Phenol Adsorption on Nitrogen-enriched Activated Carbon Prepared from Bamboo Residues

Ji Zhang, Xiao-Juan Jin,* Jian-Min Gao, and Xiu-Dong Zhang

Nitrogen-enriched activated carbons prepared from bamboo residues were characterized by means of BET, XPS, and elemental analysis. Then adsorption experiments were carried out to study the effects of various physicochemical parameters such as contact time, temperature, pH, and initial concentration. Adsorption equilibrium was achieved within 120 min at a phenol concentration of 250 mg/L. When the pH was 4 and 0.1 g of the carbon absorbent and 100 mL of phenol solution at 250 mg/L were used, the phenol adsorption of the ACs with melamine and urea modifications were 219.09 mg/g and 214.45 mg/g, respectively. Both were greater than the capacity of unmodified AC, which was 163.82 mg/g. The Langmuir isotherm adsorption equation well described the experimental adsorption isotherms. The adsorption kinetics was well explained by pseudo-second-order kinetics rather than the pseudo-first-order. In conclusion, the nitrogen-enriched activated carbon proposed as adsorbents of the phenol wastewater were shown to be effective, which also means that bamboo residues have promise as activated carbon precursors for liquid phase adsorbents for environmental protection.

Keywords: Activated carbon; Nitrogen-enriched; Bamboo residues; Phenol adsorption

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INTRODUCTION

Phenol is a widespread persistent organic pollutant that is commonly present in industrial effluent streams such as those of the petrochemical industries, kraft pulp mills, olive oil production, and other chemical industries. Since phenol is harmful to organisms even at low concentrations and is difficult to be degraded biologically, it has been classified as a priority pollutant in wastewater by the European Union. An environmental concentration limit of 0.02 μgL^{-1} has been suggested for water by the World Health Organization (Sathishkumar *et al.* 2009). Therefore, removal of phenols from industrial effluents is required before release of treated wastewater. For that purpose, various environment-friendly methods have been suggested. Several physicochemical and biological treatment techniques (solvent extraction, ion exchange by resins, *etc*.) already exist today, but the most effective and frequently used procedure for phenol removal is adsorption on activated carbons (Girods *et al.* 2009).

EXPERIMENTAL

Materials

Bamboo residues were collected from wood processing factory in Beijing Forestry University as the carbon source. Potassium carbonate (K_2CO_3), phenol, HCl, melamine, urea, and distilled water were analytical grade and were purchased from Beijing Lanyi Chemical (Beijing, China).

Preparation of activated carbon

The bamboo residues based ACs were prepared using a two-step process. In the carbonization step, samples were cut into 20 cm × 10 cm × 3 cm pieces, and then these pieces were heated up to a carbonization temperature of 500 °C at a heating rate of 100 °C/h, and were held at this temperature for 1 h. The samples were then ground and screened out with sieves. The fraction in the particle diameter ranged from 40 mesh to 60 mesh. The particle samples were dried in a 105 °C oven for 6 h. (Wu *et al.* 2013).

In the activation step, the oven-dried samples were blended with K_2CO_3 at a mass ratio of samples to K_2CO_3 of 1:3 (AC0). At the same time, the oven-dried samples were still blended with melamine at mass ratios of samples to melamine of 3:1, 3:2, and 3:3 (AC1-M, AC2-M, AC3-M), or were blended with urea at the mass ratio of samples/ urea of 3:0.5, 3:1, 3:2, and 3:3 (AC1-U, AC2-U, AC3-U, AC4-U). Namely, 3 g of oven-dried samples were soaked in 50% K_2CO_3 and modifiers solution for 16 h. The soaked samples were dried in 105 °C for 6 h. Then the oven-dried samples were activated at 850 °C for 60 min. The samples were repeatedly washed with hot distilled water until the pH of the solution reached about 6 to 7. Finally, these activated carbons were dried at 105 °C for 6 h. Each sample was replicated with a parallel sample; then the two results were averaged to obtain the reported result.

Methods

Structural and textural characterizations

The surface physical properties of the activated carbons were characterized with a Micromeritics ASAP 2020, using N_2 as the adsorbate at 77 K. The surface area (S_{BET}) was calculated with the BET equation, and the pore volume (V_{T}) was obtained from the adsorption isotherm at $P/P_0 = 0.95$. The micropore area (S_{mic}) and volume (V_{mic}) were obtained using the t-plot method. The mesopore volume (V_{mes}) was the deduction of V_{mic} from V_{T}, and the mesopore area (S_{mes}) was the deduction of S_{mic} from S_{BET} (Altenor *et al.* 2009)

Carbon, hydrogen, and nitrogen contents were determined using a CHNS type Vario El elemental analyser. Surface properties of activated carbons were characterized by X-ray photoelectron spectroscopy (XPS). The XPS spectra were collected by using a Mg Kα X-ray source (1253.6 eV). The C1s peak of the sample was analyzed in the binding energy range of 275 to 295 eV. N1s core level spectra were recorded on a RIBER MAC 2 XPS spectrometer using the Al Ka radiation (12 kV and 25 mA). The accuracy of the binding energy is ±0.1 eV.

Phenol Adsorption Process

In equilibrium experiments, selected amounts of activated carbons and phenol were placed in 250 mL glass-stopper flasks and swirled for 3 h using a water bath controlled in the appropriate temperature. The phenol in the supernatants was determined. At the end of the adsorption process, adsorbent particles were filtered out using 0.45-μm membranes. The residual phenol concentration was measured by UV-vis spectrophotometer at the appropriate optimum UV wavelengths of 270 nm. The amount of adsorption at equilibrium q (mg/g), was obtained from,

$$q = \frac{c_o - c_e}{m_s} \times V \qquad (1)$$

where c_0 and c_e are the initial and equilibrium liquid phase concentrations (mg/L), V is the volume of solution (L), and m_s is the weight of dry activated carbons used (g) (Terzyk *et al.* 2007). Each condition was tested twice, and the replicated results were averaged.

RESULTS AND DISCUSSION

Textural Characterization

Figures 1 and 2 show nitrogen adsorption and desorption isotherms of prepared activated carbons measured at 77 K. These N_2 adsorption isotherms were found to exhibit type I characteristics, indicating their microporous features (Jia *et al.* 2002).

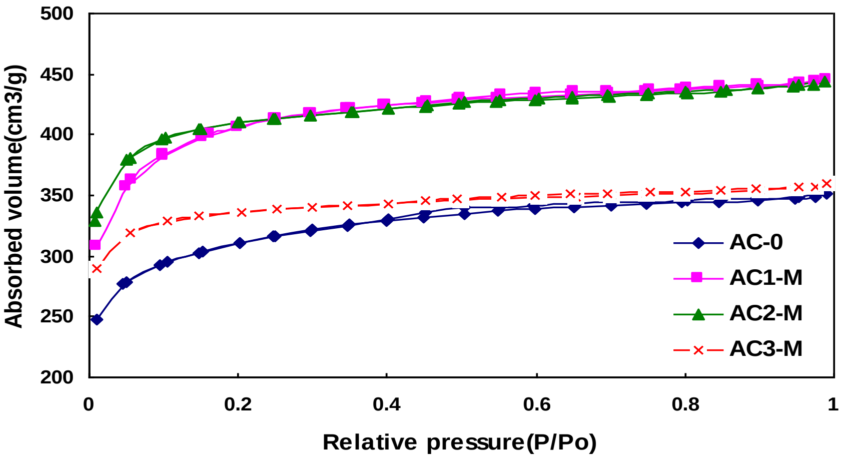

Fig. 1. Nitrogen adsorption–desorption isotherms for activated carbons samples with different melamine ratio

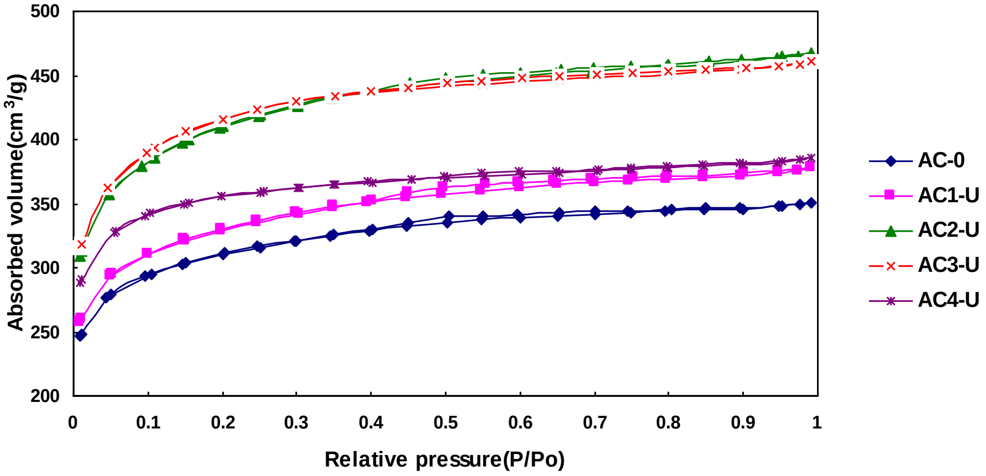

Fig. 2. Nitrogen adsorption–desorption isotherms for prepared activated carbons with different urea ratio

Figures 3 and 4 show the corresponding pore size distribution curves of prepared activated carbons. As for prepared activated carbons, relatively sharp peaks at pore diameter less than 2 nm were evident, which confirms the predominant pore size of the prepared activated carbons was in the microspore range.

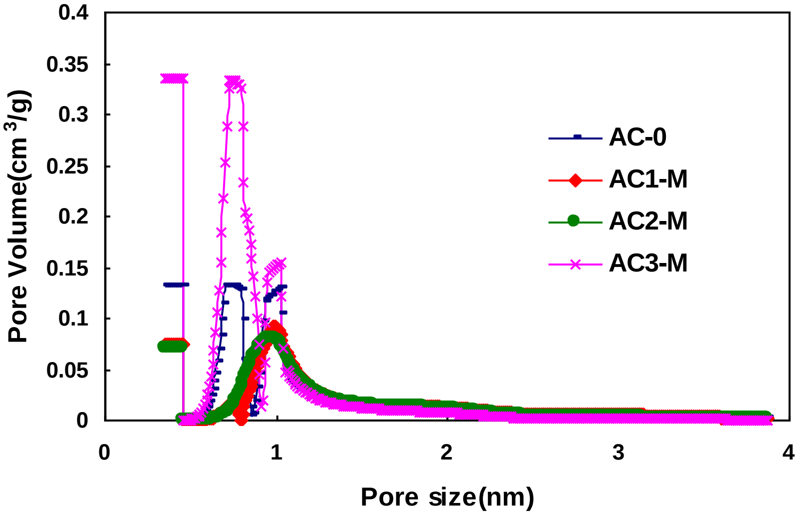

Fig. 3. Pore size distributions of prepared activated carbons with different melamine ratio

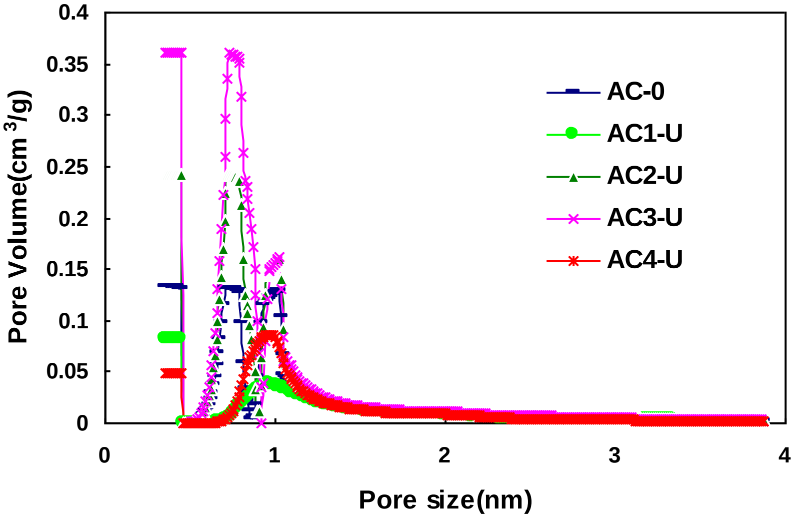

Fig. 4. Pore size distributions of prepared activated carbons with different urea ratio

The textural properties of prepared activated carbons are summarized in Table 1. The BET surface area rose with increasing melamine addition, from 1120 m^2/g (AC-0) to 1511 m^2/g (AC2-M, samples/melamine ratio of 3:2) and with increasing urea addition to 1438 m^2/g (AC3-U, samples/urea ratio of 3:2). Thereafter, the surface area began to decrease after blending with excessive melamine or urea. This is mainly attributed to filling of the pores of the activated carbon with residues resulting from the disintegration of excess modifiers under the high temperature conditions. Liu *et al.* (2010) prepared bamboo-based activated carbon with a microwave-induced activation process using phosphoric acid as the activating agent; the BET surface area in that case was in the range of 970 to 1432 m^2/g.

Table 1. Pore Structure Characteristics of Prepared Activated Carbons

Samples	Sbet （m^2/g）	Smic （m^2/g）	Smec （m^2/g）	Vt （cm^3/g）	Vmic （cm^3/g）	Vmes （cm^3/g）	Wp （nm）
AC-0	1120	1013	38.36	0.543	0.448	0.053	1.939
AC1-M	1397	1318	31.69	0.6889	0.611	0.049	1.971
AC2-M	1511	1452	37.17	0.687	0.619	0.056	1.817
AC3-M	1294	1247	24.61	0.556	0.505	0.039	1.720
AC1-U	1170	1080	45.15	0.584	0.495	0.064	1.996
AC2-U	1415	1316	48.90	0.723	0.623	0.071	2.045
AC3-U	1438	1372	34.03	0.7134	0.640	0.055	1.985
AC4-U	1307	1246	32.75	0.597	0.532	0.048	1.827

Elemental Analysis

Table 2 shows the results of elemental analysis of the prepared activated carbons. With the growth of the compound ratio of melamine or urea, the nitrogen contents of prepared activated carbons also increased. The results show that the content of the incorporation of nitrogen is affected by the modifiers (Stavropoulos *et al.* 2008). Seredych *et al.* (2008) prepared nitrogen-enriched AC treated with melamine and urea at elevated temperatures; the content of N was 2.9%, and the BET surface area was 732 m^2/g.

Table 2. Elemental Composition of Prepared Activated Carbons

Samples	C（%）	N（%）	H（%）	(C/N)
AC-0	81.59	1.491	0.524	54.72
AC1-M	87.97	2.205	0.328	39.90
AC2-M	86.24	3.836	0.458	22.48
AC3-M	86.54	4.717	0.819	18.35
AC1-U	85.93	2.219	0.649	38.72
AC2-U	90.12	2.786	0.553	32.35
AC3-U	87.17	4.686	0.683	18.60
AC4-U	81.81	6.269	1.082	13.05

XPS Study

Figure 5 and Table 3 show results of the XPS surface analysis and the resulting spectral plot for N1s. A complex XPS N1s spectrum was fitted by four components of binding energies of about 398.7, 400.3, 401.4, and 403 eV, which can be attributed to pyridinic (N-6), pyridonic and pyrrolic (N-5), quaternary (N-Q), and pyridine N-oxide (N-X). The dominant nitrogen species in the prepared activated carbons was N-5, yet the pentagonal ring of polypyrrole chemical structure (N-5) are converted to two types of N atoms (N-X and N-6) after mixed with melamine or urea (Hulicova *et al.* 2005).

The amount of pyrollic nitrogen decreased for either melamine-treated or urea-treated samples, but all the prepared activated carbons showed similar distribution of nitrogen atoms. This result indicates that at the same temperature, the different modifiers can only affect the relative quantities of the nitrogen-containing groups, but they could not affect the composition of the N1s spectrum (Lota *et al.* 2010).

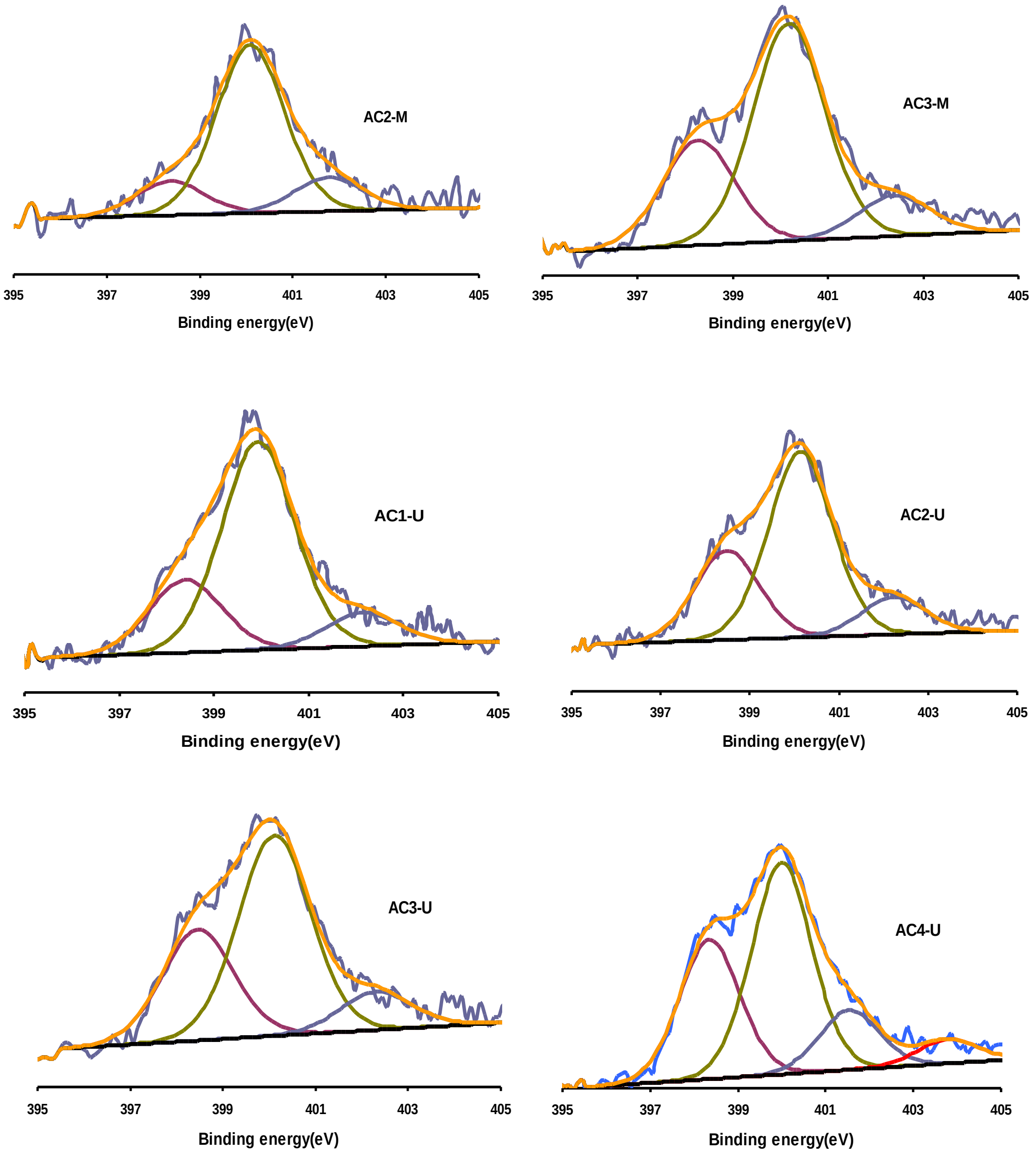

Fig. 5. XPS analysis of prepared activated carbons

Table 3. Distribution of N Species Obtained from the Deconvolution of the N1s Peaks

Sample	N-6, Pyridinic nitrogen (398.7±0.3eV)	N-5, Pyrollic nitrogen,pyridone (400.3±0.3eV)	N-Quaternary nitrogen (401.4±0.4eV)	N-X, Oxidized Nitrogen (402-405eV)
AC-0	14.80	74.17	-	11.02
AC1-M	21.13	68.16	-	10.71
AC2-M	14.44	72.21	-	14.35
AC3-M	28.96	59.81	-	11.22
AC1-U	23.08	66.14	-	10.78
AC2-U	28.66	59.31	-	12.01
AC3-U	31.74	57.36	-	10.9
AC4-U	32.06	48.64	-	19.3

Effect of Contact Time

The results of the experiments performed to determine the optimum contact time for phenol adsorption are summarized in Fig. 6. The 0.1 g activated carbon sample was put in 250 mL flasks containing 100 mL of unbuffered aqueous phenol solution. The initial concentration of phenol was 250 mg/L.

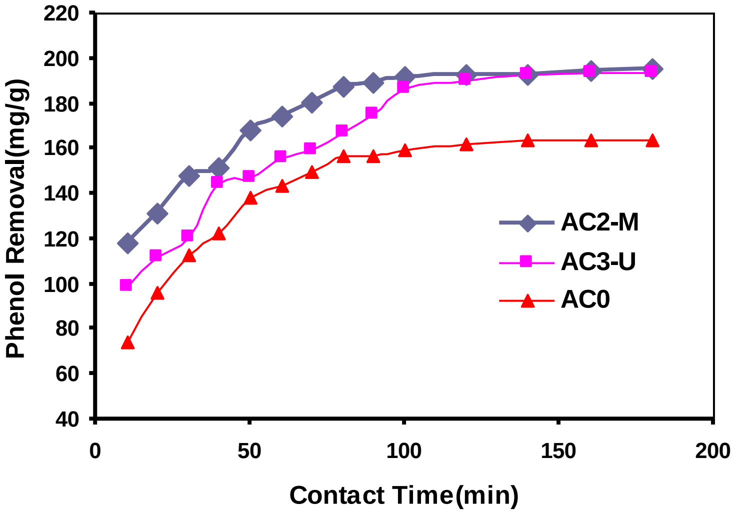

Fig. 6. Effect of contact time on adsorption of phenol (temperature=35 °C; pH=7; carbon dosage= 0.1 g; phenol concentration=250 mg/L)

There was rapid uptake kinetics within 80 min, and adsorption equilibrium was attained within 120 min (Fig. 6). Therefore 120 min contact time was chosen as the optimum equilibrium time for the experimental studies (Castillejos-Lopez *et al.* 2008).

The phenol adsorption capacity of AC0 was determined as 163.8 mg/g. After being imbued with nitrogen, the phenol adsorption capacity of AC2-M and AC3-U rose to 195.6 mg/g and 193.6 mg/g, respectively. This is due to the introduction of nitrogen-containing groups, making the activated carbon more alkaline and hydrophobic. This is conducive to the adsorption of phenol, which is predominantly hydrophobic and faintly acid. In a study reported by Mohanty *et al.* (2006), capacity of activated carbon from *Sterculia alata* nutshell could reach 140 mg/g, and phenol removal achieved 80% in 300 min.

Effect of pH

The effect of pH on the adsorption of phenols on prepared activate carbon was studied by changing the pH of the solution from 4 to 9, and the results are shown in Fig. 7. It can be observed that the removal of phenol decreased with the increase of pH. Maximum adsorption of phenol at all the concentrations took place at pH 4.

The pH dependence of phenol adsorption can largely be related to the type and ionic state of these functional groups and also on the phenol chemistry in solution. Adsorption of phenol above pH 4 suggests that the positively charged $C_6H_5OH^{2+}$ ions bind through electrostatic attraction to negatively charged functional groups on the surface of activated carbon. This is because at pH 4 more functional groups carrying negative charge would be exposed at the carbon surface (Terzyk 2003).

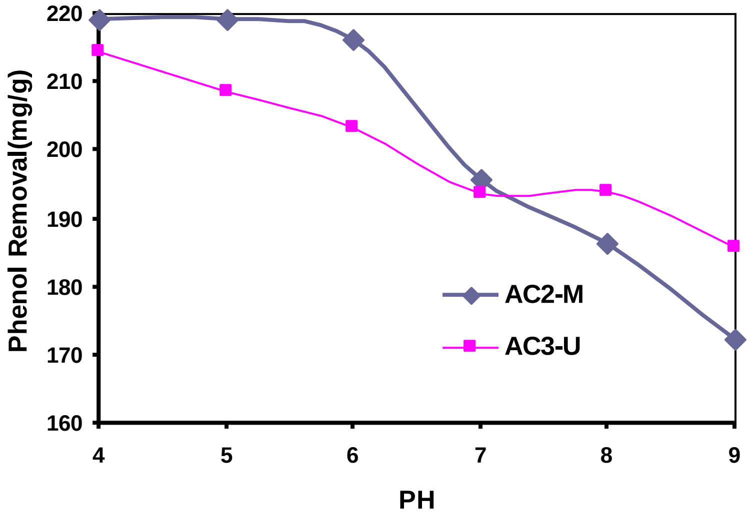

Fig. 7. Effect of pH on adsorption of phenol (temperature=35 °C; carbon dosage= 0.1 g; phenol concentration =250 mg/L)

Effect of Temperature

The adsorption studies were carried out at different temperatures in the range from 20 °C to 45 °C, and results are shown in Fig. 8. It can be observed that the removal of different phenols increased with increasing temperature from its minimum at lower temperature 20 °C to its maximum at temperature of 35 °C, where the maximum removal was observed. This implies that adsorption is an endothermic process. Thus, with an increase in temperature, adsorption capacity increased within the corresponding range (Toth *et al.* 2009). When temperature increased beyond 35 °C, phenol removal decreased sharply. Similar trends were found in research of adsorption of phenol from aqueous solution on activated carbon at 298, 303, and 323 K (Lua and Jia 2008).

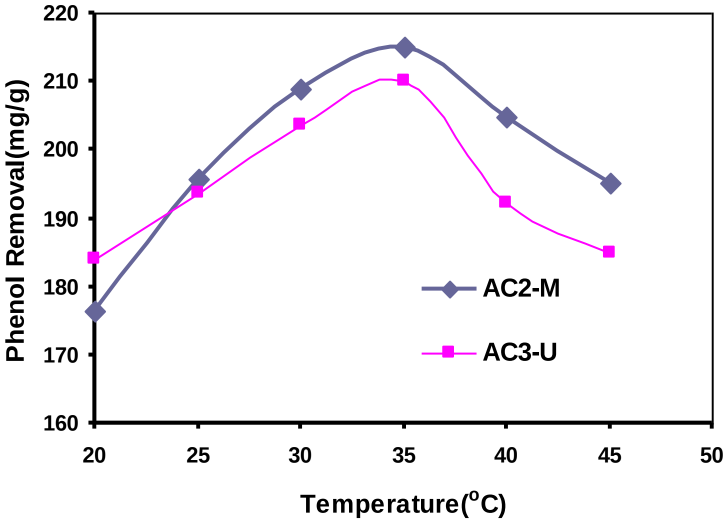

Fig. 8. Effect of temperature on adsorption of phenol (pH=7; carbon dosage= 0.1 g; phenol concentration =250 mg/L)

Effect of Initial Concentration

Figure 9 summarizes the experiments performed to determine the suitable initial concentration for phenol adsorption. For the same amount of adsorbent, the sorption efficiencies decreased with the increase of phenol concentration in solution, while the amount of phenol adsorbed increased as the initial concentration increased. Considering the sorption efficiency and the amount of phenol adsorbed, the 250 mg/L initial concentration was found to be optimal. Similar trends of initial concentration effect were also found from adsorption of phenol on carbonaceous material (brown coal) (Hofman and Pietrzak 2012).

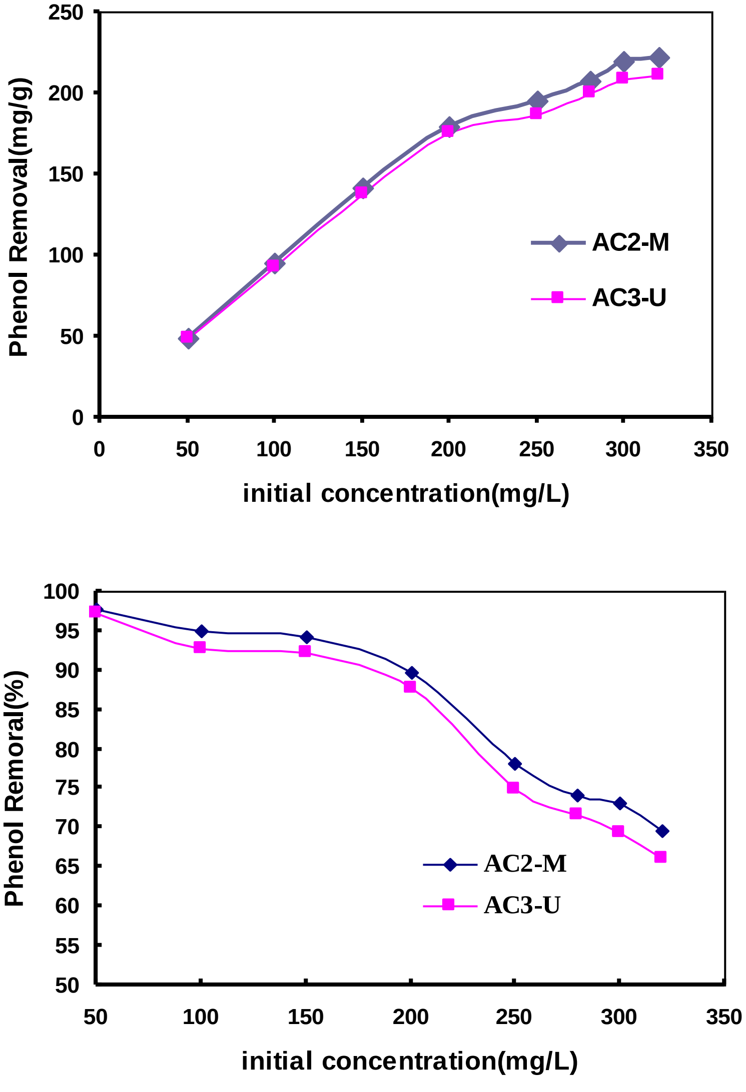

Fig. 9. Effect of initial concentration on adsorption of phenol (temperature=35 °C; pH=7; carbon dosage= 0.1 g)

Adsorption Kinetic Studies

The pseudo-first order model and pseudo-second order model were used to examine the mechanism of adsorption. The pseudo-first-order equation is represented by the following equation,

$$\ln(q_e - q) = \ln q_e - k_1 t \qquad (2)$$

where q (mg/g) is the amount of phenol sorbed at time t (min), q_e (mg/g) is the amount of phenol sorbed at equilibrium, and k_l (1/min) is the first-order adsorption rate constant. The linear plot is shown in Fig. 10.

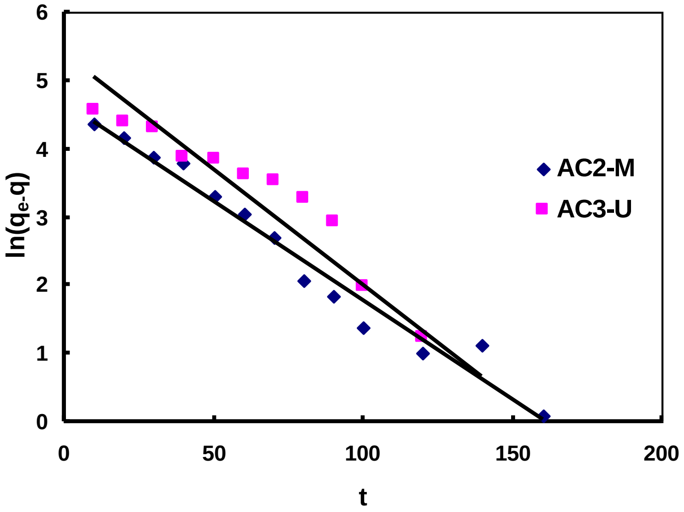

Fig. 10. First-order adsorption rate of phenol on activated carbon

The pseudo-second-order equation based on equilibrium adsorption is expressed as,

$$\frac{t}{q} = \frac{1}{k_2 \times q_e^2} + \frac{t}{q_e} \qquad (3)$$

where k_2 is the rate constant of second-order adsorption. Also, the linear plot is shown in Fig. 11 (Polat *et al.* 2006).

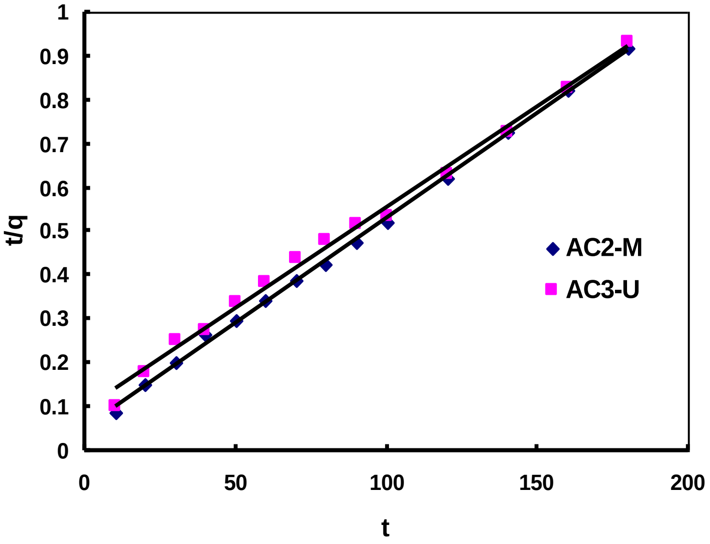

Fig. 11. Second-order adsorption rate of phenol on activated carbon

The kinetic parameters acquired from fitting results are summarized in Table 4. The values of correlation coefficient R^2 for the pseudo-second-order adsorption model is relatively high, and the adsorption capacities calculated by the model are also close to those determined by experiments. It is concluded that adsorption of phenol on the activated carbon follows the pseudo-second-order model. As expected, the overall rate of the adsorption process was controlled by chemisorption (Tancredi *et al.* 2004).

Table 4. Parameters of Different Adsorption Rate of Phenol on Activated Carbon

Samples	Pseudo-first-order equation			Pseudo-second-order equation		
	q_e (mg/g)	k_1 (min^{-1})	R^2	q_e (mg/g)	k_2 (g.mg.min^{-1})	R^2
AC2-M	109.191	0.0291	0.9698	208.333	0.000433	0.9987
AC3-U	254.561	0.0339	0.9048	217.391	0.000226	0.995

Adsorption Isotherms

Both the Langmuir and the Freundlich models were employed to fit the experimental adsorption data. The Langmuir isotherm can be represented by the following equation,

$$\frac{C_e}{q_e} = \frac{1}{q_m \times b} + \frac{C_e}{q_m} \tag{4}$$

where q_m is the amount of adsorption corresponding to complete monolayer coverage and b is the Langmuir constant related to the energy or net enthalpy of adsorption. Linear plots of C_e/q_e *vs.* C_e are shown in Fig. 12.

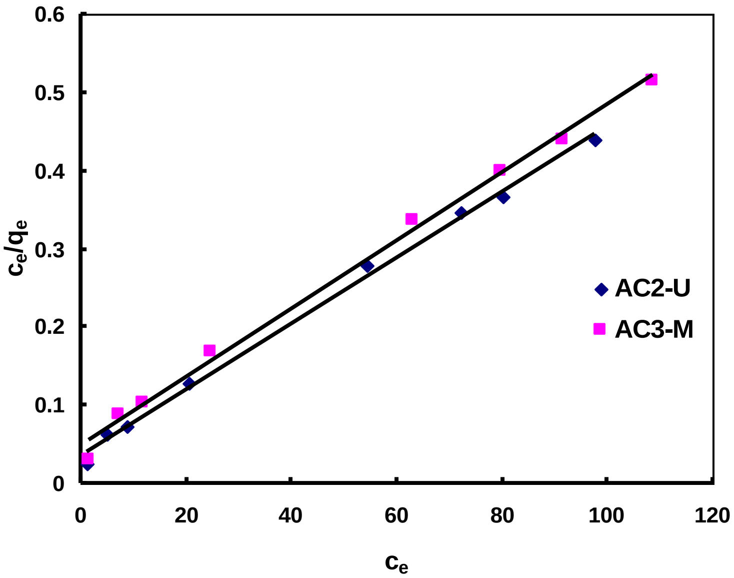

Fig. 12. Linear plot from Langmuir equation of phenol adsorption on activated carbon

The Freundlich isotherm model has the following form,

$$q_e = K_f \times C_e^{\frac{1}{n}} \tag{5}$$

where K_f and n are Freundlich constants related to adsorption capacity and adsorption intensity; n represents the degree of adsorption dependence at equilibrium concentration (Haydar *et al.* 2003). The corresponding plot is shown in Fig. 13.

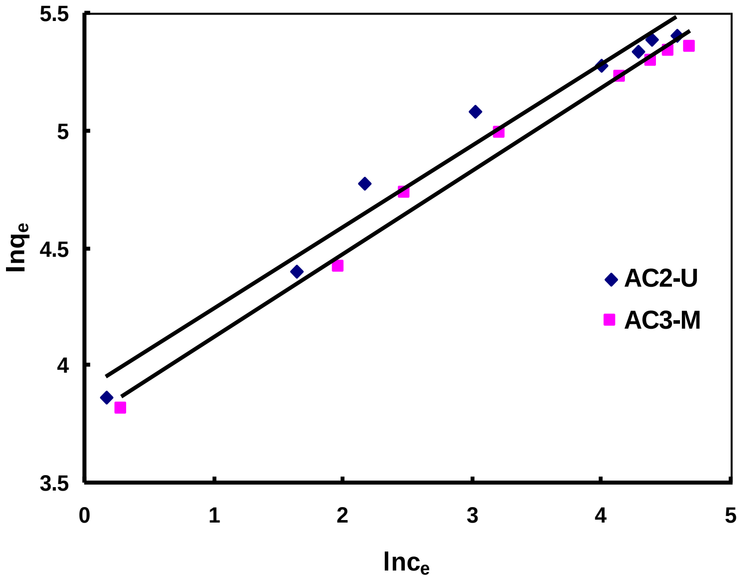

Fig. 13. Linear plot from Freundlich equation of phenol adsorption on activated carbon

The Langmuir and Freundlich constants obtained by fitting the adsorption equilibrium data are listed in Table 5. It is clear that the Freundlich adsorption isotherm gave a best fit for adsorption of phenols on to activated carbon for the entire range of concentrations. Because the value of 1/n was less than 1, it indicated a favorable adsorption. The fact that the Langmuir isotherm model gave a somewhat more accurate estimation of maximum adsorption capacity implies that ACS seemed to have high monolayer adsorption capacities. Similar trends were found by Fierro *et al.* (2008), who showed that the Langmuir model gave a better fit in adsorption of phenol.

Table 5. Parameters of Different Isotherms of Adsorption of Phenol on Activated Carbon

Samples	Langmuir			Freundlich		
	q_m (mg/g)	*b*	R^2	*n*	K_f	R^2
AC2-M	238.0952	0.117318	0.9963	2.884338	49.05785	0.9739
AC3-U	227.2727	0.090722	0.9952	2.836879	43.25446	0.9867

CONCLUSIONS

The present investigation showed that the activated carbon prepared from bamboo waste by chemical activation with K_2CO_3 and modified with melamine or urea yielded active carbons of very well developed surface area and pore structure, with the dominant contribution of micropores. The maximum BET surface area of 1511 m^2/g was obtained for condition AC2-M. Moreover, the results indicated that nitrogen-rich activated carbon made from bamboo waste had significantly higher phenol adsorption capacity compared with other non-nitrogen-containing activated carbons. The phenol adsorption of ACs made from bamboo residues increased from 163.82 mg/g to 219.09 mg/g after the treatment to introduce nitrogen groups. The introduction of surface functional groups of ACs depended upon the different modifiers. The maximum phenol adsorption capacity was 219.09 mg/g for AC2-M with an N content of 3.84% modified with melamine and 214.45 mg/g for AC3-U with an N content of 4.69% modified with urea, when the adsorption was carried out at a temperature of 35 °C and a pH of 4, with the carbon dosage of 0.1 g, and 250 mg/L initial phenol concentration. The adsorption process followed the Langmuir isotherm model well. The adsorption kinetics followed a pseudo-second order model. This study still suggested that the activated carbon prepared from bamboo waste by chemical activation with K_2CO_3 and modified with melamine or urea can be a promising for treatment of phenol wastewater, and also meant that the bamboo residues considered are promising activated carbon precursors for adsorbents for environmental protection.

ACKNOWLEDGMENTS

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