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Dr W.H. **Cheung**

Department of Chemical and Biomolecular Engineering, Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong

School of Chemical and Biomolecular Engineering, The University of Sydney, NSW, Australia 2006

Email: keawhc@ust.hk

Miss L.S. **Chan**

School of Chemistry and Chemical Engineering, Queen's University of Belfast, Northern Ireland, UK BT7 1NN

Department of Chemical and Biomolecular Engineering, Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong

Email: conniechan@palhk.com

Mr Ken S.T. **Lau**

Department of Chemical and Biomolecular Engineering, Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong

Email: kenlau98@hotmail.com

Professor Stephen J. **Allen**

School of Chemistry and Chemical Engineering, Queen's University of Belfast, Northern Ireland, UK BT7 1NN

Email: s.allen@qub.ac.uk

Professor Gordon **McKay** FHKIE, FIChemE

Department of Chemical and Biomolecular Engineering, Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong

Email: kemckayg@ust.hk

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W.H. Cheung<sup>a,b</sup>, L.S. Chan<sup>a,c</sup>, K.S.T. Lau<sup>a</sup>, S.J. Allen<sup>c</sup> and G. McKay<sup>a,\*</sup>

<sup>a</sup>Department of Chemical and Biomolecular Engineering, Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong

<sup>b</sup>School of Chemical and Biomolecular Engineering, The University of Sydney, NSW, Australia

<sup>c</sup>School of Chemistry and Chemical Engineering, Queen's University of Belfast, Northern Ireland, UK

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\* Corresponding author: Fax: +852 23580054. E-mail address: [kemckayg@ust.hk](mailto:kemckayg@ust.hk) (G. McKay)

## **Abstract**

Bamboo, indigenous to Hong Kong and China, is widely used as scaffolding in construction and building projects. Over 50,000 tonnes of bamboo scaffolding waste is disposed as landfill waste each year. These wastes can be used as a sustainable raw material for the production of a range of high value added activated carbons. Activated carbons were produced by thermal activation of bamboo with phosphoric acid. Surface areas up to 2500m<sup>2</sup>/g were produced. In addition, two acid dyes with different molecular sizes, namely Acid Yellow 117 (AY117) and Acid Blue 25 (AB25), were used to evaluate the adsorption capacity of the produced carbons. It was found that the dye with smaller molecular size, AB 25, was readily adsorbed onto the produced carbon, nearly three times higher than a commercially available carbon while the larger size dye, AY117, showed little adsorption.

## **1. Introduction**

The discharge of effluents containing toxic materials such as metal ions and dyestuffs from a wide range of industries – electroplating, microelectronics, metal forming, paper, textiles, chemicals – is of concern to the public, industry and government alike. The main treatment processes include: chemical precipitation, membrane separation, adsorption and ion exchange. Adsorption is one of the most effective in addressing the stringent requirements for pollution abatement. Numerous adsorbents including inorganic, agricultural and shell-fish by-products have been considered for adsorption (Garg, et al., 2003; Juang, et al., 1996; McKay, et al., 1998; Minamisawa, et al., 2005; Poots, et al., 1978; Walker, et al., 2003). The use of activated carbons however, has been widely favored because of their high adsorption capacities and amphoteric properties which enables their adsorption of both cationic and anionic pollutants in effluents (Al-Degs, et al., 2000; Corapcioglu and Huang, 1987). A challenge in the field of activated carbon production is to produce specific materials with given properties including pore size distribution and surface area from low cost precursors and at low temperature. In recent years, considerable research has focused on low cost alternative materials as precursors for the production of active carbons from agricultural wastes such as fruit stones, oil-palm shell and bagasse (Lua and Guo, 2001; Puziy, et al., 2005; Valix, et al., 2004).

Bamboo is a tropical plant and is indigenous to Southern Asia, including China, Hong Kong, Thailand and Vietnam. It has a rapid growth rate and consumes little energy (0.5MJ/kg). It is a sustainable product and is widely used in Hong Kong's construction industry as scaffolding. Over 50,000 tonnes of bamboo scaffolding each year is dumped as construction waste from Hong Kong's building and construction projects. Bamboo waste can be used as a raw material for the production of a range of activated carbons and carbon chars due to its high carbon content (44%). The bamboo can be carbonized in a furnace at high temperature in the

absence of oxygen to produce carbon chars. The chars can be further treated using various chemicals and over a range of temperatures to produce a selection of carbons for various uses (Asada, et al., 2002; Ohe, et al., 2003; Wu, et al., 1999). Bamboo-based activated carbon has the potential to be a sustainable and commercially available for the treatment of 1) gaseous pollutants, 2) liquid pollutants in industrial effluents, 3) in drinking water filtration applications and 4) fuel cell and electronic applications.

In the present study, the preparation and characterization of high surface area activated carbon from waste bamboo scaffolding by low temperature chemical activation will be described. In addition, dye adsorption is conducted on the produced carbon and compared with a commercially available carbon, Calgon F400.

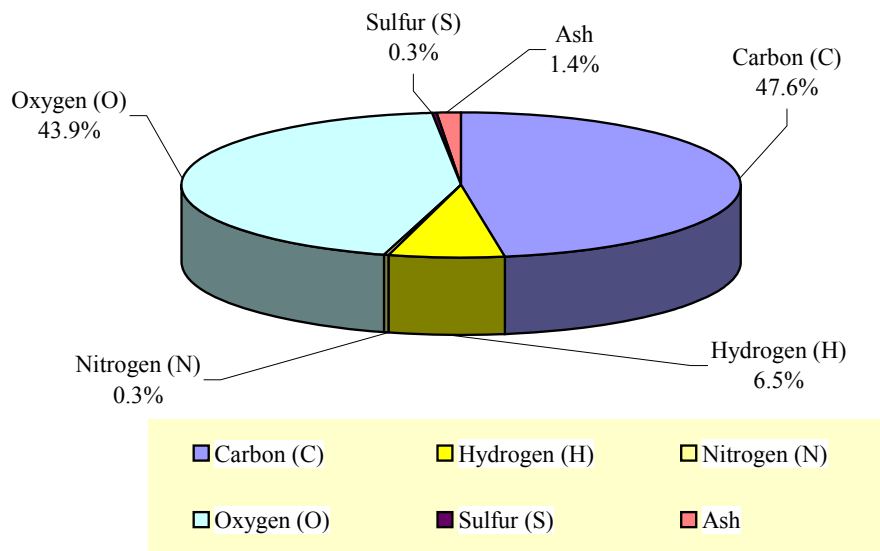
Commonly, two types of activation process are used for the production of activated carbon, namely, chemical and physical routes. For low temperature activation, the chemical route has an advantage over the physical process. Phosphoric acid activation is a conventional method for the preparation of active carbon from lignocellulosic materials (Laine, et al., 1989). Phosphoric acid induces important changes in the pyrolytic decomposition of the lignocellulosic materials as it promotes depolymerization, dehydration and redistribution of constituent biopolymers (Jagtoyen and Derbyshire, 1993). So far, only limited research has been carried out on bamboo as precursor utilizing the high temperature physical activation pathway. BET-nitrogen surface area ranges from 41 to 1038 m<sup>2</sup>/g have been reported (Abe, et al., 2001; Asada, Ishihara, Yamane, Toba, Yamada and Oikawa, 2002; Kannan and Sundaram, 2001; Wu, Tseng and Juang, 1999).

## **2. Experimental**

### **2.1 Materials**

The received waste bamboo scaffolding was washed with water, dried and reduced to size by hammer milling prior to experiment. A particle size of 500 – 710 µm was used throughout

the present study. The average chemical composition of bamboo is measured by an elemental analysis and is shown in Figure 1. The major elements are carbon (47.6%) and oxygen (43.9%) accounting for around 91% of bamboo. Other elements include hydrogen (6.5%), nitrogen (0.3%), sulfur (0.3%) and ash (1.4%). This raw material has been pretreated by soaking and saturating with ortho-phosphoric acid ( $H_3PO_4$ ) at different acid to bamboo ratios (Xp). The mixture has been stirred thoroughly to ensure homogenous mixing of the bamboo and  $H_3PO_4$ . Then, the samples were subjected to a two-step heating process. Firstly, the mixture was heated at 150°C for two hours and followed by either 400 or 600°C in a furnace under flowing nitrogen for a further four hours. After heating, samples were cooled, washed and dried for further analysis and characterization.



**Figure 1** Elemental composition of bamboo.

## 2.2 Characterization

The apparent surface area of the activated carbon was determined from nitrogen adsorption at 77K in a Quantachrome Autosorb 1-MP. The carbon analyses of activated bamboo were conducted using Elementar vario EL III elemental analyzer.

Chemical activated carbons were characterized by surface area, pore size distribution, elemental carbon analysis and dye adsorption equilibrium capacity. Surface areas were calculated using the Braunauer, Emmett and Teller (BET) equation (Brunauer, et al., 1938). The molecular area of the nitrogen adsorbate was taken as  $16.2 \text{ \AA}^2/\text{molecule}$ . The total pore volumes were calculated by converting the nitrogen gas adsorbed at a relative pressure 0.98 to the volume of liquid adsorbate. Micropore volume was estimated using the t-plot method (Lippens and de Boer, 1965). The mesopore volume was obtained as the difference between total pore volume and micropore volume. Pore size distribution was obtained using the Barrett, Joyner and Halenda (BJH) method (Barrett, et al., 1951).

### 2.3 Equilibrium Adsorption Isotherm Study

The acid dye adsorption test was used to determine the adsorption capacity of the products using two acid dyes, Acid Yellow 117 (AY117) and Acid Blue 25 (AB25) from Ciba Special Chemical and Sigma-Aldrich, respectively. The molecular structure and properties are shown in Figure 2 and Table 1. A fixed mass of activated carbon, 20.0 mg was weighed into 75 mL glass bottles and brought into contact with 50mL of dye solution with predetermined initial dye concentrations. The flasks were sealed and agitated continuously at 200 rpm in the thermostatic shaker bath and maintained at a temperature of  $25 \pm 1 \text{ }^\circ\text{C}$  until equilibrium was reached. At time  $t = 0$  and equilibrium, the dye concentrations of the solutions were measured by Varian Cary 1E UV-Vis Spectrophotometer. These data were used to calculate the adsorption capacity,  $q_e$ , of the adsorbent. The adsorption capacities ( $q_e$ ) of the each activated carbon were determined by:

$$q_e = (C_0 - C_e)V/m \quad (1)$$

where  $q_e$  = the dye concentration on the adsorbent at equilibrium (mmol/g)  $C_0$  = the initial dye concentration in the liquid phase (mmol of dye / L),  $C_e$  = the liquid-phase dye concentration at equilibrium (mmol of dye / L),  $V$  = the total volume of dye-activated carbon

mixture (L),  $m$  = mass of adsorbent used (g). Finally, the adsorption capacity,  $q_e$ , was plotted against the equilibrium concentration,  $C_e$ .

**Table 1** physical property of AB25 and AY117.

	Acid Blue 25	Acid Yellow 117
Color Index	62055	24820
Abbreviation	AB25	AY117
Molecular Weight (g/mol)	416.4	848
Dye Content (%)	45	60
Chromophore	Anthraquinone	Disazo
Maximum Wavelength, $\lambda_{\max}$ (nm)	601	438
Charge	-1	-2
Width (Å)	12.452	29.055
Depth (Å)	10.165	20.207
Thickness (Å)	2.530	4.010

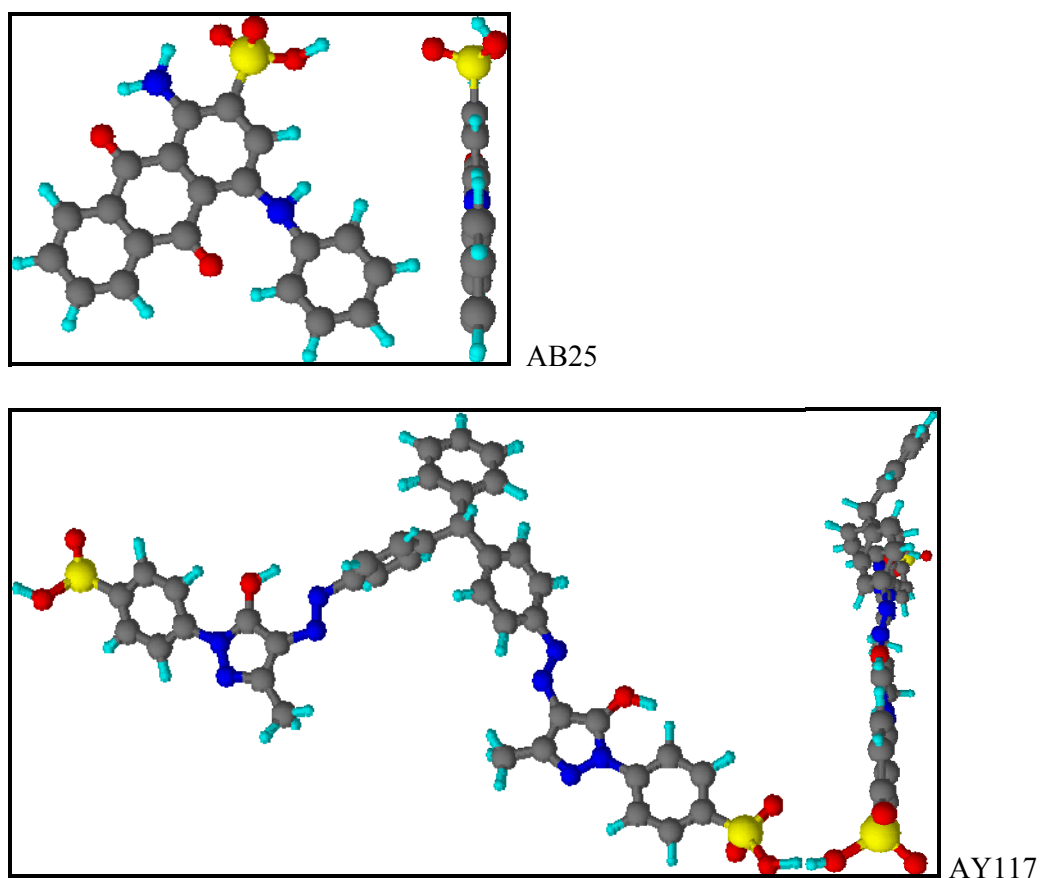
### 3. Results and Discussion

#### 3.1 Activated Carbon Production

Phosphoric acid activated bamboo carbons were produced at two temperatures, 400 and 600°C with different acid to bamboo ratio. Figure 3 shows the plot of the BET surface areas of the produced carbon against the acid to bamboo ratio,  $X_p$  and the two temperatures. The surface areas from the activated carbons produced from waste bamboo scaffolding were comparable to the commercially available carbons such as F400 (790 – 1100 m<sup>2</sup>/g), F300 (950 – 1050 m<sup>2</sup>/g) and Witco 517 (1050 m<sup>2</sup>/g) and other lignocellulosic based carbons. In some cases, depending on the  $X_p$ , higher values were obtained, thus, indicating that the produced bamboo carbon would have similar dyes adsorption capacities as the commercial



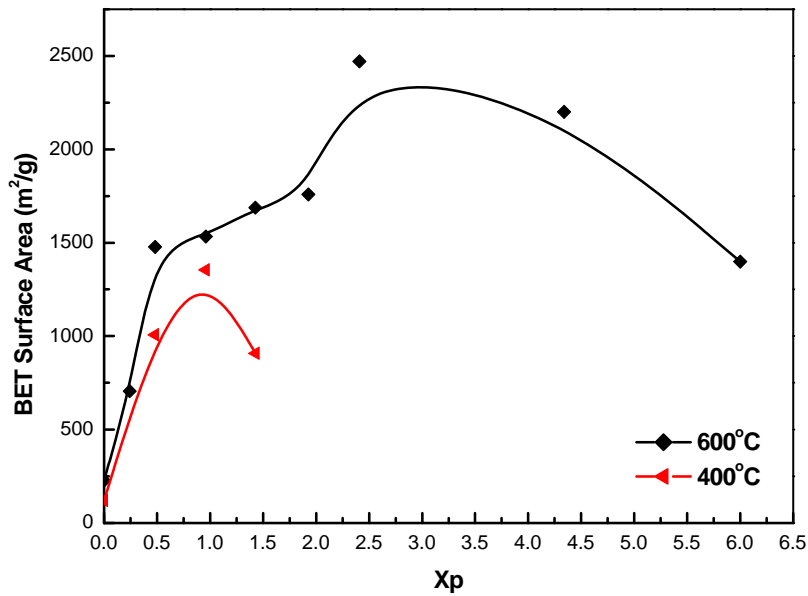
variety. Furthermore, the surface area of the carbon increased with increasing temperature for all  $X_p$  values. A maximum was found at  $X_p=2.41$  at  $600^\circ\text{C}$ , while at  $400^\circ\text{C}$ , the maximum surface area was reached at lower  $X_p$  of 0.96. This is in close agreement with published works that maximum carbon properties for lignocellulosic materials activated with  $\text{H}_3\text{PO}_4$  at  $450^\circ\text{C}$  with  $X_p$  ranging from 1.2 to 1.4 (Castro, et al., 2000; Girgis and El-Hendawy, 2002; Girgis, et al., 1994; Khalil, et al., 2000; Philip and Girgis, 1996).



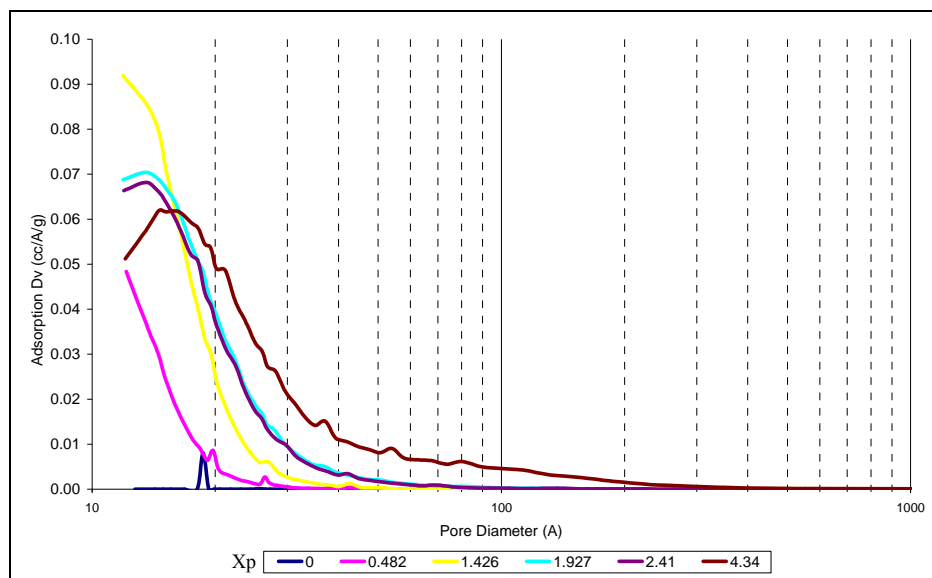
**Figure 2** The molecular structure of AB25 and AY117.

The pore size distribution using the BJH method (Barrett, Joyner and Halenda, 1951) of the produced carbons show the mixture of microporosity and mesoporosity. Figure 4 shows the pore size distribution of the produced carbon with different  $X_p$  activated at  $600^\circ\text{C}$ . The average pore diameter of the produced carbons ranged from 15 to  $55\text{\AA}$  and was found to increase with increasing  $X_p$ . The pore size distribution was found to be playing a major role

in competitive adsorption as it determined the degree of direct site competition and pore blockage mechanisms (Pelekani and Snoeyink, 2000; 2001). These results suggested that the prepared carbon could remove the dyes selected in the present study effectively because the molecular dimensions of the dyes were within the pore size range of the produced bamboo carbon.



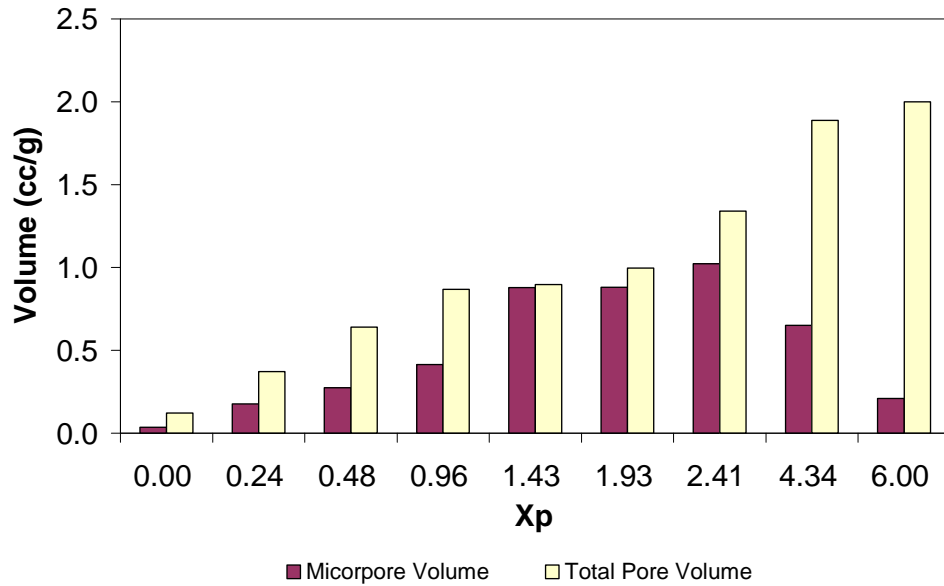
**Figure 3** The BET surface areas of the produced bamboo carbons at different acid to bamboo mass ratio,  $X_p$ , and activation temperatures.



**Figure 4** Pore size distribution of the bamboo carbon with different  $X_p$  and activated at  $600^\circ\text{C}$ .

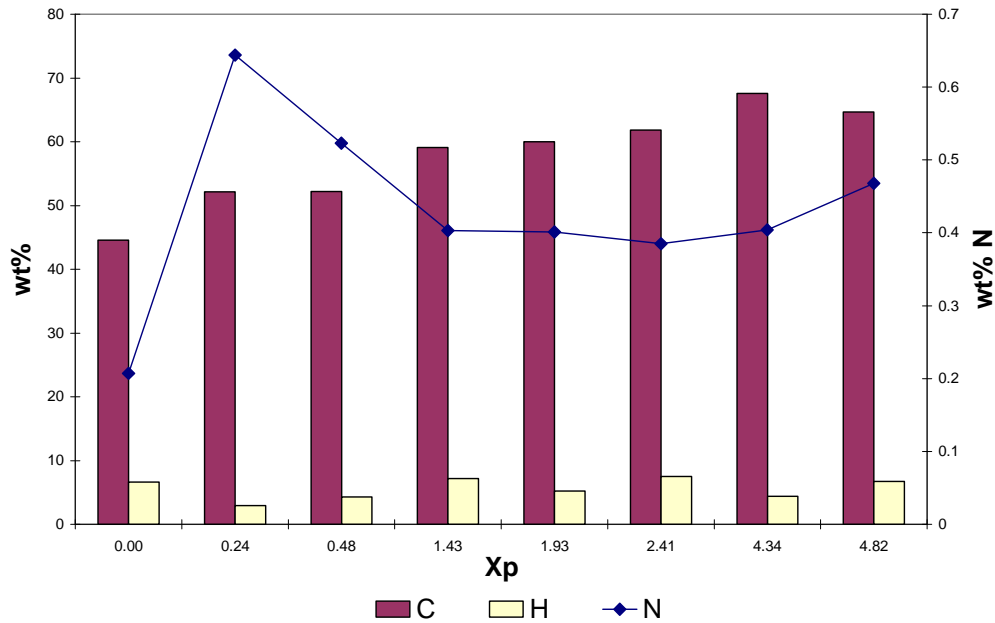
In addition, Figure 5 suggests the total pore and micropore volumes of the produced bamboo carbons are affected by  $X_p$ . The highest micropore volume was found to be at  $X_p = 2.41$  and the total pore volume was maximized at  $X_p = 6.0$ . Increasing the impregnation ratio seems to open up the porous structure of the produced carbon, creating larger pores (mesopore and macropore) with increasing  $X_p$ . This was in agreement with the surface area measurement as micropore provides the majority of surface area in activated carbon.

The elemental analysis of the produced carbon at a temperature of  $600^\circ\text{C}$  is shown in Figure 6. There was little effect on the amount of C, H and N at high  $X_p$  values. As can be seen from Figure 6, the carbon content gradually increased from 44 wt% of the raw bamboo to 68 wt% at an  $X_p$  of 4.34. The amount of nitrogen increased rapidly when  $\text{H}_3\text{PO}_4$  was introduced and then dropped and leveled at around 0.4 wt%. However, there was no general trend for the hydrogen content in the produced carbon. Furthermore, there was no sulfur detected in the produced carbon.



**Figure 5** Pore volume of the produced bamboo carbon at various Xp and activation temperature of 600°C

As can be seen in Figure 3, the BET surface area of the produced carbon increased from 227 to 2471 m<sup>2</sup>/g with the increase of acid to bamboo ratio (Xp) from 0 to 2.41 and then decreased after that to 1400 with an Xp of 6.00 at 600°C. At the lower temperature (400°C), the BET surface area is lowered at the same Xp values. Similarly, Figure 5 shows the total pore volume increases with increasing acid to bamboo ratio to a maximum with Xp = 6.0. However, the optimum micropore volume occurs at Xp = 2.41.



**Figure 6** C, H and N content of the produced bamboo carbon with different Xp and activation temperature of 600°C.

### 3.2 Equilibrium Adsorption Isotherm Study

Three produced carbons, namely, HSA1, HSA2 and LSA, were compared with a commercial carbon (F400) for dye adsorption. The physical properties of these carbons are shown in Table 2. Two acid dyes were used, namely Acid Blue 25 (AB25) and Acid Yellow 117 (AY117). The AB25 adsorption capacities for HSA carbons are nearly three times higher than that of F400. A similar adsorption capacity is observed for AY117 between HSAs and F400. On the other hand, the LSA carbon shows poor adsorption for both acid dyes. The removal of the acid dyes can be related to the carbons' porosity characteristics which determine the accessibility of the dye molecules. As shown in Table 2, AB25 is a small molecule compared to AY117; its adsorption would mainly take place in the smaller pores. By comparing the physical properties of the produced carbons, HSAs have very high surface areas and micropore volumes compared to the other two carbons. Thus, it would provide the higher

capacity for AB25. On the other hand, AY117 is too large to be adsorbed by the micropores. Although LSA is also a microporous carbon, its surface area and pore volume are relatively lower, hence, less AB25 is able to be adsorbed. The adsorption of AB25 depends on both the surface area and pore volume (micropore and mesopore) of the carbon as well.

The experimental data were fitted into Langmuir (1918), Freundlich (1906) and Redlich-Peterson (1959) equations to determine which isotherm gives the best correlation to experimental data.

### *3.2.1 Langmuir Isotherm*

Langmuir (1918) proposed a theory to describe the adsorption of gas molecules onto metal surfaces. The Langmuir adsorption isotherm has found successful application for many other real sorption processes of monolayer adsorption. Langmuir's model of adsorption depends on the assumption that intermolecular forces decrease rapidly with distance and consequently predicts the existence of monolayer coverage of the adsorbate at the outer surface of the adsorbent. The isotherm equation further assumes that adsorption takes place at specific homogeneous sites within the adsorbent. It is then assumed that once a dye molecule occupies a site, no further adsorption can take place at that site. Moreover, the Langmuir equation is based on the assumption of a structurally homogeneous adsorbent where all sorption sites are identical and energetically equivalent. Theoretically, the sorbent has a finite capacity for the sorbate. Therefore, a saturation value is reached beyond which no further sorption can take place. The saturated or monolayer (as  $C_t \rightarrow \infty$ ) capacity can be represented by the expression:

$$q_e = \frac{K_L C_e}{1 + a_L C_e} \quad (2)$$

where  $q_e$  is solid phase sorbate concentration at equilibrium (mmol/g),  $C_e$  is aqueous phase

sorbate concentration at equilibrium ( $\text{mmol}/\text{dm}^3$ ),  $K_L$  is Langmuir isotherm constant ( $\text{dm}^3/\text{g}$ ),  $a_L$  is Langmuir isotherm constant ( $\text{dm}^3/\text{mmol}$ ).

**Table 2** The physical properties of the produced carbon used for equilibrium adsorption study of acid dyes.

<b>Properties</b>	<b>HSA1</b>	<b>HSA2</b>	<b>LSA</b>	<b>F400</b>
BET surface area ( $\text{m}^2/\text{g}$ )	2471	2200	758	747
Micropore area ( $\text{m}^2/\text{g}$ )	2172	1398	724	673
External surface area ( $\text{m}^2/\text{g}$ )	245	802	34	74
Average pore diameter ( $\text{\AA}$ )	22.20	34.31	22.33	25.18
Total pore volume ( $\text{cc}/\text{g}$ )	1.341	1.887	0.423	0.470
Micropore volume ( $\text{cc}/\text{g}$ )	1.023	0.651	0.377	0.348

The Langmuir equation is applicable to homogeneous sorption where the sorption of each sorbate molecule onto the surface has equal sorption activation energy. The Langmuir equation obeys Henry's Law at low concentration; when the concentration is very low,  $a_L C_e$  is far smaller than unity, it implies  $q_e = K_L C_e$ , hence, it is analogous to Henry's Law.

### 3.2.2 Freundlich Isotherm

The Freundlich (1906) equation is an empirical equation employed to describe heterogeneous systems, in which it is characterized by the heterogeneity factor  $1/n$ . When  $n=1$ , the Freundlich equation reduces to Henry's Law. Hence, the empirical equation can be written:

$$q_e = K_F C_e^{\frac{1}{n}} \quad (3)$$

where  $q_e$  is solid phase sorbate concentration in equilibrium (mmol/g),  $C_e$  is liquid phase sorbate concentration in equilibrium (mmol/dm<sup>3</sup>),  $K_F$  is Freundlich constant (dm<sup>3</sup>/mg<sup>1-1/n</sup>/g) and  $1/n$  is the heterogeneity factor. This isotherm is another form of the Langmuir approach for adsorption on an "amorphous" surface. The amount adsorbed material is the summation of adsorption on all sites. The Freundlich isotherm is derived by assuming an exponential decay energy distribution function inserted in to the Langmuir equation. It describes reversible adsorption and is not restricted to the formation of the monolayer.

### 3.2.3 Redlich-Peterson Isotherm

Redlich and Peterson (1959) incorporate three parameters into an empirical isotherm. The Redlich-Peterson isotherm model combines elements from both the Langmuir and Freundlich equation and the mechanism of adsorption is a hybrid one and does not follow ideal monolayer adsorption.

The Redlich-Peterson equation is widely used as a compromise between Langmuir and Freundlich systems. For further application of the isotherms for use in kinetic or mass transport models, it is important to have the most accurate correlating equation.

$$q_e = \frac{K_R C_e}{1 + a_R C_e^B} \quad (4)$$

where



re  $q_e$  is solid phase sorbate concentration in equilibrium (mmol/g),  $C_e$  is liquid phase sorbate concentration in equilibrium (mmol/dm<sup>3</sup>),  $K_R$  is Redlich-Peterson isotherm constant (dm<sup>3</sup>/g),  $a_R$  is Redlich-Peterson isotherm constant (dm<sup>3</sup>/mg<sup>1-1/β</sup>) and  $\beta$  is the exponent which lies between 1 and 0.

The fitness quality of the isotherms, generated from non-linear approach, to the experimental data is optimized by error function. In this project, error function – sum of squared error (SSE) (eqn. 5) is employed and the isotherm parameters are determined by minimizing the error function across the entire concentration range of studies using the Solver add-in with Microsoft's spreadsheet, Excel.

$$SSE = \sum_{i=1}^N (q_{e,cal} - q_{e,exp})_i^2 \quad (5)$$

By comparing the SSE values for the three equilibrium isotherm models as shown in Tables 3 and 4, none of the models fit the adsorption of AY117 on HSA and AB25 on LSA well. For F400, the Redlich-Peterson model fits both dye adsorption behavior well. On closer examination, for AB25, Langmuir model can be used to describe the system adequately, as  $\beta$  in the Redlich-Peterson model is very close to unity while in AY117 system, its adsorption behavior is towards Freundlich. For the bamboo carbons, the adsorption characteristics of both dyes do not follow the Langmuir model. The Redlich-Peterson model describes the dye-bamboo carbon system reasonably well. This suggests a reasonable fixed value for the sorption activation energy, which could correspond to the chelation bond energy between the dye molecule and surface of the carbon, most likely with a lone pair of electrons on the carbon surface.

For AY117 with LSA and F400, the high correlation of the Freundlich model suggests more than one mechanism with a degree of heterogeneity is possible for ionic species involved in the solution and on the carbon surface.

#### **4. Conclusions**

The preparation and investigation of high BET surface area activated carbons from scrap construction bamboo by low temperature chemical activation has been demonstrated. The produced carbon properties including surface area and porosity were affected by the ratio of the phosphoric acid to bamboo. Super-high surface area,  $\sim 2500 \text{ m}^2/\text{g}$ , carbon can be obtained by varying the acid-bamboo ratio. The high surface area carbon shows nearly three times higher adsorption capacity for small dye molecule, AB25, than the commercial carbon, F400. For AY117, it has similar capacity as F400. However, the low surface area carbon shows poor adsorption for both dyes. Both surface area and porosity of the carbon have played an important role in the adsorption of the dyes. The high adsorption capacity of the smaller dye molecule has suggested the potential of the high BET surface area microporous activated carbon as good adsorbent for separation of mix-sized colored pollutants.

#### **Acknowledgements**

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**Table 3** Values of parameters for various isotherm models for the adsorption of AB25.

Isotherm Models' Parameter				
<b>Langmuir</b>	$K_L$ (L/g)	$a_L$ (L/mmol)	$Q_m$ (mmol/g)	SSE
HSA1	687.049	403.922	1.701	0.218
HSA2	643.548	341.248	1.886	2.423
LSA	1.336	5.233	0.255	0.026
F400	344.681	498.057	0.692	0.012
<b>Freundlich</b>	$K_f$ (L/mg <sup>1-1/n</sup> /g)	n		SSE
HSA1	1.950	0.088		0.099
HSA2	1.902	0.034		2.452
LSA	0.213	0.293		0.015
F400	0.818	0.164		0.085
<b>Redlich-Peterson</b>	$K_R$ (L/g)	$a_R$ (L/mg <sup>1-1/β</sup> )	β	SSE
HSA1	2112.000	1111.000	0.933	0.071
HSA2	661.000	352.000	1.000	2.423
LSA	210.000	963.000	0.690	0.016
F400	377.170	529.143	0.979	0.010

**Table 4** Values of parameters for various isotherm models for the adsorption of AY117.

Isotherm Models' Parameter				
<b>Langmuir</b>	$K_L$ (L/g)	$a_L$ (L/mmol)	$Q_m$ (mmol/g)	SSE
HSA1	19.266	149.605	0.129	0.013
HSA2	6.788	51.698	0.131	0.003
LSA	0.134	6.047	0.022	0.000
F400	7.520	57.547	0.131	0.002
<b>Freundlich</b>	$K_f$ (L/mg <sup>1-1/n</sup> /g)	n		SSE
HSA1	0.173	0.179		0.003
HSA2	0.173	0.246		0.001
LSA	0.028	0.535		0.000
F400	0.145	0.158		0.003
<b>Redlich-Peterson</b>	$K_R$ (L/g)	$a_R$ (L/mg <sup>1-1/β</sup> )	β	SSE
HSA1	434.7	2,515.4	0.824	0.003
HSA2	94.42	564.1	0.779	0.001
LSA	0.370	13.64	0.589	0.000
F400	6.753	54.70	1.000	0.001

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