Adsorption Studies on the Removal of an Endocrine-Disrupting Compound (Bisphenol A) using Activated Carbon from Rice Straw Agricultural Waste

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In this study, we used a two-stage process and potassium hydroxide (KOH) to transform waste biomass (rice straw) into activated carbon and then evaluated the adsorption capacity of the waste for removing bisphenol A (BPA) from an aqueous solution. Activated carbon removed BPA rapidly and effectively because of its high surface area and an adsorptive capacity (181.19 mg/g) that is significantly higher than many other adsorbents that have been developed in the recent past. The native and BPA-loaded adsorbents were characterized by scanning electron microscopy, elemental analysis, and Fourier transform infrared. Different adsorption parameters, such as the initial BPA concentration, contact time, temperature, and pH for BPA adsorption, were studied using a batch system. Equilibrium adsorption isotherms (Langmuir, Freundlich, and Temkin) and kinetics were investigated. The Langmuir model fits the experimental results well, compared to the Freundlich and Temkin models, and a pseudo-second-order equation was successfully used to describe the results of the kinetic studies. This study demonstrates that activated carbon produced from rice straw can be very effective in the adsorption of BPA from aqueous solution.

Keywords activated carbon; adsorption isotherm; Bisphenol A; kinetics; Rice straw

INTRODUCTION

Endocrine-disrupting chemicals (EDCs) in bodies of water have become a social issue, as these contaminants have been frequently detected in different water sources (1–2). Bisphenol A (BPA), which is a suspected EDC, has been widely used in the production of polycarbonate and epoxy resins as well as a stabilizer for polyvinyl chloride

resins. The annual production capacity for BPA in Taiwan has exceeded 390,000 metric tons in response to the increasing demand for compact discs and construction materials (3). Studies have shown that BPA can be eluted from these products under normal conditions of use (4). Accordingly, BPA has reportedly been detected in waste landfill leachates, rivers, seas, and soil (5). Recent findings have also shown that BPA can produce estrogenic activity at a low dose of 0.23 pg/mL in culture medium (6). Therefore, there is an urgent need to develop effective technology for the rapid removal of BPA from aqueous solutions.

The conventional methods of removing EDCs from wastewater include membrane filtration (7), biological processes (8), oxidation processes (9–11), and adsorption (12–13). Among the numerous clean-up techniques available, the adsorption technique with activated carbon is the most eco-friendly and is widely used for the removal of EDCs. However, activated carbon is an expensive adsorbent. Recently, various low-cost adsorbents derived from agricultural waste or natural materials have been investigated for their abilities to remove pollutants from aqueous solutions. These adsorbents include sunflower oil cake (14), sugar beet bagasse (15), bamboo (16–17), coconut shell (9, 18), fertilizer industry waste (19), flax shive and cotton gin waste (20).

Rice is a widely grown crop in Asia and, in open fields, burning rice straw is a common practice in regions where there is only a short period of time to prepare the fields for the next crop. However, it has been observed that open-field burning of crop residues is an uncontrolled combustion process that releases pollutants into the atmosphere. These air pollutants have significant toxicological properties and are potential carcinogens (21). In this study, rice straw, an agricultural waste that is available in large quantities in Asia, was utilized as precursor for production

an activated carbon adsorbent for removing BPA from aqueous solutions through adsorption. We used a two-stage process to transform rice straw into activated carbon with a high surface area. The adsorption capacity of the rice straw carbonaceous adsorbents has not previously been tested for EDC removal from water. A batch adsorption process was used to evaluate the maximum adsorption capacity of activated carbon produced from rice straw. The effects of initial BPA concentration, contact time, temperature and pH on BPA adsorption were studied. Adsorption isotherms and kinetic parameters were also calculated. This method has the potential to not only solve the air pollution issue but to also promote the efficient removal of BPA from aqueous solution.

MATERIALS AND METHODS
Raw Materials

Straw from 5-month-old japonica rice plants (*Oryza sativa* L.), variety Tainung 67, were obtained from an experimental farm (25° 02′ 32.79″N, 121° 36′ 47.40″E; elevation: 18 m) located on the Academia Sinica campus in Taipei, Taiwan. The rice plants were transplanted to the field during the first week of March 2009 and began to flower in the first week of June. The straw (leaves and stems) was harvested after the seeds in late July. The straw was washed and dried in the sun before use.

Activated Carbon Preparation

A modified method for the preparation of activated carbon was referenced from Oh and Park (22). As shown in Fig. 1, we placed the rice straw in an ashing furnace and dried it at 110°C for 12 hrs. One hundred grams of oven-dried rice straw was carbonized in the first stage. Next, 100 g of rice straw was put into a furnace, and the

1. Oven-dried at 110 °C for 12 hr
   ↓
2. Carbonization: Heated rice straw to 280 °C, hold on 2 hr, then raise temperature to 450 °C for 2 hr.
   ↓ Cooled down to 25 °C
3. Treated with 4M KOH for 30 min
   ↓
4. Activation: Heat the mixture to 450 °C, hold on 2 hr, then raise temperature to 850 °C for 3 hr
   ↓ Cooled down to 25 °C
5. Wash by distilled water
   ↓
6. Oven-dried at 110 °C for 12 hr
   ↓
7. Activated charcoal product

FIG. 1. Procedure for the preparation of activated carbon.

temperature was increased to 280°C for 2 hrs to vaporize all organic compounds. Then, the temperature was further increased to 450°C for 2 hours to carbonize the rice straw. When the rice straw carbon had cooled from 450°C to room temperature, we used alkali. We mixed the rice straw carbon with potassium oxide (KOH) powder in a 1:2 (w/w) ratio and then added de-ionized water to dissolve the KOH and make a 4 M KOH solution.

After 30 minutes, the mixture was transferred to an ashing furnace; the temperature was increased to 450°C for 2 hours and then increased again to 850°C for 3 hours. This was the second stage of the heating process known as the activation process, which transforms rice straw carbon into activated rice straw carbon. Once the activated carbon had returned to room temperature, we washed it with a large amount of de-ionized water to reduce its alkalinity (pH $\sim$ 10) to close to neutral (pH $\sim$ 7). Finally, we dried the activated carbon product at 110°C for 12 hrs and the yield was 14.3% with ash. All the process in ashing furnace was under inert gas. The adsorbents were stored in separate vacuum desiccators until they were needed.

Activated Carbon Characterization
Specific Surface Area

The specific surface area of the activated carbon was determined from the adsorption-desorption isotherm of nitrogen gas (N_2). The Micromeritics ASAP 2010 was used to measure this parameter, and the average pore diameter was calculated from the adsorption branch of the N_2 isotherm.

Scanning Electron Microscopy (SEM)

The morphological structure of the adsorbents was obtained by using an FEI Quanta 200 scanning electron microscopy (SEM). The samples were coated with gold (Au) prior to analysis.

Elemental Analysis (EA)

Elemental analysis of samples were carried out with an Elementar Vario EL III (Germany).

Fourier Transform Infrared (FTIR)

Fourier transform infrared spectrum (NEXUS470, ThermoNicolet) analysis was performed on the native and BPA-adsorbed adsorbents to determine the surface functional groups. The spectra were recorded from 4000 to 500 cm^{-1}.

Adsorption Equilibrium Experiments and Kinetic Studies

A fixed amount of adsorbent (10 mg) was added to a set of 250-mL Erlenmeyer flasks containing 100 mL of BPA solution with different initial concentrations (7-55 mg/L). The pH of the solutions was not adjusted. The flasks were
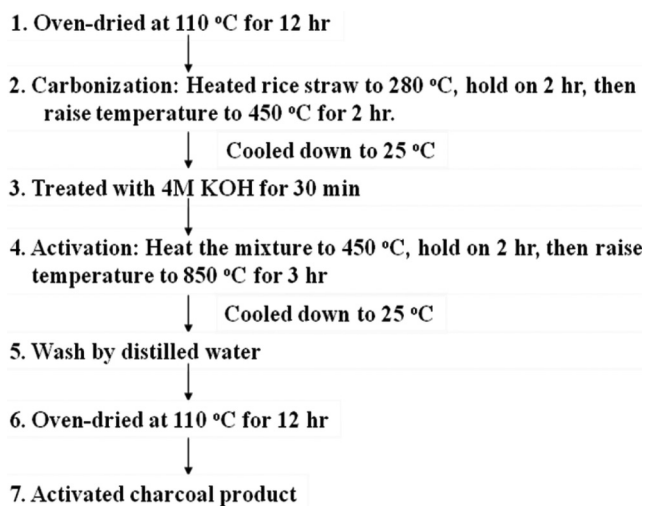
agitated in an isothermal water bath shaker at 180 rpm and 30°C for 90 min until equilibrium was reached. The solution was filtered with a Pall syringe filter (0.2-μm PTFE membrane). Subsequently, the residual BPA concentration in the filtrate was analyzed by high-performance liquid chromatography (HPLC, DIONEX, UltiMate 300) at a wavelength of 210 nm. The mobile phase for HPLC analysis was 40:60 (v/v) with acetonitrile and deionized water. The amount of adsorption at equilibrium, q_e (mg/g), was calculated by:

$$q_e = \frac{(C_o - C_e)V}{W} \qquad (1)$$

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

The amount of adsorption BPA at time t, q_t (mg/g), was calculated by:

$$q_t = \frac{(C_o - C_t)V}{W} \qquad (2)$$

Effect of Initial Concentration and Contact Time

A 10-mg sample of activated carbon was added to each 100 mL of BPA solution with initial concentrations of 7, 14, 27, 42, and 55 mg/L. The experiments were carried out at 30°C for 90 min.

Effect of Temperature

The effect of temperature on BPA adsorption was carried out in flasks sealed with Teflon-lined caps. A 10-mg sample of activated carbon was added to each 100 mL of aqueous BPA solution at different initial concentrations. The experiments were carried out at 30°C, 40°C, and 50°C for 90 min.

Effect of the Initial Ph of the BPA Solution

The pH of the solution varied from 2.35 to 11.00, and the mass of adsorbent (10 mg), the volume of solution (100 mL), the initial concentration of solution (27 mg/L), the temperature (30°C) and the shaker speed (180 rpm) were kept constant. The solution pH was adjusted with diluted 0.1 M HCl and 0.1 N NaOH.

RESULTS AND DISCUSSION
Characterization of Adsorbents

The BET surface area and the average pore diameter of the adsorbent were found to be 1,304.8 m²/g and 23.9 Å, respectively. SEM-enabled direct observation of the surface microstructures of the adsorbents. Figure 2 shows SEM micrographs for the rice straw samples at 10,000 × magnification. The rice straw had a well-developed porous
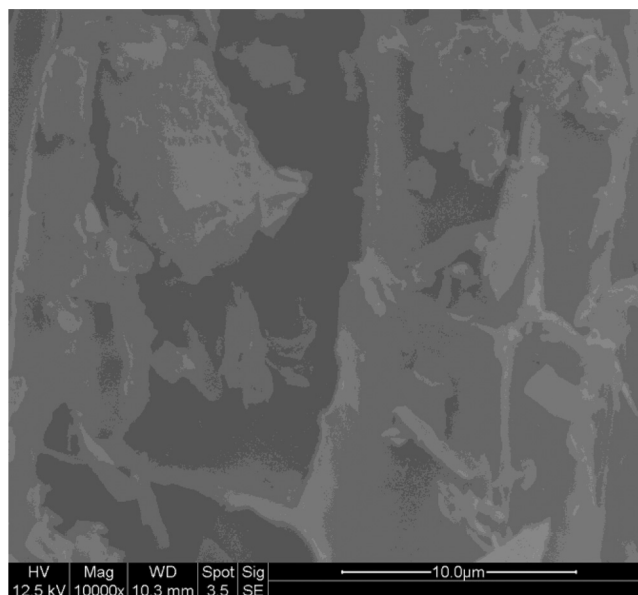

FIG. 2.   SEM micrograph of native activated carbon.

structure with good adsorption potential. The activation process resulted in the creation of pores and substantial removal of inorganic compounds in the raw material.

Table 1 shows the elemental content of the raw rice straw and the activated carbon before and after adsorption. The major elements of materials such as C, H, N, and S were examined. The activated carbon prepared from the rice straw had a higher carbon content (41.925%) compared to the raw material (36.578%) as well as a high carbon content (65.368%) after adsorption. This result indicates that some BPA was adsorbed on the activated carbon surface. FTIR analysis of the activated carbon before and after BPA adsorption showed that some peaks shifted and that some new peaks were present (Table 2). The FTIR spectrum had a broad band at 3,329 cm^{-1}, indicating bonded OH groups. In the BPA-loaded adsorbent, additional peaks at 1,742 cm^{-1} and 1,465 cm^{-1} were detected, which indicate binding with aromatic compounds. The peaks observed at 1,170 cm^{-1} and 1,086 cm^{-1} in the native adsorbent may have been produced by the stretching vibration of –OH groups that shifted to 1,163 cm^{-1} and 1,065 cm^{-1} in the BPA-loaded adsorbent. These changes in the FTIR spectra indicate that those functional groups on the surface of the activated carbon may have been involved in the adsorption process.

Effect of Initial Concentration and Time on BPA Removal

The uptake of BPA molecules by the adsorbents and the time required for the establishment of equilibrium suggest that these materials were effective for BPA treatment. To determine the equilibrium time for maximum BPA uptake, an initial concentration and contact time study were carried out. The effect of initial concentration and time

TABLE 1
Elemental content of raw rice straw and activated carbon before and after adsorption

	Elements (%)			
	C	H	N	S
Raw rice straw	36.578 ± 0.036	5.521 ± 0.038	0.851 ± 0.022	0.016 ± 0.009
Before adsorption	41.925 ± 0.283	3.542 ± 0.067	1.427 ± 0.031	1.146 ± 0.064
After adsorption	65.368 ± 0.100	3.761 ± 0.089	1.515 ± 0.126	1.069 ± 0.129

on BPA removal with activated carbon was investigated using various initial concentrations (7, 14, 27, 42, and 55 mg/L) at a constant adsorbent dose (100 mg/L), stirring speed (180 rpm), and temperature (30°C). A range of contact times was used in the study (10, 20, 30, 40, 50, 60, and 90 min). Figure 3 shows the adsorption uptake versus the adsorption time for various initial BPA concentrations. The removal of BPA by activated carbon increased with time and then reached equilibrium at approximately 90 min. When the initial BPA concentration was increased from 7 to 55 mg/L, the loading capacity of the activated carbon increased from 65.12 to 174.88 mg/g, and removal decreased from 92 to 32%. As the BPA concentration was increased, the adsorption capacity for the adsorbent increased, which may have provided the necessary driving force for overcoming the resistance to the mass transfer of BPA between the aqueous and solid phases (23). It is apparent that the efficiency of the removal of BPA was a function of the initial BPA concentration. Furthermore, in the early stages, the removal rate was rapid and then became constant. The initially high adsorption rate likely due to the abundance of free binding sites. Additionally, at lower concentrations, the ratio of the available surface area to the initial BPA concentration was higher, resulting in greater removal. However, for higher concentrations, this ratio was lower. As such, the efficiency of BPA removal was lower.

Effect of Temperature on Adsorption

Temperature has a direct influence on the adsorption process. Adsorption experiments were conducted at 30°C, 40°C, and 50°C with a constant BPA concentration (27 mg/L), adsorbent dose (100 mg/L) and contact time (90 min). The adsorption capacity of activated carbon decreased from 153.04 to 96.48 mg/g when the temperature was increased from 30°C to 50°C (Fig. 4). Gupta et al. reported that this decrease may occur due to the tendency for target molecules to escape from the solid phase to the bulk phase when there is an increase in the temperature of the solution (24). A similar observation was also reported in a study on the sorption of acid dye on activated clay and activated carbon (25).

Effect of pH on Adsorption

The pH of the solution is a critical point to the adsorption process (26). Variations in pH not only change carbon surface properties but can also affect the states of the ionic species in solution. In order to determine the adsorption

TABLE 2
FTIR spectral characteristics of rice straw before and after adsorption

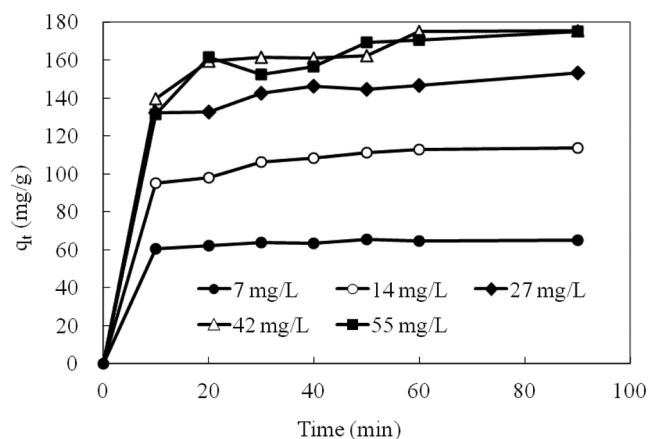| IR peak	Before adsorption	After adsorption	Difference	Assignment
	Frequency (cm^{-1})			
1	3,389	3,329	−60	Bonded –OH groups
2	–	1,742		C=C stretching vibrations
3	–	1,465		Ring stretching vibrations
4	1,170	1,163	−7	–OH stretching vibrations
5	1,086	1,065	−21	–OH stretching vibrations

FIG. 3. Variations in adsorption amount versus adsorption time for various initial BPA concentrations.
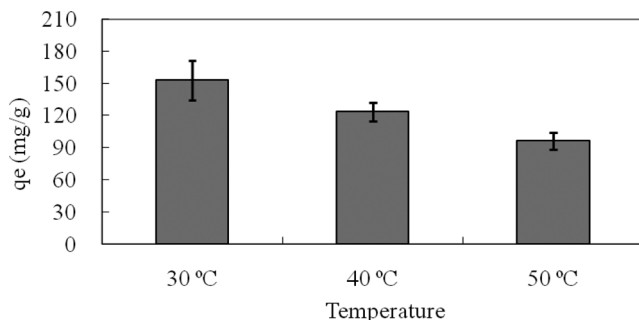
FIG. 4.   Effect of temperature on 27-mg/L BPA solutions.

behavior of BPA, experiments were conducted at an initial range of 2.35 to 11.00 pH with a contact time of 90 min, a constant BPA concentration (27 mg/L) and a constant adsorbent dose (100 mg/L). The adsorption capacities of specific adsorbents were found to be pH-dependent, as is shown in Fig. 5. Capacities decreased from 181.19 to 130.51 mg/g when the pH was increased from 2.35 to 11.00. The pH-dependence of the adsorption process may be largely related to the types and ionic states of the functional groups in the adsorbents as well as to the compound speciation in the solution (27). At low pH (acidic) of the solution the carbon surface is predominantly positively charged, whereas at basic pH, negative charges appear on the surface due to dissociation of the functional groups. Furthermore, BPA exhibits different states of pH-dependent equilibrium in an aqueous solution. BPA can be found in its molecular form at pH less than 8, and deprotonation of the bisphenolate monoanion occurs at a pH of approximately 8 (28). Therefore, due to the repulsive electrostatic interactions between the negatively charged activated carbon surface and the bisphenolate anion, this extra contribution decreased the overall uptake of BPA.

Adsorption Isotherms

Adsorption isotherms allow us to determine how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process reaches
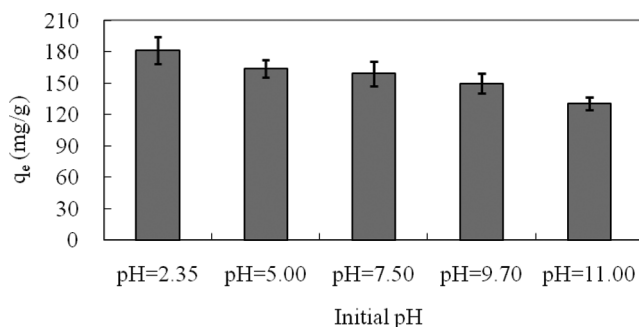

FIG. 5.   Effect of initial pH on 27-mg/L BPA solutions at 30°C.

an equilibrium state. In order to determine the most appropriate model, Langmuir, Freundlich, and Temkin isotherms were considered. The linear forms of the isotherms can be expressed with the following equations:

1. Langmuir isotherm:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \left(\frac{1}{Q_0}\right) C_e \qquad (3)$$

C_e (mg/L): Equilibrium BPA concentration
q_e (mg/g): Mass of adsorbate adsorbed per unit mass of adsorbate
Q_0: Langmuir constant related to adsorption capacity
b: Langmuir constant related to rate of adsorption

2. Freundlich isotherm:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \qquad (4)$$

q_e (mg/g): Mass of adsorbate adsorbed per unit mass of adsorbate
K_F (mg/g(L/mg)$^{1/n}$): Adsorption capacity of the adsorbent
n: An indication of how favorable the adsorption process is
C_e (mg/L): Equilibrium BPA concentration

3. Temkin isotherm:

$$q_e = B \ln K_t + B \ln C_e \qquad (5)$$

q_e (mg/g): Mass of adsorbate adsorbed per unit mass of adsorbate
B: Constant related to the heat of adsorption
K_t (L/mg): Parameter related to the heat of adsorption
C_e (mg/L): Equilibrium BPA concentration

The calculated isotherm model parameters are listed in Table 3. The Langmuir isotherm equation has been widely used as an adsorption model. It is based on the following implicit assumptions:

a. each site can accommodate only one molecule or atom,
b. the adsorption sites are localized, and
c. the adsorption energy of the adsorbents is constant for all sites (28–29).

The adsorption data were analyzed according to Eq. (3). When C_e/q_e was plotted against C_e, a straight line with a slope of $1/Q_0$ was obtained (data not shown) indicating that the adsorption of BPA on activated carbon followed the Langmuir isotherm. The nature of the adsorption process was assessed by determining the dimensionless equilibrium parameter (R_L) with the following equation:

$$R_L = \frac{1}{1 + bC_0} \qquad (6)$$

TABLE 3
Isotherm parameters for the removal of BPA by activated carbon at different temperatures

Isotherm	Parameter	Temperature (°C)		
		30	40	50
Langmuir	Q_0 (mg/g)	181.82	200.00	169.49
	b (L/mg)	0.7246	0.2128	0.2100
	R^2	0.99	0.98	1.00
Freundlich	K_F (mg/g $(L/mg)^{1/n}$)	100.72	56.68	47.50
	n	6.14	2.93	2.87
	R^2	0.97	0.97	0.89
Temkin	K_t	61.65	3.36	2.46
	B	23.185	37.808	34.581
	R^2	0.98	0.94	0.95

where b is the Langmuir constant and C_0 is the highest concentration of BPA (mg/L). In general, the favorable, irreversible, linear and unfavorable adsorption characteristics had results of $0 < RL < 1$, $RL = 0$, $RL = 1$ and $RL > 1$, respectively. In this study, the values of R_L were found to be 0.024, 0.096 and 0.094 at 30°C, 40°C, and 50°C, respectively. This result confirms that the adsorption of BPA on activated carbon is favored under the conditions studied.

The Freundlich isotherm is an empirical model that considers heterogeneous adsorption energies on an adsorbent surface (30). The Freundlich model can be used when:

a. the adsorption surface is energetically heterogeneous, and
b. the amount adsorbed increases with the concentration.

The slope and intercept obtained from a plot of lnq_e against lnC_e produced the Freundlich constants (K_F and n) for each temperature (data not shown). The larger the K_F and n values, the higher the sorption capacity. It is generally stated that n values in the range of 2–10 indicate good adsorption, values of 1–2 suggest moderately difficult adsorption, and an n value less than 1 suggests poor adsorption characteristics. Here, the favorable characteristics of

the Freundlich model are noted because of the higher value of the Freundlich constants ($n = 6.14$, 2.93 and 2.87 at 30°C, 40°C, and 50°C).

A linear plot of q_e against lnC_e (data not shown) shows that the adsorption obeys the Temkin model. Accordingly, the Temkin constants (K_t and B) were calculated and are listed in Table 3. K_t is the equilibrium binding constant (L/mg) that corresponds to the maximum binding energy, and the constant B is related to the heat of adsorption. A comparison of the average correlation coefficient (R^2) indicates that the Langmuir isotherm is a better fit ($R^2 = 0.99$) than the Temkin isotherm ($R^2 = 0.96$) or the Freundlich isotherm ($R^2 = 0.94$). The general assumption of the Langmuir adsorption isotherm is based on monolayer coverage of the adsorbate on the surface of the adsorbent (31), which is an indication of the fact that the adsorption of BPA by activated carbon generates monolayer formation.

Adsorption Kinetics Study

In the present study, two kinetic models were tested to predict the adsorption data of BPA as a function of time by using pseudo-first-order and pseudo-second-order kinetic models with different BPA concentrations. The two models led to the following equations:

1. Pseudo-first-order:

$$\log(q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303}\right)t \qquad (7)$$

2. Pseudo-second-order:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right)t \qquad (8)$$

q_e (mg/g): Mass of BPA adsorbed at equilibrium
q_t (mg/g): Mass of BPA adsorbed at time t (min)
k_1 (min^{-1}): Pseudo-first-order constant
k_2 (g/mg min): Pseudo-second-order constant

The kinetic parameters including the first-order rate constant k_1, the second-order rate constant k_2, the calculated

TABLE 4
Kinetic parameters for the adsorption of BPA by activated carbon at 30°C

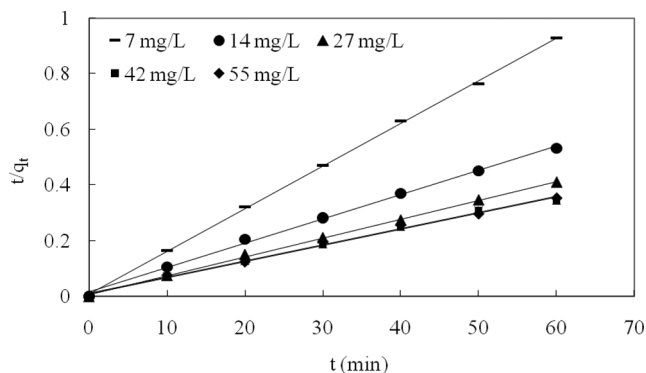| C_0 (mg/L)	Pseudo-first-order kinetics				Pseudo-second-order kinetics		
	$q_{e,exp}$ (mg/g)	k_1 (min^{-1})	$q_{e,cal}$ (mg/g)	R^2	k_2 (g/mg min)	$q_{e,cal}$ (mg/g)	R^2
7	65.12	0.0666	19.12	0.8004	0.0263	65.36	0.9950
14	113.87	0.0672	67.11	0.9412	0.0052	113.64	0.9974
27	153.04	0.0438	59.68	0.7413	0.0069	147.06	0.9989
42	175.32	0.0808	141.38	0.7094	0.0037	172.41	0.9952
55	174.88	0.0530	93.86	0.8341	0.0030	172.41	0.9941

FIG. 6. Pseudo-second-order kinetics for the adsorption of BPA by activated carbon at 30°C.

equilibrium adsorption capacity $q_{e,cal}$, the experimental equilibrium adsorption capacity $q_{e,exp}$ for BPA, and the regression coefficients (R^2) are all shown in Table 4. The values of k_1 and $q_{e,cal}$ were determined from the slope and intercept of log ($q_e – q_t$) versus time (data not shown). As shown in Table 4, the dramatic difference between $q_{e,exp}$ and $q_{e,cal}$ from the first-order model indicates that the adsorption of BPA by activated carbon did not follow first-order kinetics.

The values of k_2 and $q_{e,cal}$ were determined from the slope and intercept of t/q_t versus time, respectively (Fig. 6). The $q_{e,cal}$ for the pseudo-second-order model agrees well with the experimental $q_{e,exp}$ values. The R^2 values for the second-order kinetic model were 0.9941-0.9989, indicating that the experimental adsorption system is more appropriately described by the pseudo-second-order model.

Table 5 gives a comparison of the maximum adsorption capacity of BPA for various adsorbents. Rice straw is significantly higher than many other adsorbents that have been developed in the recent past and have a relatively high

TABLE 5
Comparison of the adsorption capacities of different adsorbents for BPA

Adsorbent	Adsorption capacity (mg/g)	Reference
Commercial activated carbon	382.12–432.34	(32)
Commercial activated carbon	263.1	(28)
Almond shell	188.9	(28)
Rice straw	181.19	This study
Commercial activated carbon	129.6	(28)
Commercial activated carbon	78.13	(3)
Carbon nanotube	61	(5)
Bamboo	58	(4)
Carbon nanomaterial	45	(33)
Minerals	0.33–0.86	(3)

adsorption capacity of 181.19 mg/g. Ultimately, rice straw may be a promising material for the removal of BPA from aqueous solution.

CONCLUSIONS

The present study revealed the potential for rice straw, an agricultural waste material, to be used as a low-cost adsorbent for removing BPA from aqueous solutions. The maximum BPA adsorption capacity, 181.191 mg/g, was observed at a pH of 2.35. In addition, the equilibrium data fit the Langmuir model very well with the experimental values. The kinetic adsorption data fit the pseudo-second-order equation better than he fit the pseudo first-order-equation. We conclude that the availability and production of carbonaceous adsorbents from rice straw biomass makes it a promising material for the removal of BPA from aqueous media.

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