Kinetics of Base Catalysed Trans-Esterification of Jatropha Oil using Potassium Hydroxide Extract from Ripe Plantain Peels.

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Abstract

Obtaining kinetic data such as the rate constant, reaction order and hence the rate law is key to a good design of reactors in which chemical reactions take place, predicting the mechanisms, rates and extent of these reactions. Temperature and concentration are the most critical of the reaction conditions for homogeneous reactions. Hence the kinetic study of the trans-esterification of jatropha oil in a batch reactor using ethanol in the presence of potassium hydroxide extracted from ripe plantain peels as the base catalyst was carried out. And from the study, it was discovered that as the temperature was raised from 75 to 83 °C, conversion of jatropha oil increased progressively within the time of 1 to 4 hrs. In 1 hr, the conversion value (0.352 mole) at 75 °C, doubled with just 8 °C rise in temperature (at 83 °C) to 0.752 mole. Conversion at 83 °C in 4 hrs recorded the highest value of 0.972 moles. The rate laws $(-r_A = 1.083C_A$ and $-r_A = C_A)$ at 75 and 83 °C respectively agree with the pseudo first order assumption made that the trans-esterification of crude jatropha oil to its biodiesel was a first order reaction with respect to crude jatropha oil while keeping the ethyl alcohol in large excess.

Keywords: Kinetics, base catalysed; trans-esterification; potassium hydroxide extract

INTRODUCTION

Trans-esterification involves the conversion of the oils to their alkyl esters by reaction with alcohol [1]. And it has been the most commonly used method for the production of biodiesel. Ethanol and methanol have been the common alcohols used in the trans-esterification of these oils to biodiesel in the presence a base or an acid catalyst [2]. Alkali catalyzed trans-esterification has been most frequently used industrially, mainly due to its fast reaction rate [3, 4]. The common alkali catalysts for trans-esterification include sodium hydroxide (NaOH) and potassium hydroxide (KOH) [5]. The challenge however,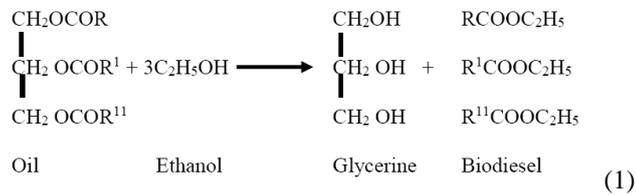 with the use of NaOH is the formation of several by-products mainly sodium salts which treated as waste most times [6]. The use of NaOH as catalyst requires in addition, that high-quality oil is used. Potassiurn hydroxide has an advantage over sodium hydroxide in that, at the end of the reaction, the reaction mixture can be neutralized with phosphoric acid resulting in potassium phosphate, which can be used as fertilizer. "Ref. [7]" reported that potassium hydroxide was superior to sodium hydroxide as a catalyst for the trans-esterification of safflower seed oil which, by extension means that potassium hydroxide is better than sodium hydroxide in base catalyzed trans-esterification reactions of vegetable oils. Alkaline-catalyzed trans-esterification proceed at considerably higher rates than acid catalyzed trans-esterification. Due to this fact and also because alkaline catalysts are less corrosive to industrial equipment, most commercial trans-esterifications are conducted with alkaline catalysts [8, 9].

The choice of the raw materials, non-edible jatropha oil and KOH extract from ripe plantain peels is key to producing low cost but high quality biodiesel. The use of oils from edible food crops for the production of biodiesel must be discouraged as shortages may arise in the long run especially in developing countries. where food grade vegetable oils are available the prices will be too high to attract interests in biodiesel production. Developing countries like Nigeria cannot afford to use edible oils to produce biodiesel as it would lead to acute food shortages. This is because larger population of her rural dwellers is mere subsistence farmers that still depend on hoe and cutlass rather than mechanized farming. Nigeria is blessed with a host of non-edible oils such as rubber seed, jatropha and castor oils which should be exploited [2].

Potassium hydroxide [KOH] used here is obtained from the agricultural wastes in ripe plantain peels ash and is renewable since it can be obtained from biomass materials. The heat of combustion of these biomass materials can be recycled in the evaporation of the extracting solvent to recover solid KOH from it and used for biodiesel production. Thus the overall

cost contributions of these raw materials result in much lower price in the final biodiesel product.

The reaction between jatropha oil and ethyl alcohol in the presence of a base catalyst may be represented by the "equation (1)" below [8, 10].

$$
\begin{array}{cccc}
CH_2OCOR & & CH_2OH & RCOOC_2H_5 \\
| & & | & \\
CH_2\,OCOR^1 + 3C_2H_5OH & \longrightarrow & CH_2\,OH\ + & R^1COOC_2H_5 \\
| & & | & \\
CH_2\,OCOR^{11} & & CH_2\,OH & R^{11}COOC_2H_5 \\
\text{Oil} & \text{Ethanol} & \text{Glycerine} & \text{Biodiesel}
\end{array} \tag{1}
$$

Where R, R^1, and R^{11} are long chain hydrocarbon.

The approximate proportions of the reaction are:

1mole of oil + 3 moles of ethanol → 1 mole of glycerol + 3 moles of biodiesel

The mechanism of the base catalyzed trans-esterification of vegetable oils is shown in "equation (1)". The first step is the reaction of the base with the alcohol, producing an alkoxide and the protonated catalyst. The nucleophilic attack of the alkoxide at the carbonyl group of the triglyceride generates a tetrahedral intermediate, from which the alkyl ester (biodiesel) and the corresponding anion of the diglyceride are formed. The latter deprotonates the catalyst and reacts with a second molecule of alcohol starting a new catalytic cycle. Diglycerides and monoglycerides are converted by the same mechanism to a mixture of alkyl esters and glycerine [9].

These chemical reactions change the molecular structure of oil and ethanol to form biodiesel (product) and glycerol (by-product) [11]. The stoichiometric quantities are given by the reaction "equation (1)" [11]. Often, the stoichiometric coefficients are imposed to be constant during the reaction; this is however, not the case in practice.

The entire reaction mechanism consists of three consecutive steps, which are reversible [9]. In the first step, the ethoxide reacts with the oil (a triglyceride) to form a diglyceride and a mole of ethyl ester (biodiesel). In the second step, the diglyceride gives a monoglyceride and a mole of biodiesel while in the third and last step, the monoglyceride yields a third mole of biodiesel and glycerine as the by-product. Theoretically, the stoichiometric ratio between the oil and alcohol is 1:3. But in practice, an excess of alcohol is usually used to drive the reaction forward in favour of the desired ethyl ester or biodiesel [9].

A study of the kinetics of trans-esterification will provide parameters that can be used to predict the extent of the reaction at any time subject to some imposed conditions. The aim of chemical kinetics is to make predictions about the changing composition of reaction mixtures as a function of time in order to understand the processes that occur during a reaction, and to identify what controls its rate [12]. The desire in chemical kinetics is the determination of the reaction order and hence its rate law from a set of experimental data. The rate law may thus be related to the reaction mechanism, and the knowledge of the mechanism of a given reaction allows the control of the reaction.
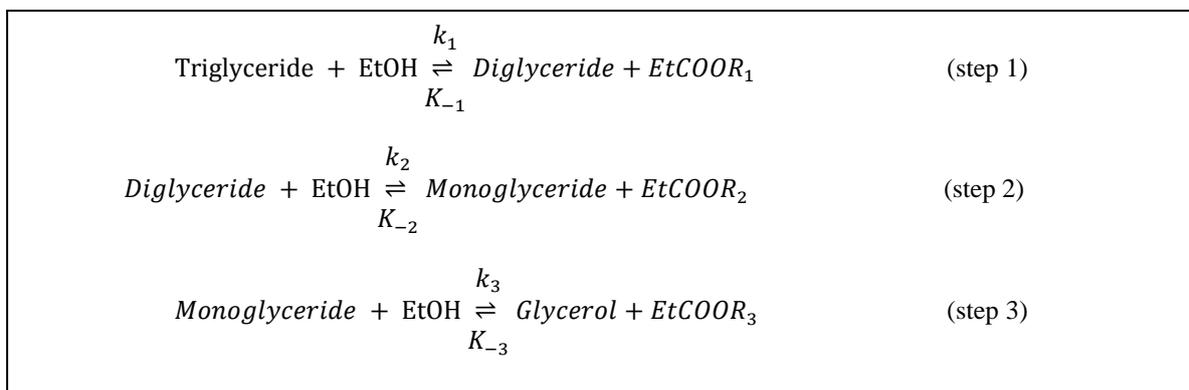
$$
\begin{array}{ll}
\text{Triglyceride + EtOH} \underset{K_{-1}}{\overset{k_1}{\rightleftharpoons}} Diglyceride + EtCOOR_1 & \text{(step 1)} \\[2em]
Diglyceride + \text{EtOH} \underset{K_{-2}}{\overset{k_2}{\rightleftharpoons}} Monoglyceride + EtCOOR_2 & \text{(step 2)} \\[2em]
Monoglyceride + \text{EtOH} \underset{K_{-3}}{\overset{k_3}{\rightleftharpoons}} Glycerol + EtCOOR_3 & \text{(step 3)}
\end{array}
$$

Figure 1: The Three Consecutive and Reversible Reaction Steps in Vegetable oil Trans-esterification Reaction [8, 11].

In this study a chemical batch reactor was used. Of the three reactor types batch, continuous stirred tank and plug flow only the batch reactor corresponds to a closed system while the remaining two correspond to open systems. In industrial set up, batch operations are well suited for the production of valuable products. The use of a batch reactor means that while the reaction is progressing, no feed is fed into it and no product is withdrawn from it until the reaction conditions such

as catalyst concentration, temperature, reaction time etc are fully satisfied. In an ideal batch reactor, it is assumed that the reacting mixture is well mixed and hence the property of the reacting mixture is uniformly distributed throughout the reactor.

MATERIALS AND METHODS

Apparatus

Analysis of the biodiesel was done using the Proton Nuclear Magnetic Resonance (^{1}H NMR) at the FAMU-FSU Tallahassee, Florida. All the glassware used were Pyrex product. They include a 3-neck flat bottom flask mounted on a hot plate magnetic stirrer (Thermo Scientific, SP 131015 IOWA, USA). To it were attached a reflux condenser and a quick fit $0 - 250$ºC thermometer. The third opening on the flask served as raw materials charging port. The set up was used for all the trans-esterification reactions. For the KOH extraction from the ash of ripe plantain peels, 500ml beakers, 250ml Erlenmeyer flasks, 50ml burette, 25ml pipettes, 500ml volumetric flasks, 500ml separating funnels 1 litre, stainless steel cups, hot plate magnetic stirrer (Thermo 57 Scientific, SP 131015 IOWA, USA), glass thermometer ($0 - 100$ ºC) and retort stands were used.

Materials

KOH extract (obtained from ripe plantain peels ash), crude jatropha oil purchased from Obeka Engineering Company in Kaduna State, Nigeria, Potassium hydroxide (>85 %purity, Sigma Aldrich analyzed) and ethanol (absolute, Sigma Aldrich analyzed) were used. Refined rapeseed oil purchased from a vegetable oil importer –Justrite, Ota, Ogun State, Nigeria, de-mineralized water obtained from Elga B114 64 (UK) de-ionizer, filter papers (125mm diameter, Whatman) and pH papers ($1 - 14$).

Procedure

The method used for the recovery of potassium oxide (K_2O) in its hydrated form (KOH) from the ripe plantain peels ash is as described by "ref. [16]".

For Biodiesel production, 50.75 g crude jatropha oil and 50.0 g ethanol (absolute) were first weighed into two separate beakers that have been previously washed and dried. 1 g of the catalyst (potassium hydroxide extract) was dissolved in the ethanol and the resultant solution (potassium ethoxide) was then charged into the reactor and pre-warmed until temperature rose to 50 ºC. The oil which, was also pre-warmed to the same temperature in a separate beaker was carefully added to the potassium ethoxide in the reactor and was then reacted at 75 and 83 ºC respectively at varied times of trans-esterification reactions.

Modelling Trans-Esterification Reaction in a Batch Reactor [13, 14].

From the reaction equation in "equation 1", a pseudo-first order reaction is assumed. The reason is that being an equilibrium reaction excess alcohol far greater than the stoichiometric quantity is needed to favour forward reaction or biodiesel production. In practice, up to 6 moles or more of alcohol is used.

Let A represent the jatropha oil which is the reactant and D represents the product (or biodiesel)

Hence

$$A \rightarrow D \tag{2}$$

$\therefore C_A$ gives the uniform concentration of the reactant A within the system at any time, t.

Hence the design equation for reactant A for the batch reactor becomes:

$$\frac{dN_A}{dt} = -r_A V. \tag{3}$$

Where N_A is the number of moles of A within the reactor at time t.

r_A is the rate of reaction of reactant A.

"Equation (3)" may be expressed in terms of conversion, x:

$x_A = \frac{N_{A0}-N_A}{N_{A0}}$, and re-arranging gives $x_A N_{A0} = N_{A0}-N_A$..

$$\Rightarrow N_A = N_{A0}(1 - x_A). \tag{4}$$

Differentiating gives $\frac{dN_A}{dt} = \frac{-N_{A0}dX_A}{dt}$. $\tag{5}$

And equating "equations (3) and (5)" gives that

$$\frac{dN_A}{dt} = \frac{-N_{A0}dX_A}{dt} = -r_A V. \tag{6}$$

By separation of variables, it gives

$$dt = \frac{-N_{A0}dX_A}{-r_A V}. \tag{7}$$

Integrating with the boundary conditions, at $t = 0, x_A = 0$ and at $t = t_f, x_A = x_{Af}$.

$$\int_{t=0}^{t=t_f} dt = \int_0^{X_{Af}} \frac{N_{A0}dX_A}{(r_A)V} \Rightarrow t_f = \int_0^{x_{Af}} \frac{N_{A0}}{(r_A)V} dX_A. \tag{8}$$

Where x_{Af} is the final conversion of A, and (r_A) should be expressed as a function of x_A in order to be able to integrate "equation (8)". The volume V is assumed to be constant.

But from "equation 2" the rate of disappearance of A or conversion of A to product may be mathematically expressed as $-r_A = K_1 C_A$. $\tag{9}$

Where K_1 is the pseudo 1st order reaction rate constant and $C_A = \frac{N_A}{V}$. $\tag{10}$

And combining "equations (9) and (10)" gives that $-r_A = K_1 \frac{N_A}{V}$.

$$\Rightarrow -r_A V = K_1 N_A. \tag{11}$$

Substituting "equations (11) and (4)" in "equation (8)" gives

that

$$t_f = \int_0^{x_{Af}} \frac{N_{A0}}{-K_1 N_{A0}(1-x_A)} dX_A = \int_0^{x_{Af}} \frac{1}{-K_1(1-x_A)} dX_A.$$

$$\therefore t_f = -\frac{1}{K_1} \ln(1 - x_{Af}). \tag{12}$$

A plot of t_f against $\ln(1 - x_{Af})$, will give a straight line with slope $= -\frac{1}{K_1}$.

Where t_f is the trans-esterification reaction time used and x_{Af} conversion value at the stipulated time.

DISCUSSION OF RESULTS

Characterization of Biodiesel

The biodiesel produced was characterized by the method described by other researchers, [15], [16]. They investigated the trans-esterification of double step process modification for ethyl ester biodiesel production from vegetable and waste oils as shown in "equation (13)".

In this research work, trans-esterification reactions using a simple batch process (described in section 2.3) were carried out with crude jatropha oil and ethyl alcohol in the presence of a base (KOH extracted from the ash of ripe plantain peels). The oil conversion to ethyl ester biodiesel was analysed using ^{1}H NMR spectroscopy. Figs. 2 – 5 give the spectra of some of the partially converted crude jatropha oil with time. Generally,

part (b) of the figures high-lights the chemical shift regions of interest. These regions or peaks of interest have been labelled Z and U for jatropha biodiesel. The high-lighted peaks were selected and their integration data used to evaluate the percentage conversion of the oils to biodiesel using the "equation (13)", [15], [16].

$$C_{EE} = \frac{I_{EE-CH_2}/2}{I_{\alpha-CH_2}/2}. \tag{13}$$

Where C_{EE} = the ethyl ester percentage conversion.

$I_{EE-}CH_2$ = the ^{1}H NMR integration values of the ethyl ester CH_2 hydrogen atoms.

The chemical shift for the ethyl ester peaks occurred between δ of 4.0 and 4.3 ppm for all the converted jatropha biodiesels.

$I_{\alpha-}CH_2$ = integration value of the methylene group hydrogen atoms that are adjacent to the carbonyl group. his occurred at δ values of between 2.1 and 2.4.

The factor '2', which divides the integration values, is the normalization factor.

The terminal methyl group hydrogen atoms of the fatty acid chains for the jatropha biodiesel are labelled as peak Y occurring between 0.71 and 0.88 ppm. The peaks T – W are attributed to the methylene internal hydrogen atoms of the triglyceride fatty acid chains. These signals occur between 1.05 and 2.77 ppm.
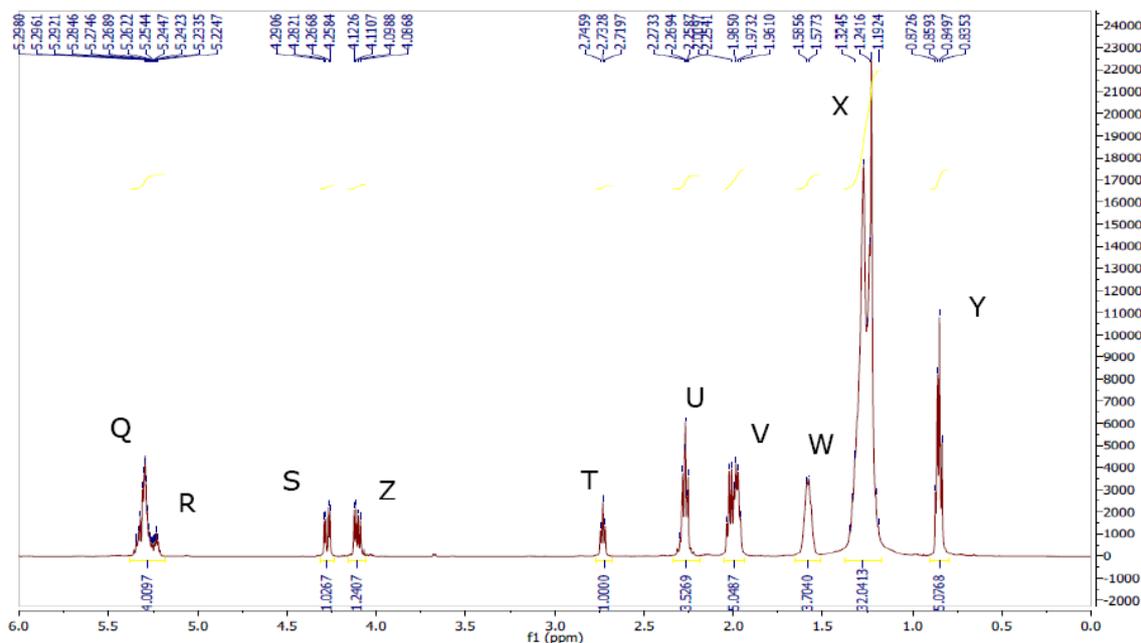

Figure 2(a): ^{1}H NMR of jatropha biodiesel (sample 1) at reaction time of 1 hr and temperature of 75°C
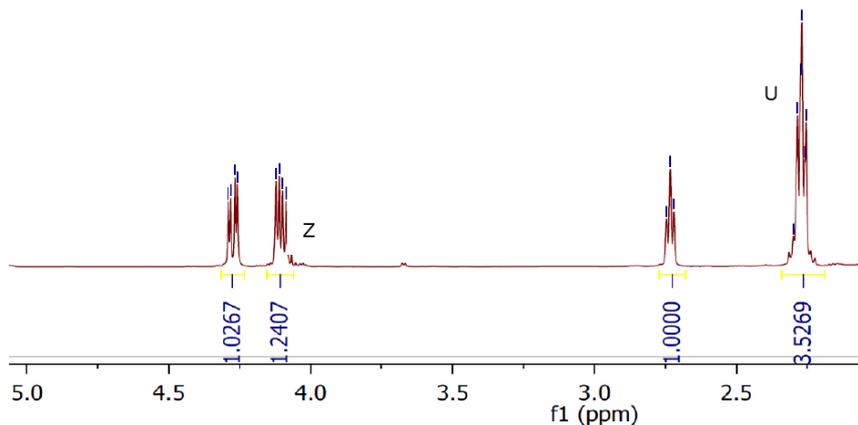
Figure 2(b): Magnified chemical shift regions for crude jatropha biodiesel: sample 1
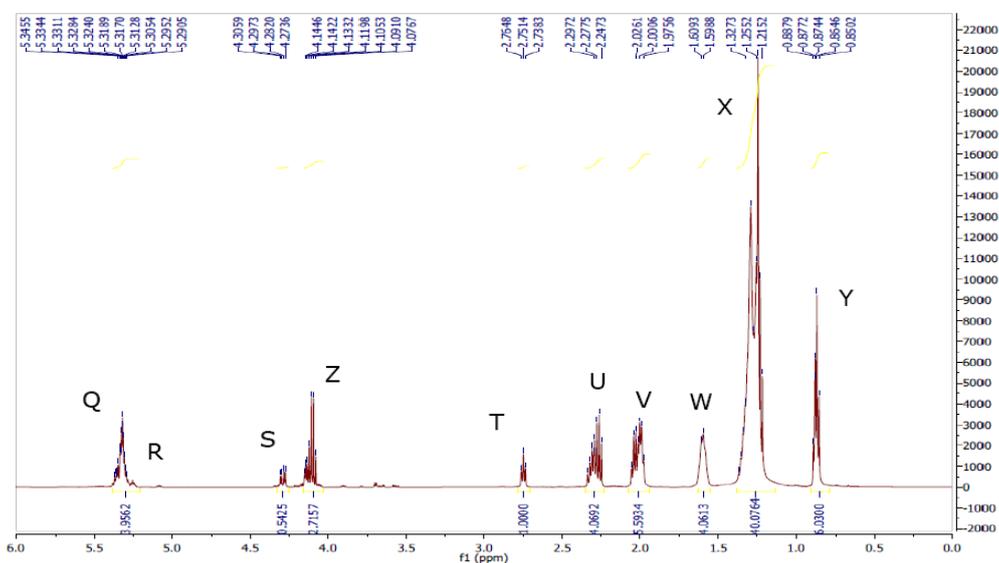

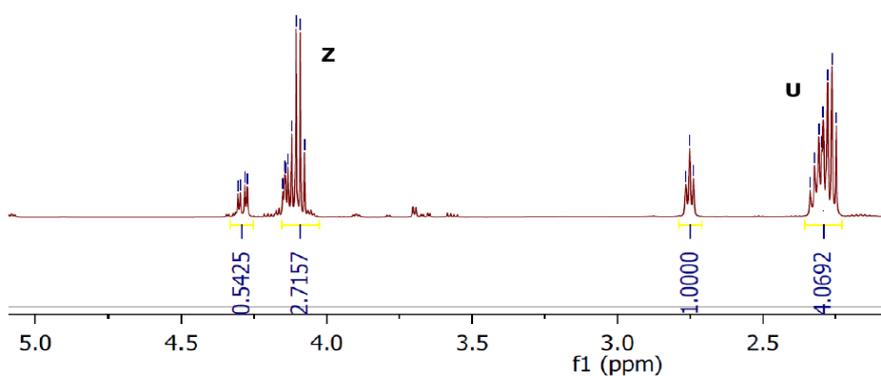**Figure 3(a):** ^{1}H NMR of jatropha biodiesel (sample 2) at reaction time of 2 hrs and Temperature of 75°C

Figure 3(b): Magnified chemical shift regions for crude jatropha biodiesel (sample 2).
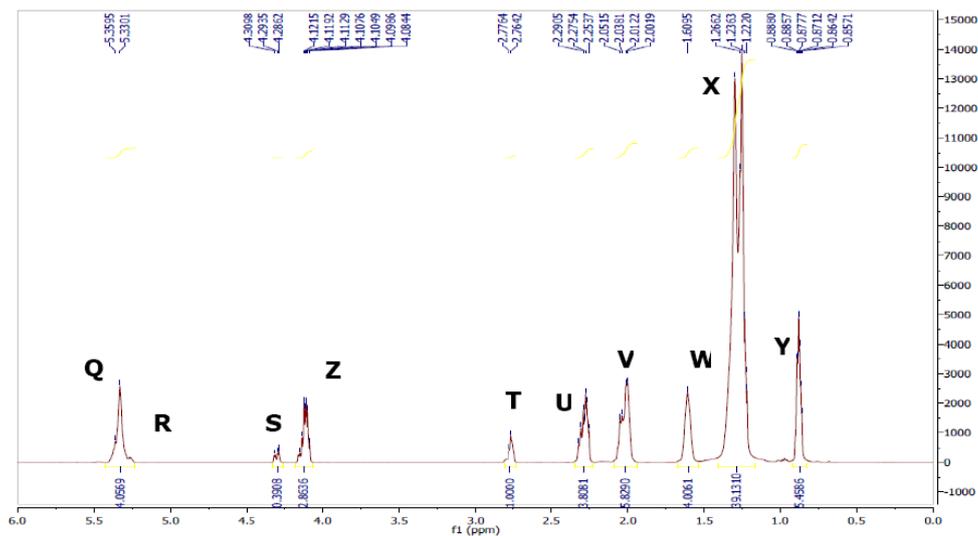
Figure 4(a): ^{1}H NMR of jatropha biodiesel (sample 3) at reaction time 1 hr and temperature of 83°C
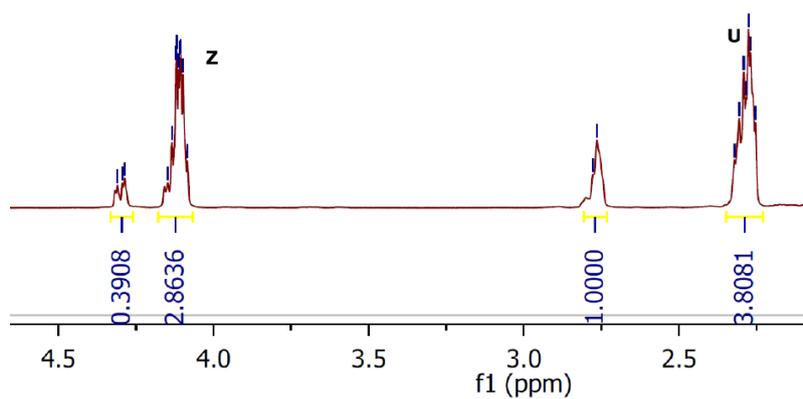

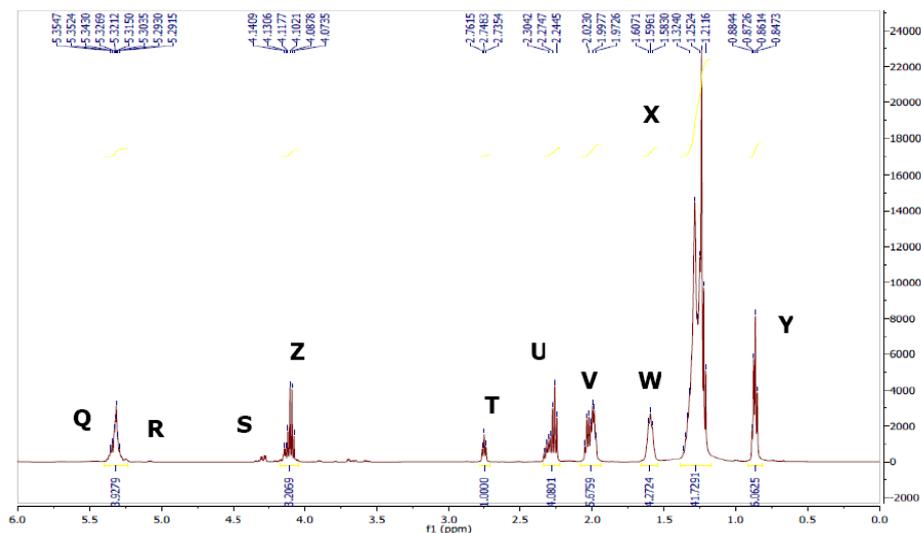**Figure 4(b):** Magnified chemical shift regions for crude jatropha biodiesel (sample 3).

Figure 5(a): ^{1}H NMR of jatropha biodiesel sample (4) reaction time 2 hrs and temperature of 83°C
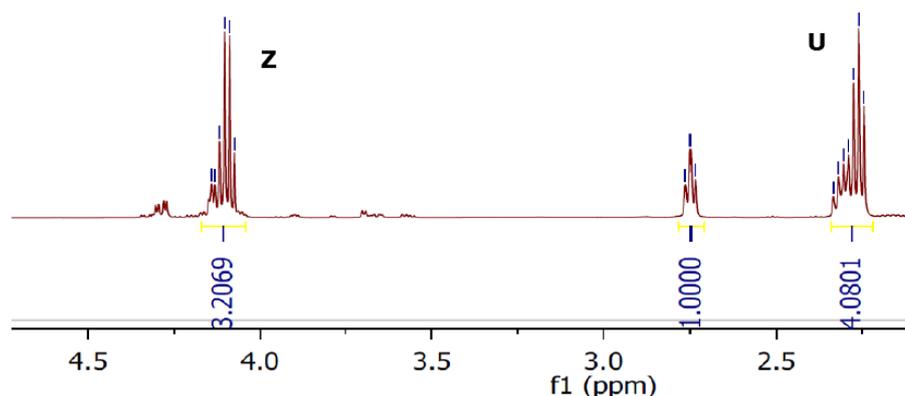
Figure 5(b): Magnified chemical shift regions for crude jatropha biodiesel (sample 4)

The external glycerol fragment hydrogen atoms (peak S) are located for jatropha biodiesel between 4.19 and 4.23 ppm. The carbon–carbon double bond olefinic hydrogen (peak Q) are located between 5.19 and 5.26 ppm and the internal glycerol fragment (gliceridic) hydrogen (peak R) at 5.15 − 5.19 ppm. These are all shown in Figures 2 − 5 for jatropha oil/jatropha biodiesel.

According to the reaction mechanism, the mono- and di-acylglycerides are the transesterification reaction intermediates. The methylene group hydrogen signals of the mono-, di- and tri-acylglycerides are located in the same region between 4.09 and 4.2 ppm [15, 16]. Peaks Z in jatropha biodiesel are due to the ethyl ester CH_2 groups [15]. Two

trans-esterification temperatures of 75 and 83°C at varied times of 1 − 4 hrs were investigated as shown in "Table 1".

The highest conversion value obtained was 0.972 mole using 4g of KOH extracted from the ripe plantain ash at trans-esterification time of 4 hours and temperature of 83 ºC. This was closely followed by conversion values of 0.970, 0.786 and 0.752 mole in 3, 2 and 1 hr(s) trans-esterification times respectively at same temperature (83 ºC). At 75 ºC, the conversion values were 0.958, 0.836, 0.668 and 0.352 moles in 4, 3, 2 and 1 hr(s) trans-esterification times respectively. Some of the spectra of the jatropha biodiesels have been displayed in Figs. 2 − 5 above.

"Table 1. Conversion of Jatropha Oil to Bio-Diesel at Varied Time,".

Reaction Temperature (^{0}C)	Sample No	Conversion x_{Af}	$1 - x_{Af}$	$ln(1 - x_{Af})$	Time (t_f) of conversion (hrs)
75 ^{0}C	1	0.352	0.648	-0.43386	1
	2	0.668	0.332	-1.10262	2
	3	0.836	0.164	-1.80789	3
	4	0.958	0.042	-3.17009	4
83 ^{0}C	1	0.752	0.248	-1.39433	1
	2	0.786	0.214	-1.54178	2
	3	0.970	0.0298	-3.51325	3
	4	0.972	0.028	-3.57555	4

It was discovered that higher conversion values were generally achieved at higher temperature of 83 than at 75 °C. The conversion value (0.352 mole) at 75 °C in 1 hr, doubled with just 8°C rise in temperature (at 83°C) also in 1 hr to 0.752 mole. It was also observed that conversion values increased as trans-esterification time and temperature increased (1 to 4 hrs) and (75 to 85 °C) respectively. Using "equation (12)", a plot t_f versus $\ln(1 - x_{Af})$ gave a straight line with negative slopes $(-\frac{1}{K_1})$ at both temperatures. The negative slope is only indicative of the reducing moles of jatropha oil remaining or unconverted as the reaction progressed. Slope obtained respectively from the two graphs are equated to $(-\frac{1}{K_1})$ from which k values are calculated.

From Fig. 6 the slope of the graph $= \frac{\Delta y}{\Delta x} = \frac{3.1 - 1.8}{-2.2 + 1.0} = \frac{1.3}{-1.2} = -1.08333 \quad -\frac{1}{K_1} = -1.08333 \, hr, \Rightarrow K_1 = 1.0833 hr^{-1}$,

and thus from "equation (9)", $-r_A = 1.083 C_A$. (14)

"Equation (14)" gives the reaction rate law of the pseudo first order trans-esterification of jatropha oil at 75⁰C to bio-diesel using potassium hydroxide extracted from the ash of ripe plantain peels.

Similarly, from Fig. 7, the slope of the graph $= \frac{\Delta y}{\Delta x} = \frac{2.6 - 1.2}{-2.6 + 1.2} = \frac{1.4}{-1.4} = -1.00 \quad -\frac{1}{K_1} = -1.00 \, hr, \Rightarrow K_1 = 1.00 hr^{-1}$,

and thus from "equation 9", $-r_A = C_A$ (15)

"Equation (15)" gives the reaction rate law of the pseudo first order trans-esterification of jatropha oil at 83⁰C to bio-diesel using potassium hydroxide extracted from the ash of ripe plantain peels. Thus from "equation (14)", "equation (15)" and the experimental plots (Figs. 6 and 7) the experimental data correlates well with the pseudo first order rate constant and rate law. The trans-esterification of jatropha oil to its biodiesel in the presence of KOH extracted from the ash of ripe plantain peels is a first order reaction with respect to jatropha oil keeping the molar ratio of ethyl alcohol practically constant by using it in large excess.
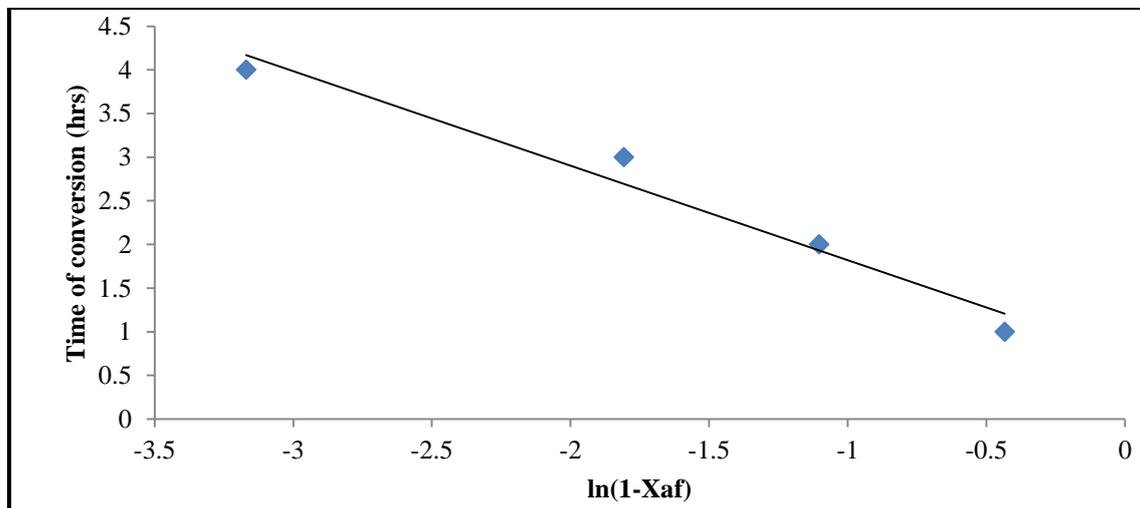

Figure 6: Psuedo First Order Plot for the Conversion of Jatropha Oil to Ethyl Biodiesel at 75 Deg. C using KOH from Ripe Plantain Peels.
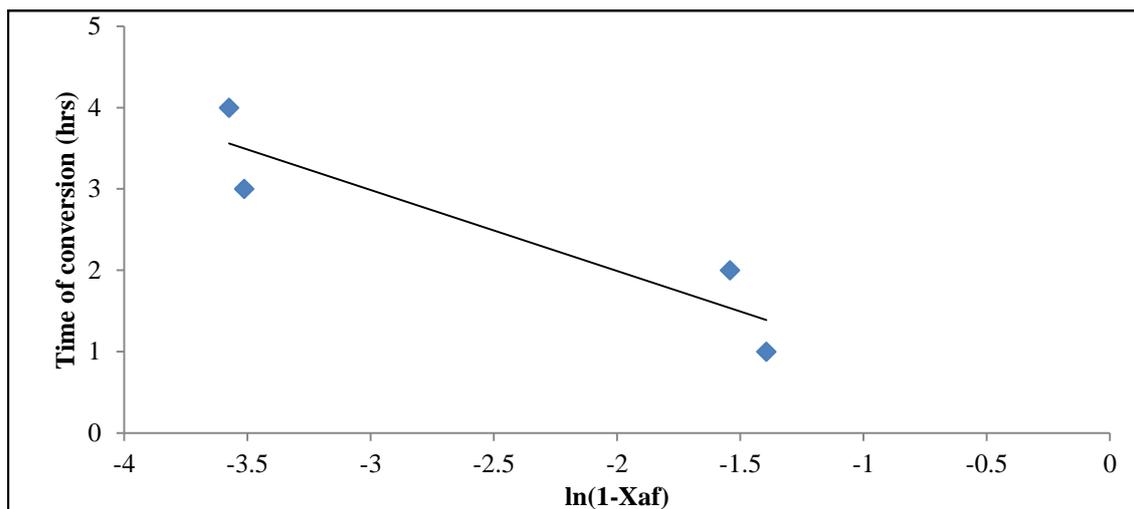
Figure 7: Psuedo First Order Plot for the Conversion of Jatropha Oil to Ethyl Biodiesel at 83 Deg. C using KOH from Ripe Plantain Peels.

CONCLUSION

The kinetic data obtained from the conversion of crude jatropha oil to its corresponding biodiesel depict good correlation: Linear graphs were obtained from the generated conversion values at both temperatures of 75 and 83 ℃. The reaction rate constants for the two reaction at 75 and 83 ℃ respectively gave 1.0833 and 1 hr^{-1}. The rate laws ($-r_A = 1.083C_A$ $and -r_A = C_A$) at 75 and 83 ℃ agree with the pseudo first order assumption made that the trans-esterification of crude jatropha oil to its biodiesel was a first order reaction with respect to crude jatropha oil while keeping the ethyl alcohol in large excess. It was established that the higher the temperature the higher the vegetable oil conversion to its biodiesel. In 1 hr, the conversion value (0.352 mole) at 75 ℃, doubled with just 8 ℃ rise in temperature (at 83 ℃) to 0.752 mole. It was also observed that conversion values increased as trans-esterification time increased from 1 – 4 hrs.

ACKNOWLEDGEMENT

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