

PRODUCTION OF ACTIVATED CARBON FROM SAWDUST USING FLUIDIZED BED REACTOR

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ABSTARCT

Activated carbon was produced from sawdust by using steam activation in a high temperature muffle furnace. Fast pyrolysis process was carried out prior in fluidized a bed furnace to produce char before activation process. Experiments were conducted to investigate the influence of various process parameters such as particle size, pyrolysis temperature and activation time on the quality of the activated carbon. In addition, the batch adsorption studies were done by using methylene blue. The experimental data were analyzed by the Langmuir and Freundlich adsorption isotherms and Langmuir isotherms shows satisfactory fit to the experiment of data. The rate of adsorption shows that the pseudo-second-order rate fitted the adsorption kinetics better than the pseudo-first-order rate equation. The best quality activated carbon was obtained with particle size of 0.5mm at pyrolysis temperature 500°C and activated with steam at 800°C for 2 hours. The maximum adsorption capacity of carbon produced from sawdust was found to be 370.37 mg/g for the methylene blue dye. In addition, the maximum BET surface area obtained was 1091.5m²/g. The surface morphology of the best performing activated carbon was examined using scanning electron microscopy (SEM). The characteristic of the prepared activated carbon was compared with the other researchers and commercial activated carbon. The results indicated that the sawdust carbon could be employed as a low cost alternative to produce commercial activated carbon.

Keywords: Sawdust, Activated carbon, Methylene blue, Adsorption isotherm, Kinetics

INTRODUCTION

Activated carbon is one of the most important microporous adsorbents due to its tremendous adsorptive capacity, an affinity for variety of dissolved organics and ability to be custom-tailored to suit specific application (Ismadji et al., 2005) Various materials are used to produced activated carbon and some of the most commonly used are agriculture wastes such as coconut shell, pistachio shell, saw dust, walnut shell, tropical wood and almond shell (Adinata, 1998).The major uses of activated carbon are in waste water treatment, water purification, gas purification, desulphurization and mercury removal.

It is particular interest to select the sawdust as the raw material to make activated carbon instead of other abundant sources such as clay minerals, rice husks, nut shells, etc. In Malaysia, residues from oil palm and wood based industries are the main biomass source. Currently, large volume of these residues in the form of sawdust, offcut and wood barks were produced by wood based industries. Due to the shortage of wood supply,

some of the waste minimization programs were implemented in these industries in order to maximize the use of the wood residue. Thus, one of the cost effective way is to convert the wood residue to activated carbon.

Pyrolysis is the process where the carboneous source materials are heated, decomposed and converted to carbonized material in the absent of air . Then, the process is continued by activation step which will increase the surface area of the carbonized material. At present, there are 2 different activation processes: (1) steam activation, (2) chemical activation. In steam activation, steam is introduced in temperature range 600-1200°C, whereas in chemical activation, raw material is impregnated with strong dehydrating agent such as phosphoric acid (H_3PO_4) or zinc chloride ($ZnCl_2$) and then heat to 500-800°C to activate the carbon (M.A. Lillo-Rodenas et al., 2003)

Steam activation method is selected in this study because the chemical activation method not only expensive but also relatively polluting the environment due to usage of strong acid or base. In steam activation, it used the concept of water-shift reaction with water vapour (steam) as carrier in order to enhance the porosity of the carbon matrix making them very valuable activated carbon.

From the environment pollution point of view, effluent from the textile industries contain high amount of colored species which are toxic to some organisms, especially to aquatic wildlife and hence indirectly harmful to human health (Lee et al., 1999). Therefore, removal of the dyes before disposal of the waste water is necessary and one of the effective way is by adsorption of dye onto the activated carbon. Therefore, the main focus of this study was to evaluate the adsorption performance of methylene blue (MB) on sawdust-based activated carbon since MB is strong adsorbed onto solid and is often serves as model compound for removing organic contaminants and coloured bodies from aqueous solutions (Husseien, M., et al, 2007)

MATERIALS AND METHODS

Preparation of Activated Carbon. The waste sawdust was collected from a local sawmill. The sawdust was sieved in order to obtain a desirable size fraction (0.5 and 1mm). Then, the sieved sawdust was washed with distilled water to remove any residues or impurities such as ash and dust. Subsequently, it was dried in an oven for 12 hours at 80°C.

After the sawdust was dry, the material was pyrolyzized in a fluidized bed furnace at different temperature range. The pyrolysis process was undergone at temperature 300, 400 and 500°C for half an hour. Then, the char produced was discharged from the first cyclone of the fluidized bed furnace. This fast pyrolysis method produced variety of char at different temperature.

Activation of the char was done by using steam average flowrate 300cc/min at temperature 800 °C in a muffle furnace for 1 and 2 hours. The activated carbon was then ground and dried in an oven at 100 °C for overnight.

Batch Adsorption Studies. The activated carbon was characterized for its adsorption capacity using MB. MB was supplied by Sigma-Aldrich (M) Sdn. Bhd. and was not purified prior to use. Various concentration of MB solution were prepared from 50 to 250ppm by dissolving MB crystal in deionized water. Then, 0.2g of the synthesized

activated carbon was added into 200ml prepared MB solution. The solution was kept in shaking incubator at 30°C with rotation speed 150rpm for 24 hours. The samples will be withdrawn at appropriate time interval using glass syringe to determine the residue concentration of the solutions. Spectrophotometer was used to determine the residual concentration of the methylene blue at 664nm wave length.

The adsorption capacities at time t (q_t) and equilibrium adsorption capacities (q_e) at different concentrations were determined by:

$$q_t = \frac{(C_o - C_t)V}{W} \quad (1)$$

$$q_e = \frac{(C_o - C_e)V}{W} \quad (2)$$

where C_o , C_t , C_e (mg/L) are the liquid-phase concentrations of dye at initial, at time t and at equilibrium, respectively. V is the volume of the solution (L) and W is the mass of dry adsorbent used (g).

RESULTS AND DISCUSSIONS

Effect of Initial Dye Concentration and Size of Adsorbent. The experimental results of sorption of MB on different samples of activated carbon at various concentrations were presented in Figure 1 and Figure 2. Based on the figures, the amount of MB adsorbed per unit mass of activated carbon increased with the increasing concentration of MB. This is mainly due to the increasing in the driving force of the concentration gradient since the initial concentration of MB solution is increasing.

Figure 1 and 2 also shows the adsorption capacity at equilibrium increases from 49.5 to 242.9 mg g⁻¹ and 48.3 to 210.5 mg g⁻¹, respectively. The result indicates that the AC0.5-500-2 has the highest adsorption capacity when compare to AC1-500-2. This is because adsorption being a surface phenomenon, the smaller adsorbent size offered comparable large surface area and hence higher adsorption occurs at equilibrium. It is expected that smaller adsorbent size has well-developed micropores as well as mesopores compared to larger size of adsorbent (Kumar et al., 2006). However, according to Weber (1967), breaking of larger adsorbent size tends to open tiny cracks and channel on the adsorbent surface, providing added surface area which can be employed in the adsorption process. Therefore, much energy might be needed to do so.

On the other hand, the contact time required in attending equilibrium was higher as the initial concentration of MB solution was increasing. For example, in Figure 1, the time required to achieve equilibrium was about 1 hour for 50ppm and 4 hours for 250ppm initial dye concentration. During adsorption process of MB, the MB molecules migrate through the solution to the exterior surface of activated carbon by molecule diffusion (film diffusion), followed by pore diffusion of molecules into interior site of adsorbent, and finally the molecules are adsorbed into the active sites at the interior of the adsorbent. Therefore, this phenomenon takes relatively long contact time (Hameed et al., 2007).

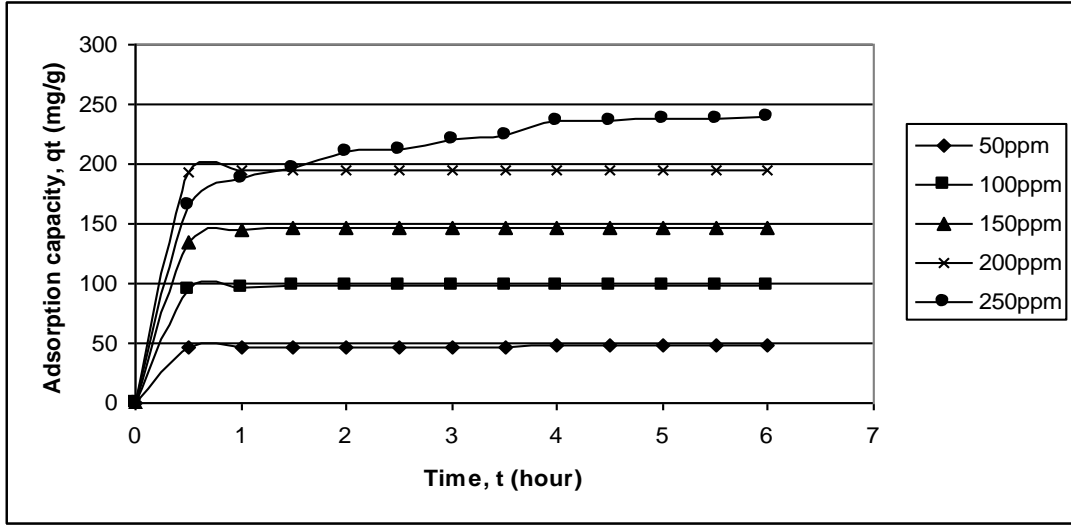


Figure 1: Effect of initial concentration on the removal of MB dye from AC0.5-500-2

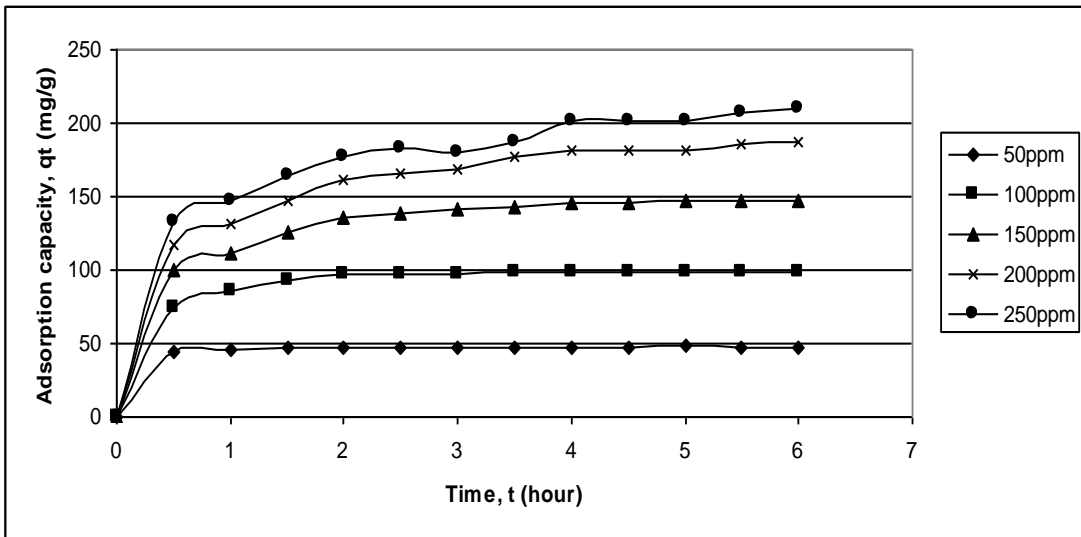


Figure 2: Effect of initial concentration on the removal of MB dye from AC1-500-2

Adsorption Isotherms. Equilibrium behavior of adsorbents usually determined the adsorption isotherm at which the functional equilibrium distribution of adsorption with concentration of adsorbate in solution at constant temperature. The adsorption isotherm indicated how the adsorption molecules distribute between the liquid phase and solid phase when the adsorption process reached an equilibrium stage. The analysis of the isotherm data by fitting them to different isotherm models is an important step to find the suitable model that can be used for design purpose. Several equilibrium models have been developed to describe adsorption isotherm relationships. Two isotherm equations were tested in this work; there were Langmuir and Freundlich isotherm. The applicability of the isotherm models was evaluated by the correlation coefficients, R^2 value of each plot. The higher the R^2 value, the better fit to the model.

Langmuir Isotherm. Langmuir isotherm equation has been widely applied to describe experimental adsorption data based on the assumptions that adsorption energy is constant and independent of surface coverage; that adsorption occurs on localized sites with no interaction between adsorbate molecules, and the maximum adsorption occurs when the surface is covered by a monolayer adsorbate (Malik, 2004). The theoretical Langmuir Isotherm equation can be expressed as (Weber, 1974):

$$q_e = \frac{Q_o b C_e}{1 + b C_e} \quad (3)$$

where Q_o is the maximum amount adsorbed per unit mass of adsorbent corresponding to complete monolayer coverage on the surface bond, b is the constant related to the affinity of binding sites, q_e is the amount adsorbed at equilibrium time (mg/g) and C_e is the equilibrium concentration of the adsorbate (mg/L).

The constant Q_o and b can be determined from the following linearised form of Eq (4):

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{1}{Q_o} (C_e) \quad (4)$$

A plot of C_e/q_e versus C_e will obtain a straight line with a slope of $1/Q_o$ and an intercept of $1/Q_o b$.

The essential features of Langmuir adsorption isotherm can be expressed in terms of a dimensionless constant called separation factor or equilibrium parameter (R_L), which is defined by the following relationship (Weber, 1974):

$$R_L = \frac{1}{1 + b C_o} \quad (5)$$

where C_o is the initial dye concentration (mg/L). The R_L value indicates the shape of the isotherm to be irreversible ($R_L=0$), favorable ($0 < R_L < 1$), linear ($R_L=1$) or unfavorable ($R_L > 1$) (Malik, 2004).

Freundlich Isotherm. Freundlich isotherm is the earliest known relationship describing the sorption equation. This empirical isotherm can be used for non-ideal sorption that involves heterogeneous surface energy systems and is expressed by the following equation (Malik, 2004):

$$q_e = K_F C_e^{1/n} \quad (4)$$

where q_e is the amount of dye adsorbed (mg/g) at equilibrium, C_e is the equilibrium liquid-phase concentrations of dye (mg/L), K_f is the adsorption capacity of the adsorbent $(\text{mg/g})(\text{L/mg})^{1/n}$ and the $1/n$ indicates the adsorption intensity. Values of $n > 1$ represent favorable adsorption condition.

Normally, if the K_F value increased, the adsorption capacity will increased too. The magnitude of n gives the favorability of adsorption. Value of $n > 1$ represent favorable adsorption condition. Eq. (4) can be linearised as following equation:

$$\log q_e = \frac{1}{n} \log C_e + \log K_F \quad (5)$$

A plot of $\log q_e$ versus $\log C_e$ will obtain a straight line with a slope of $1/n$ and an intercept of $\log K_F$.

From Table 1, the experimental data can be fitted well into the Langmuir isotherm model due to the R^2 values lie in the acceptable range ($R^2 > 0.90$). This eventually indicates that the homogeneous nature of the sawdust carbon surface and each dye molecule on sawdust carbon adsorption surface has equal adsorption activation energy. The result also showed the formation of monolayer coverage of dye molecule at the outer surface of sawdust carbon. Values of Q_0 and b were obtained from the slope and interception of the linear plots, as presented in Table 1. The maximum adsorption capacity for AC0.5-500-2 was the highest, 370.37mg/g, as compare to AC1-500-2 which exhibits 294.12. From the value of R_L , it was confirmed that the 3 samples of activated carbon were favorable for adsorption of MB solution since all the R_L values were in the range between 0 and 1.

For Freundlich isotherm, the experimental data can be fitted well only for AC0.5-500-2 due to the R^2 value was 0.9995 which lies in the acceptable range ($0.9 < R^2 < 1.0$). Consequently, it indicated that AC0.5-500-2 also exhibits a multilayer adsorption phenomenon instead of monolayer coverage. However, for AC1-500-2 the R^2 values were 0.6767 which shown poor agreement with Freundlich isotherm. On the other hand, the results suggest that MB dyes were favorably adsorbed by all prepared samples of activated carbon since the values of constant n were greater than the unity ($n > 1$).

Based on the overall adsorption performance, the Langmuir isotherm was the best fit for the adsorption of MB on sawdust activated carbon. This was because most of the correlation coefficient value (R^2) for Langmuir isotherm were more than 0.90, whereas for the Freundlich isotherm, the values were fluctuated and less than 0.90. Apart from that, for size 0.5mm with pyrolysis temperature 500°C and activation time 2 hours, the adsorption capacity was among the highest although the size was smaller. It was predicted that more micropores as well as mesopores were developed at this condition and the morphology surface of the carbon were more uniform.

Table 1: Langmuir and Freundlich constants for the adsorption methylene blue on the prepared activated carbon.

Sample	Langmuir Isotherm				Freundlich Isotherm		
	Q_0 (mg/g)	b (L/mg)	R_L	R^2	K_F ((mg/g)(L/mg) ^{1/n})	n	R^2
AC0.5-500-2	370.37	0.2348	0.0168	0.9475	71.75	1.6287	0.9995
AC1-500-2	294.12	0.2099	0.0187	0.9146	63.83	2.089	0.6767

Adsorption Kinetics. The mechanism and the efficiency of the adsorption process can be inferred from their kinetic studies. Consequently, the kinetic data was processed in order to understand the dynamics of adsorption process in terms of order of rate constant.

Pseudo-First-Order. Kinetic data were treated with the pseudo-first-order kinetic model and the linearized equation is as the following (Malik, 2004):

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (6)$$

where q_e is the amount of dye adsorbed (mg/g) at equilibrium whereas q_t refer to the amount of dye adsorbed at any time, t (min). k_1 is the equilibrium rate constant of pseudo-first-order adsorption in min^{-1} . A linear graph of $\log(q_e - q_t)$ versus t can be plot and the constant value of k_1 , equilibrium adsorption capacity, q_e can be obtained from the slope and interception of the graph respectively.

Pseudo-Second-Order. Kinetic data were further treated with the pseudo-second-order kinetic model and the linearized equation is as the following (Malik, 2004):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (7)$$

where k_2 is the equilibrium rate constant of pseudo-second-order model ($\text{g mg}^{-1} \text{min}^{-1}$). A linear graph of t/q_t versus t can be plotted and the k_2 and the q_e can be calculated from the interception and slope of the graph.

The result of constant values obtained in the linearized graph is shows in Table 2. Based on the value of linear regression coefficient, R^2 , the values were non-constant and shown tremendous different at different initial dye concentration. For example, AC0.5-500-2 has the R^2 value between 0.2185 and 0.9528. Moreover, the experimental q_e did not agree well with the calculated q_e that obtained from the linear plot. As a result, the adsorption of MB onto activated carbon was not the Pseudo-first-order kinetics. Undoubtedly, the R^2 values for second-order kinetic model were higher and more constant than first-order kinetic. Most of the R^2 values were more than 0.99. In addition, the calculated equilibrium adsorption capacity, $q_{e,cal}$ agreed very well with the experimental data, $q_{e,exp}$. This indicates that the adsorption of MB onto sawdust activated carbon obeys the pseudo-second-order kinetic and that the overall adsorption rate was controlled by the chemisorptions process.

Apart from that, the applicability of both kinetic models can be verified by Sum of Error Square equation (SSE %) instead of R^2 as shown at below:

$$SSE \% = \sqrt{\frac{\sum (q_{e,exp} - q_{e,cal})^2}{N}} \quad (8)$$

where N is the number of data points. The higher the value of R^2 and the lower the value of SSE% will indicates the best fit for the kinetic model. Based on Table 2, most of the SSE% values were lower for second kinetic model. Therefore, it was an added proved that Pseudo-second-kinetic model was best fit for MB adsorption onto sawdust activated carbon.

Table 2: Kinetics parameter for adsorption of dye on different sample sawdust carbon

Activated Carbon	Concentration (ppm)	First-order kinetic model					Second-order kinetic model			
		q_e, exp (mg/g)	q_e, cal (mg/g)	k_1 (1/min)	R^2	SSE (%)	q_e, cal (mg/g)	k_2 (g/mg min)	R^2	SSE (%)
AC0.5-500-2	50	49.45	5.58	0.2540	0.3077	11.73	47.85	1.4560	0.9999	0.43
	100	98.36	3.16	0.3300	0.202	25.44	99.01	1.0201	1	0.17
	150	146.72	4.58	0.8160	0.4958	37.99	147.06	0.5138	1	0.09
	200	194.81	2.25	0.4924	0.2185	51.46	196.08	2.6010	1	0.34
	250	242.90	133.54	0.6372	0.9528	29.23	250.00	0.0145	0.9965	1.90
AC1.0-500-2	50	48.31	5.07	0.4645	0.5171	11.56	47.85	0.8736	0.9999	0.12
	100	98.20	12.34	0.7082	0.5345	22.95	100.00	0.1111	0.9995	0.48
	150	147.54	93.26	0.9546	0.9637	14.51	153.85	0.0282	0.9980	1.69
	200	192.08	105.90	0.5265	0.9485	23.03	196.08	0.0145	0.9955	1.07
	250	230.55	124.05	0.3201	0.8782	28.46	217.39	0.0118	0.9926	3.52

Scanning electron micrograph (SEM) studies. The surface morphologies of activated carbon produced was determined using SEM. Figure 3(a) and (b) shows the SEM photograph of AC0.5-500-2 at 400x and 1000x magnifications, respectively. Figure 4(a) and (b) shows the SEM photograph of AC1.0-500-2 at 500x and 1000x magnifications, respectively.

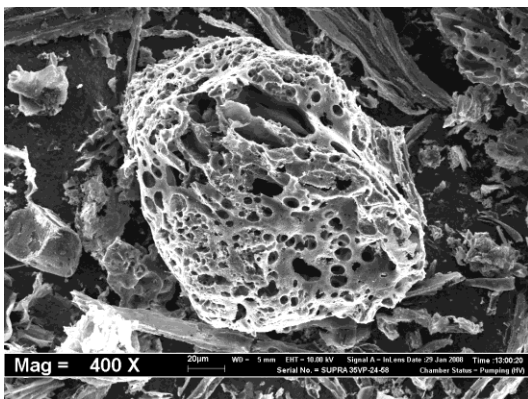
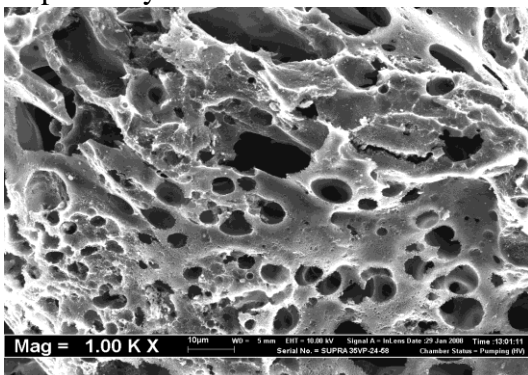


Figure 3 (a) and (b) SEM of AC0.5-500-2

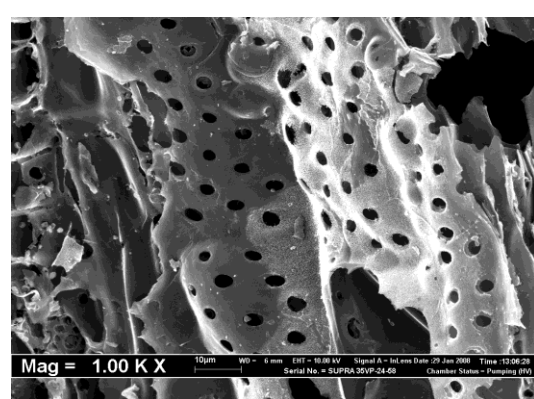
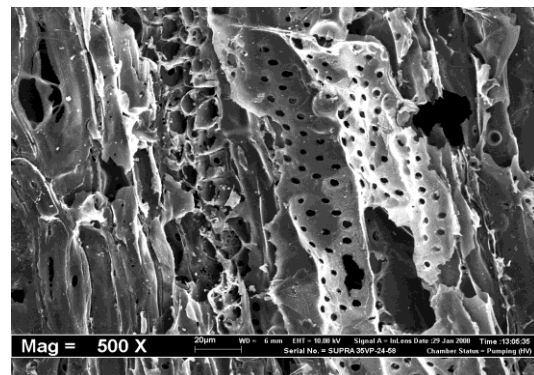


Figure 4 (a) and (b) SEM AC1-500-2

It can be observed that AC0.5-500-2 has developed more pores than the AC1-500-2; however, the pores developed were not in the uniform shape. It can be seen that although AC1-500-2 has uniform pores developed, however the adsorption capacity was poorer than AC0.5-500-2. This is because adsorption is a surface phenomenon; therefore smaller adsorbent particle size offers a comparatively larger and more accessible surface area (more pores developed) and hence higher adsorption occurs at equilibrium (Krishnan, 2008)

BET surface area studies

The AC0.5-500-2 had been selected for the BET surface area test since it has the highest adsorption capacity and the BET surface area was $1091.5\text{m}^2/\text{g}$ for the carbon. The result was comparable and even better with other early works as shown in Table 3. From the table, the highest value of BET surface area obtained by Kumar et al., 2005 was $1150\text{m}^2/\text{g}$. Although this value was slightly higher than this study, but the optimum activation holding time was 5 hours which was 3 hours longer time than this study. The BET surface area obtained in this study showed the activated carbon synthesized was good which offered shorter activation time and simpler process than other early works, especially early works with chemical activation.

CONCLUSION

Sawdust was found to be a viable raw material for the preparation of activated carbon. However, the quality of the activated carbon synthesized was highly dependent on the preparation condition. From the result of the present work, it can be said that smaller size of the sawdust, higher pyrolysis temperature and longer activation time will exhibits a better result in the removal of methylene blue. The optimum condition in this study for the preparation of activated carbon was 0.5mm size of sawdust, pyrolyzed at 500°C and activated with steam at 800°C for 2 hours.

In addition, the present study showed that sawdust was considerably efficient in removal of methylene blue from aqueous solution. The experimental data were fitted well with the Langmuir model of adsorption, indicates that monolayer coverage of dye molecules at the outer surface of sawdust carbon. The value for the maximum adsorption capacity, Q_0 , was 370.37 mg/g , and the maximum BET surface area was $1091.5\text{m}^2/\text{g}$. These results were significantly high when compared to other researchers and commercial activated carbon. The kinetics of methylene blue on sawdust carbon follows the pseudo-second-order model, indicated that the adsorption was controlled by chemisorption process. Since sawdust is freely available, the utilization of sawdust to become activated carbon seems to be economical.

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Table 2.3: Summary of early works of activated carbon synthesized from wood and their maximum BET surface area.

No	Author	Raw Material	Experiment Conditions	BET Surface Area (m ² /g)
1	Krishnan, 2008	Sawdust	Sawdust was blended with 20% K ₂ CO ₃ solution. Carbonization in the presence of steam was performed in muffle furnace at 600°C.	686.3
2	Mohanty et al., 2005	Tectona grandis sawdust	Sawdust was blended with 100-300% ZnCl ₂ solution at 50°C for 1 hour. Carbonization step was performed at 300-600°C for 1-3 hour(s) holding time under nitrogen flow rate of 150cm ³ /min.	585
3	Kumar et.al, 2006	Rubberwood sawdust	Sawdust was carbonized in fluidized bed reactor at 600°C for 1 hour. The carbonized char then activated with steam at 400-750°C for 1-4 hours.	1092
4	Hameed et.al, 2006	Rattan sawdust	Carbonized at 700°C under nitrogen for 1 hour. The char then soaked with potassium hydroxide (KOH) and activated using carbon dioxide at 850°C for 2 hours.	1083
5	Ismadji et al., 2005	Teak sawdust	The sawdust was pyrolysis at 600°C for 1 hour under nitrogen flowrate 150mL/min. The char then activated with steam (0.02g/min) at 750-900°C for 2-10 hours holding time. The optimum condition occurred at activation temperature 850°C for 5 hours holding time.	1150
6	Wu et. al, 2004	Fir wood	Fir wood was sealed in ceramic oven and heated to 550°C. In the mean time, steam generated from deionized water was poured into the oven at 3cm ³ /min for 3 hours. Subsequently, the oven was heated to 890-900°C with the same steam flow rate for holding time 5 hours.	1016
7	Wu et. al, 2004	Fir wood	Fir wood was sealed in ceramic oven and heated to 550°C. In the mean time, steam generated from deionized water was poured into the oven at 3cm ³ /min for 3 hours. The resulted char was mixed with KOH solution and dried at 130°C for 24 hours. Then, the carbon was placed in oven and heated to 780°C for 1 hour.	1064
8	Dalai et.al, 2008	Whitewood (Spruce)	The biochar was produced from whitewood (Spruce) using fast pyrolysis process by Dynamotive Energy Systems Corporation. The char was activated by steam at 600-900°C for 0.9-4 hours activation time, under mass ratio of steam to char 0.4-2.	664

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