

Determination Of Optimum Reaction Temperature And Reaction Time For Biodiesel Yield From Coconut (*Cocos nucifera*) Oil.

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ABSTRACT

The determination of optimum reaction temperature and time for biodiesel yield from Coconut (*Cocos nucifera*) oil was studied. The oil was transesterified using potassium hydroxide (KOH) in methanol as catalyst to obtain the crude biodiesel. For the reaction time, the biodiesel production was carried out at 30 min, 45 min, 60 min, 75 min and 90 min while their temperature (60°C), oil to alcohol ratio (6:1) and catalyst concentration (0.65%) were kept constant. For the reaction temperature, the reaction was carried out at 45°C, 50°C, 55°C, 60°C and 65°C, while their reaction time (60 min), oil to alcohol ratio (6:1) and catalyst concentration (0.65%) were kept constant. The crude biodiesel were purified by washing with water severally to obtain the relatively pure biodiesel. They were dried using solar dryer for a period of four (4) days to obtain moisture-free pure biodiesel. They were subsequently analyzed for various parameters; Kinematic viscosity, acid value, free fatty acid (FFA), specific gravity, iodine value, ash value and flash point. The different variants gave results of the parameters analyzed comparable to International standards of biodiesel (ASTM) methods. The results of the study showed that for the reaction time, the optimum yield was obtained at 45 min giving a biodiesel yield (after purification) of 88% while for the reaction temperature, the optimum yield was obtained at 45°C with a biodiesel yield of 87.4%. General results obtained showed that the optimum yield of biodiesel obtainable from Coconut oil when transesterified using KOH/methanol is about 88%. Overall results indicate that Coconut oil can serve as a veritable feedstock for biodiesel production.

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Keywords: Transesterification, biodiesel production, biodiesel yield, Coconut oil, reaction time, reaction temperature

1. INTRODUCTION

Fuel and energy crises and the concern for the depleting world's non-renewable energy resources have led to a renewed interest in the quest for alternative fuels [1]. Majority of the world energy needs are supplied through petrochemical sources (coal and natural gases) with the exception of hydroelectricity and nuclear energy. Of all these sources that are finite, the current usage rates are expected to be depleted in a couple of decades. Non-renewable energy sources such as petroleum are related to several drawbacks including; increased green house gas emissions, high cost of processing the crude petroleum and energy demand during the process etc. This has provided the incentives to seek alternative sources for the fossil-based fuels [2]. Biodiesel is a fuel produced from the reaction of vegetable oil and fats with alcohol in the presence of a catalyst usually alkali (NaOH, KOH etc.). Biodiesel is becoming an increasingly acceptable alternative to fossil diesel because of the narrowing gap between worldwide oil production and consumption, increasing reality of the Hubbert peak and the imperatives for carbon-neutral fuels [3]. Biodiesel is biodegradable, non-toxic and has low emission profiles when compared to fossil fuel and its usage will allow balance between agriculture, economic development and the environment [4]. Biodiesel is produced through a chemical process known as transesterification. Transesterification of vegetable oils with low molecular weight simple alcohols (methanol, ethanol, propanol, butanol and amyl alcohol) has been established as the best option to reduce the high viscosity, low volatility, heavy engine deposits and toxic substance formation associated with the direct use of vegetable oils [5,6]. A wide variety of feedstock have been identified as suitable for biodiesel production including; soybean, canola, sunflower, safflower, *Jatropha curcas*, peanut, tigernut, coconut etc. [7, 8]. However, some of these oil sources are commodities whose prices are strongly dependent on the International market. Coconut (*Cocos nucifera*), is commonly found in the western part of Nigeria and will find good use as a feedstock for biodiesel production. Coconut oil is a fat consisting of about 90% saturated fat. The oil contains predominantly medium chain triglycerides with 86.5% saturated fatty acids, 5.8% monounsaturated fatty acids and 1.8% polyunsaturated fatty acids [9].

58 **Table 1: Fatty acid composition of Coconut oil**

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Fatty acids	Composition (%)
Lauric	48.0
Myristic	16.0
Palmitic	8.5
Oleic	6.5
Stearic	3.0
Linoleic	2.0

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Source Kincs [9]

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63 Some research works have been carried out using coconut oil for biodiesel production.
64 Alamu *et al.*, [10] considered the use of coconut oil for the production of renewable and
65 environmentally friendly biodiesel as an alternative to the conventional diesel fuel. Test
66 quantities of coconut diesel were produced through transesterification reaction using 100g
67 coconut oil, 20% w/v ethanol, 0.8% potassium hydroxide catalyst at 65°C reaction
68 temperature and 120 min reaction time. The experiment was carried out in triplicates and
69 average results showed that low yield of the diesel (10.4%) was obtained. Mathiyazhagen
70 and Ganapathi [11] found that higher reaction temperature increased reaction rate and
71 shortened reaction time due to the reduction in viscosity of oils. However, Leung and Guo
72 [12] and Eevera *et al.*, [13] found that increase in reaction temperature beyond the optimal
73 level led to decrease of biodiesel yield because higher reaction temperature accelerated the
74 saponification of the triglycerides. Awaluddin and Wahyuningshi [14] studied the optimum
75 conditions of cocodiesel production by methanolysis reaction between coconut oil and
76 methanol using a heterogeneous catalyst (CaCO₃) calcined for 1.5 h at 90°C. Experimental
77 parameters studied include catalyst concentration (1 – 3 wt %) and oil to alcohol ratio (4:1 –
78 12:1). They concluded that the catalyst concentration of 2% and oil to alcohol ratio of 8: 1 at
79 60°C produced the highest conversion of cocodiesel of 75.02%. Jansri and Prateepchaikul
80 [15] studied the comparison of biodiesel production from high free fatty acid crude coconut
81 oil via saponification followed by transesterification or a two stage process. The results
82 obtained showed that the esterification followed by transesterification (TSP) gave lower
83 reaction time (14.25 h), higher yield (86.6% methyl ester) and was cheaper in cost (32.02
84 THB/L) whereas sponification followed by transesterification (SFT) had higher reaction time
85 of 26 h, yield of 45.6% methyl ester and operating cost of 57.09 THB/L. The present work

seeks to determine the optimum reaction temperature and reaction time for improved biodiesel yield from crude coconut oil using the single stage process of transesterification.

2. MATERIAL AND METHODS

2.1. Materials

The fresh, extracted coconut (*Cocos nucifera*) oil was procured from Badagry in Lagos state Nigeria. The methanol (analytical grade, Scharlau Chemie S.A, Sentmenat, Spain) and potassium hydroxide (Kalium hydroxide, Riedel-DeHaeneg, Seelze- Hannover) were used as purchased without further purification. This study was carried out in the National Centre for Energy Research and Development, University of Nigeria Nsukka between April and June, 2012. Other materials used were; 1 liter biodiesel reactor, thermo- regulator heater equipped with stirrer (Heizung Chauffage, MGW- LAUDA, D6970, Lauda- Königshofen, Germany), electronic digital weighing balance (Ohaus, Adventurer, model- AR 3130), separatory flasks, Oven (BTOV 1423), Vecstar furnace LF3, Ferranti portable viscometer model VL, Abbe refractometer (Searchtech Instruments No:820310110289, England), SYD-3536 Cleveland open cup flash point tester (Shanghaichangi Geological Instrument Co. Ltd), A greenhouse type passive solar dryer (constructed at The National Center for Energy Research and Development, UNN), operating at a temperature of 50 – 65°C.

2.2. Experimental procedure

Potassium hydroxide (2.9 g) was added to absolute methanol (125 ml) in a 250 ml conical flask and tightly stoppered. It was swirled and kept overnight to ensure complete dissolution of the KOH in methanol. 500 ml of coconut oil was measured into the 1 liter biodiesel reactor. The oil was heated to 60°C while stirring at the same time. At the set temperature of 60°C, the potassium methoxide was gradually poured in the reactor and stirring continued at a constant speed at the same temperature. For the reaction time study, the stirring time was carried out at reaction times of 30 min, 45 min, 60 min, 75 min and 90 min. For the reaction temperature, the reaction was set at temperatures of 45°C, 50°C, 55°C, 60°C and 65°C. For the two parameters studied, the speed of stirring, alcohol to oil ratio (6:1), catalyst concentration (0.65%) were kept constant. At the end of the reaction time, the mixtures were poured into different separatory flasks and left overnight to bring about gravity separation of the two layers. The lower layer (glycerol) was drawn off carefully leaving the methyl ester upper layer. The crude biodiesel was collected and measured. Their pH was recorded. Purification of the biodiesel was carried out by washing with distilled water (20% by volume of the oil used). The washing was done by pouring the measured quantity of water and

stirring vigorously to ensure proper removal of residual catalyst and methanol including soaps and other impurities. The washing was carried out four times to obtain very clear water. The purified biodiesel was put in the solar dryer for a period of four (4) days to dry, after which the samples were measured and the final yield obtained while their final pH were also taken.

2.3. Physicochemical Analysis

Odour, colour and physical state of the oil was estimated by sensory evaluation. The oil and biodiesel samples were characterized for specific gravity using specific gravity bottle, pH were determined using pH paper, moisture and ash content by AOAC (2010) method [16], kinematic viscosity using a viscometer, refractive index using Abbe refractometer, and flash point using the SYD-3536 Cleveland open cup flash point tester [17]. Other properties analyzed were the saponification values determined by titrimetry using the methods of the Palm Oil Research Institute of Malaysia [18]. Acid value, iodine value, peroxide value and free fatty acid (% FFA as lauric) were determined by titrimetry according to FAO [19]

3. RESULTS AND DISCUSSION

Acid value and free fatty acid of the oil was higher than that observed from some other feedstock including *Jatropha curcas* kernel oil (2.26%, 4.47 mgKOHg⁻¹), *Arachis hypogea* oil (2.61%, 1.31 mgKOHg⁻¹) and *Luffa cylindrica* (2.47%, 1.23 mgKOHg⁻¹). It was however lower than that of *Azadirachta indica* (6.07%, 12.08 mgKOHg⁻¹) [20, 21] (Table 2). Acid value measures the presence of corrosive free fatty acids and oxidation products. This is actually an important variable in considering the quality of oil because the lower the free fatty acid, the better the quality of oil. The acceptable limit for edible oils for human consumption is ≤10 [22]. FFA concentration of the oil was somehow higher than the maximum limit of 2.0% reported for high-grade Codex Alimentarius [23]. Vegetable oils containing high free fatty acids have significant effects on the transesterification with methanol using alkaline catalyst. It also interferes with the separation of fatty acid ester and glycerols [24]. This indicates that the oil would be better converted to biodiesel using the two-stage process of esterification followed by transesterification, saponification followed by transesterification [25, 15] or using heterogenous catalysts [26]. Specific gravity of all the oils was in the range of 0.85–0.93, which is close to the ASTM standard range of 0.87–0.90 for biodiesel [27]. Density and other gravities are important parameters for diesel fuel injection systems. The values must be maintained within tolerable limits to allow optimal air to fuel ratios for complete combustion. High-density biodiesel or its blend can lead to clogging of fuel lines due to poor conversion of

triglycerides to methyl esters [28]. The saponification value was high and therefore implies the possible tendency to soap formation and difficulties in separation of products if utilized for biodiesel production. This would also suggest that using the oils for biodiesel production would lead to low yields in the methyl esters. The peroxide value of the oil was okay indicating that the oil possesses good stability properties and would not easily go rancid as oils high in peroxide value are known to easily go rancid and therefore have short shelf life [29].

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Table 2: Physicochemical properties of the crude coconut oil

Parameters	Results
Viscosity (Cpoise)	11.57
Acid value (mg/KOHg ⁻¹)	9.87
Free fatty acid (%)	4.96
Specific gravity (g/cm ³)	0.922
Iodine value (g/100g)	63.70
Peroxide value (Meq/g)	3.76
Saponification value (mg/KOHg ⁻¹)	240.25
Moisture content (%)	2.00
Ash content (%)	0.009
Flash point (°C)	190

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The flash point of the biodiesel was well within the standard specification of 120°C min (Table 2). This indicates that their use will eliminate the fear of fire outbreaks on storage. The iodine value was also within the standard specified which is also 120 max. High iodine value indicates high un-saturation in oils and fats. This shows that the transesterification took care of the unsaturation in the oil. The saponification value was lower than that obtained for peanut oil (224.74 and 218.09 mg/KOH) and from jatropha oil (193.55 mg/KOH) as reported by Arkbar *et al.* [30]. The ash content was lower than the maximum limit indicating that the biodiesel would leave minimal or no deposits during combustion in the exhaust pipe. The specific gravity was well within the standard limit. The free fatty acid was high indicating that the two stage process of esterification or saponification before transesterification may be the preferred production route for this oil type.

Table 3: Biodiesel physicochemical properties for the Reaction temperature and time variants

Parameters	Results	ASTM D6751 - 20
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Kinematic viscosity (mm^2/g)	5.31	1.9 – 6.0
Acid value (mg/KOHg^{-1})	2.31	0.8 max
Free fatty acid (%)	1.22	-
Specific gravity (g/cm^3)	0.8687	0.875 – 0.9
Iodine value (g/100g)	72.514	120 max
Saponification value (mg/KOHg^{-1})	126.225	-
Moisture content (%)	0.85	0.05
Ash content (%)	0.014	0.02
Flash point ($^{\circ}\text{C}$)	123	120 min

The result of the experiment described the effect of reaction temperature and time on the biodiesel yield of coconut oil. Figures 1 and 2 show the biodiesel yields at those different parameters. Table 3 shows the biodiesel yield properties. The difference between the yield of the crude biodiesel and the purified samples indicate a lot of impurities like the KOH, residual methanol, glycerol and some un-reacted materials in the biodiesel which was washed out. The final yield after purification showed that the conversion of oil to methyl ester were not too high.

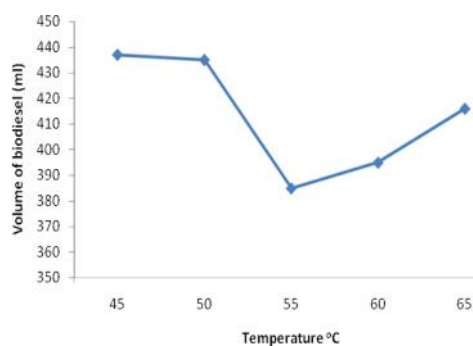
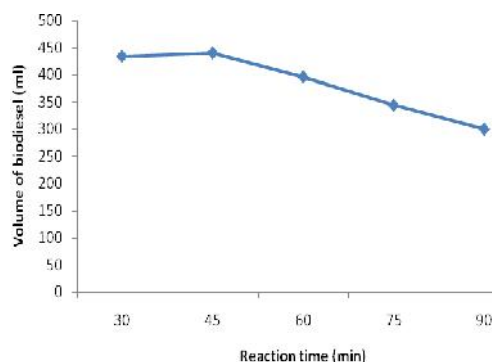


Fig. 1: Biodiesel yield at different temperatures

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210 **Fig. 2: Biodiesel yield at different reaction times.**

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212 From Fig. 1, it was observed that the product volume reduced after 45°C and dipped further
 213 at 55°C but however started increasing from 60°C. It has been reported that at high
 214 temperatures of 70°C and above, the methanol has the tendency to evaporate except if the
 215 reaction is carried out under reflux conditions. This may be explored to investigate the
 216 suitability of higher temperatures for higher yields of the product, although costs can also be
 217 a very important consideration here. In Fig. 2, the reverse was the case. There was a slight
 218 increase in volume between 30min and 45min but a steady decline in product volume from
 219 then till 90min. It is clear therefore that increase in reaction time would not yield further
 220 volume increase in biodiesel obtainable from coconut oil under the prevailing conditions of
 221 reaction. This could be explained from the viewpoint of the reversible nature of the
 222 transesterification process as reported by Darnoko [31]. Reaction times greater than 45mins
 223 may have favoured the backward reaction- the formation of glycerine, hence the yield of
 224 methyl ester at higher reaction times gave lower volumetric yield of product.

225 Some fatty substances observed during the purification by water washing lend credence to
 226 the high free fatty acids found in the oil analysis. Reports on biodiesel production from some
 227 other oil sources have obtained yields above 90%, but that of coconut oil in this study were
 228 below 90%. For instance, Highina et al. [2] reported 97% biodiesel yield from *Jatropha*
 229 *curcas*, while Meka et al. [32] reported 96.85 biodiesel yield from safflower. A test of the pH
 230 of the oil and biodiesel showed that it was slightly acidic and may contain high levels of free
 231 fatty acids. This also indicates that the conversion efficiency was poor and may require the
 232 two stage transesterification process.

233 However, Alamu *et al.*, [10] looked at the single stage transesterification of coconut oil using
 234 ethanol (20%), KOH (0.8%) at 65°C and at reaction time of 120 min. The yield obtained was
 235 10.4%. The report by Awaluddin and Wahyuningshi [14] obtained the yield of 75.02% from
 236 coconut biodiesel using methanol and CaCO₃. Again, the work of Jansri and Prateepchaikul

[15] on high free fatty acid coconut comparing the two stage methods showed that the highest yield achieved was 86.6% methyl ester with the TSP (esterification followed by transesterification). The processing conditions used were 0.5% NaOH at 60°C and reaction time of 14.25 h. In this study, the variant with 45°C gave the highest yield followed by that of 50°C. This indicates that at that catalyst concentration, methanol quantity and reaction time, 45°C is the optimum processing temperature for the highest yield of 87.4%. Results for the reaction time also showed that 45 min had the highest yield followed by that with 30 min reaction time. This indicates that at that catalyst concentration, methanol quantity and reaction temperature, 45 min is the optimum reaction time for the highest biodiesel yield of 88%. Beyond that time, the yield kept decreasing.

Table 4: Biodiesel yield properties

Parameters			Results		
Temperature(°C)	45	50	55	60	65
Crude biodiesel yield (ml)	510	510	490	510	486
pH of crude biodiesel	9.0	9.0	9.0	9.0	9.0
Pure biodiesel yield (ml)	437	435	385	395	416
pH of pure biodiesel	6.5	6.5	6.5	6.5	6.6
Biodiesel yield (%)	87.4	87.0	77.0	79.0	83.2
Reaction time (min)	30	45	60	75	90
Crude biodiesel yield (ml)	500	484	458	492	486
pH of crude biodiesel	9.0	9.0	9.0	9.0	9.0
Pure biodiesel yield (ml)	434	440	396	344	300
pH of pure biodiesel	6.5	6.5	6.5	6.5	6.5
Biodiesel yield (%)	86.8	88.0	79.2	68.8	60.0

4. CONCLUSION

The study has shown the optimum reaction temperature and time for a single stage transesterification of biodiesel from coconut oil using methanol and potassium hydroxide. The results have shown higher yields of biodiesel obtainable from coconut oil than those reported in literature from the same coconut oil. This indicates that coconut oil is a good feedstock for biodiesel production. The results further indicate that applying these reaction temperature and time to the two stage process may give a better yield of the biodiesel. The effect of catalyst concentration in the single stage and combination of these operating conditions in the two stage process will constitute separate reports.

266 **COMPETING INTERESTS**

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268 Authors have declared that no competing interests exist.

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270 **AUTHORS' CONTRIBUTIONS**

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272 The first and corresponding author designed the study, wrote the protocol, and wrote the first
273 draft of the manuscript. 'Authors 3' and 4' carried out some aspects of the experimental
274 work. 'Author 1, 2' 3' and 4' managed the analyses of the study. All authors read and
275 approved the final manuscript.

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