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Application of Taguchi Technique to Investigate the Effect of Process Parameters on Product Yield and Carbon Content in Empty Palm Oil Fruit Bunch Carbonization

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Abstract: Carbonization is an important process which is used in a number of industries where, raw material from coal or agricultural origin are treated to remove moisture, volatile matter or undesirable material to yield a charcoal like product which may be further processed to produce valuable products such as activated carbon. In the current study, selected carbonization parameters are studied to investigate their effect on product yield and carbon content. Empty Palm Fruit Bunch (EPFB) was selected as the process feedstock for its wide availability; being a byproduct from palm oil industry. The studied parameters are carbonization temperature, nitrogen flow rate and soaking time. Taguchi technique for design of experiments and offline process analysis/optimization was used to build the approach of experiments by utilizing a modified L9 orthogonal array. The significance of each parameter was calculated by analyzing the results statistically to obtain the signal to noise ratio for each run as well as each level of parameter. The results showed that temperature had the most significant effect over product yield followed by nitrogen flow rate. The soaking time effect was statistically lower than that of gas flow rate but quantitatively close in value. For the carbon content of the product, soaking time showed to have the most significant effect. Temperature effect was second while gas flow rate seems to have the least effect statistically and quantitatively. Proposed carbonization conditions based on the results obtained in the current study are temperature of 650°C, nitrogen flow rate of 0.6 L min^{-1} and 30 min soaking time which gave 60.4% carbon content and 26.7% product yield.

Key words: Empty palm oil fruit bunch, carbonization, carbon content, taguchi technique

INTRODUCTION

Carbonization involves thermal decomposition of the carbonaceous raw material, eliminating of non carbon species and producing a fixed carbon mass and rudimentary pore structure. The process is usually carried out in tubular furnaces or kilns of multiple furnaces at temperatures below 800°C under continuous stream of an inert gas to prevent feedstock oxidation or burning during the process. Some reported parameters that determine the quality and yield of carbonized product are (Mackay and Roberts, 1982a):

- The rate of heating, the final temperature
- The soaking time at the final temperature
- The nature and the physical state of the raw material

Lower heating rate is preferred during the process since, it results in lower volatilization and higher char yield since it increases dehydration and enhances stabilization of the larger molecular structure component. The heating rate however, does not affect the product porosity and is independent from the composition of raw material (Mackay and Roberts, 1982a).

The above mentioned parameters were found to influence post carbonization processes as well. In the case of activation treatment to produce activated carbon, these parameters have also a marked influence on the quality of the final products (Mackay and Roberts, 1982b). When the products were prepared at a carbonization temperature lower than the activation temperature they underwent further pyrolitic decomposition during activation, resulting in weight loss independent of the activating gas. Thus low-temperature chars gasify at a much faster rate in the initial stage of the activation process resulting in a weight loss above 20-30% during activation (Mackay and Roberts, 1982b).

In the current study, different parameters are studied for the carbonization of empty palm oil fruit bunch. The selected parameters include final temperature and total soaking time as they have been noted to have the most effect on the process. The rate of heating is held constant while that of inert gas flow, nitrogen, is varied to have a better understanding on its effect on product yield and carbon content.

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MATERIALS AND METHODS

Methodology

Feed stock preparation: Empty palm oil fruit bunch was obtained locally from a plantation in Perak, Malaysia. The material was initially washed with deionized water to remove mud and dirt followed by additional washing with n-hexane to ensure the feedstock is free from organic contaminants. The size of the bunch was then reduced using a grinder and made uniform to meet a maximum size of 2 mm. Then the samples were dried in a furnace at 110°C over night.

Carbonization procedure: A tubular furnace was used to conduct the carbonization experiments. A schematic diagram for the setup is shown in Fig. 1. A measured amount of prepared empty palm oil fruit bunch is weighted inside a crucible which is then placed inside the horizontal furnace. Nitrogen (>99%, Malaysian Oxygen MOX) flow rate is controlled using a digital gas controller. The furnace is flooded with the inert gas for 10 min prior running the experiments to remove air and any gases that might be present inside the furnace and prevent any side processes to occur during carbonization. The heating rate is fixed during all experiments at steady increments. Once the final temperature is reached, the heating is remained constant at the desired temperature and held up to the desired soaking period. Flue gases are cooled to reduce their temperature using water cooled condenser. Once the process is complete, heating is discontinued and the furnace as allowed to cool down. The final weight is measured.

Yield calculation and carbon content analysis: The product yield is calculated based on Eq. 1 from the initial weight of the fed raw material and final weight after carbonization as follows:

$$\text{Yield (\%wt)} = \frac{\text{Weight of product}}{\text{Weight of EPOFB}} \times 100 \quad (1)$$

The amount of carbon in the final product is measured using a CHNS analyzer (LOCO, USA). A fixed amount of the product, usually in the range of 1.5-2.0 mg, is placed in the sample holder of the analyzer that is initially calibrated with standard (suifamethazin, c%51.78) provided by the manufacturer. The test is then initiated where the samples are injected to CHNS furnace. The results are finally obtained from the printed report.

Design of experiments: The investigated parameters in the current study along with the levels for each are shown in Table 1.

The selection of the levels was based on the studies reported elsewhere (Mackay *et al*., 1982a, b; Ozcimen and Ersoy-Mericboyua, 2008; Mlaouhi *et al*., 1999; Byrne and Nagle, 1997; Rodrýguez-Reinoso *et al*., 2000), who recommended the temperature range below 800°C. Higher temperature however is further tested at 950°C to evaluate the yield and carbon content at this temperature. The time range selected for the soaking was based on previous studies as well. Nitrogen flow rate was tested in the given range within the constraints of the gas flow meter and literature (Ozcimen and Ersoy-Mericboyua, 2008).

Taguchi method for design of experiments was used to establish the test tables based on the methodology studied from the literature (Ross, 1995) which provided the base on selecting the appropriate orthogonal array and to perform the qualitative analysis on the results obtained. L9 orthogonal arrays was selected and modified based on the number of factors and levels studied and are shown in Appendix 1. Each run was repeated to provide

Table 1: Parameters involved in the study with their relevant levels

	Parameters		
	Temperature (°C)	N2 flow rate (L min^{-1})	Soaking time (min)
Level	A	B	C
1	350	0.2	30
2	650	0.4	90
3	950	0.6	150

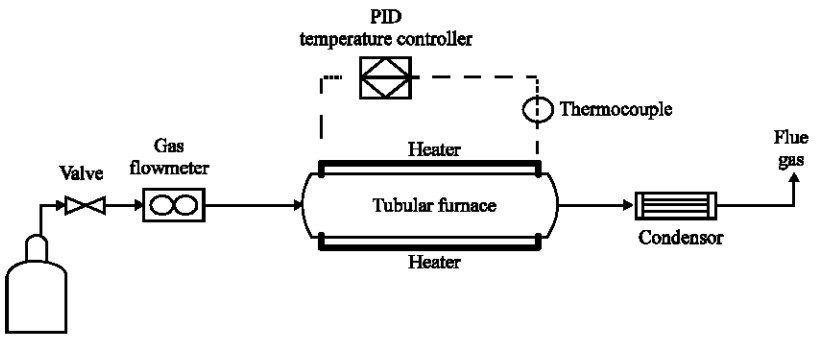

Fig. 1: Schematic diagram of carbonization setup

as much reliable data s possible and the statistical deviation between the repetitions was calculated.

Analysis of results to find the effect of parameters (not accurate): The effect of each parameter on product yield and carbon content in carbonizations is studied from the results obtained. Signal to noise ratios were used to obtain the effect of each parameter level on a given test result by isolating the effect of other factors with relation to the performance characteristic, yield and carbon content in the current study. Signal to noise ratios were calculated as shown in Eq. 2.

$$SNi = 10\log\frac{\bar{y}_i^2}{s_i^2} \quad (2)$$

Where:

$$\bar{y}_i = \frac{1}{N_i}\sum_{u=1}^{N_i} y_{i,u} \quad (3)$$

$$s_i^2 = \frac{1}{N_i - 1}\sum_{u=1}^{Ni}(y_{i,u} - \bar{y}_i) \quad (4)$$

When, the objective is to maximize the targeted performance characteristic, which is the goal in current study for both yields and carbon content, the signal to noise ratio is given by Eq. 5:

$$SN_i = -10\log\left(\frac{1}{N_i}\sum_{u=1}^{N_i}\frac{1}{y_u^2}\right) \quad (5)$$

The mean square value, sum of square, standard error and variance are calculated, as shown in Eq. 6-9, for each experiment to find the signal to noise ratio:

$$S_{mi} = \frac{\sum y_i^2}{n} \quad (6)$$

$$S_{Ti} = \sum y_i^2 \quad (7)$$

$$S_{ei} = S_{Ti} - S_{mi} \quad (8)$$

$$V_{ei} = \frac{S_{ei}}{N - 1} \quad (9)$$

The average signal to noise ratio is then calculated for each parameter as shown in Eq. 10:

$$SN_{xj} = \frac{\sum SN_{xj,i}}{n} \quad (10)$$

Calculated values are then tabulated and the effect of each parameter is studied from the difference between the highest and lowest average signal to noise ratio for each parameter. The effect of a given parameter on the performance characteristic depends on the difference magnitude.

RESULTS AND DISCUSSION

Effect of process parameters on product yield: The calculated product yield from the performed experiments is shown in the Appendix 2. Calculated standard deviation showed that the results are acceptable within the confined error. The highest yield could be achieved in Trial number 3 while the lowest was observed in number 7.

General observation of the results shows that product yield increases with the temperature. As it had been discussed previously, increasing the temperature results in increased pyrolisis of the feedstock releasing certain fractions of material at different temperatures. At 950°C however it was reported that the product was totally composing of ash even at shorter soaking time. This agrees well with the statement given by prior studies (Mackay and Roberts, 1982a, b) where carbonization temperature was recommended to be set below 800°C.

Nitrogen flow rate is shown to have the opposite effect over the yield when compared with temperature. Although, the noted change was not significant, increasing the inert gas flow rate increased the yield. Increasing soaking time on the other hand did not result in noticeable change even when, it was extended from 30 to 150 min. The reduction in raw material constituents seems to reach equilibrium within the initial 30 min, resulting in the close yield results at different soaking time for a given temperature. The deviation from such pyrolisis behavior was noted however at 950°C where, increased soaking time lowered the yield significantly as a result of empty palm oil fruit bunch ashing at the given temperature.

The results of signal to noise ratio calculations are shown in the Appendix 3. Table 2 shows the final signal to noise values for the parameters against each level. The significance of each parameter is found by comparing the maximum and minimum values for the same parameter. As

Table 2: Signal to Noise Ratio (SN) calculated for product yield and the ranking of significance of each factor on results

	Parameters		
Level	Temp (°C)	N_2 flow rate (L min^{-1})	Soaking time (min)
1	44.959	32.346	36.980
2	40.466	43.480	44.430
3	39.231	41.597	36.012
Δ	12.962	11.134	8.417
Rank	1.000	2.000	3.000

Table 3: Signal to Noise Ratio (SN) for each parameter and ranking of significance over product carbon content

	Parameters		
Level	Temp. (°C)	N_2 flow rate	Time
1	38.898	32.475	30.549
2	36.732	36.215	44.525
3	25.115	32.055	25.671
Δ	13.783	4.160	18.854
Rank	2.000	3.000	1.000

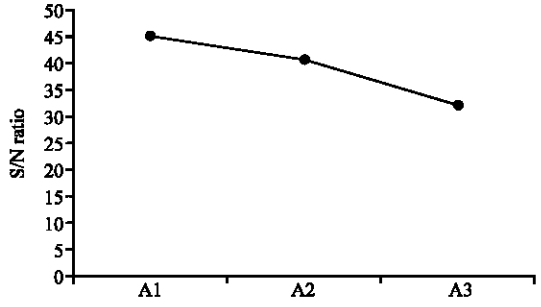

Fig. 2: Yield S/N ratio for temperature levels

it is shown in Table 3, the ranking of the parameter showed that temperature had the most effect over carbonization yield followed by nitrogen flow rate and finally, soaking time. This is comparable to the qualitative analysis of the yield results shown in Table B.

To further notice the effect of temperature, signal to noise ratio values were plotted against each level as shown in Fig. 2. It can be seen that the decreasing line is followed by temperature increase which supports the argument previously stated. Similarly, other studies performed on a number of material have shown the same effect of temperature over yield (Mackay and Roberts, 1982a, b; Rodrýguez-Reinoso *et al*., 2000). Although, 300°C resulted in the highest yield, the temperature might not be sufficient to remove some components which might not be desirable in the product; this however, depends on its final application. The highest temperature tested in the current study is not recommended since, it resulted in high yield losses resulting in total ashed product.

Nitrogen flow rate seems to reduce product losses when, it is increased from 0.2 to 0.4 L min^{-1} as it's shown in Fig. 3. Further increase did not give similar increase in the yield. This shows that the required inert condition to prevent side reaction during carbonization process does not need to exceed the mid level proposed in the current study. Other studies however have showed that the yield will be further enhanced with higher nitrogen flow rate (Ozcimen and Ersoy-Mericboyua, 2008).

The plot in Fig. 3 shows that the flow rate of the inert gas increases product yield at 0.4 L min^{-1} but did not

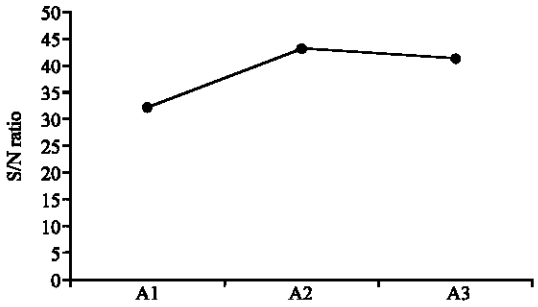

Fig. 3: Yield S/N ratio for N_2 flow rate levels

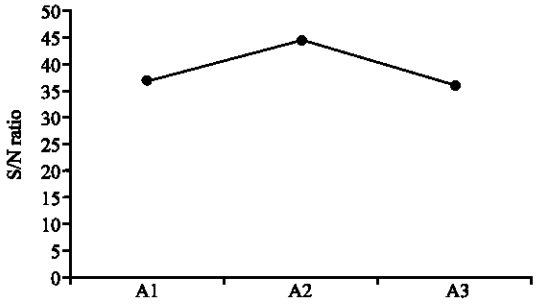

Fig. 4: Yield S/N ratio for soaking time levels

achieve higher result at 0.6 L min^{-1}. Figure 4 shows that soaking time had nominal effect to carbonization product yield. Shorter soaking time did not result in higher yield when compared to 90 min. More time might be required to achieve higher stability during pyrolisis or to reduce yield reduction that might result from furnace cooling during post carbonization. Longer soaking time however, gave lower yield as it is expected to result in further breaking down of fruit bunch constituents.

Effect of process parameters on carbon content of product: Carbon content for the products was measured experimentally and the results are shown in the Appendix 4. Results showed that the highest carbon content achieved was 62.4% at 950°C. However, at this temperature the product was ashed, thus, may not be utilized generally for further applications. Carbon content of 60.4% was achievable at 650°C without high losses in product yield. Carbonization was successful, in most experiments, in increasing the carbon content of the product from the initial value of 42.39% by removal of other matter present in the raw material.

The significance and effect of each parameter on product's carbon content was found based on signal to noise ratio calculation given in the Appendix 5. Table 3 shows a summary of the calculation along with the

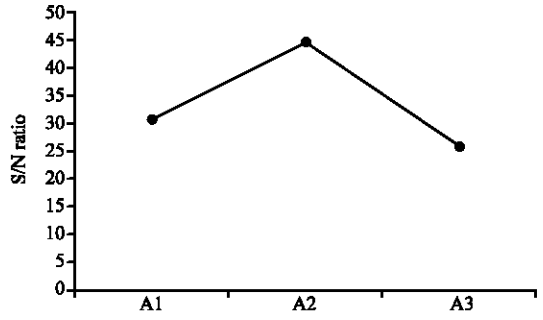

Fig. 5: Carbon content S/N ratio for soaking time levels

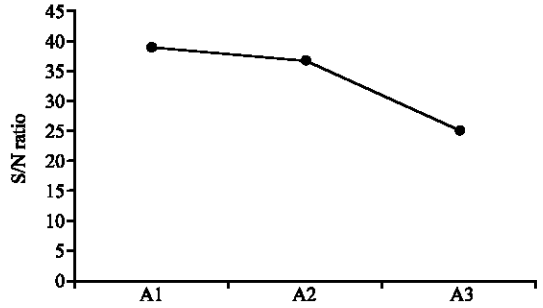

Fig. 6: Carbon content S/N ratio for temperature levels

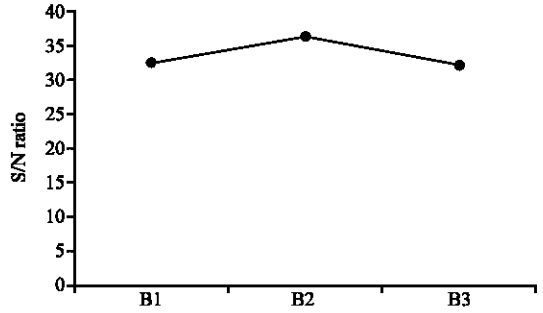

Fig. 7: Carbon content S/N ratio for N_2 flow rate levels

ranking of the parameters in terms of their effect on carbon content.

Soaking time had the highest effect among the other parameters considered in carbonization on carbon content of the result. As noted in Fig. 5, the magnitude between the highest and lowest value is the highest among the rest of the parameters which provides the perspective over its influence. Soaking time in mid range has shown to give better carbon content. A previous study (Mlaouhi *et al*., 1999) have shown carbon content in the range between 53-56% after 1 h of carbonization which is close to the values of 60.4% obtained in the current study at 1.5 h.

Temperature effect on carbon content was lower than that of soaking time but had considerable magnitude (Fig. 6). Higher temperatures were found to increase the carbon content but only when the soaking time was shorter. At higher temperature and longer soaking time, carbon content decreased noticeably. Similar behavior was reported elsewhere (Mlaouhi *et al*., 1999). Carbon content in the products following carbonization seems to be less and was almost independent from nitrogen flow rate variation throughout the experiments as shown by magnitude in Table 3 and Fig. 7.

CONCLUSION

Carbonization parameters were investigated based on the fractional factorial design of experiment utilized the Taguchi technique. The temperature, nitrogen flow rate and soaking time were studied to find their effect on carbonization product yield and carbon content. Temperature was found to have the most effect on yield while carbon content seems to have higher dependency on soaking time. Product yield was also affected by nitrogen flow rate which did not affect carbon content is significantly as the temperature. The highest temperature which exceeded 800°C was found to reduce the feedstock to ash. The recommended conditions observed to give a carbon content of 60.4 and 26.7% yield were in Trial number 6 at 600°C, 30 min soaking time and 0.6 L min^{-1} nitrogen flow rate.

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NOMENCLATURE

EPOFB = Empty palm oil fruit bunch
S/N = Signal to noise
i = Experiment number
N = Trial number
SNi = Signal to noise ratio for an experiment
y = Performance characteristic (i.e., yield or carbon content)
s = Variance
x = Carbonization parameter
j = Carbonization parameter level
n = No. of trials for each experiments; No. of levels of experiments for each level
Sm = Mean square

ST = Sum of square
Se = Standard error
Ve = Variance
SNxj = Average signal to noise ratio for a parameter
Δ = Difference between the maximum and minimum average signal to noise ratio for a parameter

APPENDIX

Appendix 1: Modified L9 orthogonal arrays

Trial No.	Temp. (°C)	N2 flow rate (L min^{-1})	Time (min)
1	350	0.2	30
2	350	0.4	90
3	350	0.6	150
4	650	0.2	90
5	650	0.4	150
6	650	0.6	30
7	950	0.2	150
8	950	0.4	30
9	950	0.6	90

Appendix 2: Carbonization experiments yield results

No.	Temp. (°C)	N_2 flow rate (L min^{-1})	Time (min)	Trial 1 pre-carbonization (g)	Trial 2 pre-carbonization (g)	Trial 1 post-carbonization (g)	Trial 2 post-carbonization (g)	Trial 1 yield (%wt.)	Trial 2 yield (%wt.)	Avg. yield (%wt.)	STD
1	350	0.2	30	0.5052	0.5065	0.2060	0.2108	40.776	41.619	41.197	0.596
2	350	0.4	90	0.5075	0.5081	0.2145	0.2138	42.266	42.078	42.172	0.133
3	350	0.6	150	0.5044	0.5070	0.2175	0.2174	43.121	42.880	43.000	0.170
4	650	0.2	90	0.5038	0.5030	0.1349	0.1333	26.776	26.501	26.639	0.195
5	650	0.4	150	0.5041	0.5054	0.1352	0.1370	26.820	27.107	26.964	0.203
6	650	0.6	30	0.5006	0.5060	0.1355	0.1340	27.068	26.482	26.775	0.414
7	950	0.2	150	0.5055	0.5052	0.0175	0.0211	3.462	4.176	3.819	0.357
8	950	0.4	30	0.5050	0.5048	0.0678	0.0690	13.426	13.669	13.547	0.172
9	950	0.6	90	0.5057	0.5063	0.0415	0.0410	8.206	8.098	8.152	0.077

Appendix 3: Yield S/N ratio calculation

No.	Temp. (°C)	N_2 flow rate (L min^{-1})	Time (min)	Trial 1 yield (%wt)	Trial 2 yield (%wt.)	Sm	St	Se	Ve	SN
1	350	0.2	30	41.619	40.776	3394.458	3394.814	0.355	0.355	36.790
2	350	0.4	90	42.266	42.078	3556.984	3557.002	0.018	0.018	50.042
3	350	0.6	150	43.121	42.880	3698.019	3698.048	0.029	0.029	48.045
4	650	0.2	90	26.501	26.776	1419.246	1419.284	0.038	0.038	42.718
5	650	0.4	150	26.820	27.107	1454.078	1454.119	0.041	0.041	42.463
6	650	0.6	30	27.068	26.482	1433.787	1433.958	0.171	0.171	36.217
7	950	0.2	150	3.462	4.177	29.173	29.429	0.255	0.255	17.530
8	950	0.4	30	13.426	13.669	367.057	367.086	0.030	0.030	37.934
9	950	0.6	90	8.206	8.098	132.917	132.923	0.006	0.006	40.529

Appendix 4: Carbonization experiments carbon content results

No.	Temperature (°C)	N_2 flow rate (L min^{-1})	Time (min)	Trial 1 C%	Trial 2 C%	AVG C%	STD
1	350	0.2	30	51.285	52.433	51.859	0.812
2	350	0.4	90	53.995	53.815	53.905	0.127
3	350	0.6	150	44.475	47.035	45.755	1.810
4	650	0.2	90	39.215	38.910	39.063	0.216
5	650	0.4	150	40.785	39.485	40.135	0.919
6	650	0.6	30	61.505	59.415	60.460	1.478
7	950	0.2	150	1.438	1.791	1.614	0.250
8	950	0.4	30	65.425	59.400	62.413	4.260
9	950	0.6	90	33.710	34.485	34.098	0.548

Appendix 5: Carbon content S/N ratio calculation

No.	Temperature (°C)	N_2 flow rate (L min^{-1})	Time (min)	Trial 1 C%	Trial 2 C%	Sm	ST	Se	Ve	SN
1	350	0.2	30	51.285	52.433	5378.746	5379.406	0.659	0.659	36.105
2	350	0.4	90	53.995	53.815	5811.498	5811.514	0.016	0.016	52.537
3	350	0.6	150	44.475	47.035	4187.040	4190.317	3.277	3.277	28.051
4	650	0.2	90	39.215	38.910	3051.758	3051.804	0.047	0.047	45.159
5	650	0.4	150	40.785	39.485	3221.636	3222.481	0.845	0.845	32.801
6	650	0.6	30	61.505	59.415	7310.823	7313.007	2.184	2.184	32.235
7	950	0.2	150	1.438	1.791	5.210	5.272	0.062	0.062	16.161
8	950	0.4	30	65.425	59.400	7790.640	7808.791	18.150	18.150	23.306
9	950	0.6	90	33.710	34.485	2325.279	2325.579	0.300	0.300	35.878

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