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# Optimization of the Product Yields of the Pyrolysis of Palm Kernel Shell in a Fixed-Bed Reactor Using Response Surface Methodology (RSM)

**[1]*Joseph O. O., [2]Akinola A. O., [3]Ogedengbe T. I., [4]Akinnuli B. O.**

[1]Department of Mechanical Engineering, Federal Polytechnic, Ilaro, Nigeria

[1,2,3]Department of Mechanical Engineering, Federal University of Technology, Akure, Nigeria

[4]Department of Industrial and Production Engineering, Federal University of Technology, Akure, Nigeria

*Corresponding Author's E-mail: olakunle.joseph@federalpolyilaro.edu.ng

***Abstract* - Pyrolysis is a method used for the thermo-chemical conversion of biomass and agro wastes into three major by-products namely, biochar, bio-oil, and biogas in the absence of oxygen. Global waste generation is increasing at an alarming rate. Palm kernel shell (PKS) is among the palm wastes accumulating in many palm oil mills around the globe with the attendant problems in waste management. The utilization of coal for energy generation is more expensive and not environmentally friendly. Bio-oil refineries and industries require the utilization of minimum resources (input) to achieve optimal response (output) in the production process. However, more than 30% of the energy content derivation from the pyrolysis process is wasted due to non-optimization of the process parameters. This study investigated the optimization of the product yields of the pyrolysis of PKS in a fixed-bed batch reactor using response surface methodology (RSM). Masses of 1 kg, 2 kg, and 3 kg were pyrolyzed in batches at process temperature variations of 200 $^{O}$C, 300 $^{O}$C, 400 $^{O}$C, and 500 $^{O}$C. Design Expert 12 software (Version 12.0.3.0 Stat-Ease Inc. MN, USA) and Analysis of Variance (ANOVA) were used for the statistical analysis. Lack-of-fit, adjusted, and predicted multiple correlation coefficients and coefficient of variation of the different polynomial models were compared to select the best-fitting polynomial model. The optimal feedstock parameters were 2.66 kg of PKS and 410.0 $^{O}$C process temperature. The predicted results were 0.986 kg of biochar, 1.205 kg of bio-oil, 0.481 kg of biogas, 1116.5 cm$^3$ of bio-oil, and 140.6 minutes. The experimental results were 0.894 kg of biochar, 1.316 kg of bio-oil, 0.498 kg of biogas, 1208.9 cm$^3$ of bio-oil, and at 162.8 minutes through validation. The validated quadratic model was suitable for the optimization and RSM is a numerical, statistical, and mathematical tool for modeling and optimization of the pyrolysis process in a fixed-bed batch reactor.***

***Keywords:*** Bio-oil, Design Expert 12, Fixed-bed reactor, Modelling, Optimization, Palm kernel shells, Product yields, Pyrolysis, Response Surface Methodology.

## 1. INTRODUCTION

Pyrolysis is a method used for the thermo-chemical conversion of biomass into three major by-products i.e. biochar, biogas, and bio-oil in the absence of oxygen [1]. It is a thermo-chemical technique employed to recover energy from any valued biomass [2] [3]. Pyrolysis of wood is one of the first chemical processes adopted by humans to produce charcoal from wood and coke from coal. Pyrolysis is a process for the combustion of most solid fuels. Bio-crude is a synthetic fuel under investigation as a substitute necessary for petroleum.

Global waste generation is increasing at an alarming rate. Synthetic materials, plastics, nylon, and poly products are adversely non-biodegradable and they negatively affect the environment through global warming and pollution. Palm kernel shell (PKS) is one of the palm wastes that have accumulated in numerous palm oil mills in the nation and pose challenges in waste management [4] [5]. Hence, proper utilization of this solid biomass waste is necessary for environmental and economic reasons [4]. In some cases, PKS is just recycled by direct burning into charcoal while the bio-oil is wasted.

Bio-crude refineries contribute to a huge quantity of waste such as empty fruit bunches, palm kernel shells, palm mesocarp fiber, and other waste products [6] [7]. Various techniques and methods have been duly utilized to provide different forms of energy for human needs and development, yet energy demands are yet to be met. The use of high-heating value materials such as coal is an additional expense and often results in environmental damage [7]. Though, during the process of thermal-decomposition of biomass in boilers, numerous disadvantages such as fouling, low heating value, storage, and handling of the biomass have become limitations

 

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to this common technique when the waste is usually utilized as fuel sources to produce the energy needed for operating mills [8].

Despite the effort towards the complete exploitation of renewable energy (RE) sources to deliver a large percentage of the global energy need, the world's usage of fossil fuels and their related dangers have been constantly increasing over the years [9]. However, more than 30% of the energy content derivation from the pyrolysis process is wasted due to non-optimization of the process parameters and results from over-utilization of a particular resource (heavily) while some resources are under-utilized (idle) [8] [10]. Bio-oil refineries and industries need the integration and utilization of minimum resources of the independent variables (input) to achieve optimal response variables of the production process.

[2] revealed that a designed and fabricated fixed-bed batch reactor was used to pyrolyze waste tyres for the production of bio-oil. Optimization of the result shows that the reactor yielded a maximum pyro oil of 42 wt% at the temperature of 400 oC with a feed size of 15 cm3 [2]. [11] used Fourier transform infrared spectroscopy (FTIR) and gas chromatography–mass spectroscopy (GCMS) to optimize the pyrolysis of PKS the optimal yield was 38.40% at 450 °C with a heating rate of 50 °C min-1 and a nitrogen sweep gas flow rate of 50 ml min-1. [12] optimized Paper Waste Sludge (PWS) to give results of optimal bio-oil yields of 44.5 ±1.7 daf.wt% at 400oC and 59.9±4.1daf. wt% at 340 oC, when the intermediate pellet size of 4.84 ±0.15mm was used. [8] study the optimization of char production from PKS through microwave-assisted pyrolysis procedure using response RSM being the closest to this research. The optimal condition of reaction time (A), sample mass (B), and nitrogen gas flow rate (C) were at 31.58 min, 30 g, and 100 ml/min, producing calorific value, fixed carbon content, volatile matter content and yield percentage of 29.9 MJ/kg, 59.8 wt%, 36.4 wt%, and 40.0 wt% respectively [8].

Previously, the practical application of optimization has been utilized for cost-effectiveness, thermal conversion efficiencies, and optimal product yields depending on several design criteria to various degrees. However, no study has been done on the optimization of palm kernel shell wastes via response surface methodology (RSM) using Design Expert 12 version software without any aided reagent or process operations. Thus, this study becomes novel among the research lists in optimization and modeling. Hence, this research was designed to investigate the effect of the process parameters of the feedstock (independent variables) on the product parameters (response variables) for optimal values of the product yields using response surface methodology for the pyrolysis of palm kernel shell. The optimum design parameters were selected and methodically investigated to familiarise them with the thermal regime of optimum performance under various operating conditions [13].

Modeling the features and units of reactors during the pyrolysis process provides the data and information needed for performance evaluation to obtain optimal operation of the reactor [14]. Optimization of the process parameters of reactors during the pyrolysis process provides the data and information needed for modeling the developed reactor for a high and optimal yield of the end products [14]. This study investigated the optimization of the product yields of the pyrolysis of palm kernel shell in a fixed-bed batch reactor using response surface methodology (RSM) to achieve a set of optimal values of the product yield that satisfy the optimal utilization of the feedstock resources for optimal reactor economy. The relationship between the two independent factors (mass of feedstock and process temperature) and the five variable responses (mass of biochar, mass of bio-oil, mass of biogas, volume of bio-oil, and process time) were investigated in this study.

## 2. MATERIALS AND METHODS

### 2.1 Materials

Palm kernel shells were obtained from a community in Ilaro, Ogun State, Western part of Nigeria. The PKS were cleaned thoroughly, drained, and sundried at an average temperature of 42 $^{o}$C. foreign materials were removed from the PKS. Furthermore, the sample was pulverized and weighed into various batches of 1 kg, 2 kg, and 3 kg. The PKS were loaded into the reactor in batches for the pyrolysis to be carried out.

### 2.2 Experimental Set-up

Experimentation and testing of the developed reactor were carried out by variation of the input parameters (mass of feedstock and process temperature). Feedstock masses of 1 kg, 2 kg, and 3 kg were pyrolyzed in batches at process temperature variations of 200 oC, 300 OC, 400 OC, and 500 OC. The three major by-products (biochar, bio-oil, and biogas) were obtained at the end of the pyrolysis of each batch. The biochar product was obtained from the reactor and the mass was measured and recorded. The bio-oil was collected in a calibrated measuring cylinder and the volume was recorded while the mass was also measured with the aid of a beam balance. The mass of the biogas was obtained using the simple difference method of equation (1)

$$G = A + B + C \qquad (1)$$

Where: G is the mass of the PKS

 

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A is the mass of biochar
B is the mass of the bio-oil
C is the mass of the biogas

The reactor temperature and the ambient temperatures were recorded at an interval of 10 minutes. The heating rate of the electric heating element was also calculated using equation (2). The ambient temperature was used to calculate the heating rate,

$$H = \frac{T_f - T_i}{t_f - t_i} \qquad (2)$$

Where:
$H$ is the heating rate
$T_f$ is the temperature at the end of the pyrolysis process of each batch
$T_i$ is the temperature at the beginning of the pyrolysis process of each batch
$t_f$ is the time at the end of the pyrolysis process of each batch
$i_t$ is the time at the beginning of the pyrolysis process of each batch

[14] Stated that experimental test procedures are used for the pyrolysis of palm kernel shells for the performance evaluation of the reactor as shown in Figure 1.

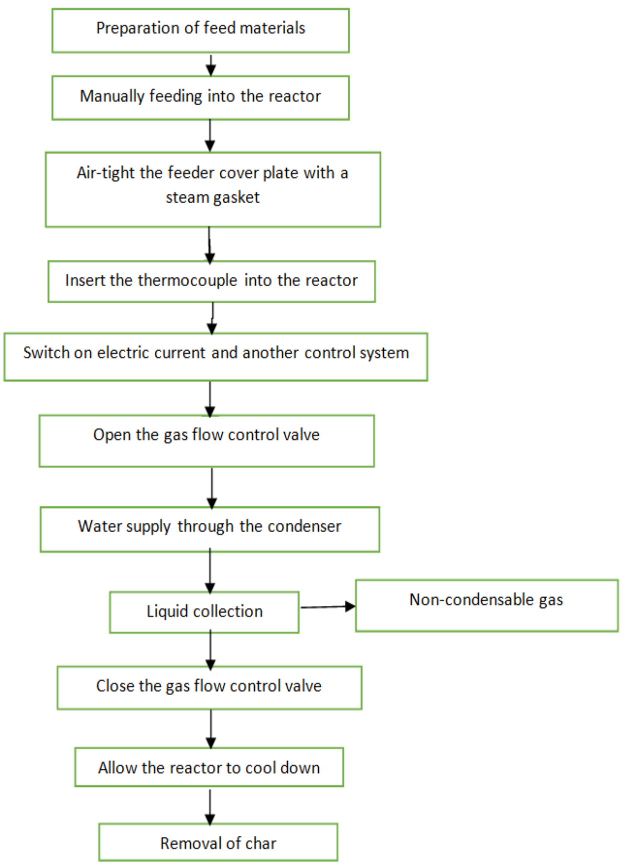


**Figure 1: Operational procedure for the pyrolysis process of the PKS**

### 2.3 Sample Analysis

Table 1 shows the approximate, ultimate analysis, and calorific values of the raw sample of PKS. The proximate analysis of moisture, volatile matter, fixed carbon, and ash content can be determined by percentages using a Thermogravimetric Analyzer, DTA/DSC TA Model SDT Q600 according to the ASTM D2974 [1]. [8] and [16] stated that the calorific value of PKS is determined using the calorimeter Leco AC-350 model according to ASTM D5468. [17] reported that the values of the physical and chemical characteristics of PKS by percentages can be determined through the proximate analysis presented in Table 1.

**Table 1: Physical and chemical characteristics of PKS**

| **Physical and Chemical Characteristics of PKS** | **Values** |
|---|---|
| Moisture mass fraction (%) | 12 |
| Particle size (mm) | 7-15 |
| Bulk density (kg m_3) | 0.56 |
| Proximate analysis | |
| Volatile mass fraction (%) | 74 |
| Fixed carbon mass fraction (%) | 23 |
| Ash mass fraction (%) | 3 |
| Ultimate analysis (daf)[a] | |
| **C** | 45.10 |
| **H** | 5.10 |
| **O** | 49.20 |
| **N** | 0.56 |
| **S** | 0.04 |
| An ultimate analysis is calculated on a dry and ash-free basis. | |

### 2.4 Experimental Design

The Response Surface Methodology (RSM) was utilized to design the coded and un-coded levels in the experimental design. This revealed the effect of the variation of the independent variables, which included the mass of palm kernel shell ($X_1$), and process temperature ($X_2$) on the response variables, such as the mass of biochar ($Y_1$), the mass of bio-oil ($Y_2$), the mass of biogas ($Y_3$), volume of bio-oil ($Y_4$), and process time ($Y_5$) to obtain a viable model for the pyrolysis process. Design Expert searches for a combination of factors that simultaneously satisfy the requirements placed on each of the responses and factors [18]. Central Composite Rotatable Design (CCRD) provided desired information with few numbers of experiment hence it was employed for the Design of Experiment (DoE). However, 13 experiments were carried out to obtain the needed data for the optimization. This experiment was a randomly performed investigation that included four factorial points, four axial points, and five central points. This experiment depended on two dependent variables which were used to design the number of

 

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experiments to be performed for the optimization. The number of experiments was derived from equation (3).

$$N = 2^n + 2n + 5 \quad (3)$$

Where:

$N$ is the number of experiments

$n$ is the number of independent variables

The CCRD with a full factorial was developed using DE as presented in Table 2. Real levels of independence variables were evaluated using equation (4).

$$U = Uo - Uc/\Delta U \quad (4)$$

Where:

$U$ is the coded level of independence variables

$Uo$ is the actual coded level of independence variable,

$Uc$ is the real value at the central point.

$\Delta U$ is the step change

The precise equations for each independence variable were obtained through equation (4) to derive their actual values. Hence, the coded levels which gave the values are expressed in equations (6) and (7).

$$Z_1 = (MP - 2)/1 \quad (5)$$

$$Z_2 = (PT - 350)/150 \quad (6)$$

Where:

$MP$ represents the mass of PKS

$PT$ represents the process temperature

**Table 2: Coded levels of the PKS with the corresponding independent variables**

| Independent variables | Symbols | Coded levels | | | | |
|---|---|---|---|---|---|---|
| | | -α | -1 | 0 | +1 | +α |
| Mass of palm kernel shell (kg) | $X_1$ | 0.585 | 1 | 2 | 3 | 3.414 |
| Process temperature (oC) | $X_2$ | 137.868 | 200 | 350 | 500 | 562.132 |

The second-order polynomial equation was used to relate the predicted responses (mass of bio-char, mass of bio-oil, mass of bio-gas, volume of bio-gas, and process time) as a determinant of an independent variable as given in equation (8). The polynomial order was non-aliased.

$$Z = \partial_o + \partial_1 X_1 + \partial_2 X_2 + \partial_{11} {X_1}^2 + \partial_{22} {X_2}^2 + \partial_{12} X_1 X_2 \quad (7)$$

Where Z is the response values, $\partial_j \, \partial_{jj}$ and indicates the values of direct and quadratic coefficients respectively, and $\partial_0$ is a constant. The values of the statistical significance of coefficients of determinations were evaluated.

### 2.5 Statistical Analysis

Data analysis and the significance of coefficients of determination were done with the aid of the DE. Statistical parameters (lack-of-fit, adjusted and predicted multiple correlation coefficients, and coefficient of difference) of various polynomial models were related to choosing the best-fitting polynomial model. Analysis of variance was used to determine the significant difference by expressing the F-value at the probability of 0.01, 0.1, and 0.5. The effects of the mass of the feedstock and the process temperature on the response variables were presented through the response plots.

## 3. RESULTS AND DISCUSSION

### 3.1 Fitting the Model

[19] reported that the relationship between two independent factors (mass of feedstock and process temperature) and five variable responses (mass of biochar, mass of bio-oil, mass of biogas, volume of bio-oil, and process time) can be investigated.

RSM is a mathematical, theoretical, and statistical technique for fitting models and model building for optimizing independent variable levels in an experimental design. [20] [21]. The effect of the mass of palm kernel shell (X1) and process temperature (X2) on the responses which included: the mass of biochar (Y1), the mass of bio-oil (Y2), the mass of biogas (Y3), the volume of bio-oil (Y4), and process time (Y5) for the pyrolysis experimentation is given in Table 3.

 

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**Table 3: Experimental design for the product yields with independent variables, experimental and predicted values of responses**

| | Mass of Palm Kernel Shell | Process Temperature | Mass of Bio Char | Mass of Bio-Oil | Mass of Bio Gas | The volume of Bio-Oil | Process Time |
|---|---|---|---|---|---|---|---|
| | Kg | °C | Kg | Kg | Kg | $cm^3$ | min |
| 1 | 3 | 500 | 0.89 | 1.44 | 0.65 | 1375 | 258 |
| 2 | 1 | 500 | 0.34 | 0.52 | 0.19 | 530 | 170 |
| 3 | 3 | 200 | 1.91 | 0.62 | 0.47 | 631 | 48 |
| 4 | 1 | 200 | 0.65 | 0.23 | 0.15 | 241 | 25 |
| 5 | 2 | 350 | 0.91 | 0.82 | 0.29 | 781 | 75 |
| 6 | 3.41421 | 350 | 1.31 | 1.45 | 0.68 | 1276 | 112 |
| 7 | 2 | 350 | 0.89 | 0.84 | 0.3 | 786 | 80 |
| 8 | 2 | 350 | 0.9 | 0.83 | 0.28 | 789 | 82 |
| 9 | 2 | 350 | 0.93 | 0.81 | 0.29 | 780 | 78 |
| 10 | 2 | 350 | 0.94 | 0.84 | 0.26 | 778 | 75 |
| 11 | 2 | 137.868 | 1.58 | 0.27 | 0.15 | 276 | 45 |
| 12 | 2 | 562.132 | 0.52 | 1.04 | 0.38 | 960 | 271 |
| 13 | 0.585786 | 350 | 0.23 | 0.28 | 0.09 | 308 | 52 |

The experiment values were utilized to compute the coefficients of polynomial equations to forecast the data obtained from the output variables. Regression equations from RSM are given in equations (8) to (12).

The coded factor equations are given as follows:

$$\text{Mass of biochar} = 0.914 + 0.417169X_1 - 0.353633X_2 - 0.1775X_1X_2 - 0.062625{X_1}^2 + 0.077375{X_2}^2 \quad (8)$$

$$\text{Mass of bio-oil} = 0.828 + 0.370579X_1 + 0.274868X_2 + 0.1325X_1X_2 + 0.004125{X_1}^2 - 0.100875{X_2}^2 \quad (9)$$

$$\text{Mass of biogas} = 0.284 + 0.201798X_1 + 0.0681586X_2 + 0.035X_1X_2 + 0.0605{X_1}^2 + 0.0005{X_2}^2 \quad (10)$$

$$\text{Volume of bio-oil} = 782.8 + 325.495X_1 + 250.04X_2 + 113.75X_1X_2 + 1.9125{X_1}^2 - 85.0875{X_2}^2 \quad (11)$$

$$\text{Process time} = 78 + 24.4816X_1 + 84.3265X_2 + 16.25X_1X_2 + 3.3125{X_1}^2 + 41.3125{X_2}^2 \quad (12)$$

[19] stated that statistical analysis results of experimental data via ANOVA could be presented probably with a quadratic polynomial model with coefficient of determination ($R^2$) values. Hence, the value of $R^2$ for the mass of biochar ($Y_1$), the mass of bio-oil ($Y_2$), the mass of biogas ($Y_3$), the volume of bio-oil ($Y_4$), and process time ($Y_5$) being 0.9931, 0.9881, 0.9853, 0.9979 and 0.9955. The result of the lack of fit was non-significant ($p \leq 0.05$) relative to the recorded error for all variables, which indicates that the developed model is statistically accurate [21]. [21] reported that as the value of $R^2$ advances to unity, the fitting results for modeling become better. However, [21] reported that the contrary view of lesser values of $R^2$ indicates that response variables are not suitable to describe the difference in performance. As stated by [19] and confirmed in this research, $R^2$ advanced to unity to show that the effects of the mass of palm kernel shell ($X_1$) and process temperature ($X_2$) on the response variables possibly will be effectively evaluated and explained via a quadratic polynomial model. ANOVA revealed a significant level of coefficients for sufficient analysis. The smaller the F-value and P-value are the more highly significant the effects on the terms of output variables.

 

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### 3.2 Effect of Independent Variables on the Response Variables

The pyrolysis of the PKS was successfully carried out by using the different batches of mass of PKS and the process temperature. The significance of the dependent variables on the mass of biochar ($Y_1$), the mass of bio-oil ($Y_2$), the mass of biogas ($Y_3$), the volume of bio-oil ($Y_4$), and process time ($Y_5$) are presented in Table 4. The product yield coefficients of regression are presented.

**Table 4: Regression coefficient values for the product yield of the pyrolysis process**

| Regression Coefficient | Mass of Bio Char | Mass of Bio-Oil | Mass of Bio Gas | Volume of Bio-Oil | Process Time |
|---|---|---|---|---|---|
| Intercept | 0.9140 | 0.8280 | 0.2840 | 782.80 | 78.00 |
| A-Mass of Palm Kernel Shell | 0.4172 | 0.3706 | 0.2018 | 325.49 | 24.48 |
| B-Process Temperature | -0.3536 | 0.2749 | 0.0682 | 250.04 | 84.33 |
| AB | -0.1775 | 0.1325 | 0.0350 | 113.75 | 16.25 |
| $A^2$ | -0.0626 | 0.0041 | 0.0605 | 1.91 | 3.31 |
| $B^2$ | 0.0774 | -0.1009 | 0.0005 | 85.09 | 41.31 |
| $R^2$ | 0.9931 | 0.9881 | 0.9853 | 0.9979 | 0.9955 |

### 3.2.1 Mass of bio-char

The mass of biochar yield depended on the mass of the palm kernel shell due to its significant effect on the mass of biochar. In the fit summary test the lack of fit p-value linear ($p<0.0001$), quadratic 0.0028 was suggested and the cubic level was aliased. [22] stated that a rise in the mass of palm kernel shells increased the mass of biochar formation in the pyrolysis process. Other independent variables that had a significant effect on the mass of biochar were determined by ANOVA for a quadratic model with a P-value of 201.03 which is significant, P-value ($p < 0.05$) specifies that the model terms are important and lack of fit F-value (12.69) shows it is also significant to the response of mass of biochar.

However, the significance of the mass of the PKS and the process temperature on the mass of biochar is shown in Figure 4. The two inputs (PKS and process temperature) influenced a quadratic consequence on the mass of biochar [23]. As soon as the mass of palm kernel shell is increased the mass of biochar was observed to also increase. In contrast, an increase in the process temperature produced a reduction of the mass of biochar due to further dryness of the biochar resulting in loss of weight [22]. Additionally, higher heat energy is required through the increase of the process temperature to further extract bio-oil and gas and produce biochar of lightweight [22]. Figure 2 shows the result surface response of the mass of biochar from the influence of the independent variables which include: the mass of the palm kernel shell and process temperature.

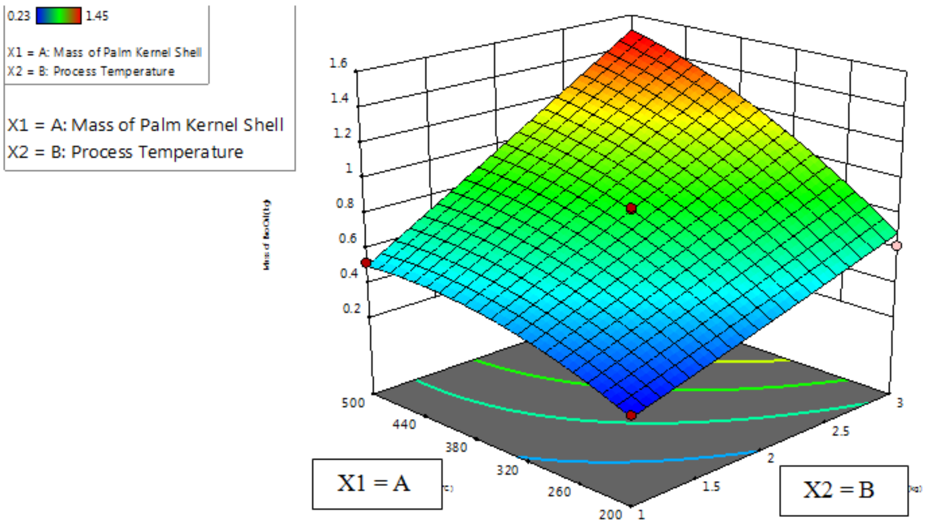


**Figure 2: Surface response of the mass of biochar from the independent variables**

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### 3.2.2 Mass of bio-oil

The mass of bio-oil yield is dependent on the independent variables which are the mass of palm kernel shell and the process temperature. The Fit summary linear value is < 0.0001, the quadratic model is proposed and the cubic is aliased. The independent variables that had a substantial result on the mass of biochar were the ANOVA for quadratic Model F-value of 116.46 indicates that the model is significant. Hence, only a 0.01% probability that an F-value was much enough to occur. P-values < 0.05 indicated that the terms of the model are effective. P-values > 0.1 indicate that the terms of the model are insignificant. The Lack of Fit F-value of 42.18 indicates the Lack of Fit is significant. Hence, 0.17% probability that a Lack of Fit F-value is great and the model might be established. The combined effects of the mass of the palm kernel shell and the process temperature on the mass of bio-oil computed data are shown in Figure 3 which explains the direct and proportional result of two input parameters (mass of PKS and process temperature).

The response of mass of bio-oil was obtained from the independent variables of mass of palm kernel shell and process temperature. A rise in the mass of PKS strengthens the mass of bio-oil produced [24]. Advancement in process time increases the mass of the bio-oil. [25] reported that both independent variables have a direct significance on the mass of the bio-oil values. A significant upward trend was observed in the mass of bio-oil yielded through a significant rise in the mass of the palm kernel shell and the process temperature. Figure 3 illustrates the surface response of the mass of bio-oil concerning the independent variables.

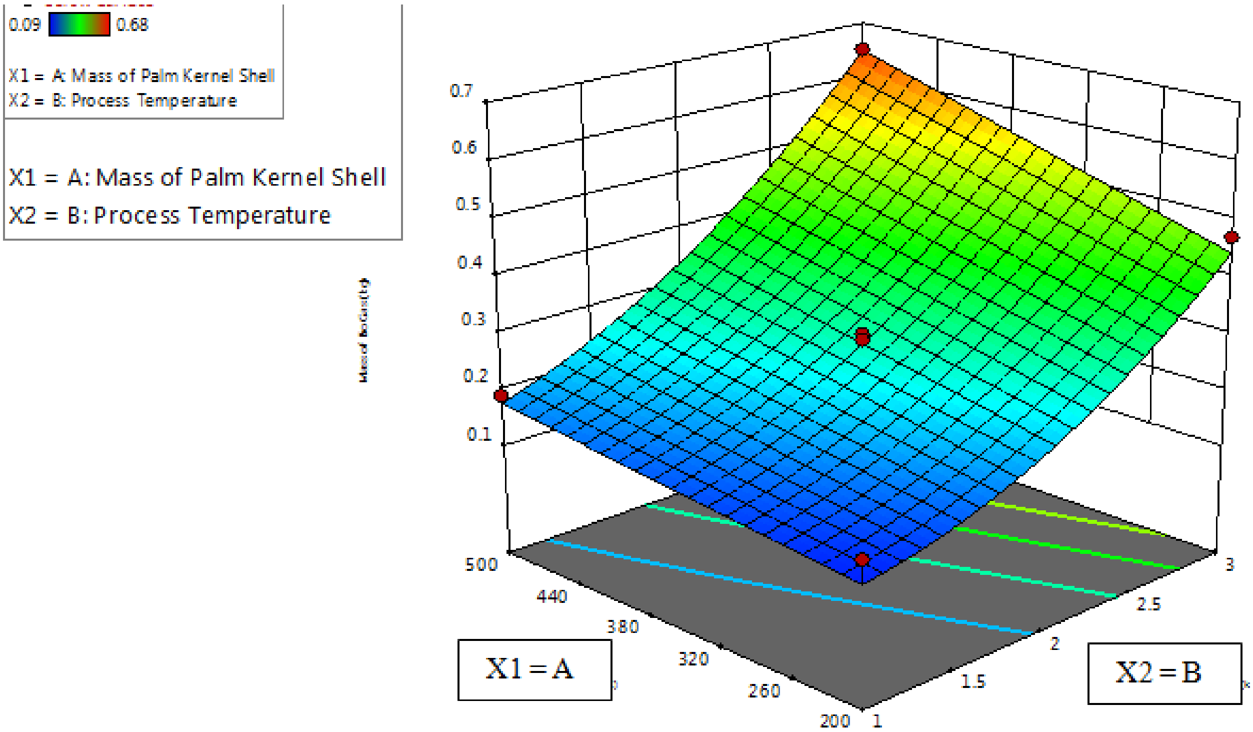


**Figure 3: Surface response of the mass of bio-oil from the independent variables**

### 3.2.3 Mass of biogas

The mass of biogas mainly depended on the mass of the palm kernel shell as it had a remarkable result on the biogas product at direct (< 0.0001), quadratic (0.0027), and cubic value of 0.4118 for the Fit summary analysis. The biogas is released at an average temperature of 60 $^{o}$C. The ANOVA for the Quadratic model indicated an F-value of 93.82 meaning the model is substantial and a P-value of < 0.0001. Additionally, a P-value < 0.05 shows terms of the model are important. If the values are higher than 0.1 (> 0.1) shows that the terms of the model terms are insignificant. The Lack of Fit F-value of 7.18 means that the Lack of Fit is significant with a probability of 4.35%. The response of the biogas was influenced significantly by both independent variables which are the mass of the palm kernel shell and the process temperature. [26] stated that the increase in the mass of biogas was caused by a linear increase in the mass of the palm kernel shell and the process time and vice versa. The response on the biogas is presented in a model graph in Figure 4 for the graphic surface optimization. Figure 4 illustrates the surface response of the mass of biogas from the independent variables during the pyrolysis of the PKS.

 

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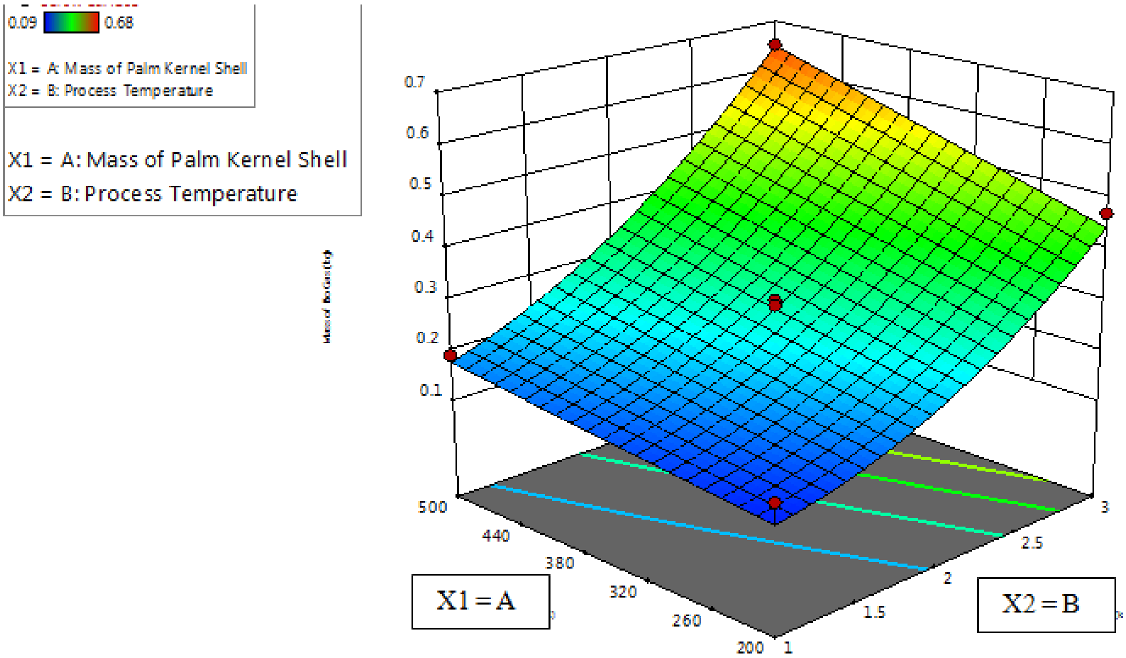


**Figure 4: Surface response of the mass of biogas from the independent variables**

### 3.2.4 Volume of bio-oil

The response of the volume of bio-oil is linearly dependent on the independent variables of the mass of the palm kernel shell and the process time as presented in Table 5. The fit summary test presented results of linear of < 0.0001for sequential p-value, quadratic level of < 0.0001 as suggested, and cubic value of 0.0033 which was aliased. The AVOVA for Quadratic model analysis presented results of Model F-value of 656.11 which means it is effective with a probability of 0.01% that the F-value is high enough for significance. [27] reported that when P-values are smaller than 0.05 reveal that the model terms are remarkable. Hence, when the obtained data are greater than (0.100), then it shows that the model terms are insignificant. An upward trend was observed in the volume bio oil values with additional mass of PKS and the process time which signified a proportional relation of both the response and independent variables. The result of the surface response for the volume of bio-oil for the optimization is presented in Figure 5.

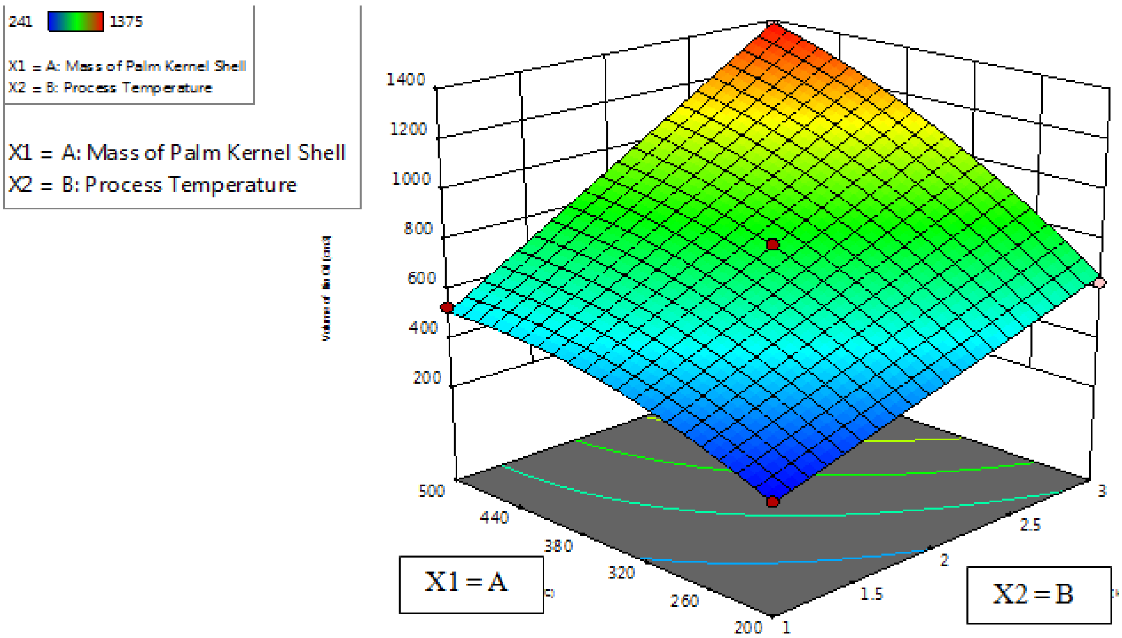


**Figure 5: Surface response of the volume of bio-oil from the independent variables**

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### 3.2.5 Process time

The processing time is a response that was generated from the duration of the pyrolysis operation. The Fit summary analysis indicates a sequential p-value of linear 0.0002, quadratic value of < 0.0001, and cubic value of 0.0407. The ANOVA for quadratic model analysis results showed that model F-value of 311.82 which means that the model is a determinant. However, there is a singular 0.01% probability that the model is certain and high enough to take place. The P-value < 0.05 displays that the terms of the model are significant. Hence, values > 0.1 imply that the terms of the model are insignificant. The Lack of Fit F-value of 10.43 shows it is significant and a probability of 2.32% value implies is large enough to occur. The process time response is dependent on the independent variables of the mass of the palm kernel shell and the process temperature. [24] reported that Figure 6 shows that the result of the surface response of the processing time is in linearity with the mass of the palm kernel shell and the process temperature.

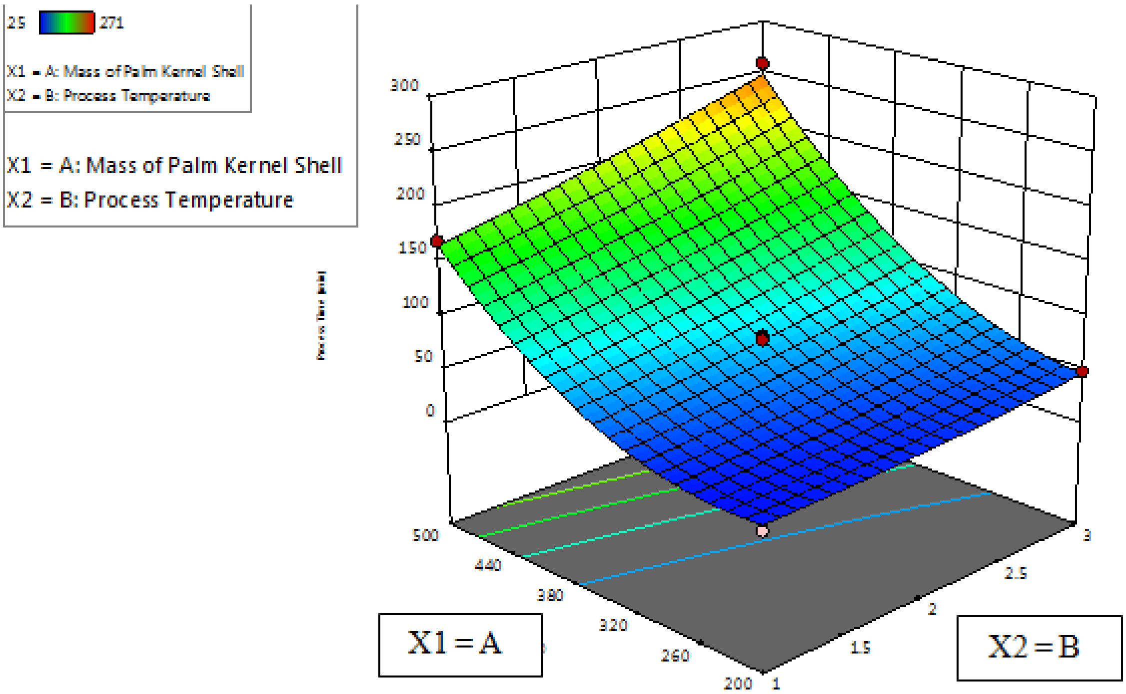


**Figure 6: Surface response of the processing time for the pyrolysis process of PKS**

### 3.3 Optimization of Independent Variables

Design Expert 12 software was utilized to illustrate the influence of the mass of the palm kernel shells and process temperature on the response variables through response surface graphs. Thereafter, mathematical optimization was done through the tool of desirability function to obtain the results shown in Tables 6 and 7. The criteria for the optimization of the product yields were identified and selected on the software interface. Various outcomes were obtained by varying the goals in the criteria option.

The results of the optimization contain different levels of independent variables concerning the goals [21]. The product yields were three namely: biochar, bio-oil, and biogas. The bio-oil is the most useful and most needed among the three products. Hence, maximum goals for the bio-oil were selected to ensure adequate secondary thermal cracking of the palm kernel shell which consequently left the biochar with no further bio-oil and insignificant chemical energy. The optimization of the product yields was in preference of the mass of the bio-oil and the volume of the bio-oil. The selected goals for the optimization of the bio-oil yield were at maximum level to give an enhanced and high production of bio-oil. The maximum level of process temperature and minimum level of biochar were utilized which allowed sufficient secondary thermal cracking of palm kernel for complete extraction of bio-oil. Also, the maximum level of the mass of bio-oil and minimum level of bio-gas was selected, as the gas was not significant in the study. The maximum level of the volume of bio-oil was utilized because it is the most significant product amongst the pyrolysis products. Ultimately, the maximum level of the processing time was utilized to allow adequate time required for the pyrolysis process. The results of the eight different optimization solutions that were found are shown in Table 5.

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**Table 5: Outcomes of the eight different optimization solutions of the product yields**

| S/N | Factors/Responses | Optimization Solutions | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| 1 | Mass of PKS | 2.44 | 1.03 | 1.59 | 1.63 | 1.84 | 2.54 | 2.66 | 2.54 |
| 2 | Process Temp. | 413.7 | 500.0 | 477.0 | 337.0 | 418.0 | 500.0 | 410.0 | 330.8 |
| 3 | Mass of Bio Char | 0.873 | 0.596 | 0.583 | 0.779 | 0.713 | 0.749 | 0.986 | 1.181 |
| 4 | Mass of Bio-Oil | 1.143 | 0.919 | 0.777 | 0.671 | 0.863 | 1.276 | 1.205 | 0.984 |
| 5 | Mass of Bio Gas | 0.430 | 0.315 | 0.246 | 0.213 | 0.282 | 0.499 | 0.481 | 0.400 |
| 6 | Volume of Bio-Oil | 1065.8 | 875.6 | 745.5 | 644.2 | 818.4 | 1186.6 | 1116.5 | 918.5 |
| 7 | Process Time | 151.5 | 197.3 | 136.4 | 62.8 | 119.9 | 226.7 | 140.6 | 81.0 |

The optimal product yield of mass of bio-oil and volume of bio-oil was obtained through the optimal desirability. Combined optimized factors and raw materials for the enhanced product yield were 2.66 kg of waste palm kernel shell and 410.0 $^{o}$C of the process temperature. The optimized outcome (predicted) values were 0.986 kg of biochar, 1.205 kg of bio-oil, 0.481 kg of biogas, 1116.5 cm$^3$ volume of bio-oil, and 140.6 minutes of process time. The results of the optimum conditions and the predicted values of the response at the optimized conditions are presented in Table 6.

**Table 6: Optimum conditions and predicted value of response at optimized conditions**

| Optimum Conditions | Coded Levels | Actual Levels |
|---|---|---|
| Mass of bio-oil (kg) | -0.08 | 2.66 |
| Process Temp. (oC) | -1.00 | 410.0 |
| Responses | Predicted Values | Experimental Values |
| Biochar (kg) | 0.986 | 0.894 |
| Bio-oil (kg) | 1.205 | 1.316 |
| Biogas (kg) | 0.481 | 0.498 |
| Bio-oil (cm$^3$) | 1116.5 | 1208.9 |
| Process Time (min) | 140.6 | 162.8 |

### 3.4 Verification of Response Surface Methodology

The desirability function was used to obtain the optimal values of the product yield. [28] reported that optimized product yields were utilized to control the suitability of the model for the predicted response values. Optimized product yields were Validation of the optimized product yields was carried out through experimentation using predicted values. The response values of the product yields at the optimized pyrolysis process were 0.986 kg of biochar, 1.205 kg of bio-oil, 0.481 kg of biogas, 1116.5 cm$^3$, and 140.6 minutes. The experimental values obtained were 0.894 kg of biochar, 1.316 kg of bio-oil, 0.498 kg of biogas, 1208.9 cm$^3$, and 162.8 minutes. The experimental response values certainly agreed with the predicted response values according to [28] as presented in Table 7.

## 4. CONCLUSIONS

The pyrolysis of palm kernel shells using a fixed-bed batch reactor to obtain an enhanced high-yield product has been evaluated. Palm kernel shells were successfully prepared and fed into the reactor. The research also revealed that RSM is a valuable means for the optimization of the pyrolysis process of palm kernel shells. RSM tool explored the connection between the dependent and independent variables of the pyrolysis process. The outcome of this study showed that the mass of the PKS and the process temperature have a substantial effect on the mass of bio-oil and volume of bio-oil. The optimized feedstock parameters were 2.66 kg of waste palm kernel shell and 410.0 $^{o}$C process temperature. Hence, the study revealed that the quadratic model was adequate to define and predict the responses of the mass of biochar ($Y_1$), the mass of bio-oil ($Y_2$), the mass of biogas ($Y_3$), the volume of bio-oil ($Y_4$), and process time ($Y_5$) when there are changes in the mass of PKS and process temperature.

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