Biomass-Based Activated Carbon from Areca Catechu for Dye Wastewater Treatment

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ABSTRACT: *The synthesis of activated carbon from Areca Catechu or Pinang frond (PFAC) through physicochemical activation has been successfully investigated. The physicochemical activation was carried out by using potassium hydroxide (KOH) impregnation and carbon dioxide (CO_2) gasification. The adsorption performance of PFAC was evaluated through the removal of methylene blue (MB) dye from synthetic wastewater. By using an experimental design, the results revealed that the activation temperature, activation time and KOH impregnation ratio (IR) were significant factors influencing the adsorption performance for all pollutants. The optimum preparation conditions for PFAC were; activation temperature 771 °C with activation time of 1.48 h and impregnation ratio of 1.94, respectively. The effects of adsorbate initial concentration (50-500 mg/L) and contact time (0-24 h) were evaluated through batch adsorption test. All dye adsorption uptakes increased with increasing initial concentration and contact time.*

KEYWORD: *activated carbon, Areca Catechu, physicochemical activation, methylene blue, adsorption*

1.0   Introduction

Textile wastewater that directly released into the environment can induce potential risk to water bodies as the effluent is toxic to the environment and living organisms (Ahmad and Hameed, 2009). Therefore, these wastes need to be treated before discharging it into the environment. In Malaysia, the wastewater containing dyes have been classified as schedule wastes under the Environmental Quality Regulation 1989 (Ahmad, 2010). In response to such event, stringent legislation on discharge quality as per requirement of Environmental Quality Act of Malaysia further constrains the industry to comply. Therefore, continuous study and research on the textile wastewater treatment is needed for sustainable the environment.

Dyes have been extensively used in textile industries. Dyes can be classified according to their type such as basic, acid, disperse, direct, reactive, solvent, sulfur and vat dyes. The most common types used in textile industries are basic, reactive and acid dyes. Basic dye, such as methylene blue (MB), has chromophore with positive ion and amino groups with high color intensity (Rafatullah *et al*., 2010). The MB is usually used for cotton and silk coloring. Unfortunately, the MB is difficult to be degraded or removed from aqueous solution due to its high stability to light, heat, and reducing agents (Robinson *et al*., 2001). The MB can be lethal to human and animal eye and vision. It can also cause breathing difficulty when accidentally inhaled. In addition, ingestion in mouth can cause, vomiting, nausea, mental confusion and profuse sweating (Tan *et al*., 2008a).

Inappropriate treatment of spent dyes may bring hazards to the aquatic environment since they can cause undesirable color effect to the water, reduce light penetration and prevent photosynthesis (Ioannidou *et al*., 2007). The discharge of untreated textile effluent can cause natural waters to be unfit as potable water source. It is common that 70-80 % of all illness affecting developing country is caused by water contamination (Pang and Abdullah, 2013a).

Therefore, the removal of dyes from industrial effluent is important before discharging it into receiving water bodies. Various methods of dyes removal exist, but adsorption on activated carbons derived from agricultural wastes appears to be efficient and relatively easier to be procured to purify polluted water (Mahmoodi *et al*., 2011; Rafatullah *et al*., 2010). Synthesis of AC from biomass have attracted growing interests such as coffee husk (Ahmad and Rahman, 2011), cocoa shell (Ahmad *et al.*, 2011), palm shell (Arami *et al.*, 2012), spent tea (Auta and Hameed, 2011), banana stalk (Bello *et al.*, 2011a), cocoa pod husk (Bello *et al.*, 2011b), mangosteen peel (Ahmad and Alrozi, 2010) and rambutan peel (Ahmad and Alrozi,

2011). The utilization of easily obtain and renewable biomass which is well available such as residues betel nut (Areca catechu) was applied.

Areca catechu is commonly known as areca palm or areca nut palm (betel palm), found in parts of east Africa, Asia, and much of tropical Pacific. The palm is originated in Malaysia. The word *Areca* is come from Malabar Coast of India and *catechu* is a Malay name for this palm, *caccu*. It is grown commercially for its seed crop which is the areca nut. Nowadays, *Areca catechu* can be found in India, Bangladesh, Taiwan and other Asian countries for their seeds. In fact, the areca palm is also utilized for landscaping. In this research, the betel nut fronds were utilized as precursors for activated carbons production for dyes removal from aqueous solution.

1.1   Research Objectives

i) To optimize preparation conditions by using response surface methodology (RSM) for development of PFAC.
ii) To evaluate the effects of adsorbate initial concentration and contact time through batch adsorption test for MB onto optimized PFAC.

2.0   Literature Review

Textile industry consumes water intensively in every stage of wet processing from sizing, de-sizing, fibers scouring and bleaching to the dye coloring, finishing and fabric printing. The water consumed are discharged equally as effluent wastewater. Commonly, the textile industry prefers vegetable-based fibers such as cotton, animal-based fibers such as wool and silk, and synthetic materials such as polyester, nylon, and acrylics (Arumai Dhas, 2008). Currently, the industry covers polymerization and man-made fiber production, spinning, texturizing, weaving, knitting, dyeing, printing and finishing of yarn and fabrics, manufacture of made-up garments and other made-up textile goods such as carpets, bed and table linen and ropes (Alrozi, 2010).

Various physicochemical and biological methods that have been discussed in previous sections offer wide range of pollutants removal with respect to their treatment mechanisms. Moreover, different treatment techniques for wastewater laden with organic pollutants and heavy metals have been developed in recent years are aimed to reduce excessive concentration of various pollutants as well as to improve the quality of treated effluent as manifested from various literature. In addition to that, the discussed treatment strategies have their inherent advantages and drawbacks in applications, accordingly. Major physicochemical methods including chemical precipitation, advanced oxidation processes (AOPs), ion exchange, membrane filtration and adsorption are the most employed methods in textile wastewater treatment. In terms of biological methods, aerobic and anaerobic processes are mostly employed in textile wastewater treatment as a pretreatment step.

Activated carbon (AC) is a microporous materials used as commercial adsorbents to mainly remove liquid and gaseous pollutants. It is a carbonaceous adsorbent with amorphous structure. Its internal pore structure is extensively well-developed for adsorption. Activated carbon is effective in the removal of organic compounds such as volatile organic compounds, benzene, pesticides, some metals, chlorine and radon, as well as drinking water contaminants when combined with other types of treatment systems (Rivas *et al.,* 2003). The AC adsorption capacity is closely related to its surface area, pore volume, and pore size distribution (Demirbas *et al*., 2009). The structure of micropores and mesopores allow the AC to adsorb large amounts of various adsorbates (Mui *et al.,* 2009). Besides, textural properties of activated carbons and their functional groups are two of the principal characteristics which should be enhanced by certain modification processes in order to make them exhibit high pollutant removal efficiencies

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Philipines or Malaysia. The word *Areca* is come from Malabar Coast of India and *catechu* is a Malay name for this palm, *caccu*. It is grown commercially for its seed crop which is the areca nut. Nowadays, *Areca catechu* or *pinang* can be found in India, Bangladesh, Taiwan, Malaysia and other Asian countries for their seeds. In fact, the areca palm is also utilized for landscaping. Areca catechu has a long history as a medicinal plant in China based on its wide spectrum of biological to kill parasites and promote digestion (Peng *et al.,* 2015).

In terms of its application in, areca catechu has widely employed in various industrial sectors. For example, biosorption for metal ions (Chakravarty *et al.,* 2010), anti-aging extract (Lee *et al.,* 1999), polyester composite (Lai and Mariatti, 2008), polypropolene composite (Hassan *et al.,* 2010) and anti-oxidant (Bhandare *et al.,* 2010). Therefore, in this present work, fronds of Areca catechu which is considered abundant, inexpensive and unexploited plant material, was choose as starting materials for activated carbon production.The residues of areca palm especially their stalks and fronds are considered a waste. In this work, *Pinang* fronds was utilized as precursor for AC production for MB removal.

3.0 Research Methodology

Areca catechu (Pinang) fronds (PF) was used as precursor to prepare AC. The precursor was obtained from Kota Kuala Muda, Kedah, Malaysia. The chemicals used were listed in Table 1, including the supplier, purity and its usage. Properties of methylene blue (MB) was used for preparation of synthetic dye wastewater, Technical nitrogen (purity 99.99 %) and carbon dioxide (purity 99.80 %) gases were supplied by Air Product (M) Sdn Bhd.

Table 1
List of chemicals

Chemical	Supplier	Purity (%)	Usage
Potassium hydroxide pellet (KOH)	Merck, Germany	85	Activating agent
Hydrochloric acid (HCl)	Merck, Germany	37	Adsorbent wash, pH adjustment
Sodium hydroxide (NaOH)	Essex, UK	99	pH adjustment
Methylene blue (MB)	Sigma-Aldrich (M) Sdn Bhd, Malaysia	82	Adsorbate

The AC preparation rig consists of stainless steel (SS) vertical tubular reactor equipped with programmable temperature controller. The gas flow meter was used to control the flow rates of N_2 and CO_2 supplied to the reactor. The piping system was mainly made of Teflon and SS fittings and pipes. The vertical stainless steel reactor with dimension of 150 mm long and 25 mm inner diameter were used to carry out carbonization and activation processes. A SS wire mesh was positioned at the bottom part of the tubular reactor to hold the sample intact.

The reactor was stand and placed inside a vertical tubular furnace with programmable controller (Model Carbolite, USA). The dimension of the furnace was 500 mm length and 82 mm inner diameter. A K-type thermocouple was used to measure the temperature of the sample inside the reactor. The maximum tolerance of the thermocouple is 1000 °C. The excess flue gas was released to a condenser.

Batch adsorption studies was carried out using an Erlenmeyer flask (250 ml) added with 200 ml adsorbate solution (Methylene Blue (MB), placed in the flask, with known initial concentration (1000

mg/L). Weight of adsorbent was fixed at 0.3 g per flask. Isothermal water bath shaker (Model Protech, Malaysia) was used at fixed 120 rpm at constant temperature until a period of time. Double-beam UV-Visible spectrometer (Model Shimadzu UV-1800, Japan) was used to measure the concentration of the dye adsorbates. Beer's law suggests linear relationship between concentration of the absorbing species and their absorbance as represented by the equation:

$$A_i = \varepsilon_\lambda b_c C \qquad (1)$$

where ε_λ is the molar absorptivity coefficient of solute at wavelength λ (nm), A_i is the measured absorbance for component i, C is the solute concentration (mg/L) and b_c is the path length of the cell (1 cm). The absorbance, A_i was obtained by the double-beam UV-Visible spectrophotometer.

The maximum wavelength of the MB was 664 nm. Calibration curve for MB dyes concentration were measured to assure the homogeneity of the absorbance reading. The replication for calibration curve was done for at least three times and the average was used for further experimental work.

The Pinang fronds (PF) samples was washed and cleaned dried in an oven (Model Heraeus Series 6000 Oven, Germany) at temperature of 120 °C for 2 hours. The dried samples were cut into 2-3 cm and kept in a hermetic container.

The activated carbon preparation procedures were divided into three main stages consists of:
i) Carbonization of the precursors
ii) KOH impregnation of char
iii) CO_2 gasification of the KOH-impregnated chars

A thirty gram (30 g) of precursor was put inside a vertical reactor continuously purged with nitrogen gas at 150 mL/min. The reactor temperature was ramped to 400 °C and held for 2 h. After that, the char produced was cooled down to room temperature. Then they were stored inside a desiccator about 2 h for further treatment.

The impregnation of char in powder form (250 μm) with potassium hydroxide (KOH) was done in various impregnation ratio (IR) calculated in Eq. 2;

$$IR = \frac{w_{KOH}}{w_{char}} \qquad (2)$$

where w_{KOH} is the dry weight (g) of KOH pellet and w_{char} is the dry weight (g) of char. A specific amount of char and KOH pellet (depending on the IR) was mixed together with deionized water in a 250 mL beaker. The mixture was stirred thoroughly until completely dissolved and have been put in oven overnight at temperature 105 °C for dehydration.

The KOH-impregnated char was then heat-treated with continuous flow of nitrogen at 150 mL/min. The required temperature was increased at 10 °C/min. Once the desired activation temperature was reached, the nitrogen was switched to carbon dioxide (CO_2) at 150 mL/min and held for a time duration which suggested by Response Surface Methodology (RSM) analysis. The CO_2 was switched back to N_2 (1 h) for the sample to cool to room temperature.

The AC was then washed with deionized water and hydrochloric acid to obtain the neutral value of ph. It was measured by using pH meter (Model Delta 320, Mettler Toledo, China). The AC was dried in oven at 105 °C for 24 hours. The dried AC was stored in desiccator for characterization and further adsorption studies.

This study utilized response surface methodology (RSM) with central composite design (CCD) analysis to optimize the studied parameters. The independent variables include;
    i. x_1, Activation temperature (°C)
    ii. x_2, Activation time (h)

iii. x_3, KOH: char (IR)

These variables and their respective ranges were selected based on the literature and the results obtained from the preliminary studies. As a matter of facts, this analysis used Design Expert software version 7.1.5 (STAT-EASE Inc., Minneapolis, USA) to develop regression model and evaluate the significance of responses. Table 2 shows the range levels of each factor (variable) studied.

Table 2
Independent variables and their coded levels for the CCD

Variables (factors)	Coded variables level				
	$-\alpha$	-1	0	+1	$+\alpha$
Activation temperature (°C)	514	600	725	850	935
Activation time (hour)	0.32	1.00	2.00	3.00	3.68
IR	0.07	0.75	1.75	2.75	3.43

As a result, the complete design matrix of the experiments is shown in Table 3. For three variables; 8 factorial points, 6 axial points and 6 replicates at the center points were employed, as calculated from Equation 3 (Azargohar and Dalai, 2005):

$$N = 2^n + 2n + n_c = 2^3 + 2^*3 + 6 = 20 \qquad (3)$$

where n is the number of factors and N is the total number of experiments.

The center points are used to measure the experimental error and the data reproducibility (Tan *et al.,* 2008b). The independent variables were coded x_1, x_2 and x_3. The low and high level are coded as -1 and +1, respectively. The axial points were located at ($\pm\alpha$, 0, 0), (0, $\pm\alpha$, 0) and (0, 0, $\pm\alpha$) where α is the distance between the center and the axial point. The α value was constant at 1.682 (rotatable). The sequence of experiments was randomized to minimize the extraneous variable effects. The responses include MB removal (Y_1) and activated carbon yield (Y_2). An empirical model was developed based on the response to correlate the response to the independent variables. A second-degree polynomial equation was used and as given by Equation 4:

Table 3
Experimental design matrix

Run	Activated carbon preparation variables		
	Activation temperature, x_1 (°C)	Activation time, x_2 (h)	IR, x_3
1	600(-1)	1.00(-1)	0.75(-1)
2	850(+1)	1.00(-1)	0.75(-1)
3	600(-1)	3.00(+1)	0.75(-1)
4	850(+1)	3.00(+1)	0.75(-1)
5	600(-1)	1.00(+1)	2.75(+1)
6	850(+1)	1.00(+1)	2.75(+1)

7	600(-1)	3.00(+1)	2.75(+1)
8	850(+1)	3.00(+1)	2.75(+1)
9	514(-1.682)	2.00(0)	1.75(0)
10	935(+1.682)	2.00(0)	1.75(0)
11	725(0)	0.32(-1.682)	1.75(0)
12	725(0)	3.68(+1.682)	1.75(0)
13	725(0)	2.00(0)	0.07(-1.682)
14	725(0)	2.00(0)	3.43(+1.682)
15	725.(0)	2.00(0)	1.75(0)
16	725(0)	2.00(0)	1.75(0)
17	725(0)	2.00(0)	1.75(0)
18	725(0)	2.00(0)	1.75(0)
19	725(0)	2.00(0)	1.75(0)
20	725(0)	2.00(0)	1.75(0)

$$Y = b_o + \sum_{i=1}^{n} b_i x_i + \left(\sum_{i=1}^{n} b_{ii} x_i \right) + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} b_{ij} x_i x_j \qquad (4)$$

where Y is the predicted response, b_o the constant coefficient, b_i the linear coefficients, b_{ij} the interaction coefficients, b_{ii} the quadratic coefficients and x_i, x_j represents the independent variables. The responses removal was calculated based on batch adsorption studies that performed in 20 sets of 250 mL Erlenmeyer flasks.

The effect of particular factor is represented by the coefficient with one factor. The interaction between two factors and quadratic effect are represented by the coefficient with two factors with second order terms. An analysis of variance (ANOVA) was used to summarize analysis performed. The "prob > F" value of less than 0.05 indicates that the model is significant (Ahmad and Hameed, 2010). It is desirable to indicate the influence of particular model terms that have significant effects on the response.

Batch equilibrium tests were conducted to study the adsorption performance of adsorbates on the ACs. The effects of adsorbate initial concentration and contact time were studied. The stock solutions (1000 mg/L) of MB was prepared by dissolving accurately weighed 1 gram of the respective dyes in 1000 mL of deionized water (Model USF Maxima version 14.15, England) in a 1000 mL volumetric flask. The stock solutions were stored in dark place to prevent direct sunlight which might cause decolorization and degradation. The solution was withdrawn to measure the concentration (C_o and C_e) by UV-Visible spectrophotometer at the maximum wavelength of the respective adsorbate. The amount of adsorbate adsorbed at equilibrium, q_e (mg/g) was calculated in Equation 5:

$$q_e = \frac{(C_o - C_e)V}{W} \qquad (5)$$

where V is the volume of the solution (L), C_o and C_e (mg/L) are the liquid-phase concentrations of adsorbate at initial and equilibrium, respectively and W is the mass of adsorbent used (g). The adsorbate percent removal was calculated as:

$$\text{Removal \%} = \frac{C_o - C_e}{C_o} \times 100 \qquad (3.6)$$

4.0   Results And Discussions

In this study, one set of experimental designs was applied using RSM for the preparation of activated carbons from biomass materials namely PF. The three variables studied were activation temperature (x_1), activation time (x_2), and KOH/char IR (x_3) with two responses; MB removal (Y_1), and AC yield (Y_2) for synthetic dyes.

During activated carbon preparation, high adsorption capacity and high activated carbon yield were profoundly desirable in production of activated carbon (Auta and Hameed, 2011). However, high adsorption capacity always comes with low activated carbon yield. Activation temperature, activation time and IR were significant effect to the adsorption capacity. However, increasing these three variables result in lower carbon yield, thus optimization of variables was done to identify the optimum preparation conditions for high adsorption capacity and lower carbon yield. Design Expert Software version 7.1.5 (STAT-EASE Inc., Minneapolis, USA) was utilized to optimize the parameters with all the responses were set as maximum values, while the three preparation variables were maintained within the study range.

Optimized parameters were selected with higher desirability. Optimum preparation conditions were selected and verified by experimental of PFAC. The finding shows that the error between the experimental values and predicted values was small. The optimum MB removal by PFAC were obtained by using activation temperature, activation time and IR of 771 °C, 1.48h and 1.94, respectively. The maximum percentage removal of MB obtained was 96.94 % for PFAC.

Figures 4.1 showed that the MB percentage removals was found increase with time and became constant after 22 hours indicating saturation of dyes uptake by the ACs. In the early stage, the surface sites available for adsorption are large in numbers. When the adsorption takes place and start occupying the available sites, the surface sites remained after some time elapsed, becomes difficult for further occupation due to the strong repulsion between the solute molecules of bulk and solid phases (Khattri and Singh, 2009).

As the initial concentration increased from 50 to 500 mg/L the adsorption uptakes at equilibrium were increased from 48.55 to 361.64 mg/g for MB onto PFAC. Higher MB concentration provides stronger driving force of the concentration gradient and resulting higher adsorption capacity. At lower concentration, large AC's surface was able to adsorb dye as ratio of MB to available sites was small. But at higher concentration, the ratio of MB to available sites were higher, thus it reduced the MB removal. Results achieved were in good agreement with adsorption capacity recorded using coconut husk-based AC as reported by Tan *et al*. (2008b). Shaarani and Hameed (2010) also reported that a longer contact time was needed to adsorb dye with high initial concentration by using oil palm empty fruit bunch based AC.
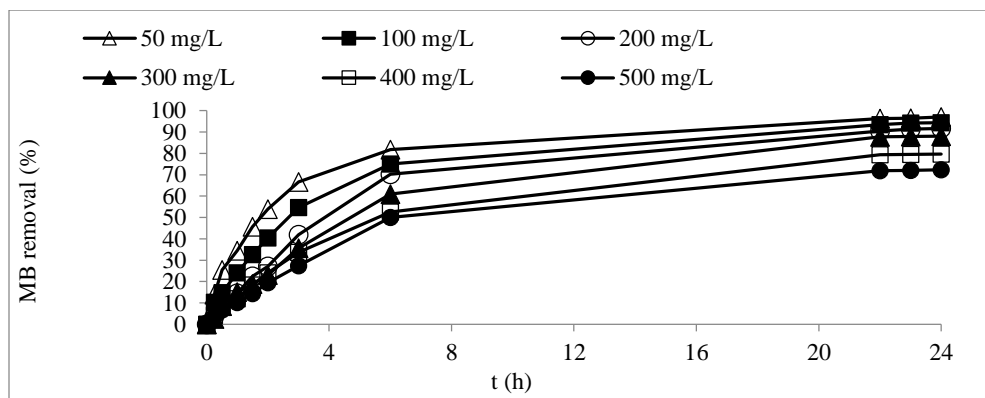

Figure 4.1 MB Percent Removals Versus Adsorption Time at Various
Initial Concentrations at 30°C (PFAC)

5. 0 Conclusion

Areca catechu fronds were successfully used to produce PFAC using physiochemical activation method. The optimum preparation conditions of PFAC were 771ºC, 1.48 h and 1.94 for PFAC. Optimized PFAC gave removal of MB, 96.94 % while the adsorption uptakes of the PFAC increased with increased in adsorbates initial concentration and contact time.

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