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Influence of Operating Parameters on the Product Yields During Pyrolysis of Oil Palm Husk

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Abstract

The influence of operating parameters on the product yields during the pyrolysis of Oil Palm Husk (OPH) was investigated in this study. 0.5 kg of dried sample of OPH was loaded into a steel retort, and the retort interior was rendered airtight. The retort was then placed into the furnace chamber and the OPH 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 model terms. It was observed that the experimental data fitted better because of the Pred R-Squared of 0.8283 is in reasonable agreement with the Adj R-Squared of 0.9690. 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 1.54% was achieved at pyrolysis temperature and duration values of 300 °C and 10 min respectively. The optimum conversion yields of oven-dried weight of OPH char, tar and gas products at their respective pyrolysis conditions were 97 wt% char at 302 °C and 10 min., 51 wt% tar at 559 °C and 25 min., and 47 wt% gas at 700 °C and 30 min. The results obtained showed that OPH can be readily pyrolised to obtain optimum yield of bio fuels (gas, tar and char).

Keywords: Pyrolysis, Oil-palm Husk, Bio fuels, Gas, Tar, Char and Response surface methodology.

1.0 Introduction

The world's dependence on fossil fuel as the main source of energy is continuously increasing through time because of the rapid industrial and economic development and increase in population. With the depletion of fossil fuel reserves, renewable energy development should be focused on to renovate the energy source structures and keep sustainable development safe (Qi *et al*., 2007). Bio-fuel produced from biomass 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. Biomass is considered to be the oldest and one of the most promising renewable energy sources. It already provides approximately 14 % of the total worldwide energy needs (Funda, *et al*., 2006). Also, biomass produces less emissions of SO_2, NO_X, soot and net emission of CO_2 (Qi *et al*., 2007). Biomass resources such as forest residues, low-grade plants, agricultural residues, and municipal solid wastes are composed of organic raw materials that can be converted to energy (Caglar and Demirbas, 2000). In addition, biomass wastes 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*., 2007; Tsai, *et al*., 2006). In countries of excess production of agricultural residues such as Nigeria, eco-friendly and more energy efficient utilization alternatives must be developed. The high production of agricultural residues in Nigeria could provide considerable amount of energy recovery if properly utilized (Williams and Nugranad, 2000; Lucas, *et al*., 2014).

Application of thermo-chemical conversion processes such as combustion, fermentation, gasification and pyrolysis, on biomass could produce fuels with higher heating values efficiently and economically, rather than just burning the biomass directly to produce heat or power (Caglar and Demirbas, 2000). Pyrolysis is one of the most promising thermo-chemical processes, producing solid fuel (char), Liquid fuel (tar) and gas, all of which have potential end uses. Certain factors determine the distributions and qualities of these products so that pyrolysis conditions can be optimized for either char, tar, or gas production (Encinar *et al*., 1997; Williams and Nugranad, 2000; Zanzi *et al*., 2002; Bridgewater and Peacock, 2000). Pyrolysis of biomass is normally carried out in the absence of oxygen at temperature above 400-500 °C (Lucas and Itabiyi, 2012). Conventional pyrolysis consists of the slow, irreversible, thermal decomposition of the organic components in biomass. Slow pyrolysis was traditionally used for the charcoal production. Fast pyrolysis was used to obtain high yield of liquid at moderate temperature and short residence time.

Pyrolysis operating parameters, such as pyrolysis temperatures, heating rates, duration, particle size and moisture contents are key factors affecting the three fractions (Baumlin *et al*., 2006). For example, higher char yields are obtained at low temperatures or low heating rates than at higher temperatures or fast heating rates. To use biomass such as wood as an alternative carbon source for producing novel carbon-based materials, studies on the effects of pyrolysis process conditions will be necessary to maximize the yields of the most economically valuable products.

In the conversion of coal to char, it has been discovered that both raw material and operating conditions such as the heating rate, temperature and duration influence char yield (Guerrero *et al*., 2005). Studies on the influence of the coal feedstock and the experimental conditions on the characteristics of the char from co-pyrolysis of coal and petroleum residue indicated that char yields increased as temperature and pressure increased (Suelves *et al*., 2002). The yield of chars from the pyrolysis of maize stalk decreased from 22 to 16.3 percent as the temperature increased from 600 to 900 °C. The decrease in H (hydrogen)/C (carbon) was more than twofold that in 0 (oxygen)/C (Fu *et al*., 2009). The elemental composition of char from the pyrolysis of the pinewood chips varies with the pyrolysis temperature (Demirbas, 2004). Previous studies on biomass and coal pyrolysis have shown that the increase in temperature leads to decrease in char yields because the gasification reactions take place at higher temperatures (Encinar *et al*., 2000).

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 higher ash content in agricultural residues favors the charring reactions. The higher lignin content in olive waste results in a higher char yield in comparison with straw. Therefore, this work investigated the influence of operating parameters on the product yields during the pyrolysis of Oil Palm Husk.

2.0 Methodology

2.1 Oil-palm husk material preparation.

Oil-palm husk used for the pyrolysis experiments in this study was obtained from an oil-palm industry in Otamokun, Oyo State, 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 balance (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 influence of operating parameters on the product yields from OPH. 0.5 kg of dried OPH was 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 a water-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 condensate 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 $$Y = \frac{mass\ of\ product}{mass\ of\ sample} \times 100 \quad (1)$$

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 OPH. 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 (^{0}C)	300	500	700
B (Min)	10	20	30

2.4 Analysis of Data and Response Equations.

Regression Models were developed for OPH 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 OPH 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.

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

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.

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

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 OPH Product Yields.

The effect of FFD on the OPH 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 0.9690, 0.8934 and 0.8937 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 76.02, 21.10 and 21.19 respectively. These values were significant at $p< 0.05$ indicating good model fit.

$$Y_{C_{OPH}} = 38.78 - 16.98A - 25.32B + 6.29A^2 + 14.13B^2 \qquad R^2 = 0.9690 \tag{4}$$

$$Y_{t_{OPH}} = 47.42 + 7.53A + 13.63B^2 - 17.08A^2 - 13.12B^2 \qquad R^2 = 0.8934 \tag{5}$$

$$Y_{g_{OPH}} = 13.74 + 9.45A + 11.69B + 10.83A^2 \qquad R^2 = 0.8937 \tag{6}$$

where: $Y_{C_{OPH}}$ = Yield of char from OPH (wt%)
$Y_{t_{OPH}}$ = Yield of tar from OPH (wt%)
$Y_{g_{OPH}}$ = Yield of gas from OPH (wt%)
A = Temperature (^oC)
B = Time (minutes).

3.2 *Optimization of Pyrolysis Process.*

Response surface methodology was used for the optimization of the pyrolysis process of the feedstocks (OPH) 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 values obtained for OPH pyrolysis product yields and their respective pyrolysis conditions are: 97.14 % char at A = 301.97 ^oC and B = 10.05 minutes, 51.48 % tar at A = 559.40 ^oC and B = 24.93 minutes and 47.22 % gas at A = 700 ^oC 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 time as a single factor was the most influential factor, because of its higher F-value. The time at which pyrolysis process was conducted is highly significant ($p < 0.05$) with an F-value of 222.39 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 OPH 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 OPH decreases as the pyrolysing temperature and time increase. 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 residues or through the greater primary decomposition of the OPH at higher temperatures.

The cubic response surface plot shown in Figure 2(a) depicts the effect of pyrolysing temperature and time on the OPH 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. 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 OPH 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 OPH

	Coded Level		**Actual Values**		**Responses**		
	A(°C)	**B(min)**	**Temp. (°C)**	**Time(min)**	$Y_{C_{OPH}}$	$Y_{t_{OPH}}$	$Y_{g_{OPH}}$
1	0	0	500	20	37.96	48.85	13.13
2	1	0	700	20	24.49	38.60	36.91
3	1	1	700	30	17.72	33.68	48.60
4	-1	-1	300	10	96.42	1.54	2.04
5	-1	0	300	20	69.78	14.94	15.28
6	-1	1	300	30	48.23	25.71	26.06
7	0	0	500	20	37.96	48.85	13.13
8	0	1	500	30	29.43	50.24	20.33
9	0	0	500	20	37.96	48.85	13.13
10	0	0	500	20	37.96	48.85	13.13
11	0	0	500	20	37.96	48.85	13.13
12	1	-1	700	10	70.33	15.10	14.57
13	0	-1	500	10	80.53	11.23	8.24

A = Temperature (°C)
B = Time (min)
$Y_{C_{OPH}}$ = Yield of char from OPH (wt%)
$Y_{t_{OPH}}$ = Yield of tar from OPH (wt%)
$Y_{g_{OPT}}$ = Yield of gas from OPH (wt%)

Table 4: Parameter Estimation from Regression Analysis of OPH

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

Responses	Model Factors	Coefficients	F-Values	p-Values
	Model	38.78	76.02	0.0001*
Yield of Char $Y_{C_{OPH}}$	A	-16.98	100.06	0.0001*
	B	-25.32	222.39	0.0001*
	A^2	6.29	6.32	0.0402*
	B^2	14.13	31.90	0.0008*
	AB	-1.10	0.28	0.6115*
	R^2	0.9690		
	Model	47.42	21.10	0.0004*
Yield of Tar $Y_{t_{OPH}}$	A	7.53	10.23	0.0151*
	B	13.63	33.47	0.0007*
	A^2	-17.08	24.21	0.0017*
	B^2	-13.12	14.28	0.0069*
	AB	-1.40	0.23	0.6429
	R^2	0.8934		
	Model	13.74	21.19	0.0004*
Yield of Gas $Y_{g_{OPH}}$	A	9.45	32.69	0.0007*
	B	11.69	50.02	0.0002*
	A^2	10.83	19.75	0.0030*
	B^2	-0.98	0.16	0.6981
	AB	2.50	1.53	0.2562
	R^2	0.8937		

* Significant at $p<0.05$ level

MODEL EQUATION OF OPH PRODUCT YIELDS

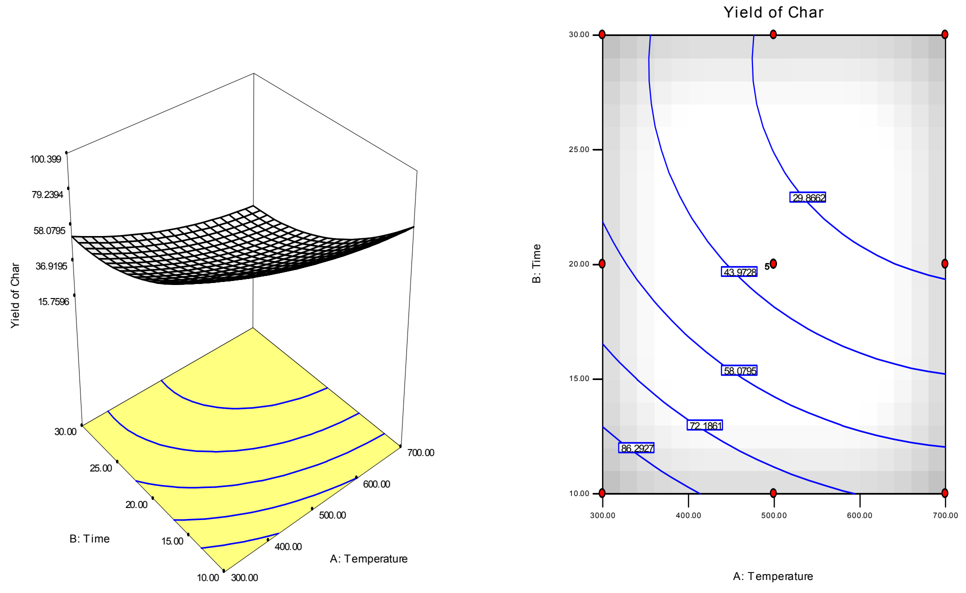
$$Y_{C_{OPH}} = 38.78 - 16.98A - 25.32B + 6.29A^2 + 14.13B^2 \qquad R^2 = 0.9690$$

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

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

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

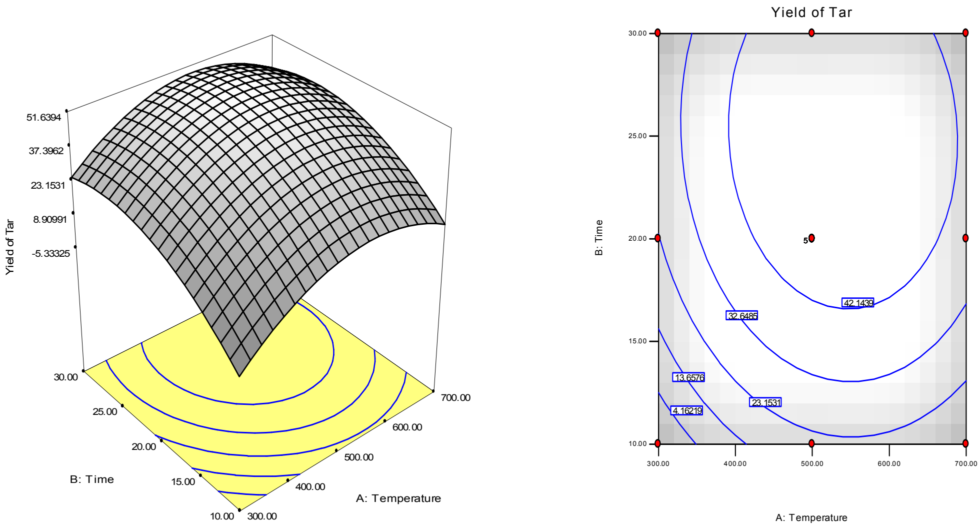
Responses	Source of Variance	Degree of Freedom	Sum of Squares	Mean Square	F	Adjusted R^2
$Y_{C_{OPH}}$	Regression	5	6572.58	1314.66	76.02	0.9690
	Residual	7	121.05	17.29		
	Total	12	6693.63			
	Lack of fit	3	121.05	40.35		
$Y_{t_{OPH}}$	Regression	5	3512.49	702.50	21.10	0.8934
	Residual	7	233.00	33.29		
	Total	12	3745.49			
	Lack of fit	3	233.00	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)

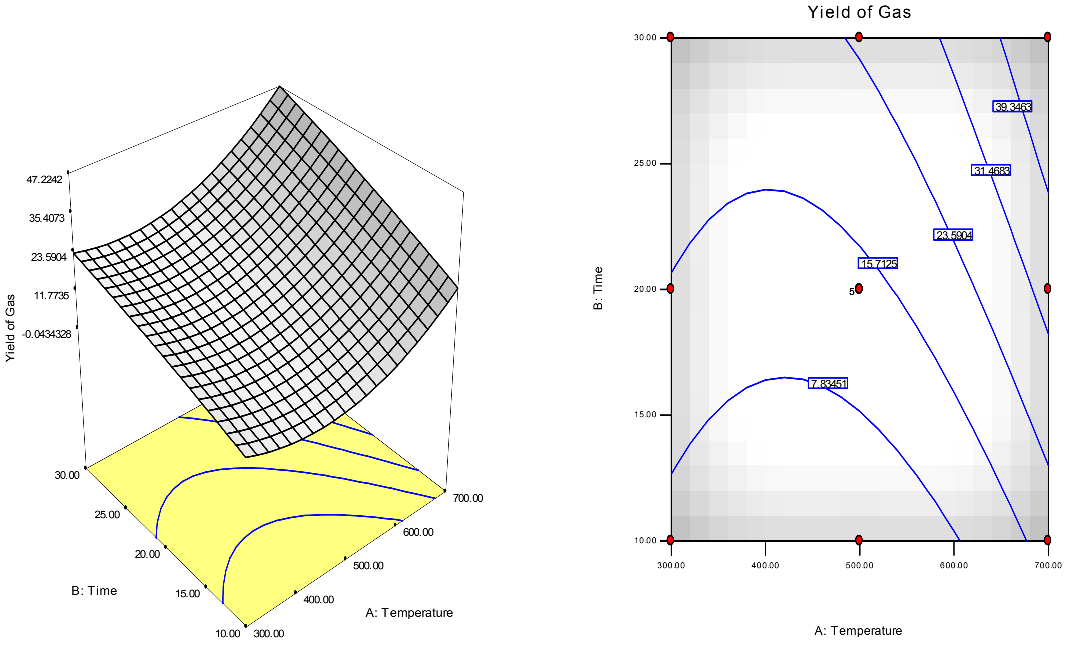
(b)

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

4.0 Conclusion

This study has shown that pyrolysis of OPH under different pyrolysis conditions (temperatures and time) 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 selectingg pyrolysis parameters that maximises product yields from OPH. Pyrolysis of OPH gave the optimum char yield of 97.14 % at 301.97 ^{o}C, optimum tar yield of 51.48 wt% at 559 ^{o}C and optimum gas yield of 47.22 wt% at 700 ^{o}C.

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