than biodiesel, due to the biodiesel high saturated fatty acids content (Wan Ghazali *et al.*, 2015). Cloud and pour points of *Datura metel* oil biodiesel are 4.85℃and 8.61℃ respectively which higher than the values reported by Mathiarasi and Partha (2016) from Datura metel Linn oil biodiesel produced using SO_2Cl_2 catalyst with ultrasound wave from sonicator using conventional transesterification method. Hence, biodiesel produced may not cause filters clogging in the engine fuel operating system at 8.61℃ and above temperature (Verma *et al.*, 2016). Therefore it could be a suitable alternative fuel for both hot and cold weather conditions in tropical region.

Cetane number is a fuel quality parameter used to estimate the ignition quality of diesel fuels. The *Datura metel* oil biodiesel cetane number 70.24 is higher than ASTM biodiesel minimum standard limit (47). *Datura metel* oil biodiesel might have good cold start properties and reduced white smoke formation due to high cetane value (Jahirul *et al.*, 2015). It may have shorter delay interval and will have higher combustibility.

High heating value is an important factor in fuel selection which determine its energy content (Sivaramakrishnan and Ravikumar, 2011; Atabani *et al.*, 2013). Thus, the amount of fuel require to covered a long distance drive (Mukhtar and Dabai, 2016) The *Datura metel* oil biodiesel high heating value 38.05 MJ/kg obtained as shown in table, 1, is higher than the minimum standard specified by EN 14213(35MJ/kg). This indicates that *Datura metel* oil biodiesel has high energy content. Therefore, this property had uplift it use as alternatives to conventional diesel.

Table 3: GC-MS fatty acids methyl esters profile of *Datura metel* seed oil biodiesel

	Possible Compounds	%composition
FAME		
	Methyl Hexadecanoate	14.10
	Methyl-11-Octadecenoate	43.23
	Methyl Octadecanoate	11.34
	Methyl -Eicosanoate	0.44
Total FAME		**66.86**
FFA		
	Hexadecanoic acid	4.65
	Oleic acid	21.04
Total FFA		25.69
Other compounds		7.45

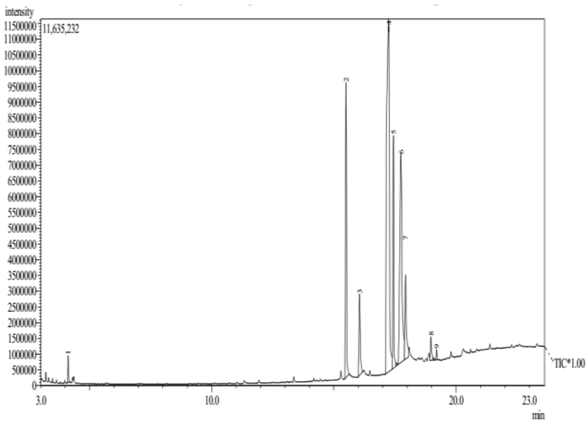

Fig. 1: GC-MS chromatogram of *Datura metel* oil fatty acid methyl esters

Table 3 and Fig. 1 presented the fatty acid methyl esters of *Datura metel* oil biodiesel. It revealed that 11-Octadecenoic acid, methyl ester (43.23%), Hexadecanoic acid, methyl ester (14.10%) and Octadecanoic acid, methyl ester (11.34%) are the major fatty acid methyl esters detected in *Datura metel* oil biodiesel, while other minor fatty acid methyl ester are Eicosanoic acid, methyl ester (0.44%).Free fatty acids found present are Hexadecanoic acid (4.65%) and Oleic acid (21.04%) This results indicates that *Datura metel* oil biodiesel content 66.86% fatty acid methyl esters. Overall fatty acids profile indicate that *Datura metel* oil biodiesel predominantly consist of 64.27% unsaturated fatty acids. This indicates a good cold flow properties (Evangelos, 2013; Verma *et al.*, 2016). It is in good agreement with the cloud and pour points presented in table1 which indicate high cold flow properties. The absent of polyunsaturated esters indicate that *Datura metel* oil biodiesel may have higher storage oxidation stability.

Conclusion

High grade CaO was prepared from snail shell which produce high biodiesel yield of 91%. This indicates high CaO catalyst activity. GC-MS result revealed that *Datura metel* oil biodiesel contained high proportion of unsaturated fatty acid methyl esters coupling with cloud and pour points indicates that *Datura metel* oil biodiesel could be a good alternatives fuel in both hot and cold climate conditions. All the tested fuel physicochemical properties are within the recommended standard limits with exception of acid value which revealed that it requires acid treatment or blend with petroleum diesel before use in ignition compression engine. Hence CaO derived from Snail shell is a potential catalyst for biodiesel production from *Datura metel* oil.

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