Screening the factors affecting torrefaction of palm kernel shell by using Plackett-Burman Design

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Abstract. Torrefaction is a thermochemical treatment for the production of solid fuel from biomass which promotes the increasing of fuel quality. This process involves the heating of lignocellulose at temperature range 200°C – 320°C in the absence of oxygen. The decomposition of cellulose, hemicellulose and lignin depends on several parameters. This research aims to investigate the screening of factors affecting torrefaction of palm kernel shell (PKS) which is a waste from oil palm tree. A Plackett–Burman experimental design is applied for screening the influence of operation parameters such as temperature, time, oxygen feed ratio, heating rate and biomass size. Mass yield (MY), moisture content (MC), volatile matter (VM), fixed carbon content (FC), ash content (AC) and calorific value (CV) are analyzed as the response to evaluate the performance of torrefaction conditions. The significant parameters are selected based on the analysis of variance (ANOVA) results. The results show that the temperature, time, oxygen feed ratio and biomass size are significant parameters. The fuel properties of torrefied product were compared with the standard for solid fuel. These results show that the quality of torrefied PKS satisfies the standards. Therefore, the torrefaction process can be used as the potential technique for improve the quality of PKS fuel properties.

1. Introduction
Energy consumption of Thailand has severely increased in the last decade. Its growth trend is expected to continue in the future. Although the energy requirement has still continuous increased, the providing of domestic source are limited. Fuels imports are the way of overcome this issue. Biomass is interesting to use as the alternative source of energy because can regrow in a relatively short period of time. Moreover, it can be converted to be fuel into all form: solid, liquid and gas. An oil palm waste is considered to be one type of biomass which is the potential energy resource.

The palm oil industry in Thailand generates large quantity of biomass wastes whose disposal is a challenging task. PKS are the shell fractions left after the nut has been removed after crushing in the palm oil mill. For the past many years, the PKS was not only sold in bulk, but also used as alternative fuel around the world. However, it is hygroscopic substance and has high moisture content which effect to the heating value. The higher the moisture content, the lower the heating value. In order to overcome these problems, the pre-treatment process is suggested to improve the quality of biomass.

Torrefaction is thermochemical process to improve the quality of fuel. In the process, biomass is heated in the range of 200 °C – 320 °C under atmospheric and absence of oxygen with low residence time. This is the efficient process to enhance fuel properties by eliminating the moisture, reducing the hygroscopic character and improving grindability. The research and development trend of many studies were conducted on the fuel property improvement of waste biomass via torrefaction and had reported
the process efficiency. Gan et al. [1] examined the torrefaction of de-oiled Jatropha seed kernel. The results showed that temperature was highly affected more than holding time for torrefaction performance. Cardona et al. [2] performed the torrefaction of eucalyptus-tree residues to evaluate the significant of temperature and time on process. The energy densification of produced solid fuel increased 29% compared to the raw material. Wang et al. [3] also reported the effect of torrefaction on physicochemical of Norway spruce stem wood, stump and bark. The fuel properties of torrefied product significantly improve compared to untreated biomass. Li et al. [4] studies the torrefaction of corncob under nitrogen and carbon dioxide atmospheres to change the physicochemical properties of its torrefied. The obtaining solid product showed the fuel quality higher than raw corncob. The temperature effect showed more significance than atmosphere. The impact of various factors like temperature, time, oxygen feed ratio, heating rate and biomass size, it becomes difficult to ascertain factors affecting the torrefaction process. Screening designs are commonly used to find the significant factors from a large number of two – level factors. Typically, it used in the initial stages of experimentation because it requires fewer experimental numbers than other designs. It is relatively inexpensive and efficient way to begin improving a process. A Plackett – Burman designs (PBD) are used for screening unimportant factors. In the PBD, experiments are generated at various combinations of low and high values of the process variables and analyzed for their effect on the process.

This research aims to screening the significance factors and improving the fuel properties by torrefaction process of PKS. A PBD is applied for screening the influence of operation parameters such as temperature, time, oxygen feed ratio, heating rate and biomass size. MY, MC, VM, FC, AC and CV are analyzed as the response to evaluate the performance of torrefaction conditions. The significant parameters are selected based on the ANOVA results.

2. Experimental

2.1. Materials
The palm kernel shell (PKS) was used as raw material and was collected from palmdeesrinakorn Co., Ltd. The PKS was grinded and sieved to size of 0.3 - 1.5 cm. Then, the obtained materials were kept in air-tight container to prevent the adsorption of moisture from the air.

![Figure 1. Schematic diagram for torrefaction process](image)

2.2. Torrefaction process
Figure 1 illustrated the schematic diagram for torrefaction process. Torrefaction of the PKS sample was carried out at an atmospheric pressure. The torrefaction reactor is a stainless steel horizontal tubular with an internal diameter of 5 cm and length 50 cm. Approximately 80 g of PKS was placed in the reactor. Throughout the experiment, the total flow rate of N\textsubscript{2} and O\textsubscript{2} gas was set constant a 500 ml/min to provide the oxygen flow rate of 0 - 20%. The operating conditions for the torrefaction temperature were set as 200 °C - 320°C. The residence times were 5 – 50 min. Also, the heating rates were 5 – 30 °C/min. The mass yield of the torrefied PKS can be determined by equation (1) as follows,
Mass yield (%) = \frac{\text{mass of PKS after torrefaction (g)}}{\text{mass of PKS before torrefaction (g)}} \times 100 \quad (1)

2.3. Plackett–Burman design
The PBD is very frequently used to screen the influence of operation parameters. Each variable was tested at a low level (-1) and a high level (+1) as depicted in Table 1. The Design – Expert software trial version 7.0.0 was used for experimental design, data processing and statistical analysis.

| Factor                  | Levels       |
|-------------------------|--------------|
|                         | Low (-1)     | High (+1)   |
| Temperature : A         | 200          | 320         |
| Time : B                | 5            | 50          |
| oxygen feed ratio : C   | 0            | 20          |
| heating rate : D        | 5            | 30          |
| biomass size : E        | 0.3          | 1.5         |

2.4. Proximate analysis
Proximate analysis was performed to examine the quantity of MC, VM and AC based on the standard number D3173, D3175 and D3174, respectively, of American Society for Testing Materials (ASTM). The high temperature furnace model ELF11/6B+301 Controller was used as the heating source.

Approximately 2 g of PKS sample was placed in the furnace at 105°C for 24 h. After that, the remaining sample was reweighted and the measurement of MC in PKS was calculated based on the loss in the weight of moist after drying. Then, the dried sample was heated at 900°C for 7 min to explore %VM. After VM examination, the residual sample was heated at 750°C for 6 h to measure the %AC. Finally, FC was analyzed by subtracting the summation of %MC, %VM and %AC from total of 100%.

2.5. Calorific value analysis
Calorific value was measured with automatic calorimeter model Leo AC-500. In the experiment, about 0.2-0.4 g of sample was compressed to be tablet and carried on in the bomb calorimeter. The released heating from the burning of sample with pure oxygen was determined.

3. Results and discussion
3.1. Characterization of PKS and Torrefied PKS
The quantity of cellulose, hemicellulose and lignin of PKS raw material were 35.56%, 16.67% and 46.67%, respectively. Figure 2(a) and 2(b) illustrated the physical appearance of PKS and torrefied PKS sample. The PKS was a brown colored with high density of lignocellulosic biomass. The %MC, %FC and CV were 9.89%, 8.15% and 4,791.12 Cal/g, respectively. When it was torrefied, the physical color became deeper and finally turned to black.

3.2. Plackett–Burman design results
In present study, PBD was used for the screening of process parameters for the response of %MY, %MC, %VM, %FC, %AC and CV. The matrix of total 12 experimental responses were given in Table 2. %MY were decreased when the torrefaction temperature increased. The torrefaction results of PKS showed that the highest and lowest of %MY were 98.13% and 43.50%, respectively. Moisture content is a significant property of biomass fuels. High %MC decreases the value of CV because it affects the energy conversion. When it is torrefied, the %MC were 0.30% and 0.13% at low and high temperature. The volatile matter supports the burning performance of the combustion process because high %VM indicates that the biomass ignited easily. The range of %VM of torrefied PKS were 83.34% to 47.61%. Fixed carbon is the remaining combustible solid that is residue after a biomass particle is heated until
the moisture and the volatile matter is eliminated. %FC were increased when the torrefaction temperature increased. The %FC of torrefied PKS were in the range of 15.07% to 49.16%. Ash is the residue of metallic elements after combustion. High %AC in biomass is usually attributed to the high concentration of non-combustible contained in the biomass. Therefore, high %AC can also reduce CV. The torrefied PKS had the quantity of ash between 0.97% - 3.08%. CV is the most important property for biomass as a fuel. The CV of torrefied PKS increases in direct proportion to the applied temperature. The highest and lowest of CV for torrefied PKS was 6915.11 Cal/g and 4801.73 Cal/g which increased from the value of raw materials.

The decreasing of MC and the increasing of FC and CV were observed when the temperature and residence time of torrefaction increased. The comparison of obtained results with biomass fuel standard shows that the performance of the torrefied PKS are very competitive. Based on this study, torrefaction process was found significantly improves the suitability of PKS’s fuel quality.

Table 2. PBD of factors with a response of torrefied PKS

| Run | A | B | C | D | E | %MY | %MC | %VM | %FC | %AC | CV   |
|-----|---|---|---|---|---|-----|-----|-----|-----|-----|------|
| 1   | +1| +1| -1| -1| -1| 43.50| 0.15| 47.61| 49.16| 3.08| 6815.91|
| 2   | +1| +1| -1| +1| +1| 48.25| 0.13| 49.81| 47.73| 2.33| 6915.11|
| 3   | -1| +1| +1| -1| +1| 90.63| 0.17| 82.57| 16.13| 1.14| 5068.03|
| 4   | -1| +1| +1| -1| -1| 90.00| 0.16| 83.34| 15.07| 1.43| 5102.66|
| 5   | +1| -1| +1| +1| -1| 52.63| 0.13| 50.80| 46.78| 2.29| 6117.72|
| 6   | -1| -1| -1| -1| -1| 95.25| 0.30| 75.64| 22.81| 1.25| 4979.69|
| 7   | +1| -1| +1| +1| +1| 55.12| 0.13| 49.94| 48.49| 1.44| 5828.95|
| 8   | +1| +1| -1| -1| +1| 43.63| 0.15| 52.68| 44.70| 2.47| 6120.72|
| 9   | +1| -1| -1| -1| +1| 56.13| 0.15| 46.96| 50.95| 1.93| 6698.46|
| 10  | -1| +1| -1| +1| +1| 93.75| 0.17| 81.72| 16.53| 1.58| 4941.85|
| 11  | -1| -1| +1| -1| -1| 95.63| 0.29| 79.47| 18.94| 1.31| 4910.32|
| 12  | -1| +1| +1| -1| +1| 98.13| 0.17| 80.55| 18.30| 0.97| 4801.73|

Biomass fuel standard - <10.0 - - <6.0 > 4179.80

Table 3. ANOVA for the regression model

3.3. Analysis of variance (ANOVA)
The ANOVA applied to evaluate the significant of the parameter for the response of %MY, %MC, %VM, %FC, %AC and CV. The statistical results of ANOVA were showed in Table 3. The significant of all variable was evaluated at 95% confidence level. The parameters were considered to be significant when p-value < 0.0500. The results showed that the response of %MY and %AC were significantly influenced by temperature, time and biomass size. %MC was significantly influenced by temperature, time, oxygen feed ratio and biomass size. %VM and %FC were significantly influenced by temperature, time and oxygen feed ratio. CV was also influenced by temperature and oxygen feed ratio. Therefore, the results show that the temperature, time, oxygen feed ratio and biomass size were significant parameters for the torrefaction of PKS.
%MY = + 75.56 - 7.71 A - 1.51 B + 0.75 E \quad (2)
%MC = + 0.18 - 0.012 A - 0.0087 B - 0.0094 C - 0.0091 E \quad (3)
%VM = + 67.67 - 5.42 A + 0.50 B + 0.65 C \quad (4)
%FC = + 30.47 + 5.26 A - 0.59 B - 0.58 C \quad (5)
%AC = + 1.69 + 0.17 A + 0.099 B - 0.085 E \quad (6)
CV = + 5571.03 + 253.80 A - 77.84 C \quad (7)

### 3.4. Analysis of perturbation

The perturbation plot is used to describe the effects of all parameters at a specific range. The plot of response was constructed by varied only one parameter in the study range while the other variables were fixed constant. The sensitivity of each parameter was expressed by its steep slope. The greater the slope, the higher sensitivity of that parameter to the response. Moreover, a slope of perturbation graph also corresponds to the coefficient of main factor in the equation in terms of coded factor. The higher the coefficient of main affect, the greater the slope of perturbation graph.

Figure 3 showed the individual effect of all factors by perturbation plot. Figure 3(a) and 3(e) were revealed that temperature was more sensitive to %MY and %AC than time and biomass size. Figure 3(b) showed that temperature was the highest effect on %MC following by oxygen feed ratio, biomass size and time, respectively. Figure 3(c) was revealed that temperature was more sensitive to %VM than oxygen feed ratio and time. Temperature was the highest influence on %FC following by time and oxygen feed ratio, respectively which was shown in figure 3(d). Figure 3(f) showed that temperature was more sensitive to CV than oxygen feed ratio. Consequently, temperature, time, oxygen feed ratio and biomass size had sensitive effect on %MY, %MC, %VM, %FC, %AC and CV which temperature was the highest influence compared to other factors.

### 4. Conclusions

This research aims to investigate the screening of factors affecting torrefaction of PKS. A PBD was applied in order to assess the significance of process parameters against %MY, %MC, %VM, %FC, %AC and CV of torrefied biomass. The ANOVA was employed to test the significant process parameters. The results showed that the temperature, time, oxygen feed ratio and biomass size were significant parameters for the torrefied of PKS. The perturbation graphs clearly showed that the
temperature has a major role in the improvement process of fuel quality of PKS. The quality of torrefied produced was compared with standard fuel and the results showed that it was standard. From the results of this work, it can be concluded that the torrefied PKS can be used as a potential alternative to solid fuel and is an environment-friendly fuel.

![Diagram](image-url)

**Figure 3.** Perturbation graph of (a) %MY (b) %MC (c) %VM (d) %FC (e) %AC (f) CV

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