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#### Umeuzuegbu JC

Department of Chemical Engineering, Chukuemeka Odumegwu Ojukwu University, Uli, Anambra State, Nigeria

#### Arokeshi IA

Department of Chemical Engineering, Chukuemeka Odumegwu Ojukwu University, Uli, Anambra State, Nigeria

#### Onukwuli OD

Department of Chemical Engineering, Nnamdi Azikiwe University, Awka, Anambra State, Nigeria

Corresponding Author: Umeuzuegbu JC Department of Chemical Engineering, Chukuemeka Odumegwu Ojukwu University, Uli, Anambra State, Nigeria

### Single step methanolysis of sesame seed oil using calcined cement clinker catalyst

#### Umeuzuegbu JC, Arokeshi IA and Onukwuli OD

#### Abstract

The need for renewable, sustainable and environmentally friendly energy sources has directed researchers into harnessing energy from biomass. Bio-fuels, the energy sources derived from biomass comprise principally of biodiesel, bioethanol and biogas are now preferred alternative to fossil fuel. This research work focused on a single step methanolysis of sesame seed oil using cheap, locally sourced, calcined cement clinker catalyst. Sesame seed oil was solvent extracted using n-hexane. The physiochemical properties of the sesame seed oil (SSO) was characterized base on American Standards for Testing Materials (ASTM) method. The fatty acid profile and the functional groups of the oil were respectively determined using gas chromatography mass spectroscopy (GC-MS) and Fourier Transform Infra-red spectroscopy. The effect of process parameters, methanol to oil molar ratio, catalyst concentration, reaction time and temperature and agitation speed on sesame seed oil fatty acid methyl ester (SSOFAME) or biodiesel yield were determined based on one factor at a time method. The fuel properties of the SSOFAME produced were obtained based on ASTM standards. The physiochemical properties of SSO were obtained as density 890 kg/m<sup>3</sup>, saponification value 191.4 mgKOH/g, iodine value 86.5 gI<sub>2</sub>/100 g, peroxide value 2.01 mEq/kg, kinematic viscosity 32.5 mm<sup>2</sup>/s, fire point 187.1 °C, flash point 200 °C, cloud point 2.0 °C, pour point 0.5 °C, refractive index 1.465, specific gravity 0.890, moisture content 2.86%, acid value 4.3 mgKOH/g, free fatty acid 2.15%, calorific value 20.5 MJ/kg. The optimum process parameters for obtaining the highest biodiesel yield of 87% were methanol to oil molar ratio7:1, catalyst concentration 1.0 wt%, reaction temperature 60 <sup>o</sup>C, reaction time 60minutes, and agitation speed 300 rpm. Results show the fuel properties to be density 850 kg/m<sup>3</sup>, kinematic viscosity 4.72 mm<sup>2</sup>/s, cetane number 58.8, flash point 170 °C, cloud point 2.5 °C, water content 0.05%, acid value 0.25 mgKOH/g, calorific value 38.5 MJ/kg, iodine value 86.4 gI<sub>2</sub>/100 g, pour point o.5 <sup>O</sup>C, specific gravity 0.85, free fatty acid 0.13%, refractive index 1.466. The various data obtained for transformation of SSO to SSOFAME points to the fact that the biodiesel is ostensibly suitable alternative to diesel fuel.

Keywords: Characterization, cement clinker, sesame seed oil, sesame seed oil fatty acid methyl ester, transesterification

#### 1. Introduction

The rapid depletion of fossil fuel coupled with its environmental impact have necessitated research into various alternative energy sources such as solar, hydro, geothermal, hydrogen, nuclear and biomass. Among the various alternatives to fossil, energy from biomass called bio-fuels comprising of biodiesel, bio-ethanol, biogas, etc. are now most attractive worldwide because of their renewable nature and environmentally friendliness <sup>[1, 2, 3]</sup>. Biodiesel is a better lubricant than diesel fuel as a result of its relative high oxygen content. The advantages of biodiesel over fossil fuel are, being renewable, biodegradable, environmentally friendly, high cetane number and high lubricity <sup>[4]</sup>. However, biodiesel has its own drawbacks which include high density and viscosity, poor cold-flow properties <sup>[4]</sup>. Diesel engines are the prime movers in transportation, power generation, construction industries etc. It is now the practice blending biodiesel with petro-diesel to give a better lubricating effect than the sulphur compounds in diesel <sup>[5]</sup>.

The ideal vegitable oil for biodiesel production must be readily available, its plant should be easy to cultivate, and its fatty acid composition must be a proportionate monounsaturated, polyunsaturated and saturated fatty acids <sup>[6]</sup>.

A proper selection of feedstock for production of biodiesel is critical for viable alternative fuel to petro-diesel. Although, biodiesel is gaining popularity, more than 95% of the renewable resources used for its production are edible oils <sup>[7]</sup>, which will in a long term have serious implications on food availability and the cost of biodiesel as it may be more expensive than petro-diesel. Worldwide, biodiesel production is mainly from edible oils such as soya bean, sunflower, canola, palm oils etc. as these oils contain less free fatty acid than non-edible oils and most times do not require esterification or pretreatment before transesterification. Much research efforts are now geared towards identifying and evaluating non-edible seed oils as suitable feed stock. However, the cost of non-edible oil biodiesel purification is high because of their high free fatty acid content which must be considered before making a good choice of the feedstock.

Different feed stocks consisting of edible and non-edible oils have been successfully used in biodiesel production, including soybean oil <sup>[8]</sup>, sunflower oil <sup>[9]</sup>, rapeseed oil <sup>[10]</sup>, palm oil <sup>[11]</sup> jatropher oil <sup>[12]</sup>, camelina oil <sup>[13]</sup>, sesame oil <sup>[14]</sup>, neem seed oil <sup>[15]</sup>.

*Sesamum indicum* commonly called beniseed or sesame is an important oilseed crop cultivated in many parts of the world with oil content of 50-52%. Sesame oil is extracted from sesame seed which consist of proportionate mixture of mono-unsaturated, poly-unsaturated and saturated fatty acids [6] that make the oil suitable for biodiesel production.

The cost of biodiesel will be further reduced by the use of low cost catalyst. The oil from sesame seed has relatively high free fatty acid, of which biodiesel yield is not favored by alkaline catalysis alone without first of all esterifying the oil with an acid catalyst. This research work, in order to circumvent the two step process of esterification with acid catalyst before transesterification with alkali catalyst, investigated the use of heterogeneous catalyst of cement clinker for single stage transesterification of the SSO into SSOFAME. Heterogeneous base catalyst has the advantages of being reusable, tolerant to free fatty acid and water content of the oil feedstock, improved yield and purity of biodiesel, simpler purification process of glycerol <sup>[16, 17, 18]</sup>. The advantages of use of CaO catalyst for transesterification include low cost, high activity, mild reaction conditions and being reusable. Some researchers <sup>[19, 20]</sup> have reported biodiesel yield of 95% and 96.6% respectively from soya bean oil using CaO catalyst.

#### 2 Materials and Methods

#### **2.1 Materials**

Sesame seeds (plate 1), reagents, glass wares, equipments including gas chromatography mass spectrometer (GC-MS), Fourier transform infrared spectroscopy (FTIR), viscometer, magnetic hot plate, soxhlet extractor, specific gravity bottle; electrostatic oven etc.

#### 2.2 Experimental Methods

#### 2.2.1 Sample preparation

The sesame seeds used in this research work was bought

from Ochanja market Onitsha, Anambra state Nigeria. 10kg of the sesame seeds was sorted and washed with water to remove sand, dirt and other impurities. The seed was then dried and de-shelled manually. The main seed was sundried for three days and further oven-dried at temperature of 50 °C for 4 days in other to eliminate the moisture content and then size-reduced by grinding with mechanical grinder. The sample was then kept for further analysis.

#### 2.2.2 Extraction of oil from the dry ground sesame seed

The oil content of sesame seed was determined using soxhlet extractor with n-hexane solvent. The use of nhexane solvent is in agreement with the finding of [21-25] who reported enhancement of oil yield from sesame seed using n=hexane. The oil content of the seed was evaluated using soxhlet extractor and n-hexane when the oil was extracted for 18 hours for complete extraction of the oil. For the oil used in the work, 3kg of the dried, ground sesame seed was measured into a plastic container containing 3 liters of nhexane. The mixed content of the container were vigorously shaken after covering the container. The container was made air tight to prevent evaporation of the solvent and then kept to macerate for a day. Then the dissolved oil in n-hexane was decanted and the slurry filtered. The filtrate was distilled to recover the solvent at 65 °C <sup>[47]</sup>. The percentage oil content was calculated as:

% oil yield = weight of oil obtaind  $\div$  weight of seed sample x 100 (1)



Plate 1: Sesame seed

#### 2.2.3 Characterization of sesame seed oil

The physiochemical properties of the oil extracted from sesame seed was characterized based on American Society for Testing Materials, ASTM 6751 (1973) method. Analytical equipments, GC MS (QP2010 plus Shimadzu, Japan) and FTIR (M530 Bulk scientific FTIR) were used to determine the fatty acid profile and the functional groups of the oil respectively.

#### 2.2.4 Preparation of the clinker catalyst

The clinker used was obtained from DANGOTE Cement Factory in Nigeria. The cement clinker was washed with 1%

solution of sulphuric acid to remove dirty stains on its surface. It was then pulverized and sieved using 80-100 mesh in order to obtain large surface area of the catalyst particles for efficient catalysis. The chemical composition of the clinker was obtained from X-ray florescence with inbuilt XRD (ARL 8660S which shows the major constituent of clinker as the base CaO responsible for the catalysis of transesterification reaction. The clinker was activated by soaking with methanol in the ratio of 1:1(w/w), followed by calcinations at 700 °C for 7 hours in the furnace. After cooling on a water bath the catalyst was ready for use.

#### 2.2.5 Effect of process parameters on biodiesel yield

The effects of process parameter on biodiesel yield from sesame seed oil were investigated using one factor at a time method involving keeping a factor constant at a time and varying the others in turn. The five factors investigate are, molar ratio of methanol to oil, catalyst concentration, reaction time, reaction temperature and agitation speed

# 2.2.6 Synthesis of sesame seed oil fatty acid methyl ester (SSOFAME)

The free fatty acid in sesame seed oil 2.15% is in excess of 1.0% of the maximum required for optimum yield of biodiesel with basic catalyst involving esterification or pretreatment of the oil first with concentrated sulphuric acid and methanol to reduce the free fatty acid below 1.0wt % before being transesserified with sodium hydroxide. The two stage homogeneous transesterification process was circumvented by employing of one stage heterogeneous transesterification using cheap, calcined cement clinker catalyst.

The biodiesel in this work was produced using the normal laboratory method of preparation. The amount of oil required for the transesterification was run into a 500 cm<sup>3</sup> three-necked round bottomed flask. Onto the side arms of the three-necked flask used as the reactor, were fitted a thermometer and a receiver respectively, while the central arm was fitted with a condenser. The amount of oil specified for the reaction was run into the flask and the oil heated to the specified temperature for the reaction. Specified amount of calcined cement clinker catalyst (4% w/w of oil) with methanol were added onto the flask content. The hot plate stirrer was switched on after setting the stirrer speed at the value required for the reaction. Heating was continued and the flask content continuously stirred and refluxed. At the end of transesterification, the flask content was poured into separating funnels, allowed to settle for a day where it separated into upper biodiesel layer and the lower glycerol layer. The two layers were tapped off separately, the glycerol layer first followed by the biodiesel layer. As the biodiesel layer may contain some traces of sodium hydroxide and glycerol, they were removed by wet washing. The washed biodiesel was then dried on a laboratory hot plate at 105 °C to remove all traces of moisture remaining in it. The percentage biodiesel yield is given by the expression,

2.2.6 Determination of the fuel properties of SSOFAME

The fuel properties of the sesame seed oil biodiesel produced were characterized based on ASTM method. The properties characterized for include density, viscosity, iodine value, saponification value, cetane number acid value, free fatty acid, calorific value, flash point, cloud point, pour point etc.



Plate 2: Biodiesel production by transesterification

# Results and Discussion Oil content of sesame seed

Solvent extraction using soxhlet extractor gave the oil yield of sesame seed as 48.0%. This is in agreement with the findings of <sup>[22, 27-29]</sup> who reported the range of oil content of sesame seed as 46-48.5%. Sesame seed could therefore be graded as oil bearing, and in addition to its moderate saponification number is a suitable substitute for commercial biodiesel feedstock. The determined saponification value of 191.4 mgKOH/g for the oil is moderate which makes it suitable for biodiesel production as it will not be prone to formation of soap as compared to oils of high acid value.

#### **3.2** Characterization of cement clinker catalyst

The chemical composition of the clinker was obtained from X-ray florescence with in-built XRD (ARL 8660S) as shown in table 1. From the table it could be seen that the constituents of clinker are the oxides of the metals and non-metals including; CaO, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MgO, SO<sub>2</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, TiO<sub>2</sub> with the basic oxide CaO constituting 66.4% by weight of the catalyst are the major

constituent responsible for the catalysis of transesterification reaction.

Table 1: X-ray fluorescence of clinker catalyst

Constituent compounds	Weight %
CaO	66.40
SiO <sub>2</sub>	21.59
Al <sub>2</sub> O <sub>3</sub>	6.01
Fe2O3	3.35
MgO	0.65
SO <sub>2</sub>	0.73
K <sub>2</sub> O	0.84
Na <sub>2</sub> O	0.12
P <sub>2</sub> O <sub>5</sub>	0.03
TiO <sub>2</sub>	0.32

#### **3.3 Characteristics of SSO**

#### 3.3.1 Physiochemical properties of SSO

The physiochemical properties of sesame seed oil is as presented in table 2. The viscosity and density of sesame seed oil like any other oil is higher than that of biodiesel produced thereof. The kinematic viscosity measures the flow resistance of the fuel, while the density determines the quantity of the fuel metered as this is measured volumetrically. Oils of high viscosity and density possess difficulty in atomization in internal combustion engine as it is associated with increased engine deposits and hence cannot be used directly as biodiesel [30]. High density of oil is reduced by transesterification to enable the oil to be used in internal combustion engine. From the table, it could be seen that the free fatty acid values of the oil is moderately high (>1%) thus requiring esterification before been transesterified with a base catalyst. The experimentally determined acid value of 4.30 mgKOH/g and free fatty acid value of 2.15% respectively for sesame seed oil are high and unacceptable for direct transesterification with alkali as the excess alkali give rise to soap formation and inhibits ester separation from biodiesel <sup>[31]</sup>. The relatively low saponification value of 191.4 mgKOH/g for SSO is moderate, indicative of its suitability for biodiesel production as it will not be prone to formation of soap.

The experimentally evaluated iodine value of the oil, 86.0 gI<sub>2</sub>/100 g oil does not strictly place SSO as a drying oil. Iodine value, a measure of degree of un-saturation of the oil obtained is below 100  $gI_2/100$  g oil, indicative of the oil being nondrying and therefore suitable for biodiesel production. High iodine value of oil corresponds to high degree of un-saturation of the fatty acid in the triglyceride, and if heated, such an oil is prone to thermal oxidation and polymerization of the triglyceride causing formation of deposits. The peroxide value, an index of rancidity obtained as 1.70 mEq/Kg was relatively low, ostensibly because of high antioxidant content of SSO including sesamine, sesamol, sesamolin and tocopherol, indicative of high resistance of the oil to peroxidation during storage and handling. The determined fire point and flash point of SSO are 201 °C and 183 °C respectively. The flash point in excess of 130 °C indicates that the oil is non-flammable for

handling and storage.

The key flow properties for winter fuel specification are the cloud and pour point. The cloud point is the lowest temperature at which wax-like material begins to form on cooling the oil while the pour point is the lowest temperature at which the oil can flow The cloud point of 2.5 °C and pour point 0.5 °C for SSO are moderately low and suitable for use in warm and temperate climates but not suitable for cold climatic condition. Oils of high cloud and pour points can readily congeal and faces difficulty of handling during cold weather.

#### 3.3.2 Fatty Acid Profile of SSO (GC -MS)

The fatty acid profile of sesame seed oil shown in table 3 was obtained with the aid of gas chromatography mass spectroscopy (GC-MS). The GC MS spectra of SSO is as shown in figure 1. Sesame seed oil consist of saturated fatty acid namely palmitic, stearic and arachidic acids, monounsaturated fatty acids namely palmitoleic, oleic and eicosanoic acids, and polyunsaturated fatty acid of linoleic, and linolenic acids. The most abundant fatty acid in sesame oil are the polyunsaturated acids linoleic and linolenic acids. The fatty acid composition obtained in this work are within the range reported by <sup>[32, 33, 34]</sup> as shown in table 3. Sesame seed oil is categorized as oleic oil akin to olive oil that consist of 56-85% oleic acid <sup>[35, 36]</sup>.

Table 2: Physiochemical properties of TNO

Properties	Unit	NSO
Oil yield	%	48.0
Density	Kg/m <sup>3</sup>	890
Saponification value	mgKOH/g	191.4
Iodine value	(gI <sub>2</sub> /100g oil)	86.5
Peroxide value	mEq/Kg	1.70
Kinematic viscosity	mm <sup>2</sup> /s	32.5
Fire point	<sup>0</sup> C	201
Flash point	<sup>0</sup> C	183
Cloud point	<sup>0</sup> C	2.5
Pour point	<sup>0</sup> C	0.5
Refractive index		1.465
Specific gravity		0.890
Moisture content	%	2.86
Acid value	mgKOH/g	4.3
Free fatty acid	%	2.15
Calorific value	MJ/Kg	20.5

Table 3: Fatty acid profile of SSO

Common name	Systemic name	Lipid number	Concentration (%)
Palmitic	Cis-9 tetradecanoic acid	C16:0	12.96
Palmioleic	Hexadecanoic acid	C16:1	0.22
Stearic	Octadecanoic acid	C18:0	5.76
Oleic	Cis-9-octadecanoic acid	C18:1	41.68
Linoleic	Octadeca-9-12dienoic acid	C18:2	38.29
Linolenic	Octadeca trienoic acid	C18:3	0.48
Arachidic	Eicosanoic acid	C20:0	0.53
Eicosenoic		C20:1	0.15

## **3.3.3** Fourier Transform infrared spectra analysis (FTIR) of sesame seed oil

The Fourier transform infrared spectra of SSO was analyzed using Fourier transform infrared spectroscopy (M530 Buck scientific FTIR). This analysis was carried out in order to detect the various functional groups contained in the oil. The FTIR spectrum of SSO is as shown in figure2. The various functional groups detected at different detectable peaks of note were recorded. The peaks around 1311.6082 cm<sup>-1</sup> and 1384.538 cm<sup>-1</sup> were both assigned to C=C anti-symmetric vibration of ethane compounds respectively.

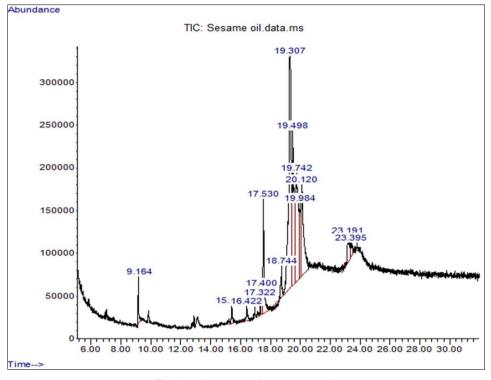


Fig 1: GC-MS plot of sesame seed oil

The absorbance band around 1614.382 cm<sup>-1</sup> was assigned to N-H stretching vibration of primary amine compound while that around 1872.325 cm<sup>-1</sup> was due to CO stretching vibration of cyclic ester compound. The peaks located at 2010.045 cm<sup>-1</sup>, 2063.472 cm<sup>-1</sup> and 2192.396 cm<sup>-1</sup> were all assigned to COO stretching vibration of carboxylic acid respectively. Also, the absorbance around 2458.883 cm<sup>-1</sup>

was assigned to CN anti-symmetric vibration of nitrile compound. The weak band around 2877.133 cm<sup>-1</sup> was assigned to C-H symmetric vibration of methylene compound. The strong band around 3141.931 cm<sup>-1</sup> and 3833.034 cm<sup>-1</sup> corresponds to OH vibration of primary and tertiary alcohols respectively.

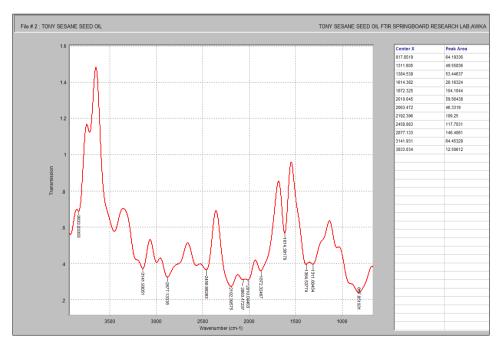


Fig 2: FTIR spectra of sesame seed oil

#### 3.4 Effect of Process Parameter on Biodiesel Yield

3.4.1 Effects of catalyst concentration on SSOFAME yield: The alternative reaction pathways for breaking of bonds created by the use of catalysts most often involve lower activation energy. The effect of catalyst concentration on the yield of SSOFAME was investigated from 0.25 to 1.5% wt catalyst concentration as shown in figure 3. It was found that the yield of biodiesel increased with increase in catalyst concentration until an optimum yield was obtained at 1% wt. of catalyst when the yield started declining. Decrease in biodiesel yield beyond the 1% wt. catalyst concentration could be explained by the fact that in the presence of excess catalyst above the optimum 1% wt., the excess catalyst react with the oil to form soap which increases the viscosity of the reaction mixture, hindering effective dispersion and mixing of the reactants mixture and also separation of glycerol from biodiesel which result in the reduction of biodiesel production. This is in conformity with the findings of <sup>[37, 38]</sup>.

3.4.2 Effects of methanol to oil molar ratio on SSOFAME yield: The molar ratio of alcohol to oil for transesterifying triglyceride to fatty acid methyl ester is one of the most vital factors that affects the yield of esters. It has been reported by researchers that the use of excess alcohol in place of the stoichiometric ratio required of 3:1 gave higher yield of bio-diesel. In this work, the effect of methanol to oil molar ratio of 1:1 to 11:1 was investigated, when other process parameters, catalyst concentration, reaction temperature, reaction time and agitation speed were kept constant. The yield of sesame seed oil biodiesel for the different molar ratio of methanol to oil is shown in Figure 4. The results indicated that methanol to oil molar ratio has significant effect on the FAME yield. The maximum ester yield was obtained at a methanol to oil molar ratio of 7:1 for SSO. SSOFAME yield reduced when the molar ratio was higher than 7:1. This trend can be explained by the fact that while the increase in methanol to oil molar ratio favors transesterification reaction, very high methanol to oil molar ratio decreased the catalytic activity of the basic catalyst, resulting in the reduction of biodiesel produced. This is in agreement with the findings of [39, 40], It has also been reported by <sup>[41]</sup> that the use of excess alcohol for transesterification reaction increased the polarity of the reaction mixture and this increased the solubility of the glycerol in the reaction mixture which retards separation of glycerol from biodiesel and thus reduce the yield of biodiesel.

3.4.3 Effects of reaction temperature on SSOFAME yield The rate of reaction is known to increase with increase in temperature. In order to investigate the effects of temperature on the yield of SSO biodiesel, the temperature was varied from 30 °C to 80 °C at a step increase of 10 °C while the other parameters, catalyst concentration, methanol to oil to oil molar ratio, reaction time and agitation speed were kept constant as shown in figure 5. From the figure, it could be seen that biodiesel yield increased with increase in reaction temperature until a maximum yield was obtained at optimal temperature of 60 °C when the yield started decreasing. The decrease in biodiesel yield beyond 60 °C may be explained by the fact that the boiling point of methanol is approximately 65 °C, and therefore ones this temperature range is exceeded, the backward reaction is favored as most of the methanol will be lost by evaporation, thus reducing the yield. This conforms with the findings of  $^{\left[ 42\right] }.$ 

**3.4.4 Effects of reaction time on SSOFAME yield:** In this work, the effects of reaction duration from 15 to 90minutes on the yield of biodiesel from SSO was investigated. It was found that reaction time of 60 minutes was needed for a maximum yield of SSOFAME investigated and beyond this time, the yield decreased as shown in Figure 6.

The decreased in yield after 60 minutes may be due to reversible reaction nature of transesterification resulting in loss of esters <sup>[43]</sup>. Also longer reaction time most times allow the fatty acid present to react with alkali and this will result to soap formation. The presence of soap retards the formation of ester <sup>[44]</sup>.

#### 3.4.5 Effects of agitation speed on SSOFAME yield

In order to study the effect of agitation speed on the yield of SSO biodiesel, agitation speed was varied from 150rpm to 400rpm while keeping the other parameters constant as shown in figure 7. Agitation is particularly important during transesterification in order to ensure homogeneity within the reaction mixture. From the figure it could be observed that SSO biodiesel yield gradually increased with increase of agitation speed until the maximum yield was attained at 300rpm when the yield starts decreasing. The decrease in yield on exceeding the optimal agitation speed of 300rpm may be explained by the fact that the backward reaction may have been favored when mixing intensity went beyond the optimal value of 300 rpm thereby retarding the formation of biodiesel. These results conformed with the observations made by Endaah et al. [43], who studied the effect of agitation speed on the transesterification of nonedible oil and concluded that higher agitation promoted the homogenization of the reactants and thus led to higher yield of biodiesel.

#### **3.5 Fuel Properties of the SSOFAME Produced**

The summary of fuel properties of SSOFAME produced are given in table 4. The experimentally determined values of density and viscosity of sesame seed oil biodiesel produced are 850 kg/m<sup>3</sup> and 4.72 mm<sup>2</sup>/s respectively. The density and viscosity of SSOFAME are lower than that of SSO (890  $kg/m^3$  and 32.4 mm<sup>2</sup>/s) from which it was produced. High density and viscosity of fuel results in poor atomization in compression ignition engine, which gives rise to carbon deposits, plugging of fuel filter and injector cocking <sup>[46]</sup> and therefore reduction of engine power output. The essence of transesterification is to reduce density and viscosity of oil in order to circumvent the above problems. However, the viscosity of fuel should not be excessively low as this produces very subtle spray which cannot properly get into the combustion cylinder, thus forming a fuel rich zone that give rise to formation of sooth [45, 46]. The flash point is a determinant for flammability classification of materials. The typical flash point of pure methyl ester is ≥130 °C, classifying them as "non-flammable". However, during production and purification of biodiesel, not all the methanol, may be removed, making the fuel flammable and dangerous to handle and store if the flash point falls below 130 °C. The experimentally determined flash point of the SSOFAME is 170 °C. This falls within the ASTM standard as shown in table 4, indicative of its safety in handling and storage.

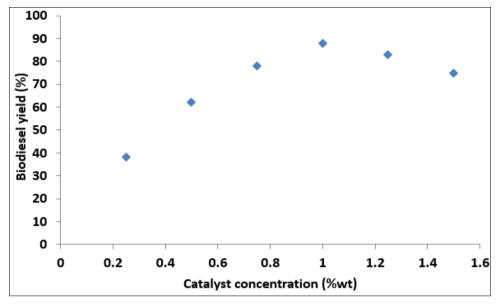


Fig 3: Effect of catalyst concentration on SSOFAME yield

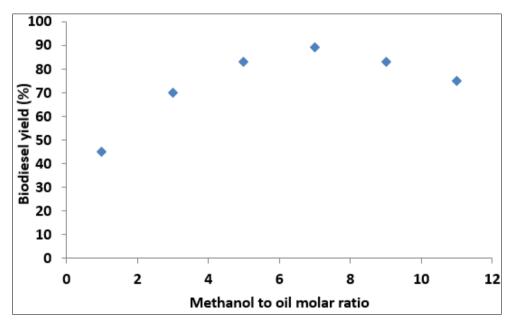


Fig 4: Effect of methanol to oil molar ratio on SSOFAME yield

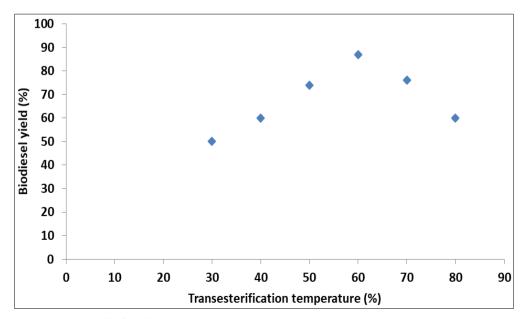


Fig 5: Effect of transesterification temperature on SSOFAME yield

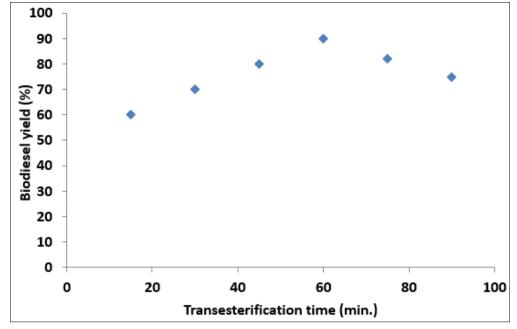


Fig 6: Effect of transesterification time on SSOFAME yield

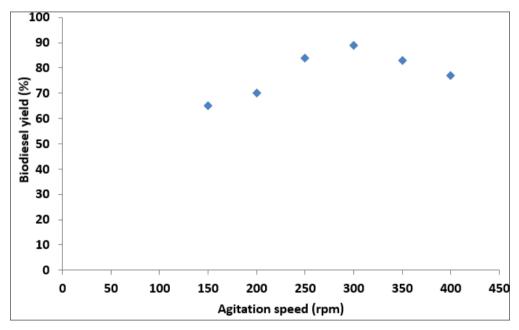


Fig 7: Effect of agitation speed on SSOFAME yield

Cetane number which serves as a measure of ignition quality of a fuel was experimentally determined as 58.8 for SSOFAME. Fuels of low cetane number shows increase in emission due to incomplete combustion. The ASTM limit for biodiesel cetane number is 47. Thus the evaluated cetane number of 58.8 for SSOFAME is within the ASTM standard, indicative of the fact that the produced biodiesel possess good ignition response.

A good quality biodiesel is expected to be of low acid value as high acidic content may corrode and damage machine parts. The acid value of SSOFAME in this work was determined as 0.25 mgKOH/g which is within the ASTM limit. Low saponification number is expected of high quality biodiesel as oil of high saponification value is prone to soap formation by alkali transesterification and thus resulting to reduction of quality and quantity of the biodiesel produced. The cloud point which is the lowest temperature of first appearance of wax-like material on cooling the biodiesel was determined as 2.5 °C for SSOFAME while the pour point which is the lowest temperature at which the fuel will still pour was determined as 0.5 °C. The cloud and pour points are moderately low but not sufficiently as not to give rise to cold flow problems in cold season especially in the cold regions. This cold flow problems however could be overcome by the addition of suitable cloud and pour point depressants or by blending with diesel oil <sup>[46]</sup>. The properties of the biodiesel produced are within the ASTM limit for biodiesel, as shown in table 4.

Properties	Unit	SSOFAME	ASTM Standards	Test method
Density	Kgm <sup>-3</sup>	850	860-900	D93
Kinematic viscosity	mm <sup>2</sup> s <sup>-1</sup>	4.72	1.9-6.0	D445
Cetane number		58.8	47min.	D613
Flash point	<sup>0</sup> C	170	100 to 170	D93
Cloud point	<sup>0</sup> C	2.0	-3 to -15	D2500
Water &sediment	%	0.05	0.05	D2709
Acid value	mgKOHg <sup>-1</sup>	0.25	0.50	D664
Calorific value	MJKg <sup>-1</sup>	38.5	42.06	D35
Iodine value	gI <sub>2</sub> /100g oil	86.4	42-166	
Pour point	<sup>0</sup> C	0.5	-10 min.	D97
Specific gravity		0.85	-	D287
Free fatty acid	%	0.13	-	
Refractive index		1.467		

Table 4: Fuel properties of SSOFAME

#### 4. Conclusion

Sesame seed oil was successfully transesterified by the use of calcined cement clinker catalyst.

The results obtained from the research work shows that sesame seed contained high percentage of oil and thus fulfilling the requirement for sustainable biodiesel feedstock which include high oil yielding seeds of high yield per hectare <sup>[47]</sup>. Cement clinker, a byproduct of manufacture of cement is envisaged to reduce the overall cost of the biodiesel as it is comparatively less expensive than the alkali catalyst, involve one step process which minimizes the cost of purification of biodiesel by two step process of homogeneous catalysis. The SSOFAME produced thereof meets the ASTM standards in terms of density, viscosity, flashpoint, cetane number etc. From the results therefore it is obvious that the biodiesel produced from sesame seed oil is a good alternative to diesel in compression ignition engine

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