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Adsorption of phenol from aqueous solutions using activated carbon prepared from crofton weed

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ABSTRACT

Activated carbon derived from crofton weed (ACCW) was evaluated for its ability to remove phenol from an aqueous solution. The kinetics study, adsorption isotherm, pH effect, and thermodynamic study were examined in batch experiments. Adsorption data for phenol uptake by ACCW were analyzed according to Langmuir, Freundlich and Temkin adsorption models. Thermodynamic parameters for the adsorption system were determined at 293 K, 313 K and 333 K ($\Delta H^\circ = -22.92$ kJ mol^{-1}; $\Delta G^\circ = -2.87$ to -5.28 kJ mol^{-1} and $\Delta S^\circ = -60.20$ J/K·mol). ΔG° values obtained were negative, indicated that the adsorption of the phenol on the surface of ACCW was a spontaneous adsorption process. The kinetics process can be described by a pseudo-second-order rate equation very well. These results show that the ACCW could be considered as a potential adsorbent for phenol in aqueous solutions.

Keywords: Phenol; Adsorption; Crofton weed; Isotherms; Kinetics; Activated carbon

1. Introduction

Phenols are generally considered among the most hazardous organic pollutants in wastewater and they are highly toxic even at low concentrations. In addition, the presence of phenol in natural waters can lead to the formation of other toxic substituted compounds during disinfection and oxidation processes [1]. Excessive exposure to phenol may cause health effects on brain, digestive system, eyes, heart, kidney, liver, lungs, skin and it can cause genetic damage. Therefore, the removal of phenols in waste effluents is of great environmental importance because they are widely used in industries such as paint, pesticide, coal conversion, polymeric resin, petroleum and petrochemicals industries [2]. The pretreatment methods such as chemical oxidation, ion exchange, solvent extraction and adsorption have been used for this pollution control [3,4]. Among all, the adsorption process has been effectively used for the removal of organic as well as inorganic compounds from wastewater because of its simplicity and safety [5].

A number of adsorbents such as titanium oxide, redmud, rubber seed coat, clay and natural zeolite, etc. have been used for phenol removal [2,6–9]. Adsorption by activated carbon (AC) is the best and most frequently used method, particularly in treating low concentrations of wastewater streams and in meeting stringent treatment levels. However, the use of effective commercial activated carbons based on relatively expensive starting materials is unjustified for most pollution control applications. It is, therefore, very important to examine the feasibility of using cheaper raw materials to prepare activated carbon. Numerous successful attempts have been made to develop activated carbons from various

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range of agricultural solid waste such as bamboo, rice husk, rubber-wood sawdust, oil palm shell, coir pith and Coconut shell [10–15].

Crofton weed (Eupatorium adenophorum spreng) is a tufty, semi-shrubby, perennial herbaceous plant of about 1–2 m in height. Originating from Mexico, Crofton weed has successfully invaded many regions on the globe ranging from Hawaii and California (USA) to New South Wales (Australia), New Zealand, northern India, northern Thailand, Vietnam, Burma and South Africa [16–18]. It invaded China in the 1940s from Burma and is now widespread in southwest China, damaging native ecosystems and causing great economic losses. Many farmlands, pasture fields and forests have been destroyed by the weed. Much attention has been paid to management of this weed and several methods have been developed to control it, including manual, chemical and biological control, however, no obvious progress is made. The weed is still spreading from southwest to northeast at a rate of 10–60 km per year in China [19]. Compared with simple control processes, utilization of the weed gives more benefits. Crofton weed chemical composition is akin to hard wood which mainly composed by lignin and cellulose. Hence, in the conversion of crofton weed onto activated carbon, particularly, the stem is most desirable.

Among the numerous dehydrating agents, zinc chloride in particular is the widely used chemical agent in the preparation of activated carbon. Chemical activation by $ZnCl_2$ improves the pore development in the carbon structure and because of the effect of chemicals the yields of carbon are usually high [20]. Therefore, in this work it is reported the results obtained on the preparation of activated carbons from crofton weed with zinc chloride activation and their ability to remove phenol from wastewater. The influence of several operating parameters for adsorption of phenol such as contact time, temperature, pH and adsorbent dose, etc. were investigated in batch mode. Kinetic models were used to identify the possible mechanisms of such adsorption process. The Langmuir, Freundlich and Temkin models were used to analyze the adsorption equilibrium.

2. Materials and methods

2.1. Instrumentations

Four equipments were used in this research: A UV/visible spectrophotometer (Shimadzu, UV-2401, Japan) at $\lambda = 270$ nm was used to determine the phenol concentrations. A pH meter (Cany, PHSJ-4A, China) was used for pH measurement.

A mechanical shaker (Jintan, SHZ-82, China) was used for agitating the samples. A microwave muffle furnace (Preekem, China) was used to heat precursor of activated carbon.

2.2. Materials and preparation of activated carbon

All chemicals and reagents used in this work are in analytical grade. In all experiments, deionized water was used for preparation, dilution and analytical purposes of solutions. The stock solution (1000 mg/l) was prepared by dissolving 1.0 g of phenol in 1000 ml of deionized water. All working solutions with different concentrations were obtained by successive dilution. The prepared phenol solutions were stored in brown color glass reservoirs to prevent photo-oxidation.

The crofton weed (Fig. 1) was collected from campus of Kunming Metallurgy College, Yunnan, China and washed with deionized water three to four times for removing dirt and dust particles. The washed weed stems were cut into 10–20 mm pieces. The stems were dried at oven at 105°C for 1 day. It was grounded and sieved to 20–50 mesh size. The powder was immersed in $ZnCl_2$ solution containing 40 wt.% $ZnCl_2$ for 18 h. Then the slurry subjected to vacuum drying at 500°C for 10 min in microwave muffle furnace. The products were washed sequentially with 0.5 mol HCl, deionized water to remove residual $ZnCl_2$ and mineral matters, and then dried at 110°C. The carbon was grounded and screened through different mesh size and stored in a closed bottle to use in adsorption studies.

2.3. Effect of contact time

The contact time of adsorbent and adsorbate is of great importance in adsorption since contact time depends on the nature of the system used. Adsorption experiments for phenol on ACCW were carried out as follows: to each of 0.1 g the ACCW sample, 50 ml of solution containing 20 mg/l of phenol was added. The samples were shaken at room temperature for periods ranging from 5 min to 1.5 h, and then centrifuged and

Fig. 1. Photo of crofton weed.

5 ml portions of liquid phases were measured. The agitation speed of the shaker was fixed at 150 rpm for all batch experiments.

2.4. Effect of pH

The pH of the aqueous solution is a significant controlling factor in adsorption mechanism. In order to optimize the pH for maximum removal efficiency, the effect of pH on the removal of phenol is investigated by varying the pH values from 2 to 12 at a temperature of $25\pm1^\circ$C. The initial phenol concentration was 20 mg/l and the adsorbent dose was kept at 0.1 g in 50 ml solution. The contact time has been fixed at 1 h for all experiments.

2.5. Effect of amount of adsorbent

Under optimum conditions of shaking time and pH, the effect of adsorbent dosage on the removal of phenol at $C_0=20$ mg/l was also studied by shaking 50 ml of solution with 0.05–0.5 g of adsorbent.

2.6. The adsorption isotherms

To investigate the sorption isotherms, three equilibrium models, Langmuir, Freundlich and Temkin isotherm were applied. The Langmuir isotherm is often used to describe sorption of a solute from a liquid solution [21]. The linear representation of this model for single-component sorption is:

$$\frac{C_e}{q_e} = \frac{C_e}{Q_0} + \frac{1}{Q_0 b} \tag{1}$$

where C_e is the equilibrium liquid phase concentration (mg/l), q_e is the amount of sorbent adsorbed per unit weight (mg/g), Q_0 and b are the Langmuir constants related to the sorption capacity (mg/g) and the equilibrium constant (l/g), respectively. Langmuir equation is probably the most widely applied model for isotherm adsorption. It considers that the adsorption energy of each molecule is the same, independently of the surface of material, the adsorption takes place only on some sites and there are no interactions between the molecules.

The Freundlich isotherm [22] provides an empirical relationship between the sorption capacity and the equilibrium concentration of the adsorbent. Linear form of this model is:

$$\log q_e = \log K + \frac{1}{n}\log C_e \tag{2}$$

where q_e is the amount of phenol adsorbed at equilibrium (mg/g), C_e is the equilibrium concentration of phenol in solution (mg/l), and K and $1/n$ are Freundlich constants which correspond to adsorption capacity and adsorption intensity, respectively.

The Temkin isotherm assumes that the fall in the heat of adsorption is linear rather than logarithmic, as implied in the Freundlich equation. The Temkin isotherm [23]:

$$q_e = A + B\mathrm{In}C_e \tag{3}$$

where A and B are Temkin constants.

A further analysis of the Langmuir equation can be made on the basis of a dimensionless equilibrium parameter, R_L [24], also known as the separation factor, given by Eq. (4):

$$R_L = \frac{1}{1+bC_0} \tag{4}$$

where b (l/mg) is the Langmuir constant and C_0 (mg/l) is the initial highest concentration of phenol. The value of R_L lies between 0 and 1 for a favorable adsorption, while $R_L>1$ represents an unfavorable adsorption, and $R_L=1$ represents the linear adsorption, while the adsorption operation is irreversible if $R_L=0$.

2.7. Thermodynamic studies

The free energy change (ΔG°), enthalpy change (ΔH°) and entropy change (ΔS°) were evaluated using the following equations [25,26]:

$$K_C = \frac{C_{Ae}}{C_e} \tag{5}$$

$$\mathrm{In}K_C = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \tag{6}$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{7}$$

where K_c is the equilibrium constant and C_{Ae} and C_e (both in mg/l) are the equilibrium concentrations for solute on the sorbent and in the solution, respectively. R is the universal gas constant (8.314 J/K·mol) and T (K) is temperature.

2.8. Adsorption kinetics

Adsorption kinetics is important from the point of view that it controls the efficiency of the process and the models correlate the adsorbate uptake rate with its bulk concentration. Experiments were also performed in order to understand the kinetics of phenol removal by ACCW. In order to analyze the sorption rate, the kinetic data were modeled using Lagergren pseudo-first-order, pseudo-second order and Elovich kinetic equations [27–31].

Lagergren first-order:

$$\log(q_e - q_1) = \log(q_e) - \frac{k_1}{2.303}t \tag{8}$$

The pseudo second-order equation:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \tag{9}$$

For the boundary conditions $t=0$ to $t=t$ and $q_t=0$ to $q_t=q_t$, the integrated form of equation is

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + k_2 t \tag{10}$$

which can also be rearranged to obtain a linear form:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{11}$$

where q_t and q_e are the amounts of phenol adsorbed (mg/g) at time t and at equilibrium, respectively, and k_1 (min^{-1}) and k_2 (mg/g·min) are the rate constants of pseudo-first-order and pseudo-second-order adsorptions, respectively.

Elovich equation:

$$\frac{dq_t}{dt} = \alpha \exp(-\beta q_t) \tag{12}$$

where α (mg/g·min) is the initial adsorption rate and β (g/mg^1) is the desorption constant related to the extent of the surface coverage and activation energy for chemisorptions. Eq. (12) is simplified by assuming $\alpha\beta > t$ and by applying the boundary conditions $q_t=0$ at $t=0$ and $q_t=q_t$ at $t=t$, as given by Eq. (13):

$$q_t = \frac{1}{\beta}\mathrm{In}(\alpha\beta) + \frac{1}{\beta}\mathrm{In}t \tag{13}$$

3. Results and discussion

3.1. Effect of contact time

The adsorption processes as a function of time to determine the point of equilibrium were studied from the adsorption experiments of phenol onto the ACCW. All experiments were run twice and a good reproducibility of the procedures was obtained. The results are shown in Fig. 2, where it is clear that adsorption of phenol into the ACCW is rather quick and after 1 h the complete adsorption equilibrium between the two phases is obtained. The amount of phenol removed reached a maximum of 90.87%. After it reached the equilibrium, there was no a significance changes in phenol concentration in the solution by placing adsorbent samples in contact with phenol solutions for 1 days of shaking time (data not shown). This behavior showed that adsorption of phenol occurred in a single step. The maximum adsorption capacity was obtained after 1 h.

3.2. Effect of pH

The surface charge of the adsorbent and the ionization degree of the adsorbate are strongly affected by the pH of the aqueous solutions, hence the uptake of phenol by the adsorbent depends on the solution pH. The initial pH of phenol solutions was adjusted from 2 to 12 with HCl or NH_4OH solutions. The pH stability was verified by periodic measurement. The results obtained are shown in Fig. 3. The phenol removal was maximum and unaffected when the initial pH of the phenol solution was in the range of 2–6. A similar trend of pH effect was observed for the adsorption of phenol on activated carbon-commercial grade and laboratory grade [32]. Phenol as a weak acid compound with $pK_a \approx 9.89$ is dissociated at $pH > pK_a$ [33]. Therefore, the adsorption decrease at high pH values due to ionization of adsorbate molecules. The reason could be also due to the electrostatic repulsions between the negative surface charge and the phenolate–phenolate anions in solution [34]. While at acidic pH, the percentage removal was higher because phenol was undissociated and the dispersion interaction predominated

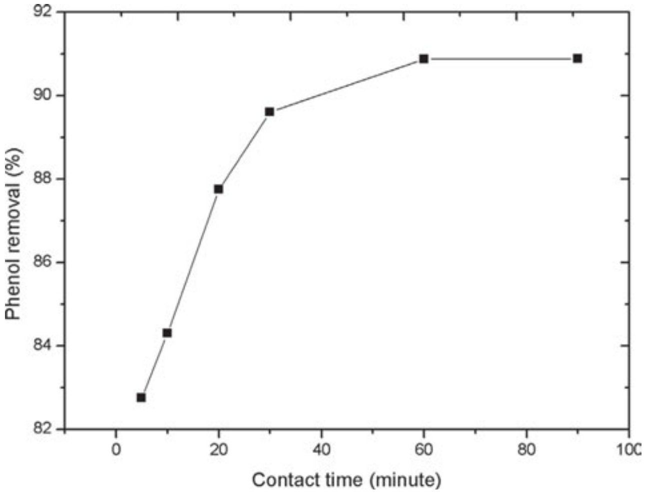

Fig. 2. Adsorption of phenol on ACCW as a function of contact time.

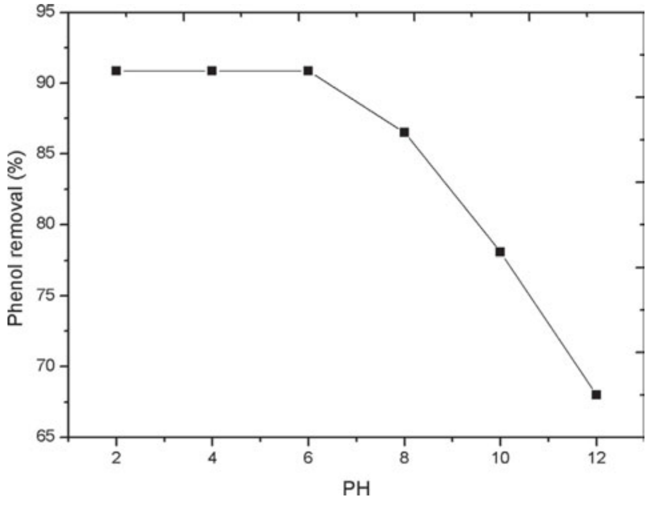

Fig. 3. Influence of initial pH on phenol adsorption.

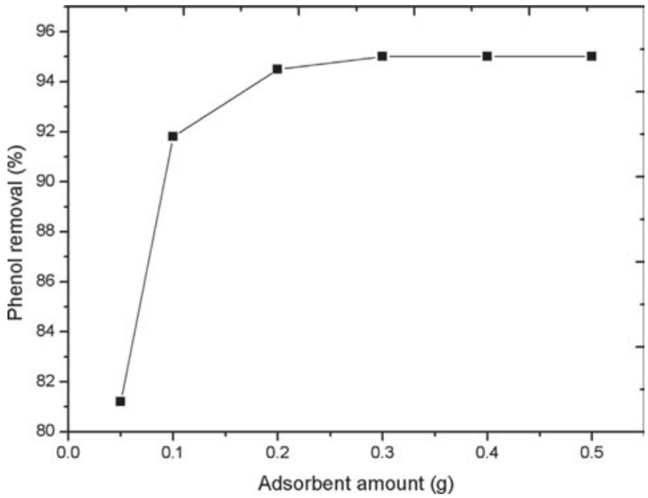

Fig. 4. Dependence of phenol adsorption on the amount of adsorbent.

3.3. *Effect of the amount of adsorbent*

The results from the dependence of phenol adsorption by variation of different amount of the ACCW in the system was carried out at pH 6.0 and equilibration time of 1 h for the adsorption process and they are presented in Fig. 4. The removal of phenol was found to increase with an increase in adsorbent dosage from 0.05 to 0.5 g. Phenol removal almost remains unchanged after adsorbent dosage 0.3 g. Increase in adsorption with increase in adsorbent dosage attributed to the availability of larger surface area and more adsorption sites. At very low adsorbent concentration, the absorbent surface becomes saturated with phenol and the residual phenol concentration in the solution is large. With an increase in adsorbent dosage, the phenol removal increases until a certain value is reached; afterwards, the removal efficiency is maintained constant even if the ACCW is added. So, 0.3 g of adsorbent is enough for the quantitative removal of phenol from waste water.

3.4. *The adsorption isotherm*

Isotherm parameters for the Langmuir, Freundlich and Temkin models for the ACCW are reported in Table 1. The correlation coefficients (R^2) values were higher for Langmuir isotherm than that of the Freundlich and Temkin isotherms. This reinforces the fact that Langmuir isotherm is useful to explain the adsorption from the solution on the current adsorbent when it follows the monolayer mode, rather than the multilayer mode. A basic assumption of the Langmuir theory is that the sorption takes place at specific homogenous sites on the adsorbent. When a site is occupied by an adsorbate, no further sorption can take place at the site. Q_0 and b were determined from the slope and intercept of the plot to be 33.94 (mg/g) and 0.23 (l/mg), respectively. This indicates the good sorbing capacity of the ACCW. The value of R_L (0.179) in the present investigation has been found $0 < R_L < 1$. Hence the sorption process was very favorable and the adsorbent employed exhibited a good potential for the removal of phenol from aqueous solution [35].

3.5. *Thermodynamic parameters*

The values of $\Delta H°$ and $\Delta S°$ values are given in Table 2. The Gibbs free energy change ($\Delta G°$) values were found to be negative at different temperatures, which indicated

Table 1
Parameters of the Langmuir, Freundlich and Temkin isotherm models

T (K)	Langmuir			Freundlich			Temkin		
	Q_0 (mg/g)	b (l/mg)	R^2	K	n	R^2	A	B	R^2
293	33.94	0.23	0.990	5.84	1.48	0.975	7.51	6.26	0.940

Table 2
Thermodynamic parameters for adsorption of phenol on the ACCW

Temperature (K)	$\Delta G°$ (kJ/mol)	$\Delta H°$ (kJ/mol)	$\Delta S°$ (J/mol·K)
293	–5.28	–22.92	–60.20
313	–4.08		
333	–2.87		

Table 3
Kinetic parameters for pseudo first order, pseudo second order and Elovich kinetic models

C_0 (mg/l)	Pseudo-First Order			Pseudo-Second Order			Elovich model			q_e (mg/g) (experimental)
	K_1	q_{e1}	R^2	K_2	q_{e2}	R^2	α	β	R^2	
20	0.0590	1.22	0.998	0.130	9.22	0.999	1×10^{11}	3.12	0.952	9.14
50	0.0561	1.23	0.943	0.116	21.38	1.000	6×10^{16}	2.04	0.843	21.29
100	0.0401	2.03	0.959	0.069	36.68	0.999	1×10^{23}	1.59	0.952	36.60

the spontaneous nature of the process. In addition, more negative value with the decrease of temperature shows that the amount adsorbed at equilibrium must increase with decreasing temperature. The enthalpy change ($\Delta H°$) was −22.92 kJ/mol, the negative value of $\Delta H°$ suggest the exothermic nature of adsorption of phenol on the ACCW. The entropy change ($\Delta S°$) was −66.20 J/mol·K, the negative value of $\Delta S°$ show the decreaseing randomness at the solid/solution interface. As the free energy changes are negative and accompanied by negative entropy changes, the adsorption reactions are spontaneous with a high affinity.

The enthalpy and entropy values can give some idea about the mechanism of bonding. Generally, the change of free energy for physical adsorption is smaller than that of chemisorption. The change in adsorption enthalpy for physisorption is between 20 and 40 kJ/mol, but chemisorption is in the range of 80–400 kJ/mol. The values of the change in enthalpy indicated that each adsorption process is physical in nature.

3.6. Adsorption kinetics

The values of pseudo-first-order and pseudo-second-order equation parameters together with correlation coefficients are given in Table 3. The correlation coefficients for the pseudo-first-order equation obtained at all the studied concentrations were low.

The correlation coefficients (R^2) for the pseudo-second-order equation were 0.999 for all concentrations. The calculated q_{e2}, values also agree very well with the experimental data. This strongly suggests that the sorption of phenol onto the ACCW is most appropriately represented by a pseudo second-order rate process.

The correlation coefficients for Elovich equation are obtained in the range of 0.843–0.952 which is found to be less than the values calculated using pseudo first-order and second-order kinetic model as shown in Table 3.

3.7. Comparison of ACCW with other adsorbents

The value of Q_0 is of importance to identify which sorbent shows the highest adsorption capacity and is useful in scale-up considerations [36]. The direct comparison of adsorbent capacity of ACCW with other sorbents is difficult due to the varying experimental conditions employed in those studies; however, the ACCW in this study possesses reasonable adsorption capacity in comparison with other sorbents (Table 4).

Table 4
The adsorptive capacities of various adsorbents for phenol

Adsorbent	Q_0 (mg/g)	Reference
Commercial activated carbon	49.72	[37]
Activated carbon-commercial grade	30.2187	[38]
Activated carbon-laboratory grade	24.6458	[38]
Activated carbons prepared from Tectona grandis sawdust	13.45	[39]
Rice husk char	7.9	[40]
Activated carbon prepared from palm seed coat	18.3	[3]
ACCW	33.94	This work

4. Conclusions

In this study, activated carbon has been prepared from crofton weed with zinc chloride activation. The feasibility of using activated carbon developed with crofton weed as treated adsorbent for the removal of phenol from aqueous solutions was successfully carried out. The results indicated that adsorption capacity of the adsorbent was considerably affected by initial pH, temperature and the amount of adsorbent. The optimum pH value was found to be 6.0. The results showed that phenol adsorption decreased with temperatures up to 60°C. The ACCW showed nearly 95% adsorptive removal of phenol under optimized conditions of dosage 0.3 g/50 ml for aqueous solutions containing 20 mg/l phenol in 1 h. The equilibrium, kinetic and thermodynamic parameters for the adsorption of phenol onto ACCW was also determined. The negative values of $\Delta G°$ confirm the spontaneous nature adsorption process. The negative value of $\Delta S°$ shows the decreased randomness at the solid–solution interface during adsorption and the negative value of $\Delta H°$ indicates that adsorption process is exothermic. The Langmuir and Freundlich adsorption equations were used to express the adsorption phenomenon of the phenol. The equilibrium data were well described by the Langmuir model. Value of Q_0 was obtained as 33.94 mg/g at 20°C. The pseudo-first order and pseudo-second order kinetic models were used to analyze data obtained for phenol adsorption onto ACCW. The results indicated that the pseudo-second order equation provided the better correlation for the adsorption data. It can be concluded that the activated carbon from crofton weed is an efficient adsorbent for the removal of phenol from dilute aqueous solutions.

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