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Effect of Different Catalyst Concentrations on Biodiesel Production from Palm Oil Mill Effluent (Pome)

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ABSTRACT

The scarcity of conventional fossils fuels, growing emissions of combustion generated pollutants, and their increasing in production cost makes biomass sources more attractive. Biodiesel has become more attractive recently because of it environmental benefits and the fact that it is produced from renewable resources.These studies therefore focus on the production of biodiesel from palm oil mill effluent using different catalyst concentration. Oil was extracted from palm oil mill effluent to produce biodiesel through transestrification with ethanol using potassium hydroxide(KOH) ascatalyst. The bio-oil to ethanol ratio of 6:1 (150 ml: 25 ml) at60 minutes reaction time and65^{0}Creaction temperature respectively. The biodiesel was produced at differentcatalyst concentration 0.25g, 0.5g, 0.75g, 1.0g, while other parameters were kept constant. The optimal biodiesel yield was obtained at catalyst concentration of 0.75g. Meanwhile the following tests were carried out on the optimal yield conditions. Viscosity 9.660 pas, specific gravity 0.885, relative density 751kg/m^3, pour point -2.1^{0}C, cloud point 6.0^{0}C. At 2500 rpm rotational speed an exhaust temperature of 450^{0}C, power output of fuel on dynamometer 24.7W and toque of 42.4 Nm were obtained. These tests were carried out according to standardsspecified by ASTM.

KEYWORDS: POME oil, KOH, transestirification, physicochemical propertiesand engine performance test.

I. INTRODUCTION:

Fossil fuels are, nowadays, the most used worldwide but that are some problems involving their utilization. First of all, the price, which is escalating often,makes petroleum nolonger economically sustainable. Secondly, emission of very hazardous pollutants such as carbon dioxide during burning of petrochemical sources which in turn causes global warming. In addition, fossil fuels are non-renewable;they last for a limited period of time. For all these reasons, vegetable oils are emerging as a great alternative source of fuel, because of their renewable nature and environmental benefits (Ferellaet al., 2010). Despite all these advantages, the use of vegetable oils as fuel source has some disadvantages. The incomplete burning during direct use in diesel internal combustion engines form deposits in the fuel injectors system,this is due to high viscosity and low volatility effects.

According to specialized literature there are five ways of reducing the problems mentioned above: blending of vegetable oil and diesel, thermal cracking (pyrolysis), micro-emulsions, esterification and transesterification (Ma & Hanna, 1999). Esterification and transesterification reactions are currently the most favored reaction pathways to produce biodiesel (Janaun& Ellis, 2010).

Biodiesel, defined as the simple alkyl monoesters of long chain fatty acids derived from renewable feedstock, is the most suitable substitute to diesel. For this reason the research on this biofuel are steadily growing all over the planet. In Brazil, the focus of research is the production of biodiesel using ethanol, since this alcohol is produced on a large scale in the country. Ethanolysis process produces a biodiesel less damage to the environment than that produced by

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methyl alcohol, since ethanol is derived from sugar cane or corn. In the rest of the world, the production takes place mostly in the methyl route and with use of heterogeneous catalysts (Pighinelli, 2010).

Biodiesel is highly biodegradable in fresh water as well as in soil and great part of it is mineralized until 28 days under aerobic or anaerobic conditions (Makareviciene&Janulis, 2003; Pasqualinoet al., 2006; Zhang et al., 1998). It is also a carbon-free fuel, as the plants that serve as raw material for its production absorb more carbon than which is released during the burning of this biofuel (Antolinet al., 2002; Lang et al., 2001; Sharma et al., 2008; Vicente &Martìnez, 2004).

Moreover, when biodiesel is burnt in diesel engines the emissions of hydrocarbons, carbon monoxide, particulate matter and sulphur dioxide are reduced with the exception of nitrogen oxides, emission increases due to the oxygen content of biodiesel (Canakciet al., 2006; Labeckas&Slavinskas, 2006; Turrio-Baldassarry, 2004).

Biodiesel sold today is still considered expensive, since the production costs involved are influenced by the raw materials, which are vegetable or animal fat and oils. It is estimated that approximately 80% of the total cost of biodiesel production is related to the acquisition of triglycerols source (Pighinelli, 2010). Another problem that has been a subject of discussion frequently is the competition between "food and energy production". Some researchers argued that, there will be food shortages if the available land is used for oilseed cultivation.

In order to reduce the production costs and to make it competitive with petroleum diesel, biodiesel producers should use a raw material readily available in their territory. Also, search for alternative crops/sources, such as non-edible oils, as CrambeAbyssinica, JatrophaCurcasand others, waste frying oils,and oil from palm oil mill effluent(POME) (Marchettiet al., 2007).

Thus, this article captured the production of biodiesel from palm oil mill effluent at different catalyst concentrations, under the influences of bio-oil to ethanol ratio, reaction time and temperature. Quality assurance of biodiesel in relation to its production as well as some post-production parameters was given proper considerations.

II. MATERIALS AND METHODS

Palm oil mill effluent (POME) was obtained from a local palm oil factory in Ogbomoso(8.1227° N, 4.2436° E) area. Ethanol, KOH and n-hexane of analytical grades were purchased from LAB-TRADEchemical storeOritaNaira area,Ogbomoso. The materials were taken to the Chemical Engineering Department'slaboratory,LadokeAkintola University of Technology where the experiments were performed.

The oil extraction was done usingsoxhlet apparatus of 250cm^3 capacity with n-hexane as solvent. The raw POME was added to solvent with ratio 3:1 (240:80 ml) in a quick fit bottle for 60 minutes and at a temperature of 75^{0}C which is above the boiling point of n-hexane, then allowed to stand for 10 minutes and the upper layer (bio-oil) was decanted into a conical flask through a filter paper. (Babatundeet al. 2016)

Preparation of homogeneous catalyst

Potassium ethoxide was prepared according to Babatundeet al. (2016) method, whereby 0.25 g of KOH was weighed using an electronic weighting balance into a plastic container through funnel and 2.5ml of ethanol was carefully added using another funnelto form potassium ethoxide.The mixture was reflux at 80˚C for 1 hour for methanol to be completely dissolved.Biodiesel was produced according to the procedure shown in the figure 1.0.

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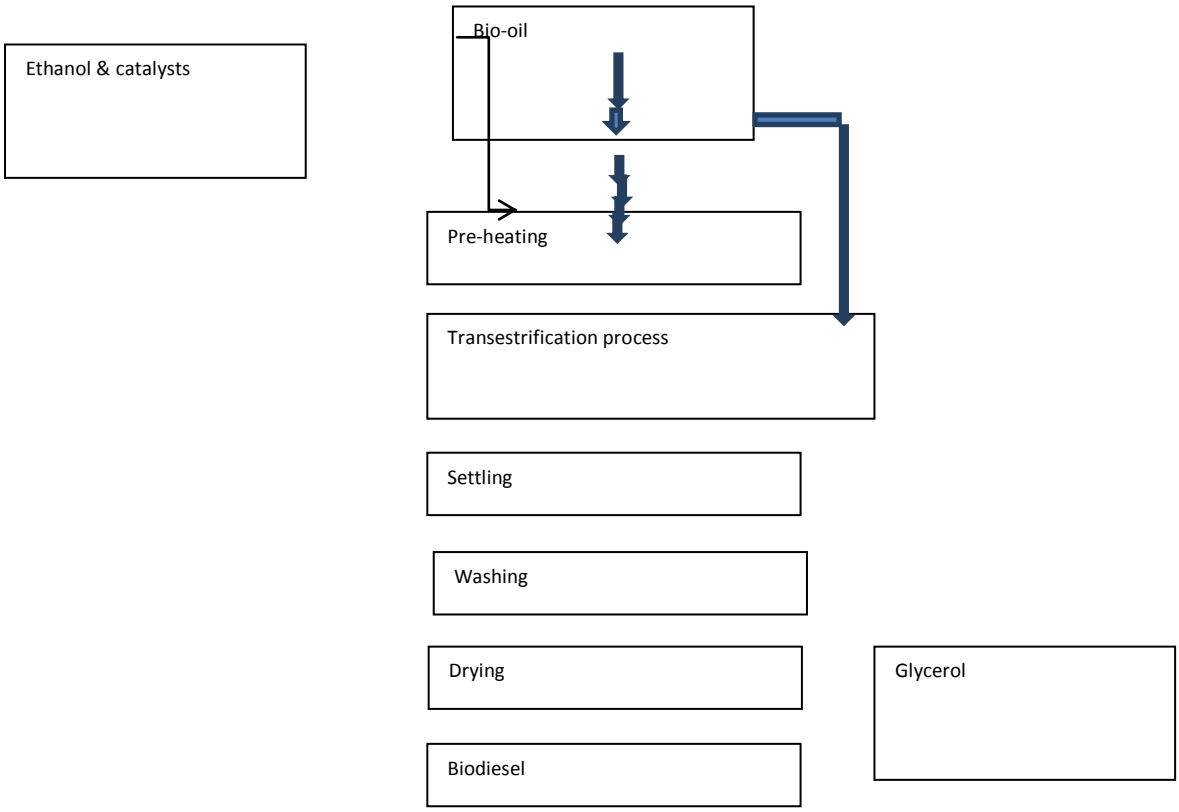

Figure 1.0: Flow chart of Biodiesel production process.

The final mixture was centrifuged at 800 rpm for 10 minutes which was used for transesterification process.

Batch process of biodiesel production

Transesterification method employed by Endalew, Yohannes and Rolando (2011) was adopted here for biodiesel production, using a three – necked round bottom flask glass reactor system as a batch reactor on a magnetic stirrer. The condenser was fixed at the center of the reactor; the agitator was placed inside the flask and the thermometer at the left hand side. 150ml of the bio-oil was measured and pre-heated in an electric mantle to about 65^{o}C and set aside for the transestrification reaction.

A prepared homogeneous catalyst was poured into the three- necked round bottom flask and a prepared 150 ml of bio-oil was carefully added then placed on a magnetic stirrer at 1000 rpm for 60 minutes.

The resulting mixture was allowed to settle for 48 hours inaseparating funnel. The biodiesel obtained was thoroughly washedusing distilled water of half the volume of the biodiesel produced. The washed biodiesel was dried in oven so as to eliminate the traces of water. The remaining biodiesel productions processes were carried out for catalyst concentration 0.5, 0.75 and 1.0g of KOH.

Characterization of Biodiesel Produced

The following fuel properties:Specific gravity, cloud point, pour point and viscosity of the biodiesel produced were carried out at each catalyst concentration and the results were compared with ASTM standards.

Determination of specific gravity

Specific gravity was determined according to ASTM D 1298 using a thermometer, the temperature of the sample was determined. An empty bottle was weighed (W_1) using electric balance; thereafter a measured volume of the sample was poured into density bottle and weighed (W_2). Also an equal volume ofdistilled water was weighed as (W_w). Thus, specific gravity of the sample was given by equation 1 (Daniyanet al., 2015).

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$$S.G = \frac{W_2 - W_1}{W_w - W_1} \quad (1)$$

Where: W_1= Weight of empty density bottle, W_2 = Weight of density bottle filled with sample
W_w= Weight of density bottle filled with water.

Determination of cloud point

Cloud point which is the temperature at which the fuel began to solidify according to ASTM D 2500. It isimportant for low temperature operations of fuel. The temperature of the biodiesel produced was taken using a low temperature thermometer and poured into test sample bottles before being transferred into a refrigerator. The bottles were removed from the refrigerator at an interval of five (5) minutes to measure the temperature at which the sample solidifies.

Determination of pour point

According to ASTM D 97 procedure, the pour point was determined at the temperature where oil ceases to flow as a result of cooling thereby resulting into an increase in the size and number of wax, which eventually change from solid to liquid state. The biodiesel produced having been solidified was removed from the refrigerator and the temperature at which it finally turns to liquid was measured as the pour point.

Determination of viscosity

According to ASTM D 445 procedure, viscosity was determined using viscometer tube and a stop watch. The biodiesel samples were placed on the base of the viscometer as shown in plate 3.4, while a sensing element named spindle was immersed in the fluid and driven at constant rotational frequency. The viscosity is related to the torque generated by the fluid resistance to the induced movement and is hence determined by measuring the tightening of the spiral spring attached to the spindle. The viscosities of the each sample were displayed on the display unit of the equipment.

Engine Performance Test for the Optimum Biodiesel Yield Produced.

The following engine performance test carried at the automobile laboratory of the mechanical engineering department, LAUTECH;Torque (Nm), Rotational Speed (RPM), Exhaust Temperature (^oC) and Power (W)

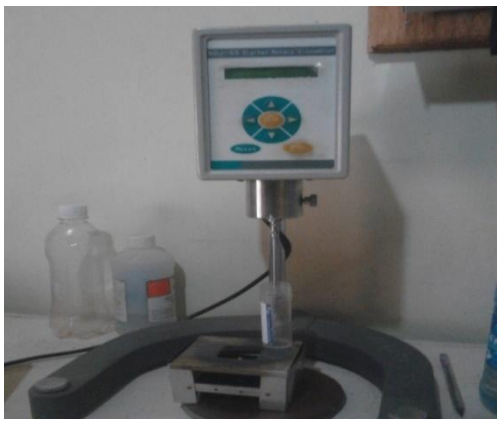

Plate1: A viscometer at the chemical laboratory, LAUTECH

Measurement of torque

Engine torque was measured by the TD115 hydraulic dynamometer and transmitted to a torque meter located on the TD114 instrumentation unit as shown in plate 2. A water brake creates a resistance by circulating water which acts as the load between a rotating impeller in a stationary shell while an electric dynamometer generates and absorbs direct current. In each case, the element that exerts the restraining influence is freely cradled so that its tendency to rotate with the rotating body can be arrested and the arresting force at a long distance from the axis of rotation. The torque is the product of spring load or weight and the distance from the axis of rotation. For each sample of biodiesel poured into diesel engine, the torque developed was indicated on the torque meter on the instrumentation unit.

Measurement of rotational speed

The engine speed was measured electronically by a pulse counting system. An optical head mounted on the dynamometer chassis contains an infra-red transmitter and receiver. A rotating disc with radial slot is situated between the

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optical source and the sensor. As the engine and slotted disc rotates, the beam is interrupted. The resulting pulse train is electronically processed to provide a readout of engine speed. For each sample of biodiesel poured into diesel engine, the rotational speed of the shaft was indicated on the rotational speed meter located on the instrumentation unit.

Measurement of exhaust temperature

Exhaust gas temperature was measured by chrome/alumel thermocouple conforming to BS1827 standard. The thermocouple is located in a (1/8'') BSP union brazed into the exhaust pipe close to the cylinder block of the engine. Colour coded leads from the thermocouple were connected to terminals underneath the TD114 instrumentation unit. The temperature is indicated on a direct reading meter scaled from 0 to 1000 ^{o}C. For each sample of biodiesel poured into diesel engine, the exhaust temperature obtained from the exhaust manifold was indicated on the exhaust temperature meter located on the instrumentation unit.

Plate 2: A TD115 hydraulic dynamometer coupled to a TD114 instrumentation unit

Measurement of power output

The power output is calculated from the torque by multiplying by the angular velocity in radians per second. Because the dynamometer acts as a brake on the engine, the power at the output shaft is referred to as the "Brake Power" (Altinet al., 2001).

$$P_B = \frac{2\pi NT}{60} \quad (2)$$

Where: P_B = Brake Power, N= Rotational Speed and T= Torque

III. RESULTS AND DISCUSSION

Result of Oil Yield from the Extraction of POME

The percentage oil yield was calculated as shown below.

Volume of extracted oil = 1800 ml, Volume of POME = 4800 ml.

$$\text{Percentage oil yield} = \frac{1800 \times 100}{4800} = 37.5\% \quad (3)$$

The result of the solvent method of extraction of oil from POME is as shown above. The volume of extracted oil of 1800 ml was obtained from 4800 ml of POME using n-hexane as a solvent. The percentage oil yield obtained at the corresponding feed/solvent ratio of 3:1, extraction temperature of 75 ^{o}C and extraction time of 60 minutes was 37.5%.

This is a good yield as compared to rapeseed of 25.02 % of dry massas reported by Ying Li et al., (2014).

Result of Biodiesel Produced from the Extracted Oil at Various Catalyst Concentrations

The production of biodiesel from the POME extracted oil usingconstant reaction temperature of 65 ^{o}C, bio-oil volume of 150 ml, ethanol volume of 25 ml, reaction time of 60 minutes and various catalyst concentration of 0.25, 0.50, 0.75, 1.0 g gave a biodiesel yield of 59.9, 61.3, 65.9 and 54.3% respectively at an average of the three runs (Table 1).This shows that biodiesel yield was optimal at a catalyst concentration of 0.75 g.The result is in accordance with the values obtained by Benjamin et al. (2007)who studied the concentration level of potassium hydroxide (KOH)catalyst that gives the optimal yield fraction of 0.67 of biodiesel from tigernut (cyperusesculentus) oil. This value was obtained as 0.9 % weight (in gram) of catalyst per volume of transesterified.

Hence, any increase above this optimal catalyst concentration does not necessarily result in an increase in biodiesel yield but could rather

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increase the cost of production of biodiesel in terms of purification. The losses accounted for were obtained during the washing process as emulsion and un-reacted reagents.

Table 1: Results of Biodiesel Produced at Various Catalyst Concentrations

Experimental Conditions	Catalyst Concentration(g)			
	0.25	0.50	0.75	1.00
Reaction temperature (^{o}C)	65	65	65	65
Volume of bio-oil (ml)	150	150	150	150
Volume of ethanol (ml)	25	25	25	25
ReactionTime (minutes)	60	60	60	60
Biodiesel obtained (ml)	**105**	**107.5**	**115.8**	**95.5**
Glycerol obtained (ml)	65.05	63.5	56.7	77.75
Losses (ml)	5.2	4.5	3.25	2.75
Biodiesel yield (%)	**59.9**	**61.3**	**65.9**	**54.3**

Physicochemical Properties of the Biodiesel Produced at Various Catalyst Concentrations.

In characterizing the biodiesel produced in this research work, the important physicochemical properties considered were viscosity,specific gravity, relative density, pour point andcloud point. The results obtained are presented in table 2 below:

Kinetic Viscosity @ 40^{0}C

Kinetic Viscosity measures the resistance to flow of liquids, it is found to be decreasing at the instance of increasing in temperature, and it calls for the temperature specification at which a particular substance is being measured. Unfavourable pumping and inefficient mixing of fuel with air which in turn contribute to incomplete combustion were found to have caused by high viscosity of vegetable oil. Hence, vegetable oil must be modified to bring its combustion related properties closer to those of diesel oil. Therefore, such fuel modification is mainly aimed at increasing the volatility and reducing the viscosity (Babatunde et al., 2019). In this research work, the feedstock increased with increase in catalyst concentration, this is found in accordance with Godwin et al., 2015 who determined the effect of biodiesel production parameters on viscosity and yield of methyl esters: Jatrophacurcas, Elaeisguineensis and Cocosnucifera.

Specific gravity at 15^{0}C

Specific gravity measures the ratio of density of a substance to the density (mass of the same unit volume) of a reference substance. Hence,it varies with temperature and pressure, specific gravity of produced POME biodiesel at different catalyst concentrations are 0.897, 0.977, 0.885 and 0.845g/mL respectively, this is in agreement with specification of ASTM D1298 for biodiesel according toHamza, 2008who obtained 0.923g/mL for red Sesamumindicum L. seed oil and Babatundeet al.(2019) who obtained specific gravity of 0.85g/mL forChrysophyllumalbidum seed oil.

Pour point

This is the temperature at which oil cease to flow: this occurs most when there is change from liquid to solid state. The pour point values of -5.1, -6.4, -2.1 and -8.2 obtained for palm oil mill effluent oil at different catalyst concentrations were found to be within the limit set by ASTM D 97 of range -15 to 10^{0}C (Tint and Mya, 2009). This is also in accordance to the work of Babatundeet al. (2019) who obtained 2.14% of raw Chrysophyllumalbidum seed oil after adopting ASTM D 97 standard.

Cloud point

This is the temperature at which dissolved solids are no longer completely soluble or precipitating as a second phase which gives the fluid a cloudy appearances. Krukaet al., 1995explained explicitly that ASTM procedure for cloud point determination are not applicable to dark crude oil and also do not account for potential sub-cooling of the wax. From the analysis done to determine the cloud point of POME biodiesel at different catalyst concentrations are as follows 3.0, 2.5, 6.0 and 7.0 ^{o}C respectively. This is found to be withinthe range of -3-120C specified by ASTM D2500 of D6751 for biodiesel production (Edgar et. al., 2005).

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Table 2: Physicochemical Properties of the Biodiesel Produced at Various Catalyst Concentrations.

	Catalyst Concentration (g)			
Property	0.25	0.5	0.75	1.00
Viscosity (PaS)	9.773	9.763	9.660	9.758
Specific Gravity	0.897	0.877	0.885	0.845
Pour Point (^oC)	-5.1	-6.4	-2.1	-8.2
Cloud point (^oC)	3.0	2.5	6.0	7.0

Results of Engine Performance Test for the Biodiesel Produced on a Dynamometer

In a bid to analyze the POME biodiesel produced further, table 3 shows engine performance tests such as: engine torque, exhaust temperature, rotational speed and power using a dynamometer, that were obtained under optimum condition of biodiesel produced under exhaust temperature of 250^0C and 24.70 W Power generated.

The optimum biodiesel yield was obtained at rotational speed of 2500 rpm, which was found to be in accordance with Wail and Khaled (2012) who obtained satisfactory performance at engine speed between 1200-2600 rpm using biodiesel extracted from waste cooking oil at steady state on a four stroke single cylinder diesel engine without any modification.

Table 3: Engine Performance Test for the Optimum Biodiesel Yield Produced

Biodiesel Yield (%)	Torque (Nm)	Exhaust Temperature (^oC)	Rotational Speed (RPM)	Power (W)
65.9	42.4	450	2500	24.70

Sugozu et al., (2011) arrived at engine torques of 35.22 Nm and 34.48 Nm are observed for diesel fuel and biodiesel fuel respectively at 1800 rpm engine speed when considering Biodiesel production from animal fat–palm oil blend and performance analysis of its effects on a single cylinder diesel engine. The result obtained here is in accordance with the results obtained above. The increase in value is due to increase in agitation speed of the experiment performed.

IV. CONCLUSION

Biodiesel was produced and found to be increasing in yield with increase in catalyst concentration until the optimal yield was obtain. The characterized POME biodiesel produced was found to be in quality with standard specified by ASTM. The limited performance tests carried out showed that POME biodiesel can successfully fuel a diesel engine becauseless power output are obtained from the POME biodiesel compared to the petroleum diesel.

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