

# Optimal Transesterification Duration for Biodiesel Production from Nigerian Palm Kernel Oil

<sup>1</sup>Alamu, O. J., <sup>2</sup>Waheed, M. A., <sup>3</sup>Jekayinfa, S. O. and <sup>4</sup>Akintola, T.A.

<sup>1</sup>Department of Mechanical Engineering, Olabisi Onabanjo University, Ibogun Campus, Ogun State, Nigeria

<sup>2</sup>Department of Mechanical Engineering, Ladoke Akintola University of Technology, Ogbomosho, Nigeria

<sup>3</sup>Department of Agricultural Engineering, Ladoke Akintola University of Technology, Ogbomosho, Nigeria

<sup>4</sup>Degree Foundation Programme Unit, College of Engineering and Technology, Olabisi Onabanjo University, Ibogun Campus, Nigeria  
tolasum@yahoo.com

## ABSTRACT

The Energy Commission of Nigeria's long term (2016-2025) plan on the nation's energy requirements is completely non-fossil. This is borne out of the global depletion of the non-renewable energy sources, its attendant negative environmental impact and the intent to utilize the hitherto neglected abundant renewable resources in Nigeria. Locally produced Palm kernel oil (PKO) is currently being investigated as an alternative source of diesel fuel amongst other efforts at executing the country's renewable energy master plan. Transesterification of PKO with ethanol was carried out using potassium hydroxide (KOH) catalyst on a laboratory scale and standard fuel characterization (ASTM fuel tests) was carried out on the resulting biodiesel. This work further considers the effect of varying mixing time on PKO biodiesel yield. Three replicated transesterification experimental runs were carried out for each of the mixing duration 30, 45, 60, 75, 90, 105 and 120min under identical typical transesterification reaction conditions of 100g PKO, 60°C temperature, 1.0% KOH concentration and 20% ethanol (wt% PKO). Results showed PKO biodiesel fuel properties (viscosity, specific gravity, cloud point, pour point and flash point) to be within international biodiesel standard specifications while average PKO biodiesel yield of 87.4, 90.1, 92.5, 94.2, 96.0, 96.0 and 96.0% was obtained for the respective mixing duration. Optimal yield of the PKO biodiesel, within the constraint of the typical transesterification process parameters used, was found to be 90min.

**Keywords:** Palm kernel oil, biodiesel, renewable fuel, transesterification, optimum duration, Nigeria.

## 1. INTRODUCTION

Recent estimates ranked Nigeria as the 11th largest oil producer in the world with 36 billion barrels oil reserves (USEIA, 2007). Also, global ranking by the US Energy Information Administration places Nigeria as having the seventh largest natural gas reserves in the world, with an estimated 182 trillion cubic feet of proven natural gas reserves (Alamu *et al.*, 2007a). Enormous as these energy sources appear, the global consensus is that fossil energy sources are

finite (Sambo, 1981; Fluck, 1992; Jekayinfa and Bamigboye, 2004; Eleri *et al.*, 2005). This is evident from available statistics on oil reserves of world oil producers.

Table 1. World oil producers with declining oil reserves

Country	Oil (billion barrels)	
	Year-end 2005	January 1, 2007
Mexico	13.670	12.352
United States	29.922	21.757
Ecuador	5.060	4.517
Peru	1.097	0.930
Trinidad and Tobago	0.809	0.728
Italy	0.731	0.600
Norway	9.691	7.849
United Kingdom	3.998	3.875
Kazakhstan	39.620	30.000
Russia	74.436	60.000
Iran	137.490	136.270
Oman	5.572	5.500
Saudi Arabia	264.211	262.300
Syria	3.000	2.500
Angola	9.035	8.000
Congo (Brazzaville)	1.784	1.600
Egypt	3.720	3.700
Equatorial Guinea	1.765	1.100
Gabon	2.205	2.000
Sudan	6.405	5.000
Tunisia	0.681	0.400
Australia	4.045	1.592
Brunei	1.120	1.100
China	16.038	16.000
India	5.919	5.625
Indonesia	4.301	4.300
Malaysia	4.200	3.000
Thailand	0.527	0.290
Vietnam	3.119	0.600

Source: USEIA,2007

As revealed in Table 1, from 2005 till date, 29 oil producing countries are already experiencing declining oil reserves (USEIA, 2007). Before Nigeria's inclusion in such list; the country is already initiating policies aimed at re-directing the nation's major energy sources from the finite crude oil to renewable sources. The various capacities of Nigeria's renewable resources have been reported (Eleri *et al.*, 2005; Alamu *et al.*, 2007b).

Vegetable oil is an important renewable feedstock in the long-term (2016-2025) vision of providing secure, abundant, cost effective and clean source of energy for Nigeria. Common vegetable oil in the country include palm oil, palm kernel oil (PKO), peanuts, cottonseed and soybean. Profile for these vegetable oils is presented in Table 2 (David, 2006).

Table 2. Nigeria vegetable oil profile (2006)

Commodity	Quantity (tons)	Percentage Share
Palm oil	800,000	50
Palm kernel oil	270,000	17
Others: peanuts, cottonseed, soybean	260,000	16
Imports	270,000	17
National requirement	1,600,000	100

Source: David, 2006

As evident from Table 3, PKO production in Nigeria rose from 272,000 tons in 2004 and 2005 to 275,000 tons in 2006. Industrial underutilization of this commodity is also evident as only 86,000 out of 275,000 tons produced in 2006 was utilized for industrial domestic consumption. Its industrial use has however been limited to soap, detergent, baker's fat and margarine. Soap manufacturers in the country are, even, increasingly turning to imported tallow. Successful reports on transesterification of some Nigerian lauric oils in the preparation of biodiesel is an indication of better industrial utilization of PKO in Nigeria, as considerable research efforts are now focusing on this alternative diesel fuel worldwide (Abigor *et al.*, 2000; Darnoko and Cheryan, 2000; Munack *et al.*, 2001; Yuan *et al.*, 2004; Conceicao *et al.*, 2005; Chitra *et al.*, 2005; Knothe and Steidly, 2005; Best, 2006; Gupta *et al.*, 2007; Singh *et al.*, 2007).

Table 3. Nigeria's Palm kernel oil profile

Commodity	USDA	Revised	USDA	Revised	USDA	Revised
	Estimate (2004)	Estimate (2004)	Estimate (2005)	Estimate (2005)	Estimate (2006)	Estimate (2006)
(1000 tons)						
Beginning stock	10	10	10	10	10	10
Production	272	272	272	272	275	275
Imports	1	1	1	1	1	1
Total supply	283	283	283	283	286	286
Exports	1	1	1	1	1	1
Industrial domestic consumption	86	86	86	86	86	86
Food use domestic consumption	186	186	186	186	189	189
Total domestic consumption	272	272	272	272	275	275

Source: David, 2006

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Biodiesel, described as a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats is oxygenated, essentially free of sulphur and biodegradable (Yuan *et al.*, 2004). It has also been widely reported to be characterized with reduced exhaust emissions, reduced toxicity and improved lubricity, higher flash point, lower vapour pressure and positive energy balance (Margaroni, 1998; Knothe and Steidly, 2005; Best, 2006). It can be used in the pure form, or blend in any amount with diesel fuel for use in compression ignition engines (Ajav and Akingbehin, 2002; Krahl *et al.*, 2005).

In the present study, Nigerian PKO biodiesel was prepared through transesterification of PKO with ethanol using KOH catalyst (Fig.1) on a laboratory scale as previously reported (Alamu *et al.*, 2007b). However, with considerable works, which have been published on the optimization of various transesterification process variables such as feedstock proportions, reaction temperature, reaction time, catalyst amount and agitation rate, as earlier reported (Chitra *et al.*, 2005; Alamu *et al.*, 2007b,c); it becomes imperative to extend the present study to some economic considerations.



Fig. 1. Transesterification process

In the optimization of methyl esters of *Jatropha curcus* oil in India, Chitra *et al.* (2005) considered reaction time range 30-120min. They found out that the biodiesel yield increased with reaction time with maximum yield occurring at 90min reaction time. Only limited reports are available on this concept, while none has been found in the literature on PKO biodiesel. This work therefore studies the effect of transesterification reaction duration on PKO biodiesel yield in the KOH-catalysed laboratory production of biodiesel from Nigerian PKO, with a view to identifying the optimum reaction time.

## 2. MATERIALS AND METHODS

### 2.1 Experimental

Transesterification process for PKO was carried out using the DFP Laboratory equipment of the Olabisi Onabanjo University, Ibo-gun, Nigeria. Values of process parameters selected (Table 4) are within ranges, which have produced successful results from earlier reports (Ma *et al.*, 1998; Alacantara *et al.*, 2000; Alamu *et al.*, 2007b). Locally produced PKO was purchased at Ogijo, Nigeria. Ethanol used, manufactured by Aldrich Chemicals was analytical grade, 99.9% pure with a boiling point of 78°C. KOH used was also an analytical grade, manufactured by Aldrich Chemicals. Other materials include a dry and wet mill blender with a clear glass (1,250 capacity) containers and stainless steel cutting blades, thermometer, pet bottles, duct tape, funnel, scales, measuring beakers and translucent white plastic container with bung and screw-on cap.

Table 4. Transesterification process parameters

Experimental conditions	Values for all the experimental runs
Palm kernel oil (PKO) quantity (g)	100
Ethanol quantity (g)	20.0
Reaction temperature ( $^{\circ}\text{C}$ )	60
KOH concentration* (%)	1.00
Transesterification duration (min.)	Varies with experimental batches (1-7)

\*(wt% PKO)

For the first batch of the experiment, 20.0g of ethanol was measured and poured into a plastic container. 0.5g of KOH was carefully added to the plastic container. The bung and the screw on the cap were replaced tightly. The container was swirled round thoroughly for about 2 minutes repeatedly about six times to enable the KOH completely dissolve in the ethanol. 100.0g of PKO was measured out in a beaker, pre-heated to  $60^{\circ}\text{C}$  and poured into the blender. The prepared potassium ethoxide from the plastic container was carefully poured into the PKO, the blender lid was secured tightly and the blender switched on. Agitation in the blender was maintained for 30minutes. The reaction mixture was poured from the blender into a PET bottle and allowed to stand overnight while phase separation occurred by gravity settling. The PKO biodiesel phase was carefully decanted from the equilibrium mixture into a PET bottle. Excess ethanol and residual catalyst were washed from the biodiesel using water as earlier detailed and reported (Alamu *et al.*, 2007b,c). The procedure was replicated three times and average yield of the biodiesel and glycerol was evaluated.

## 2.2 Variation of Transesterification Duration and Fuel Characterization

Additional experiments were conducted to study the effects of transesterification reaction duration on the PKO biodiesel yield using the same process parameters in Table 4, with reaction time varied between 30-120 min. at 15 min. step. Some physical properties of the biodiesel fuel produced were tested together with commercial grade Philips diesel fuel purchased from a fuel station at Ifo, Nigeria. Specific gravity and viscosity measurements were made using the Thermal-Hydrometer apparatus and Viscometer respectively, following ASTM standards D1298 and D445. Cloud and pour points were determined using Baskeyl Setapoint cloud and pour point apparatus following ASTM standards D25100-8 and D97 respectively. Detailed procedures for these tests are available elsewhere (ASTM, 1995).

## 3. RESULTS AND DISCUSSION

The PKO biodiesel prepared using 30 min. reaction time yielded 87.40g PKO biodiesel and 32.40g glycerol, with 1.2g losses. These losses are obviously some un-reacted alcohol, residual catalyst and emulsion removed during the washing stage of the production process. Similar observation has been reported in the alkali-catalysed transesterification of *Jatropha curcus* oil (Chitra *et al.*, 2005). The results stated are averages of three different experimental runs. Detailed results for each of the experimental runs are presented in Table 5.

Table 5. Biodiesel production results for batch 1 experiment (reaction time = 30min)

Experimental Conditions	1 <sup>st</sup> Run	2 <sup>nd</sup> Run	3 <sup>rd</sup> Run	Average
Transesterification duration (min.)			30	
PKO biodiesel obtained (g)	88.20	87.50	86.50	87.40
Glycerol obtained (g)	32.40	32.30	32.50	32.40
Losses (g)	0.40	1.20	2.00	1.20
PKO biodiesel yield (%)	88.20	87.50	86.50	87.40

### 3.1. Optimal Transesterification Duration

With the reaction time increased to 45 min (batch 2 experiment), an improved biodiesel yield of 90.10% was obtained, with 30.0% glycerol and 0.9% losses as presented in Table 6. This trend continued with reaction time 60 min (batch 3 experiment) and 90 min (batch 4 experiment) with respective biodiesel yield of 92.5% and 94.2% (Table 6). This implies that within the temperature range 30 – 90 min., PKO biodiesel yield increased with reaction time; an observation earlier made by Chitra *et al.* (2005) for *Jatropha curcus* biodiesel and Yuan *et al.* (2004) for soybean biodiesel. Beyond 90 min reaction time however, there wasn't any significant change in the biodiesel yield. As evident from Table 6, PKO biodiesel yield neither increased nor decreased with reaction time between 90 min and 120 min. Hence, the minimum reaction time corresponding to the maximum process yield was 90 min.

Table 6. Three-run average results of biodiesel production for 7 batches of experiments (30-120min.)

Experimental conditions	Batch						
	1	2	3	4	5	6	7
Transesterification duration (min)	30.00	45.00	60.00	75.00	90.00	105.00	120.00
PKO biodiesel obtained (g)	87.40	90.10	92.50	94.20	96.00	96.00	96.00
Glycerol obtained (g)	32.40	30.0	27.20	25.40	23.40	23.70	22.00
Losses (g)	1.20	0.90	1.30	1.40	1.60	1.30	2.00
PKO biodiesel yield (%)	87.40	90.10	92.50	94.20	96.00	96.00	96.00

Once the maximum biodiesel yield has been achieved at 90 min reaction duration, it may no longer be cost effective for the transesterification reaction to be continued. Fig.2 reveals the situation at close to and slightly beyond the optimum reaction time; no biodiesel yield increase was evident. This shows that the effect of reaction time on KOH-catalysed transesterification of PKO with ethanol does not agree perfectly with earlier report on NaOH catalysed transesterification of *Jatropha curcus* oil with methanol beyond 90 min reaction time.

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Possible explanation could be the different boiling points of the alcohols involved in each case. While methanol reported in Chitra *et al.* (2005) has a boiling point of 65°C, the boiling point of ethanol used in the present study is 13°C higher. Another possible explanation could be the inherent properties of the particular feedstock.

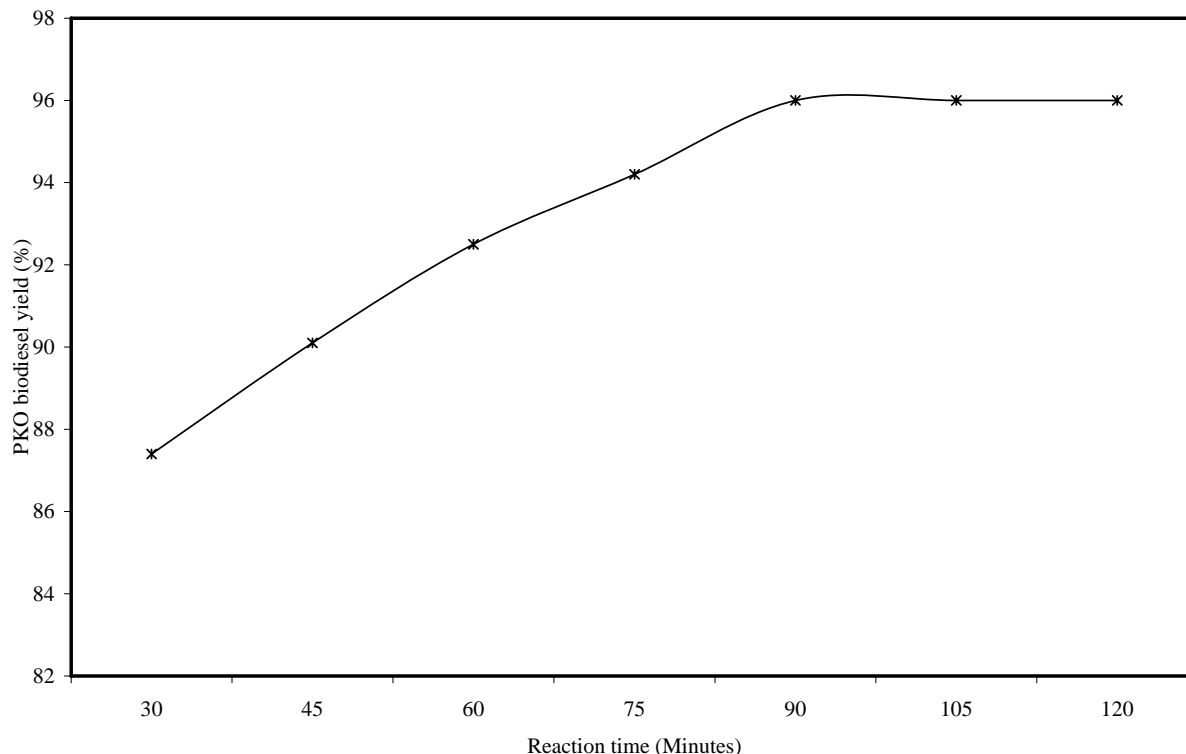


Fig. 2. Variation of transesterification reaction duration with % PKO biodiesel yield

### 3.2 Statistical Analysis of Data

Statistical analysis of the experimental data showed no difference in mean through the analysis of variance (ANOVA). The calculated  $F_{RATIO}$  ( $F_{t(calculated)} = 0.4678$ ) was less than the tabulated ( $F_{t(table)} = 4.76$ ) value at 5% level of significance. It therefore implies that the factor levels (1 to 7) investigated in the experiment has 0.95 probability of generating optimal yield of the PKO biodiesel.

### 3.3 PKO Biodiesel Fuel Characterization and Comparison with Petroleum Diesel and Biodiesel from other Oil Crops

In assessing the suitability of the PKO biodiesel produced as alternative diesel fuel, the PKO biodiesel and the commercial grade fossil diesel, were analyzed for specific gravity at reference 60°F, viscosity at 40°C, pour point, cloud point and flash point following ASTM standard test procedures. Results obtained are presented in Table 7.

Table 7. Measured PKO biodiesel and fossil diesel fuel properties

Fuel Characteristics (Properties)	Values (PKO biodiesel)	(Petroleum diesel)
Pour point ( $^{\circ}\text{C}$ )	2	-16
Cloud point ( $^{\circ}\text{C}$ )	6	-12
Flash Point ( $^{\circ}\text{C}$ )	167	74
Boiling Point ( $^{\circ}\text{C}$ )	320	191
Gross heat of combustion (MJ/kg)	40.56	45.43
Net heat of combustion (MJ/kg)	37.25	42.91
Specific gravity (at $15.56^{\circ}\text{C}$ )	0.883	0.853
Viscosity (at $40^{\circ}\text{C}$ ) ( $\text{mm}^2/\text{s}$ )	4.839	2.847

In agreement with earlier observations made by several authors (Peterson et al., 1990; Graboski and McCormik, 1998) the specific gravity recorded for the PKO biodiesel was higher than the values obtained for the fossil diesel (Table 7). At  $60^{\circ}\text{F}$  reference temperature, specific gravity obtained for PKO biodiesel was 1.034 times that of the commercial fossil diesel. Compared to other ethanol-based biodiesel from other oil crops such as rapeseed, canola and soybean, as presented in Table 8, specific gravity of PKO biodiesel obtained is in good agreement; minimum and maximum deviation being 0.227% and 0.79%. Besides, the specific gravity obtained for the PKO biodiesel falls within the limit specified for biodiesel fuel in Europe (EN14214: 0.86-0.90), Austria (ONC1191: 0.85-0.89), Czech Republic (CSN656507: 0.87-0.89), Germany (DINV51606: 0.875-0.90), Sweden (SS155436: 0.87-0.90) and Italy (UNI10635: 0.86-0.90). (Chitra *et al.*, 2005; Knothe and Steidly, 2005). The level of agreement recorded in specific gravity for the PKO biodiesel is an important pointer to suitability of the biodiesel as diesel fuel substitute as important fuel performance indicators such as cetane number, heating values, fuel storage and transportation are correlated with specific gravity (Yuan *et al.*, 2004; Ajav and Akingbehin, 2002).

Table 8. Specific gravity of PKO biodiesel compared with specific gravity of ethanol-based biodiesel from other oil crops

<i>Ethanol-based biodiesel</i>	<i>Specific gravity at <math>15.56^{\circ}\text{C}</math></i>	<i>% Deviation from S.G of PKO biodiesel at <math>15.56^{\circ}\text{C}</math></i>
Rapeseed ethyl ester	0.876 <sup>a</sup>	0.793
Canola ethyl ester	0.878 <sup>a</sup>	0.566
Beef Tallow ethyl ester	0.869 <sup>a</sup>	1.586
Soybean ethyl ester	0.881 <sup>a</sup>	0.227

a: Peterson, et al. (1990)

Also, from Table 7, PKO biodiesel has higher viscosity than conventional diesel fuel in agreement with reports from several researchers (Yuan *et al.*, 2004; Knothe and Steidly, 2005; Peterson et al., 1990; Graboski and McCormik, 1998). The PKO biodiesel viscosity of 4.839 centistokes obtained is almost twice the viscosity of the fossil diesel as found in alcohol esters of rapeseed, canola, soybean and beef tallow (Van Gerpen, 2004). The value also falls within the specified limits by ASTM D6751, ASTM D975 and BIS (India) standards.



The reported technical implication of higher viscosity of biodiesel is that it decreases the leakages of fuel in a plunger pair and in turn it changes the parameters of a fuel supply process (Lebedevas and Vaisekauskas, 2006).

Higher pour point, cloud point, flash point and boiling point obtained for PKO biodiesel compared to conventional petroleum based diesel as well as lower gross and net heat of combustion obtained for fossil diesel was found to be consistent with earlier findings on such biodiesel fuel like alcohol esters of rapeseed, canola, beef tallow, soybean and midwest biofuel methyl soyate (Abigor *et al.*, 2000; Peterson *et al.*, 1990; Graboski and McCormik, 1998; Schwab *et al.*, 1997).

#### 4. CONCLUSIONS

From the laboratory preparation, testing and optimization of transesterification reaction time of biodiesel from Nigerian PKO studied under typical transesterification reaction conditions of 60°C temperature, 100.0g PKO, 20.0g ethanol, 1.0% catalyst (KOH) concentration and 30-120 min. reaction time, it can be concluded that Nigerian PKO biodiesel gave promising results as alternative diesel fuel with acceptable specific gravity, viscosity, cloud point, pour point and flash point values. For transesterification reaction duration 30, 45, 60, 75, 90, 105 and 120 min investigated, PKO biodiesel yield of 87.4%, 90.1%, 92.5%, 94.2%, 96.0%, 96.0% and 96.0% were respectively obtained. A maximum PKO biodiesel yield of 96% was obtained at a minimum reaction time of 90 min, beyond which, no further increase in PKO biodiesel yield was achieved.

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