Study on adsorption of methylene blue on activated carbon from pinang frond using an experimental design to determine the optimum operating parameters

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Abstract. In textile industries, the dyes are commonly used and creating a waste of dye in drainage stream water. To overcome this matter, the removal of dyes are needed by using activated carbon. The precursors of activated carbon can be prepared by various organic materials with high level of carbon content. Pinang frond was being selected for precursor of activated carbon in this study, and named as PFAC. This PFAC was used to adsorb methylene blue (MB) as a media for removal of MB. The aim of this study is to determine the optimum operating parameters such as CO\textsubscript{2} flow rate (mL/min), activation time (hour) and activation temperature (°C) for the better removal of MB by using response surface methodology (RSM) as an experimental design (DoE). The 476 mL/min CO\textsubscript{2} flow rate, 6.0 hour activation time, and 867°C activation temperature were become the optimum operating parameters of PFAC in this study. The model developed by RSM shows that less than 2.5% error in comparison with the experimental data, hence it can be used for prediction of the fixed responses of operating parameters. The pseudo-first-order model was matched with the MB adsorption kinetic.

Keywords: Activated carbon; an experimental design; methylene blue

1. Introduction
Activated carbon is a material that can be functioned as an adsorbent. Some activated carbon is used in aqueous solution for elimination organic and inorganic pollutions [1], some used from a matter of gaseous condition [2]. With high surface area and high total pore volume, activated carbons become a comparative chosen for these purposes. The commercial activated carbons usually are expensive and limited to its application and usage that commonly made from bituminous coal, lignite, and petroleum coke [3]. Therefore, to overcome this situation, the other alternative precursors is need, which offers an abundantly available and inexpensive materials. This motivated to conduct the research in term of investigation of production of activated carbon. It was found that the capability of alternative precursors indeed had greater in comparison with commercial activated carbons [4]. These are some other alternative precursors that have been used as activated carbons, such as oil palm shell [5,6], coconut shells [7], kenaf core fibres [8], pinang frond [9], cocoa bean husk [10], tobacco waste [11], waste polystyrene foams [12], and palm kernel shell [13].
There are physical and chemical activation treatments applied in preparing activated carbon. The structure built with large active area and more pores is bigger chance in physical activation [7] and during the disposal stage, only less effect with respect to problem of the secondary pollution [14]. The most important parameters during preparation of activated carbon are activation temperature, activation time and CO2 flow rate that can create adsorption capacity or uptake [9, 15].

In the textile industry, annually can generate 10,000 different dyes in 0.7 million tons that have been wasted into the streaming water channel during the processes [16]. Reactive dyes, dispersed dyes, direct dyes, acid dyes, and basic dyes are such examples of dyes varieties. For these varieties of dyes, the basic dyes are widely used in textile industries due to their affinity to textile materials with net negative charge [17]. Instead of highly visible even in a very low concentration, basic dyes also have high intensity of colours. For example, methylene blue (MB) which categories as basic dye has been widely used for dying cotton, silk and manufacturing of paints and printing inks [18]. Nevertheless, MB stability to oxidizing agents, light, and heat is causing the difficulty to be removed or degraded from aqueous solution [19]. On inhalation, the difficulty in breathing in short periods also can be happened by the MB as well as profuse sweating, vomiting, mental confusion, and nausea. Even eyes of human and animal can be injured when in contact with MB. For these cases that caused as wastewaters, the removal of methylene blue (MB) become interesting to be explored. This study has similar work that studied on removal of Remazol Brilliant Blue R (RBBR) dyes instead of methylene blue (MB) [20].

The pinang frond (Areca catechu) as a waste material is usually being thrown away with no use of it [15]. In the previous literature, only a few study on preparation of activated carbon from pinang frond. Therefore, the aim of this study is used pinang frond as activated carbon that means for adsorbing MB dye for elimination in aqueous solution by determining the optimization of operating parameters such as CO2 flow rate, activation time, and activation temperature.

The experimental design (DoE) is a method that well-ordered and structured to describe the interactions among the typical variables, which can influence the result and also its process. The experimental design is the tactic of experimental data analysis for collecting empirical knowledge. DoE produces an experimental design set of all relevant variables by varying systematically and it assisted to find (i) optimal conditions and (ii) most influence factors affecting the results. Response surface methodology (RSM) is the most common method used in the experimental design. RSM is a technique by statistics and mathematical collection that intents for analysis problems and modelling. The RSM purpose is to treat numerous variables that can improve the response [21]. There are several design categorised in RSM such as face centered composite design (FCCD), hybrid design, 3-Level Factorial, and Box-Behnken design. Still, due to its flexibility and robustness, FCCD has been chosen among other RSM designs, since this method is most proper design in preparation of activated carbon.

FCCD permits curvature estimation by design using a central point and set of a star point or axial point of fractional factorial. Featuring for stabilizing the variance of the predicted response based on a measure of pure error that done at the center point by the duplication of it [22]. The estimation error can be more precise when there are more replicates. Normally, FCCD comprises of a star points (2n axial), center run (n0), and 2n points of factorial. Two parameters are required to be determine; center points (n0) from the design center and amount of the axial points distance (α0)[23]. Where n in the design based on the amount of factors and α0 is equal to one for face centered. In this study, n represents the variables number. Therefore, this work can predict the optimum parameters of activated carbon preparation for removal of methylene blue (MB) that useful for green industry of textile industries.

2. Materials and methods

2.1. Preparation of activated carbon from pinang frond (PFAC)

Pinang frond was collected from Sungai Petani (Malaysia). Pinang frond was washed and to eliminate the moisture content, it was dried for 24 hours at 110 °C. Afterward, a blanketed tube furnace was used that filling with stainless steel reactor in vertical tubular shape. The sample was crushed, then put in tubular reactor. The furnace was set to 20°C/min of heating rate up to 600-900°C of
activation temperature with streaming flow of a purified nitrogen (99.99%) at 150 ml/min. After the temperature of activation was achieved, 150-600 ml/min of CO₂ flow rates was introduced for 1-7 hours that performed as the activation agent. After finished, the reactor was cooled down using of flowing nitrogen until reach room temperature. The airtight container was used for keeping the samples that would be used for further adsorption of MB test in order to identify the operating parameters optimization. The PFAC characterization in detail can be found in Herawan et al. [9] and Ahmad et al. [15].

2.2. PFAC preparation experimental design
RSM (Design-Expert v. 6.0.6, STAT-EASE Inc.) was used for DoE optimization of PFAC preparation. Operating parameters of preparation of PFAC have been observed using RSM design, which a standard FCCD has been chosen for this purpose. The operating parameters are CO₂ flow rate ($x₀$, mL/min), activation time ($x₂$, hour), and activation temperature ($x₃$, °C) as an independent variables.

The independent variables coding using FCCD is shown in Table 1. While, Table 2 displayed an experimental design matrix based on the levels and the ranges given. By using FCCD, these three variables were implementing at the center points, which were six replicates with 6 axial points and 8 factorial points. This procedure can generate 20 experiments that obtained using Equation 1:

$$N = 2^n + 2n + n_e = 2^3 + 2 \times 3 + 6 = 20$$  \hspace{1cm} (1)

Where $N$ is the amount of experiments needed, while $n$ is a factor number.

| Table 1. Independent variables coding using FCCD |
|----------------------|----------------------|----------------------|
| Variables (factors)  | Coded variable levels |
| CO₂ flow rate (mL/min) | -1 150 375 600 |
| Activation time (hour) | 1.0 4.0 7.0 |
| Activation temperature (°C) | 600 750 900 |

Design matrix generates from the 6 replicate points (Run 15-20) as the center points is used to verify experimental error and data reproducibility. Low level is encoded as -1 and high level is +1 as a coding variable interval. MB removal ($Y$) was determined based on the response and studies of batch adsorption. As a test of this experiment, a200 mg/L initial concentration of MB solution in flask was used as a media for 0.2 g of PFAC and observed the decreasing MB concentration as representing adsorption process occurred into PFAC until equilibrium was achieved. This condition was conducted in water bath shaker with 120 rpm at 30°C. The MB concentrations before and after adsorption can be determined by using UV-Visible spectrometer with double-beam from Shimadzu (UV-1800). The MB removal in percentage was obtained from equation 2.

$$MB \text{ Removal in equilibrium (})\%\text{) } = \frac{(C_o - C_e)}{C_o} \times 100$$  \hspace{1cm} (2)

Where $C_o$ is the concentration of liquid-phase for adsorbate at before adsorption (mg/L) and $C_i$ is the saturation adsorption (mg/L).

3. Results and Discussion

3.1. Characterization of activated carbon from pinang frond
Activated carbons from pinang frond have 2.32 nm average pore diameter, 576.89 up to 958.23 m²/g of BET surface area, and total pore volume of 0.3449 up to 0.5469 mL/g [15]. Activated carbon from
pinang frond characteristics in detail including the analysis of SEM was presented in Ahmad et al. [15]. Ahmad et al. [15] found that total pore volume and BET surface area were increasing with increasing CO$_2$ flow rate, activation time, and activation temperature until reached certain values as an optimum condition of each parameter, the activated carbon performance started to decrease.

3.2. Experimental design

The experiment design of preparing PFAC in this study was using FCCD. Each MB removal response $Y$ (%), with variables of CO$_2$ flow rate ($x_1$), activation time ($x_2$) and activation temperature ($x_3$), were observed, as shown in Table 2. To analyse the experimental data, Software of Design Expert was chosen.

| Run | Variable of preparation of PFAC | Removal of MB, $Y$ (%) |
|-----|---------------------------------|------------------------|
| 1   | CO$_2$ flow rate, $x_1$ (mL/min) | 150 1.0 600 | 74.5 |
| 2   | 150 1.0 900 | 80.7 |
| 3   | 150 7.0 900 | 84.6 |
| 4   | 150 7.0 600 | 83.7 |
| 5   | 600 1.0 600 | 84.6 |
| 6   | 600 1.0 900 | 85.4 |
| 7   | 600 7.0 600 | 85.4 |
| 8   | 600 7.0 600 | 90.7 |
| 9   | 375 4.0 600 | 78.8 |
| 10  | 375 4.0 900 | 94.5 |
| 11  | 375 1.0 750 | 80.9 |
| 12  | 375 7.0 750 | 91.1 |
| 13  | 150 4.0 750 | 81.9 |
| 14  | 600 4.0 750 | 88.6 |
| 15  | 375 4.0 750 | 93.7 |
| 16  | 375 4.0 750 | 93.5 |
| 17  | 375 4.0 750 | 93.5 |
| 18  | 375 4.0 750 | 93.6 |
| 19  | 375 4.0 750 | 93.7 |
| 20  | 375 4.0 750 | 94.8 |

Since the additional terms have a significant effect, the model of highest order polynomials was being selected. The removal of MB ($Y$) response was obtained from quadratic model based on Table 2, which reveals the response based on interaction and the significance of variables, which created by FCCD consisting of coded factors. The one coefficient factor dedicates for only effect of certain factor, while the two coefficients factors dedicate to the two factors interaction. The second-order term coefficients correspond for effect of quadratic. Equation 3 shows the final response of MB removal generated from the model of coded factors using empirical strategy.

MB removal ($Y$):

$$Y = 91.97 + 4.19 x_1 + 2.62 x_2 + 3.25 x_3 - 2.58 x_1^2 - 3.23 x_2^2 - 3.98 x_3^2 + 1.5 x_1 x_2 - 1.72 x_1 x_3 - 0.05 x_2 x_3$$  (3)
In term of the significance and adequacy, ANOVA (analysis of variance) was used to verify the models. With this, the means squares was calculated by dividing the squares each variation source sum, while by the degree of freedom of relevant is dedicated for the error variance and the model. The model can be determined as having a significant effect on the response and also the result is not random if Prob> F value less than 0.05 [24]. Table 3 shows the PFAC model of removal of MB generated by the ANOVA. Since Prob> F is 0.0029 for removal of MB and the F-value is 6.87, then the model of removal of MB can be concluded has a significant effect. For this case, a significant model terms were CO$_2$ flow rate ($x_1$), activation time ($x_2$) and activation temperature ($x_3$).

The removal of MB of the three-dimensional response surface is illustrated in Figure 1with the range of CO$_2$ flow rate, activation time, and activation temperature at 300 to 550 mL/min, 3.0 to 6.0 h, and 800 to 870°C, respectively. By increasing these operating parameters, the removal of MB becomes higher due to the development of internal porous cavities that can cause the surface area increment. Apparently, this finding was also found by Bello et al. [25]. Nevertheless, by increasing temperature, the performance of activated carbon was significantly affected due to the devolatilization rate on the activated carbon pore structure [26]. Indeed, the development of pores was generated from removing the moisture volatile and components when increasing these parameters. Unfortunately, the performance of pinang frond activated carbon gradually decline in adsorbing MB for eliminating it in the solution. This condition happened, when CO$_2$ flow rate, activation time, and activation temperature exceeded the values of 50 mL/min, 6.0 h, and 870°C, respectively. These values indicate that these are an optimum condition for pinang frond activated carbon to be used as removal of MB adsorbent. Beyond these values, the surface area and percentage dyes removal would start decline due to the collapsing of some pores that affected the activated carbon performance. This condition was supported by Ahmad and Alrozi [27] that they found when activation time and activation temperature were exposed too long, the activated carbon surface area and adsorption capacity would decrease. They used mangosteen peel as a precursor of activated carbon.

Lua and Yang [28] have a similar finding that the increment of CO$_2$ flow rate, activation time, and activation temperature, could enhance the capacity of adsorption due to more pores to be created and enlarged. They also notice that when these parameters beyond the optimum value, the pore volume and surface area of activated carbon would decreasing that resulting undesirable characteristics.

3.3. Optimization of operating parameters

By using the experimental design, the optimum operating parameters can be determined on preparation of activated carbon with respects to high removal of MB. Removal of MB validation model for PFAC is shown in Table 4. The optimum parameters were selected based on the desirability values. Next, these optimum parameters would be tested by conducting the experiment to verify the result. By using Design Expert Software, optimum operating parameters can be determined by targeting the operating parameters value within the range of maximum value. The optimum operating parameters of PFAC for the removal of MB were found at CO$_2$ flow rate of 476 mL/min, activation time of 6.0 hour, and the activation temperature of 867 °C. As shown in Table 4, the experimental results were in good agreement with the predicted results by minimum error (less than 2%). Based on this finding, it can be concluded that the fixed responses of operating parameters can be predicted using this model.

3.4. Optimum PFAC pyrolysis condition and adsorption study

Based from optimization result obtain from FCCD, the optimum conditions for PFAC preparation was found at 867 °C of activation temperature, 6.0 hours of activation time, and 476 mL/min of CO$_2$ flow rate. Therefore, this sample was chosen for the further adsorption studies.
Table 3. Removal of MB by analysis of ANOVA for PFAC

| Source | Sum of Squares | Degree of Freedom | Mean Square | F-Value | Prob> F |
|--------|----------------|------------------|-------------|---------|---------|
| Model  | 744.46         | 9                | 82.72       | 6.87    | 0.0029  |
| x₁     | 175.56         | 1                | 175.56      | 14.58   | 0.0034  |
| x₂     | 68.64          | 1                | 68.64       | 5.70    | 0.0381  |
| x₃     | 105.52         | 1                | 105.62      | 8.77    | 0.0142  |
| x₁²    | 18.27          | 1                | 18.27       | 1.52    | 0.2463  |
| x₂²    | 28.64          | 1                | 28.64       | 2.38    | 0.1540  |
| x₃²    | 43.5           | 1                | 43.5        | 3.61    | 0.0865  |
| x₁x₂   | 18.0           | 1                | 18.0        | 1.49    | 0.2490  |
| x₁x₃   | 23.8           | 1                | 23.8        | 1.98    | 0.1900  |
| x₂x₃   | 0.02           | 1                | 0.02        | 1.6x10⁻³| 0.9680  |

Table 4. Removal of MB validation model for PFAC

| Dye | Model desirability | CO₂ flowrate | Activation time | Activation temperature | Removal of MB % | Error (%) |
|-----|--------------------|--------------|-----------------|------------------------|-----------------|-----------|
| MB  | 0.989              | 476 mL/min   | 6.0 h           | 867 °C                 | 94.80           | 2.10      |

3.4.1 MB Adsorption isotherm

Adsorption isotherm was important to describe how dyes adsorb onto PFAC. In this work, the Langmuir and Freundlich isotherms were employed to analyse the adsorption equilibrium data. The linear regression was selected to determine the best-fitting isotherm. The proximity of isotherm equations was achieved if R² values were closer to unity based on comparison and evaluating between the correlation coefficients that can fit better[29].

Table 5 shows the isotherm model parameters and correlation coefficients and R² values for MB adsorption on the PFAC. Langmuir isotherm model gave the higher R² value than Freundlich isotherms. This result shows that the MB adsorption was made up of homogeneous monolayer adsorption patches. Langmuir model also considers adsorption to be localized with all active sites on the surface be active sites on the surface that have the same energy. In addition, Langmuir model shows none interaction on the surface reaction as in the heterogeneous reaction, such as interaction between adsorbed molecules exists and the limiting reaction step [30].

There was also some other adsorbents that were fallen into the Langmuir isotherm models for MB adsorption, such as rattan sawdust [18] and jute fiber carbon [31]. The monolayer adsorption capacity for adsorption of MB onto PFAC was 384.62 mg/g. Whereas the value of n > 1 shows that the higher the n value, the stronger the adsorption intensity due to the favourably adsorbate that adsorbed on the adsorbent [32].

3.4.2 MB adsorption kinetics

By using pseudo-first-order and pseudo-second-order kinetic models, kinetics adsorption data of MB dye on PFAC was analyzed. Figure 2 shows linear plots of ln (qe-qt) against t with intercept of ln qe at gradient of k₁. Table 6 shows the values of k₁ and R² obtained from the plots. The value of R² obtained for the pseudo-first-order shows the highest value which indicates that this kinetic model has a good relation between the experimental and the calculated qe values. It also shows that pseudo-first-order model were close to unity, showing that the adsorption of MB on PFAC fitted well.
Figure 1. Plot of removal of MB on PFAC response surface; (a) Relationship of activation time and activation temperature with 476 mL/min of CO$_2$ flow rate; (b) Relationship of CO$_2$ flow rate and activation temperature with 6 h of activation time; (c) Relationship of CO$_2$ flow rate and activation time with 870 °C of activation temperature.

Table 5. Langmuir and Freundlich isotherm model parameters and correlation coefficients for adsorption of MB on PFAC

| Activated carbon | Langmuir | Freundlich |   |   |
|------------------|----------|------------|---|---|
|                  | $Q_o$   | $K_L$      | $R^2$ | $K_F$ | $n$ | $R^2$ |
| PFAC             | 384.62  | 0.09       | 0.991 | 6.006 | 2.61 | 0.974 |
Figure 2. Plots of pseudo-first-order for MB adsorption on PFAC

Figure 3 shows linear plots of $t/q_t$ versus $t$ with $1/k_2 q_e^2$ as the intercept and $1/q_e$ as the gradient. From Table 6, the $R^2$ value of pseudo-second-order model was shown to be chaotic without a particular trend. It also shows that the experimental $q_e$ values do not match with the calculated values obtained from the linear plots. Therefore, it can be said that the adsorption of MB on the PFAC does not follow pseudo-second-order model.

Table 6. Pseudo-first-order and pseudo-second-order kinetic model parameters for the adsorption of MB dye on PFAC

| $C_0$ (mg/l) | $q_{e,exp}$ (mg/g) | $q_{e,1}$ (mg/g) | $k_1$ (1/h) | $R^2$ | $q_{e,2}$ (mg/g) | $k_2$ (g/mg h) | $R^2$ |
|-------------|-------------------|-----------------|-------------|-------|----------------|----------------|-------|
| 50          | 49.45             | 45.993          | 0.376       | 0.990 | 48.78          | 0.014          | 0.971 |
| 100         | 95.8              | 92.916          | 0.279       | 0.995 | 91.74          | 0.0048         | 0.889 |
| 200         | 184.6             | 184.233         | 0.197       | 0.985 | 238.10         | 0.007          | 0.566 |
| 300         | 266.1             | 308.524         | 0.285       | 0.988 | 416.67         | 0.0003         | 0.382 |
| 400         | 320               | 318.684         | 0.181       | 0.997 | 357.14         | 0.001          | 0.762 |
| 500         | 363               | 359.783         | 0.154       | 0.996 | 312.50         | 0.001          | 0.770 |
4. Conclusions
The pinang frond can be used as an alternative precursor for preparing activated carbon and has a capability to adsorb MB as a function of removal of MB in textile industry. The 476 mL/min CO$_2$ flow rate, 6.0 hour activation time, and 867°C activation temperature were become the optimum operating parameters of PFAC in this study. The model developed by RSM shows that less than 2.5% error in comparison with the experimental data. Based on this finding, it can be concluded that the fixed responses of operating parameters can be predicted using this model. The pseudo-first-order model was matched with the MB adsorption kinetic.

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