Effects of Pyrolysis Variables on the Product Yields During Pyrolysis of Palm Kernel Shells

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Abstract: The effect of operating variables on the pyrolysis products when Palm Kernel Shell (PKS) was subjected to pyrolysis process was investigated in this work. 0.5 kg of dried sample of PKS was loaded into a steel retort, and the retort interior was rendered airtight. The retort was then placed into the furnace chamber and the PKS was pyrolysed at 300 °C between 10 - 30 minutes at 5 minutes interval. This was repeated for temperatures of 400, 500, 600 and 700 °C and in each case, the quantities of char, tar and pyro - gas produced were determined. Response surface methodology (RSM) was used to develop polynomial regression model and investigate the effect of changes in the level of pyrolysing temperature and duration on the product yields using Full Factorial Design (FFD). The contribution of pyrolysis temperature, duration and their squares (A, B, A^2 and B^2) to the model developed are significant. It was observed that the experimental data fitted better because of the Pred R-Squared of 0.9383. The agreement between the predicted and experimental values describe the accuracy of the model developed and can be used to navigate within the design space. The product yields minimum value of 0.89% was achieved at pyrolysis temperature and duration values of 300 °C and 10 min respectively. The optimum conversion product yields for dried PKS char, tar and gas at their respective pyrolysis conditions were 99 wt% char at 304 °C and 12 min., 35 wt% tar at 700 °C and 27 min., and 40 wt% gas at 700 °C and 30 min. The results obtained showed that PKS can be readily pyrolysed to obtain optimum yield of bio fuels (gas, tar and char).

Keywords: Pyrolysis, Palm Kernel Shells, Bio fuels, Gas, Tar, Char and Response surface methodology.

1.0 INTRODUCTION

Periodic escalation in petroleum products prices and decreasing stock of crude oil deposits had brought non-conventional and renewable energy sources into greater focus (Itabiyi, 2017). Along with the solar and the wind energy, the long neglected but potentially rich biomass residues became the focus of intensive utilization for energy generation. Bio-fuel produced from biomass residues is an alternative to petroleum products and has received great attention during the last decades due to the environmental problems associated with the usage of fossil fuel. It already provides approximately 14% of the total worldwide energy needs (Funda, et al., 2006). Aside from its abundant availability, biomass residues have negotiable contents of sulfur, nitrogen and ash which give lower emissions of SO2, NOx, soot and net emission of CO2 compared to conventional fossil fuels, thus keeping the environment and the public’s health safe (Qi, et al., 2006; Tsai, et al., 2006a, Okeunle, et al., 2018). In countries of excess production of agricultural residues such as Nigeria, eco-friendly system and more energy efficient utilization alternatives, that meets the needs of the present without compromising the ability of future generations must be developed (Itabiyi, et al., 2018).

One of the technologies by which biomass could be converted to energy is through pyrolysis. Among the thermo-chemical conversion processes, pyrolysis is the viable process for biomass upgrading by cracking polymeric structure of lignocellulosic materials and converting them into fraction consisting of a solid carbon-rich residue in form of charcoal and a range of volatile products, which include condensable and non-condensable gases. Volatile fraction can be used as a fuel or as a chemical feedstock. The remaining solid fraction can find several applications, such as in the production of activated carbon or used directly as a solid fuel. Therefore, pyrolysis is an important process that can contribute much more, to solving energy problem in the world especially developing countries (Lucas and Itabiyi, 2012). Distribution of pyrolysis products depends on such operating conditions as type of feedstock, reaction time, pyrolysis temperature and sweep gas flow rate (Choi et al., 2010; Rodjeen et al., 2006). To obtain high liquid yield, the pyrolysis conditions require high heating rate, moderate temperature (450-550 °C) and short vapor residence time (Mohan et al., 2006). Higher proportion of gas product is obtained from applying high temperature, low heating rate and long residence time, while slow heating at lower temperatures and long vapor residence time favor the formation of char product (Mohan et al., 2006; Nugranad, 1997; Onay and Koçkar, 2003). Also, cellulose composition of biomass and process variables including pyrolysis temperature and heating rate can have a profound influence on the chemical compositions of both gas and liquid products (Yaman, 2004; Branca et al., 2003; Yang et al., 2007).

Weerachanchai, et al, 2011 studied the effects of pyrolysis temperature on the product yields of palm kernel cake and cassava pulp residue and noted that the char yield decreased sharply from 300 to 500 °C followed by a slow decrease at higher temperatures and approaching a constant value at 800 °C. Temperature is also found to have an influence on the composition of element in the products. For example, studies by few researchers (Zanzi, et al. 2002) showed that carbon content increases with an increase in temperature, while the hydrogen and oxygen contents decrease.
Higher temperature, smaller particle size, and increased heating rate resulted in decreased char yield from pyrolysis of agricultural residues (Itabiyi and Lucas, 2013). The cracking of the hydrocarbons with an increase in the hydrogen content was favoured by a higher temperature and by using smaller particles (Zanzi et al., 2002). Wood gives more volatiles and less char than straw and olive waste. The effect of particle size in the range of 0.71 – 3.56 mm on product yields at 700°C was studied by Weerachanchai, et al. (2011) using palm kernel and cassava pulp residues. Their results showed that the pyrolysis of the two biomasses with the average of 2.03 mm gave the maximum in the liquid yield of 54.3 wt% and 42.4 wt% for palm kernel cake and cassava pulp residue, respectively. Their study further revealed that for particle sizes smaller than 2.03 mm, higher gas and char yields but a lowering in liquid yield were obtained as the particle size was decreased. Their study suggested that it is likely that the smaller size of biomass particles could affect greater heat transfer because of less temperature inside the particle, thus giving higher yields of released gases and volatiles. This work investigated the influence of pyrolysis temperature and time on the product yields during the pyrolysis of Palm kernel shells.

2.0 METHODOLOGY

2.1 Palm kernel shells preparation for the experiment

Palm kernel shells used for the pyrolysis experiments in this study were procured from an oil-palm industry in Iresa apa, Oyo State, South-Western Nigeria. The residues were cleaned in order to remove foreign particles such as stones, leaves, debris and other unwanted components. The weight of the sample (W_1) was measured using Ohaus top loading digital weighing scale of sensitivity ± 0.001 g (Model: PA4102, range: 0-4100 g, Ohaus company, Manufactured in Switzerland) and then oven-dried at a temperature of 105 °C until constant weight (W_2) was obtained in accordance with official methods of the ASTM D5373-02 (2005).

2.2 Methods

Pyrolysis experiments were carried out to determine the effect of operating variables on the product yields from PKS. 0.5 kg of dried PKS were fed into the retort. The retort was placed into the furnace and pyrolysed at around 300, 400, 500, 600 and 700 °C. The retort was connected through a pipe to the condensate receiver which was placed in an ice-cooling unit for the quick recovery of the condensable products (tar), and from the condensate receiver the uncondensed gases moved through a rubber hose into the gas collection unit. The char in the retort and the tar in the condensate receiver were collected and weighed using Ohaus top loading digital weighing balance. The weight of gas was evaluated by subtraction. The percentage of product yields was calculated from equation 1.

Percentage product yields \( Y = \frac{\text{mass of product}}{\text{mass of sample}} \times 100 \) (1)

2.3 Experimental Design

Full-Factorial Design (FFD) of response surface methodology was used for the experimental design to optimise the pyrolysis product yields from PKS. FFD consisted of a two-factor, three-level design comprising the pyrolysis temperature and pyrolysis duration of the feedstock as the independent variables while pyrolysis product yields consisting of char, tar and gas as the dependent variables or the responses were used as shown in Table 1. A centre point for the design was selected with factors at a level of medium standards as shown in Table 2. With the centre point design selected, the actual values of each factor were calculated. The design was based upon the symmetrical selection of variation about the centre point and levels of variations were chosen to be within the boundary range of the variables. The coded and actual values of the variables at various levels and responses are given in the Table 2. Three replications were carried out for all experimental design conditions and the average recorded. Thirteen experimental runs were carried out and the order of the experiment was fully randomised to reduce the effect of the unexplained variability in the observed responses due to extraneous factor as recommended by Singh et al (2003).

Regression Models were developed for PKS product yield and each of the product yields as a function of the two factors. The Design Expert 6.0.8 software was used to analyse the data obtained from the pyrolysis of PKS for developing response equations, Analysis of Variance (ANOVA), to generate surface plots and determine optimum pyrolysis conditions and product yield using its optimization toolbox. In multiple regressions, as in the present case, R^2, which is the square of the adjusted coefficient of determination and standard error are the indices. F statistics shows the significance of overall model while the t-statistics tests shows the significance of each of the variables of the model. The Functions was assumed to be approximated by a second degree polynomial equation as shown in equation 2.

\[ Y = b_0 + \sum_{i=1}^{m} b_i x_i + \sum_{i=1}^{m} b_i x_i^2 + \sum_{i=1}^{m} b_i x_i x_j \] (2)
where $Y$ is the predicted response, $b_i$ is the value of the fitted response at the centre point, and $b_{ij}$, $b_{i}$, $b_{i}$, $b_{ij}$ are linear, quadratic and cross product regression terms respectively. $m$ is the number of factors considered in the study which is equal to 2.

2.5 Optimization of the Product Yields.

A nonlinear programming problem of the form of equation 2 was formed from the vector of equation 2 as shown in equation 3. The optimization problem statement to maximize the product yields was formulated as shown in equation 3.

Maximize $Y = f(AB)$  

Subject to  

$L_A \leq A \leq U_A$  
$L_B \leq B \leq U_B$  

Where $Y$ is the product yields, $L_i$ is the lower limit of the factors and $U_i$ is the upper boundary of the factors. The fine search problem stated in equation 2 was embedded and solved in the optimization routine of design expert 6.0.8 version to obtain the optimal yields and the corresponding optimal process variables.

3.0 RESULTS AND DISCUSSIONS

Based on t-test, the regression coefficient that are not significant at 95% confidence level were discarded while only those ones that are significant were used to develop the final model.

3.1 Response Equations for PKS Product Yields.

The effect of FFD on the PKS pyrolysis product yields (char, tar and gas yields) is as shown on Table 3 that was subsequently used to fit the response equations for product yields. Multiple regression analysis was used as tools of assessment of the effects of two or more independent factors on the dependent variables (Boomee et al, 2010). The coefficients of determination ($R^2$) is a measure of the total variation of the observe values of the product yields about the mean explained by the fitted model (Shridhar et al, 2010). The factors of the models, their parameters estimates and the coefficients of determination ($R^2$) for the responses (char, tar and gas yields) were 0.8709, 0.8711 and 0.9383 respectively. The coefficient of determination ($R^2$) were high for response surfaces, and indicated that the fitted quadratic models accounted for more than 89% of the variance in the experimental data. Based on the $p$ values, the regression coefficient that were significant at p<0.05 were selected for the models that resulted in equations 4 - 6. Analyses of variance (ANOVA) were conducted to evaluate the adequacy and consistency of the models using F-statistic. The analysis of variance of the models is presented in Table 5. The results presented on Table 5 showed the $F$-values for char, tar and gas as 41.49, 17.22 and 61.86 respectively. These values were significant at p< 0.05 indicating good model fit.

$$Y_{C_{PKS}} = 64.77 - 22.07A - 17.06B$$

$$R^2 = 0.8709$$

$$Y_{T_{PKS}} = 25.83 + 9.77A + 9.13B - 7.52B^2$$

$$R^2 = 0.8711$$

$$Y_{G_{PKS}} = 15.23 + 11.35A + 8.09B + 5.14AB$$

$$R^2 = 0.9383$$

where: $Y_{C_{PKS}}$ = Yield of char from PKS (wt%)  

$Y_{T_{PKS}}$ = tar yield from PKS (wt%)  

$Y_{G_{PKS}}$ = gas yield from PKS (wt%)  

$A$ = Temperature (°C)  

$B$ = Time (Minutes).

3.2 Optimization of Pyrolysis Process.

Response surface methodology was used for the optimization of the pyrolysis process of the PKS and for understanding the factors affecting the pyrolysis process. The models were useful for indicating the direction in which to change the variable in order to maximise the yields of char, tar and gas. The multiple regression equations were solved using Design Expert 6.0.8. The regression equation was optimized for maximum value, to obtain the optimum conditions. The optimum actual values obtained for PKS pyrolysis product yields and their respective pyrolysis conditions are: 99.27% char at $A = 304.38^\circ C$ and $B = 12.43$ minutes, 35.22% tar at $A = 700.00^\circ C$ and $B = 26.94$ minutes and 39.81% gas at $A = 700^\circ C$ and $B = 30$ minutes.

The linear effects of temperature and time are the primary determining factors of the responses as shown in Table 4. Pyrolysing temperature as a single factor was the most influential factor, because of its higher F-value. The temperature at which pyrolysis process was conducted is highly significant (p<0.05) with an F-value of 112.85 as shown in Table 4.

Figures 1-3 show three-dimensional (3D) surface plots and accompany contour plot for the relationship between the independent and dependent variables for chosen model. The cubic response surface plot shown in Figure 1(a) depicts the effect of the pyrolysing temperature and time on the PKS char yield. From the contour plot in Fig 1(b) it is observed that the surface area decreases as the pyrolysing temperature and time increase. Fig 1(b) shows that, char yield of PKS decreases as the pyrolysing temperature and time increase. Itabiyi and Lucas (2016) ; Okekunle et al, (2016a) ; Okekunle et al, (2016b) and Adeleke et al, (2018) observed similar trend when they conducted pyrolysis experiment on oil palm trunk, cassava chaff and cassava peel respectively in a fixed bed pyrolysis reactor. Mohamad (2008) reported that, the decrease in char yield with an increase in pyrolysing temperature could either be due to secondary decomposition
of the char or through the greater primary decomposition of the PKS at higher temperatures.

The cubic response surface plot shown in Figure 2(a) depicts the effect of pyrolysing temperature and time on the PKS tar yield. It was observed from the contour plot in Figure 2(b) that the surface area increases as the pyrolysing temperature and time increases. Figure 2(a) cubic response surface indicates that the tar yield increases as the pyrolysing temperature and time increase to optimum condition while further increase in pyrolysing temperature and time led to decrease in tar yield. This shows that, there was a mutual interaction between the pyrolysing temperature and time on tar yield. Itabiyi and Lucas (2016); Okekunle et al., (2016a); Okekunle et al., (2016b) and Adeleke et al., (2018) observed similar trend when they conducted pyrolysis experiment on oil palm trunk, cassava chaff and cassava peel respectively in a fixed bed pyrolysis reactor. Pyrolysis process at higher temperature might have led to more tar cracking resulting into higher gas yield and lower tar yield.

The cubic response surface plot shown in figure 3(a) depicts the effect of pyrolysing temperature and time on the PKS gas yield. It is observed from the contour plot in figure 3(b) that the surface area increases as the pyrolysing temperature and time increased. Figure 3(a) cubic response surface indicates that the gas yield increases as the pyrolysing temperature and time increase. The increase in gaseous products as the reaction temperature increases might be due to the secondary cracking of the pyrolysis vapours at higher temperatures, or secondary decomposition of the char at the higher temperatures (Mohamad, 2008).

| Exp. No. | A(°C) | B(min) | Temp. (°C) | Time (min) | Y<sub>C_PKs</sub> | Y<sub>T_PKs</sub> | Y<sub>G_PKs</sub> |
|----------|-------|--------|------------|-------------|----------------|----------------|----------------|
| 1        | 1     | 0      | 700        | 20          | 34.98          | 36.83          | 28.2           |
| 2        | 0     | 0      | 500        | 20          | 61.1           | 25.77          | 13.12          |
| 3        | -1    | -1     | 300        | 10          | 98.13          | 0.89           | 0.98           |
| 4        | 0     | 0      | 500        | 20          | 61.1           | 25.77          | 13.12          |
| 5        | 0     | 0      | 500        | 20          | 61.1           | 25.77          | 13.12          |
| 6        | 0     | -1     | 500        | 10          | 82.13          | 5.89           | 11.98          |
| 7        | 0     | 1      | 500        | 30          | 43.16          | 31             | 26.84          |
| 8        | -1    | 0      | 300        | 20          | 94.38          | 7.12           | 4.2            |
| 9        | 0     | 0      | 500        | 20          | 61.1           | 25.77          | 13.12          |
| 10       | 0     | 0      | 500        | 20          | 61.1           | 25.77          | 13.12          |
| 11       | 1     | 1      | 700        | 30          | 29.92          | 30.19          | 39.89          |
| 12       | -1    | 1      | 300        | 30          | 79.29          | 13.15          | 7.51           |
| 13       | 1     | -1     | 700        | 10          | 74.46          | 12.77          | 12.77          |

A = Temperature (°C)
B = Time (min)

Y<sub>C_PKs</sub> = Yield of char from PKS (wt%)
Y<sub>T_PKs</sub> = Yield of tar from PKS (wt%)
Y<sub>G_PKs</sub> = Yield of gas from PKS (wt%)

### Table 4: Parameter Estimation from Regression Analysis of PKS

| Model Factors | Coefficients | F-Values | p-Values |
|---------------|--------------|----------|----------|
| Model         | 64.77        | 41.49    | 0.0001*  |

| Responses | Model Factors | Coefficients | F-Values | p-Values |
|-----------|---------------|--------------|----------|----------|
| Yield of Char Y<sub>C_PKs</sub> | A | -22.07 | 51.95 | 0.0001* |
| | B | -17.06 | 31.03 | 0.0002* |
| | R<sup>2</sup> | 0.8709 | 17.22 | 0.0008* |
| | Model | 25.83 | 17.22 | 0.0008* |
| Yield of Tar Y<sub>T_PKs</sub> | A | 9.77 | 35.56 | 0.0006* |
| | B | 9.13 | 31.05 | 0.0008* |
| | A<sup>2</sup> | -3.99 | 2.73 | 0.1427 |
| | B<sup>2</sup> | -7.52 | 9.69 | 0.0170* |
| | AB | 1.29 | 0.41 | 0.5409 |
| | R<sup>2</sup> | 0.81 | 15.39 | 0.0035* |
| Yield of Gas Y<sub>G_PKs</sub> | A | 11.35 | 112.85 | 0.0001* |
| | B | 8.09 | 57.35 | 0.0001* |
| | AB | 5.14 | 15.39 | 0.0035* |
| | R<sup>2</sup> | 0.9383 | 15.39 | 0.0035* |
MODEL EQUATION OF OPH PRODUCT YIELDS

\[
Y_{\text{COPH}} = 38.78 - 16.98A - 25.32B + 6.29A^2 + 14.13B^2 \quad R^2 = 0.9690
\]

\[
Y_{\text{TOPH}} = 47.42 + 7.53A + 13.63B - 17.08A^2 - 13.12B^2 \quad R^2 = 0.8934
\]

\[
Y_{\text{OPH}} = 13.74 + 9.45A + 11.69B + 10.83A^2 \quad R^2 = 0.8937
\]

Table 5: Analysis of Variance (ANOVA) for the Responses

| Responses | Source of Variance | Degree of Freedom | Sum of Squares | Mean Square | F | Adjusted R² |
|-----------|-------------------|-------------------|----------------|-------------|---|-------------|
| \( Y_{\text{COPH}} \) | Regression        | 5                 | 6572.58        | 1314.66     | 76.02 | 0.969       |
|           | Residual          | 7                 | 121.05         | 17.29       |     |             |
|           | Total             | 12                | 6693.63        |             |     |             |
|           | Lack of fit       | 3                 | 121.05         | 40.35       |     |             |
|           | Regression        | 5                 | 3512.49        | 702.5       | 21.1 | 0.8934      |
| \( Y_{\text{TOPH}} \) | Residual          | 7                 | 233            | 33.29       |     |             |
|           | Total             | 12                | 3745.49        |             |     |             |
|           | Lack of fit       | 3                 | 233            | 77.67       |     |             |
|           | Regression        | 5                 | 1736.29        | 347.26      | 21.19| 0.8937      |
| \( Y_{\text{OPH}} \) | Residual          | 7                 | 114.74         | 16.39       |     |             |
|           | Total             | 12                | 1851.03        |             |     |             |
|           | Lack of fit       | 3                 | 114.74         | 38.25       |     |             |

* Significant at p <0.05 level

Figure 1: (a) Response Surface Cubic Plot showing the 3D Effects of Temperature, Time and their Interaction on the Optimum Char yield from PKS. (b) Contour Plot of Figure 1a.
Figure 2 (a) Response Surface Cubic Plot showing the Effects of Temperature, Time and their interaction on the Optimum Tar yield from PKS. (b) Contour Plot of Figure 2a.

Figure 3: (a) Response Surface Cubic Plot showing the Effects of Temperature, Time and their Interaction on the Optimum Gas yield from PKS. (b) Contour Plot of Figure 3a.
4.0 CONCLUSION

This study has shown that PKS lend itself to pyrolysis process under different pyrolysis conditions and these have produced different amounts of pyrolytic products (Char, tar and gas). In general, as the pyrolysis temperature increases, the char or solid production decreases and vice versa. The study has also demonstrated the applicability of response surface methodology in selecting pyrolysis variables that maximises product yields from PKS. Pyrolysis of PKS gave the optimum char yield of 99.27 wt% at 304.28 °C, optimum tar yield of 35.22 wt% at 700 °C and optimum gas yield of 39.81 wt% at 700 °C.

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