Application of Plackett-Burman design on screening the factors affecting torrefaction of palm kernel shell

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Abstract. Plackett-Burman design (PBD) is used for screening important factors. In a PBD, the experiments are generated at various combinations of low and high values of the process variables and analyzed for their effect on the process. The purpose of this investigation was to study the statistical analysis for screening the factors affecting torrefaction of palm kernel shell (PKS). PBD was applied for screening effect of temperature, time, oxygen content, heating rate and biomass size to calorific value of torrefied PKS. The significant parameters were selected based on the analysis of variance (ANOVA) results. The results shown that the temperature and oxygen content were significant factors for the torrefaction of PKS which the respectability of received model was proved with high correlation coefficient. The result of 3D surface plots was shown that the temperature was the highest effect compared to other factors which calorific value were increased when the temperature increases. The diagnostic plots were examined for the model decency which the obtained linear model was quite good for prediction of calorific value of torrefied PKS. It was summarized that the predicted values by statistical model were close to experimental values.

1. Introduction
Torrefaction is a thermal pre-treatment process at temperature range of 200 °C - 320 °C in inert atmosphere which this process improves both physical and chemical properties of biomass. The obtained product from this process is called torrefied biomass which the advantages of torrefied biomass include lower moisture content, high calorific value, hydrophobicity, grindability and easy to be transported. Consequently, this process is appropriate for solid fuel production from biomass [1, 2].

Oil palm wastes can be generated from palm oil production process in oil palm mill and oil palm plantation. The PKS are produced from crushing process after removed palm oil process. A generated waste has approximately 0.04 tonnes of PKS per tonnes of fresh fruit palm [3]. Due to this fact, oil palm wastes seem to have great potentials to become an alternative energy source. The purpose of this investigation was to study the effect of temperature, time, oxygen content, heating rate and biomass size to the calorific value of torrefied product. The PBD was used to design the experiments, data processing and statistical analysis for screening the factors affecting calorific value of torrefied PKS.
2. Experimental

2.1 Torrefaction process
Torrefaction of PKS sample was carried out at an ambient pressure using a horizontal tubular reactor with 50 cm length and 5 cm internal diameter, made of stainless steel. The 80 g of PKS sample, which was crushed to size between 0.3 – 1.5 cm, was placed in the reactor. Throughout the experiment, the total flow rate of N\textsubscript{2} and O\textsubscript{2} mixture gas was set constant at 500 ml/min. The ratio of oxygen flow rate was varied in the range of 0 - 20%.

The variables for screening the factors affecting torrefaction were selected and given in table 1. After the torrefaction process was complete, the torrefied PKS was allowed to cool to room temperature before it was taken out from the reactor.

Table 1. Variables used in PBD

| Variable       | Unit | symbol | Low (-1) | High (+1) |
|----------------|------|--------|----------|-----------|
| Temperature    | °C   | A      | 200      | 320       |
| Time           | min  | B      | 5        | 50        |
| Oxygen content | %vol.| C      | 0        | 20        |
| heating rate   | °C/min| D     | 5        | 30        |
| biomass size   | cm   | E      | 0.3      | 1.5       |

2.2 Plackett-burman design
PBD is used for screening important factors. It can significantly reduce the number of repetitive experiments to be conducted in a further optimization study, using a general factorial, D-optimal, Taguchi OA or response surface methodology. The directly using these techniques with multiple factors are not appropriate because it increases the study cost. Therefore, a simplified design strategy is required that can be used to overcome these problems. The PBD experiments are generated at two level as low and high values of the variables and analyzed for their effect on the process. The Design - Expert 7.0.0 software (Trial version) was used to design of experiments for statistical analysis on screening the factors affecting torrefaction.

2.3 Calorific value analysis
Calorific value of PKS and torrefied PKS were measured by using automatic calorimeter model Leo AC-500.

3. Results and discussion

3.1 Characterization of raw PKS
The raw PKS was characterized its properties and was summarized in table 2. The proximate analysis informed that PKS consisted of 9.89% of moisture content, 80.90% of volatile organic, 1.06% of ash content and 8.15% of fixed carbon content. Besides, the ultimate analysis of PKS demonstrated that the percent composition of carbon, hydrogen, oxygen, nitrogen and sulfur were 46.06, 5.98, 47.68, 0.28 and <0.01, respectively. Also, the calorific value of PKS was 4,791 cal/g. All results shown fairly close values to research reported by Onochie et. al [4].

3.2 Statistical analysis
In this study, PBD was used for the screening of significant parameters for the response of calorific value. The experimental designs and results from actual values and predict values on torrefaction of PKS were presented in table 3. The regression model was used to fit the data for creating equation and it was assessed the statistical importance of the parameters by ANOVA. The linear model of calorific
value obtained from PBD regression analysis was described in terms of the actual values by equation (1).

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\text{Calorific value} = 737.899212.0730 + A - 18.5127C
\]  

(1)

### Table 2. Proximate and ultimate analysis of raw PKS

| Properties                  | This study   | Onochie et. al. [4] |
|-----------------------------|--------------|---------------------|
| %moisture content           | 9.89±0.113   | 10.23               |
| %volatile organic content   | 80.90±0.074  | 85.11               |
| %ash content                | 1.06±0.075   | 3.24                |
| %fixed carbon content       | 8.15±0.102   | 1.42                |
| Heating value (cal/g)       | 4.791        | Not report          |
| C content                   | 46.060±0.119 | 47.88               |
| H content                   | 5.979±0.133  | 5.15                |
| O content                   | 47.68±0.249  | 42.69               |
| N content                   | 0.275±0.003  | 0.94                |
| S content                   | <0.01        | 0.10                |

### Table 3. Experimental design and results on torrefaction of PKS

| Run | A  | B  | C  | D  | E  | actual | predict |
|-----|----|----|----|----|----|--------|---------|
| 1   | 320| 50 | 0  | 5  | 0.3| 6,815  | 6,601   |
| 2   | 320| 50 | 0  | 30 | 1.5| 6,915  | 6,601   |
| 3   | 200| 50 | 20 | 5  | 1.5| 5,068  | 4,782   |
| 4   | 200| 50 | 20 | 30 | 0.3| 5,102  | 4,782   |
| 5   | 320| 5  | 20 | 30 | 0.3| 6,117  | 6,231   |
| 6   | 200| 5  | 0  | 5  | 0.3| 4,979  | 5,152   |
| 7   | 320| 5  | 20 | 30 | 1.5| 5,828  | 6,231   |
| 8   | 320| 5  | 20 | 5  | 0.3| 6,120  | 6,231   |
| 9   | 320| 5  | 0  | 5  | 1.5| 6,698  | 6,601   |
| 10  | 200| 50 | 0  | 30 | 1.5| 4,941  | 5,152   |
| 11  | 200| 5  | 0  | 30 | 0.3| 4,910  | 5,152   |
| 12  | 200| 5  | 20 | 5  | 1.5| 4,801  | 4,782   |

### 3.2.1. Analysis of variance

According to the statistical analysis, the results were showed in table 4. The significant of variable was assessed at 95% confidence level. The variables were analyzed to be significant when \( p \)-value less than 0.0500 [5]. The results showed that the F-value of model was 21.08 which implies the model is significant. There is only a 0.10% chance that a model F-value this large could occur due to noise. The determination coefficient (\( R^2 \)) of the model was 0.9461. This means that 94.61% of the calorific value can be explained by the mathematical model of Eq. (1). According to the \( p \)-value of five factors, it is clear that the linear coefficient of A and C were significant (\( p<0.05 \)). In comparison, the factor B, D and E were not significant (\( p>0.05 \)), indicating that these factors had no significant effect on the calorific value of torrefied PKS. In addition, adequate precision value which indicates the ratio of signal to noise was 11.305. This value represents the adequate of model due to higher than 4 [6]. Therefore, it can be used to navigate the design space.
Table 4. Analysis of variance of the regression model for calorific value

| source  | Sum of squares | df | Mean square | F value | p-value |
|---------|----------------|----|-------------|---------|---------|
| Model   | 6.96×10^6      | 5  | 1.39×10^6   | 21.08   | 0.0010  |
| A       | 6.29×10^6      | 1  | 6.29×10^6   | 95.21   | < 0.0001|
| B       | 2.20×10^5      | 1  | 2.20×10^5   | 3.34    | 0.1175  |
| C       | 4.11×10^5      | 1  | 4.11×10^5   | 6.22    | 0.0469  |
| D       | 3.71×10^4      | 1  | 3.71×10^4   | 0.56    | 0.4818  |
| E       | 3.57×10^3      | 1  | 3.57×10^3   | 0.054   | 0.8239  |

R² = 0.9461, Adeq Precision = 11.305

3.2.2. Interactions between the factors to calorific values
The 3D surface plots were showed in figure 1(a) – 1(j). These plots presented the interaction of factors on calorific value of torrefied PKS. The calorific value of the torrefied PKS increased with an increase in the temperature. The effect of temperature was the highest when compared to other factors. While the %oxygen was rather few effects because the p-value had value almost higher than 0.05. The others factor e.g. time, heating rate and biomass size had no effect on the calorific value of torrefied PKS.
Figure 1. 3D surface plots showing the effect of two variables on calorific value while fixing the other three variables (a) temperature and time (b) temperature and oxygen content (c) temperature and heating rate (d) temperature and biomass size (e) time and oxygen content (f) time and heating rate (g) time and biomass size (h) oxygen content and heating rate (i) oxygen content and biomass size (j) heating rate and biomass size.

3.2.3. Diagnostics analysis
The diagnostic plots were examined for the model decency checking in figure 2(a) - 2(d). The relationship between normal % probability and studentized residuals was used to show the distribution pattern of the data was showed in figure 2(a). This plot showed that the obtained data was approximately straight line rather fit. The studentized residuals and predicted values plot (figure 2(b)) and the studentized residuals and run numbers plot (figure 2(c)) showed a random distribution of data points around the central line. The actual values and predicted values plot were indicated in figure 2(d). The plots of actual and predicted had linear behavior and spread close to diagonal line. Consequently, the obtained linear model was quite good for prediction of calorific value of torrefied PKS.

4. Conclusion
The statistical analysis for screening the factors affecting torrefaction of PKS using PBD. The purpose of this investigation was to study the effect of temperature, time, oxygen content, heating rate and biomass size to calorific value. The results showed that the temperature and oxygen content were significant factors for the torrefaction of PKS which the respectability of received model was proved with high correlation coefficient. While, the result of interactions between the factors to calorific values on 3D surface plots were show that the temperature was the highest effect compared to other factors which calorific value were increased when the temperature increases. The diagnostic plots were examined for the model decency which the obtained linear model was quite good for prediction of calorific value of torrefied PKS. It was summarized that the predicted values by statistical model were close to experimental values.
Figure 2. Diagnostic plots of calorific value (a) normal % probability and studentized residuals plot (b) studentized residuals and predicted values plot (c) studentized residuals and run numbers plot (d) actual values and predicted values plot

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