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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 ºC between 10 - 30 minutes at 5 minutes interval. This was repeated for temperatures of 400, 500, 600 and 700 ºC 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 0.9242 is in reasonable agreement with the Adj 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 ºC 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 ºC and 12 min., 35 wt% tar at 700 ºC and 27 min., and 40 wt% gas at 700 ºC and 30 min. The results obtained showed that PKS can be readily pyrolised 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 negligible contents of sulfur, nitrogen and ash which give lower emissions of SO_2, NO*x*, soot and net emission of CO_2 compared to conventional fossil fuels, thus keeping the environment and the public's health safe (Qi, *et al*., 2006; Tsai, *et al*., 2006a, Okekunle, *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 ºC) 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, cellulosic 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 ºC followed by a slow decrease at higher temperatures and approaching a constant value at 800 ºC. 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 7000C 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 was 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 ^oC 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 ^oC. 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 $$Y = \frac{mass\ of\ \ product}{mass\ of\ \ sample} \times 100 \qquad (1)$$

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).

Table1: Experimental Factors and Responses

Type	**Variables**	**Symbols**
Factors	Temperature	A
	Duration	B
Responses	Char yield	Y_c
	Tar yield	Y_t
	Gas yield	Y_g

Table 2: Experimental Values of Coded Levels

	Coded Levels		
Factors	**-1**	**0**	**+1**
A (^0C)	300	500	700
B (Min)	10	20	30

2.4 Analysis of Data and Response Equations.

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.

$$Y = b_0 + \sum_{i=1}^{m} b_i x_i + \sum_{i=1}^{m} b_{ii} x^2{}_i + \sum_{i \neq j}^{m} b_{ij} x_i x_j \qquad (2)$$

where Y is the predicted response, b_0 is the value of the fitted response at the centre point, and b_i, b_{ii}, 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.

$$\text{Maximize } Y = f(AB) \qquad (3)$$

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 line 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 statistics of the estimates for the best functions adopted, taking into consideration all main effects, linear, quadratic, and interaction for each model are as shown on Table 4. The coefficients of determination (R^2) for the responses (char, tar and gas) 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. Base on the p values, the regression coefficient that were significant at p<95% 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.

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

$$R^2 = 0.8709 \qquad (4)$$

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

$$R^2 = 0.8711 \qquad (5)$$

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

$$R^2 = 0.9383 \qquad (6)$$

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 °C and B = 12.43 minutes, 35.22% tar at A = 700.00 °C and B = 26.94 minutes and 39.81% gas at A = 700 °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 observe 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) the 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).

Table 3: Full Factorial Design Arrangement and Responses for PKS

	Coded Level		**Actual Values**		**Responses**		
Exp. No.	**A(°C)**	**B(min)**	**Temp. (°C)**	**Time (min)**	$Y_{C_{PKS}}$	$Y_{T_{PKS}}$	$Y_{G_{PKS}}$
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)
$Y_{C_{PKS}}$ = Yield of char from PKS (wt%)
$Y_{t_{PKS}}$ = Yield of tar from PKS (wt%)
$Y_{g_{PKS}}$ = Yield of gas from PKS (wt%)

Table 4: Parameter Estimation from Regression Analysis of PKS

Estimated Coefficient of the fitted model for properties based on t-statistics

Responses	Model Factors	Coefficients	F-Values	p-Values
Yield of Char $Y_{C_{PKS}}$	Model	64.77	41.49	0.0001*
	A	-22.07	51.95	0.0001*
	B	-17.06	31.03	0.0002*
	R^2	0.8709		
Yield of Tar $Y_{t_{PKS}}$	Model	25.83	17.22	0.0008*
	A	9.77	35.56	0.0006*
	B	9.13	31.05	0.0008*
	A^2	-3.99	2.73	0.1427
	B^2	-7.52	9.69	0.0170*
	AB	1.29	0.41	0.5409
	R^2	0.811		
Yield of Gas $Y_{g_{PKS}}$	Model	15.23	61.86	0.0001*
	A	11.35	112.85	0.0001*
	B	8.09	57.35	0.0001*
	AB	5.14	15.39	0.0035*
	R^2	0.9383		

* Significant at $p < 0.05$ level

MODEL EQUATION OF OPH PRODUCT YIELDS

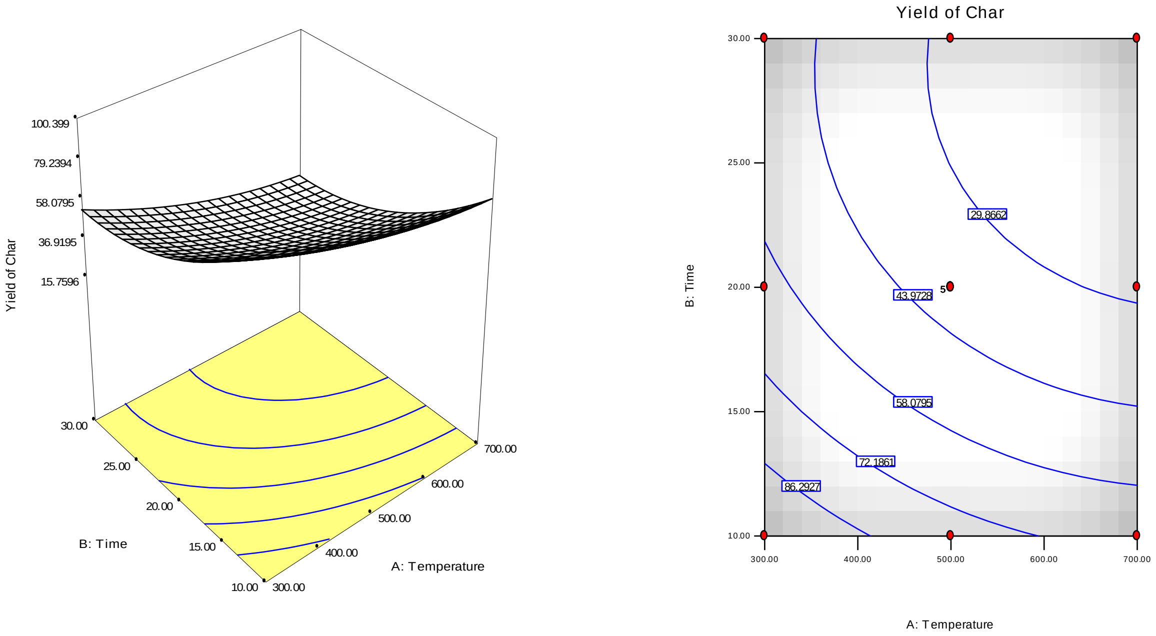
$$Y_{C_{OPH}} = 38.78 - 16.98A - 25.32B + 6.29A^2 + 14.13B^2 \qquad R^2 = 0.9690$$

$$Y_{t_{OPH}} = 47.42 + 7.53A + 13.63B^2 - 17.08A^2 - 13.12B^2 \qquad R^2 = 0.8934$$

$$Y_{g_{OPH}} = 13.74 + 9.45A + 11.69B + 10.83A^2 \qquad R^2 = 0.8937$$

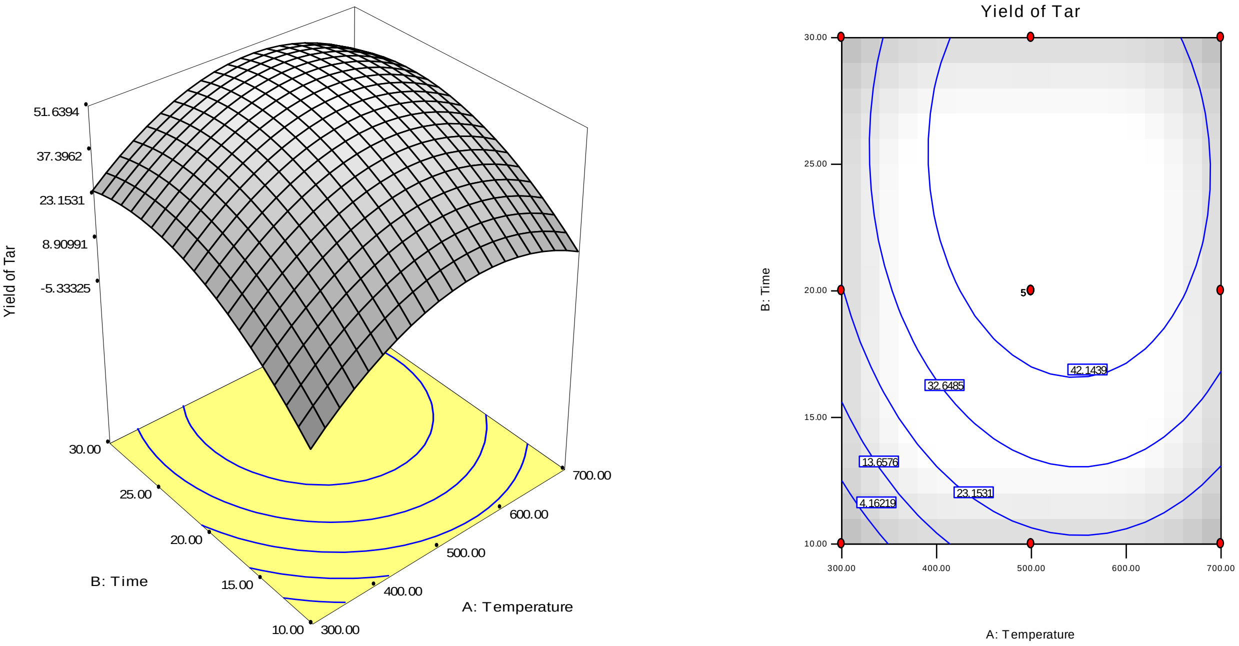
Table 5: Analysis of Variance (ANOVA) for the Responses

Responses	Source of Variance	Degree of Freedom	Sum of Squares	Mean Square	F	Adjusted R^2
$Y_{C_{OPH}}$	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		
$Y_{t_{OPH}}$	Regression	5	3512.49	702.5	21.1	0.8934
	Residual	7	233	33.29		
	Total	12	3745.49			
	Lack of fit	3	233	77.67		
$Y_{g_{OPH}}$	Regression	5	1736.29	347.26	21.19	0.8937
	Residual	7	114.74	16.39		
	Total	12	1851.03			
	Lack of fit	3	114.74	38.25		



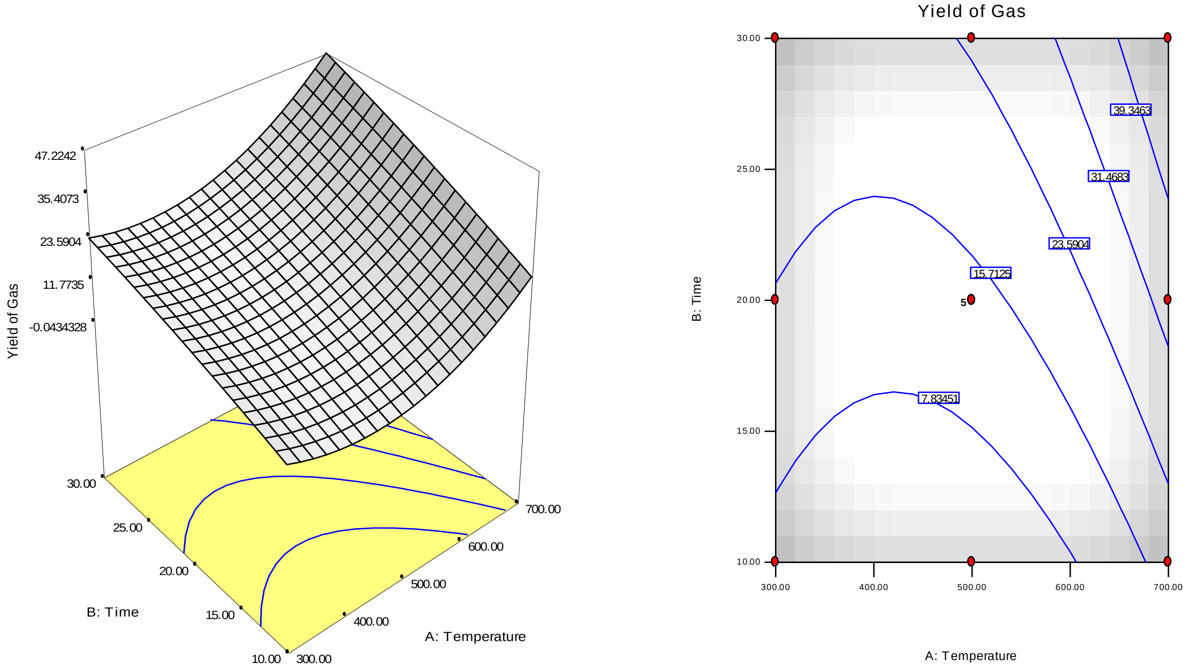
(a) (b)

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.



(a) **(b)**

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.



(a) **(b)**

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 w% at 304.28 ºC, optimum tar yield of 35.22 wt% at 700 ºC and optimum gas yield of 39.81wt% at 700 ºC.

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