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# Effect of Reaction Temperature on the Yield of Biodiesel From Neem Seed Oil

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## Abstract

This research work studied the effect of reaction temperature on the yield of biodiesel from neem seed oil by Transesterification process. The materials used are neem seeds and it was sourced from Federal University of Technology, Owerri (FUTO) and its host community (Ihiagwa). The dried seeds measured 10kg and were ground using attrition mill, the oil was extracted using solvent extraction (Soxhlet extraction method, *n*hexane) process. Biodiesel was produced by transesterification process using a biodiesel production plant designed here in FUTO. The non-edible neem seed oil was used as the feedstock with potassium hydroxide (KOH) and methanol as catalyst and alcohol respectively. The free fatty acid content of the refined oil was 8.42mg/KOH which corresponds to free fatty acid (FFA) value of 4.20%. The FFA was further reduced to less than 1% by two step acid esterification processes. The reaction temperature was varied from 30°C to 65°C while every other parameter such as reaction time, stirring speed, alcohol oil ratio and volume of oil used were kept constant at 60mins, 350rpm, 7:1 and 60m$^3$ respectively. Conversion of biodiesel was low during reaction at 30°C but increased as the temperature was increased before the decrease beyond 60°C. The maximum yield of 94% was observed at 55°C and the regression equation for yield of neem biodiesel against reaction temperature gave $R^2$-value of 0.931. The result shows that reaction temperature can influence the reaction rate and the ethyl esters (neem biodiesel) yield because the intrinsic rate constants are strongly dependent on temperature.

## Keywords

Effect, Neem Seed, Reaction Temperature, Yield, Biodiesel, Neem Seed Oil

## 1. Introduction

Biodiesel is a mixture of mono-alkyl esters of long chain (C16-18) fatty acids that can be obtained from a variety of vegetable oils and animal fat, [1] by a process known as transesterification. It is chemically a mono-alkyl ester of long chain fatty acids derived from renewable biological sources such as vegetable oils and animal fats [2-3]. It is a clean burning alternative fuel, produced from domestically grown renewable resources such as vegetable oils, edible and non-edible oils; it contains no petroleum products, but can be blended at any concentration with diesel from fossil sources to create a biodiesel blend, [4]. Biodiesel is now recognized as an alternative fuel because it has several advantages over conventional diesel including being environmentally friendly, renewable and non-toxic [5]. A wide variety of feedstock have been identified as suitable forbiodiesel production including; soybean, canola, sunflower, safflower, *Jatrophacurcas*, peanut, tigernut, coconutetc. [6-7]. More so, there are several non-edible oil seed such as thevetia (thevetia peruviana), karanja (pongomia pinnate), jatropha (jatropha curca), and neem (azadirachta india). Among these, azadirachta indica seed which contain 25%-45% oil on dry matter basis is non edible oil that can be use in biodiesel production [8].

Neem (*Azadirachta indica*) is a tree in the mahogany family *Meliaceae* which is abundantly grown in varied parts of India. The Neem grows on almost all types of soils including clayey, saline and alkaline conditions. Neem seed obtained from this tree are collected, de-pulped, sun dried and crushed for oil extraction. The seeds have 45% oil which has high potential for the production of biodiesel [9]. Neem oil is generally light to dark brown, bitter and has a rather strong odour that is said to combine the odours of peanut and garlic. It comprises mainly of triglycerides and large amounts of triterpenoid compounds, which are responsible for the bitter taste. It is hydrophobic in nature and in order to emulsify it in water for application purposes, it has to be formulated with appropriate surfactants.

Neem oil also contains steroids (campesterol, beta-sitosterol, stigma sterol) and a plethora of triterpenoids of which Azadirachtin is the most widely studied [4]. The major component fatty acids of Neem oil are Palmitic acid (19.4%), Stearic acid (21.2%), Oleic acid (42.1%), Linoleic acid (14.9%) and Arachidic acid (1.4%) [5]. Theseed crop could be utilizedas a source for production of oil [9] and can be grown in large scale on non-croppedmarginal lands and waste lands.

Methods commonly used for producing biodiesel involve various stages including oil extraction, purification (degumming, deacidification, dewaxing, dephosphorization, dehydration, etc.) and esterification and/or transesterification process. Conventionally, alkali catalyst, such as sodium and potassium hydroxides, are the most preferred catalysts in biodiesel production [10].

Vegetable oils needs to be transesterified to biodiesel because of its high viscosity and cold flow properties by reacting it with alcohol in the presence of basic or acidic catalyst to form esters and glycerol [1].

The main parameters affecting the transesterification reaction are molar ratio of vegetable oil to alcohol, catalyst type and amount, reaction time, reaction temperature, the contents of free fatty acids (FFAs) and water in substrate oil [11] and also the intensity of mixing during the chemical reaction. [12] found that higher reaction temperature increased reaction rate and shortened reaction time due to the reduction in viscosity of oils. However, [13-14] found that increase in reaction temperature beyond the optimal level led to decrease of biodiesel yield because higher reaction temperature accelerated the Saponification of the triglycerides. Except for the reaction temperature, other reaction parameters in transesterification of methyl esters have been optimized by various studies [15-16]. Therefore, the purpose of this research is to study the effect of reaction temperature on yield of biodiesel from neem seed oil.

## 2. Materials and Methods

The materials used in this research are neem seeds and it was gotten from Federal University of Technology, Owerri (FUTO) and Ihiagwaenvironments. The cleaned seed kernels still within their endocarp were sun dried in the open at 50° for three days to allow for their easy removal after soaking for 5mins and washing before removing the endocarp. Soxhlet extraction method (*n*hexane) was used during the extraction of neem oil [17]. The quantity of the extracted crude neem oil was recorded at the end of the extraction process and the percentage extracted oil was determined using equation (1).

$$\%\, oilyield = \frac{Massofcrudeoilextracted}{Totalmassofseedkernel} \times 100 \qquad (1)$$

A two-step process acid catalyzed esterification followed by alkali catalyzed Transesterification was employed before base catalyzed transesterification [18]. The alcohol used was methanol and potassium hydroxide (KOH) as catalyst. During the transesterification process, the reaction temperature was varied from 30°C to 65°C while every other parameter was kept constant; reaction time 1hr, stirring speed 350rpm, methanol-oil ratio 6:1 and catalyst 0.75wt%,. According to [19], the catalyst amount should be usually between 0.1 and 1.5% by weight of oil, therefore catalyst amount was taken as between 0.5 and 1.5% by weight of oil. The ethanol used had a boiling point of 78°C therefore reaction temperatures for trans-esterification must be below the boiling point of alcohol [20] therefore; the reaction temperature was maintained below 78°C. After the Transesterification the upper ester layer contained traces of KOH, methanol and glycerol and the biodiesel was washed by spraying hot water over it to remove unreacted methanol and residual catalysts which may corrode engine components [21]. The quantities of biodiesel yield from various transesterification processes were calculated using equation (2).

$$Yield = \frac{\text{Amount of produced biodiesel}}{\text{Amountof oil used}} \times 100 \qquad (2)$$

Generally, under transesterification with alcohol, the first step is the conversion of triglycerides to diglyceride, which is followed by the subsequent conversion of higher glyceride to lower glycerides and then to glycerol, yielding one methyl ester molecule from each glyceride at each step. Each of the production process starts with the reactor charged with a given amount of neem seed oil, which was pre-heated at different temperature ranges, meanwhile a solution of catalyst in methanol ($CH_3OH$) was added. The reaction condition based on temperature was varied while other parameters like reaction time, alcohol-oil ratio, catalyst weight *etc*. were kept constant until various amounts of methyl ester yield were gotten.

## 3. Results and Discussion

Table 1 shows the yield of neem biodiesel with respect to reaction temperatures ranging from 30 to 65°C. The first column represents the various ranges of reaction temperatures, the second column is measured volumes of biodiesel after reaction, the third column represents the measured volumes of neem biodiesel after washing and the fourth column represents the final yield of biodiesel.

***Table 1.*** *Effect of temperature on biodiesel yield from neem seed.*

| Temp.°C | Volume of biodiesel after reaction ($cm^3$) | Volume of biodiesel after washing ($cm^3$) | Final yield of biodiesel (%) |
|---|---|---|---|
| 30 | 4.30 | 4.00 | 85 |
| 35 | 4.50 | 4.10 | 87 |
| 40 | 4.60 | 4.20 | 89 |
| 45 | 4.70 | 4.50 | 90 |
| 50 | 4.70 | 4.60 | 91 |
| 55 | 4.75 | 4.65 | 94 |
| 60 | 4.80 | 4.70 | 88 |
| 65 | 4.85 | 4.75 | 84 |

Transesterification can occur at different temperatures depending on the oil used and temperature is a crucial parameter as it influences the reaction rate and yield of the methyl esters. In order to determine the effect of reaction temperature on the oil conversion, experiments were conducted at 30, 35, 40, 45, 50, 55, 60 and 65°C, the results are shown in Table 1 and Figure 1. It was observed that as reaction temperature increased, there was a corresponding increase in the conversion of the triglycerides to methyl esters, showing peak at temperature 55°C. The highest conversion was 94% at a reaction temperature of 55°C. [22] carried out the transesterification of palm oil with methanol at 60, 120 and 150°C in the presence of $K_2O$ loaded on MCM-41 synthesized from rice husk. The performance depended on the $K_2O$ loading and temperature with the highest conversion observed on 8% $K_2O$/RH-MCM-41 at 100°C. Also, [23] used edible oil for the production of biodiesel with methanol and alkaline catalyst. They employed transesterification process in order to produce biodiesel form high FFA content neem seed oil by using 100ml volume of the oil with pretreatment conditions, 45v/v ratio of methanol to oil, catalyst concentration 0.5% v/w of $H_2SO4$ and reaction time 45 minutes, at 50°C temperature. Further transesterification process continued on the pretreated neem oil with neem oil to methanol molar ratio 0.3:1, in the presence of 1% KOH catalyst for one hour, at 55°C. The result gave maximum average yield of neem biodiesel of 90 ± 2%.

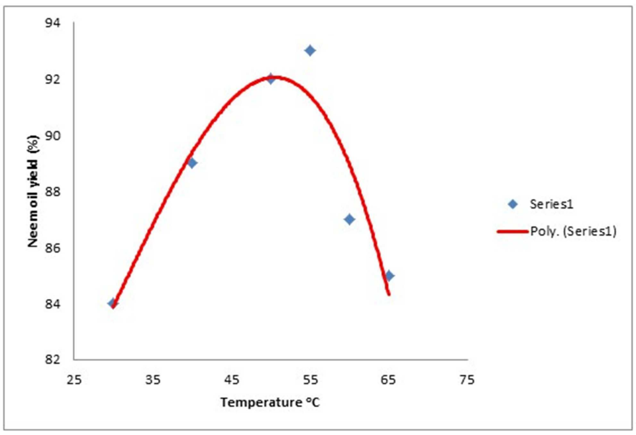


***Figure 1.*** *Effect of temperature on the yield of neem biodiesel.*

Effect of temperature on reaction rate can be explained through the theory of chemical reaction kinetics. An increase in temperature will result in increasing fraction of molecules that have ahigh speed and therefore has a high kinetic rate [9]. From the graph, it can also be observed that the reaction temperature at 55°C gave the optimum yield of 94% neem biodiesel. The yield of neem biodiesel ranges from 84 to 94% with average yield of 88.5%. The conversion of neem oil to neem biodiesel was low at 30°C and increased as the temperature increased before the decrease beyond 55°C. The reason for this is that higher temperature accelerates the side saponification reaction of triglycerides. Further increase in temperature is reported to have adverse effect on the conversion process [24]; therefore, increased reaction temperature resulted in decrease in the neem biodiesel yield as methanol is lost because methanol tends to evaporate beyond 65°C.

According to [1], the optimum temperature was 64°C (near the evaporating temperature of methanol). Increase in temperature beyond this point decreases the yield due to evaporation of methanol. Several researchers [19, 24, 25, 26] have reported that increase in temperature influence trans-esterification in a positive manner, however, the temperature must not exceed the boiling point of the reacting alcohol to prevent the volatilization of the alcohol during trans-esterification. The result was similar to that of [27] who studied the transesterification of refined soybean oil with methanol (6:1), 1% NaOH catalyst, at three different temperatures 60, 45 and 32ºC. Ester yields were found to be 94%, 87% and 64% after 30mins at reaction temperature of 60, 45 and 32ºC, respectively. Also, ester formation was found to be identical at 60 and 45ºC reaction temperature after 1hr and it became slightly lower at 32ºC, further increase in temperature decreases the conversion [28]. More so, the result of this study is also similar to reports on biodiesel production from oil sources where yields of above 90% were obtained. [29] reported 97% biodiesel yield from *Jatrophacurcas,* while [30] reported 96.85% biodiesel yields from safflower. More so, [31] focused on the feasibility of production of biodiesel from neem oil. Transesterification process was used for the production of biodiesel and optimum experimental conditions were determined, in order to obtain maximum biodiesel yield. In their study, biodiesel was produced at different temperatures by varying molar ratio of oil to alcohol and it was found that the maximum yield of biodiesel occurred at temperature 55°C. The results showed that the prepared biodiesel produced was within ASTM standards of biodiesel fuel. However, the report by [32] opined that the yield of 75.02% was obtained from coconut biodiesel using methanol and $CaCO_3$. Again, the work of [32] on high free fatty acid coconut comparing the two stage methods showed that the highest yield achieved was 86.6% methylester. It shows that temperature clearly influenced the reaction rate and yield of esters and transesterification can proceed satisfactorily at ambient temperatures, if given enough time, in the case of alkaline catalyst. It has been reported that at high temperatures beyond 65°C, the methanol has the tendency to evaporate except if their action is carried out under reflux conditions [33].

The regression equation model for the yield of neem biodiesel against reaction temperature is given below in equation (3)

$$Y = 8E - 05T^4 - 0.016T^3 + 1.151T^2 - 34.30T + 449.5(R^2 = 0.931) \quad (3)$$

Where Y is the neem biodiesel yield (%) and T is various temperature ranges (°C).

## 4. Conclusion

In this research, biodiesel was produced from neem seed oil and the effect of reaction temperature ranging from 30, 35, 40, 45, 50, 55, 60, 65°C on the yield was studied. It was found that the reaction temperature at 55°C gave the optimum yield of 94% neem biodiesel. The yield of neem biodiesel ranged from 84% to 94% with average of 88.5%. The yield was temperature dependent and increased with temperature to certain points beyond which it decreased due to increased volatility and miscibility. More so, the use of neem seed oil as biodiesel feed stock should be greatly encouraged because it is a non-edible crop and would not compete with acreage dedicated for food crops.

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