Granular Activated Carbon Adsorption Process for Removing Trichloroethylene from Groundwater

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Batch adsorption capacity experiments and continuous flow breakthrough experiments were conducted to compare the effectiveness of seven granular activated carbons (GACs) in removing trichloroethylene (TCE) from groundwater. The order of GACs' adsorptive capacities for TCE was the same as their phenol numbers (indicator for <1 nm micropores). A small amount of methanol (0.05% v/v) did not affect the pure water TCE capacities, which were notably reduced by the small organic constituents of tap water. The breakthrough results show low utilization of the available adsorptive capacity due to both slow mass transport and the need to achieve a high degree of TCE removal, and that the serial bed mode of treatment is cost effective because a very high capacity utilization can be attained. The low-cost, environmental friendly bamboo GAC is attractive as its competitive TCE capacity was readily used in actual treatment because of its favorable pore structure. © 2010 American Institute of Chemical Engineers *AIChE J,* 57: 542–550, 2011

Keywords: trichloroethylene, activated carbon, adsorptive capacity, competitive adsorption, MCRB

Introduction

Trichloroethylene (TCE) is a widely used organic solvent for electronic, machinery, metal finishing, and dry cleaning applications. As a result of the poor housekeeping and/or improper waste disposal practices, TCE has become a common groundwater pollutant because of its moderate water solubility, low biodegradability, and toxicity.[1–4] Although TCE concentration of most contaminated groundwater is rather low (<2 mg/L), high TCE concentrations (10–920 mg/L)[5] have been reported for many groundwater samples taken nearby areas of recent contamination. TCE is a suspected carcinogen and listed as a priority pollutant: its removal to below 5 μg/L is required for drinking water in many countries.[6]

Granular activated carbon (GAC) adsorption technology is often used to remove TCE and other persistent organic pollutants (POPs).[7,8] Commercial activated carbons of different raw materials and activation methods have significantly different adsorptive capacities for the target water pollutant, and that the equilibrium adsorptive capacity of a GAC for the pollutant in pure water is only partially used in actual treatment because of the mass transfer limitations, competitive adsorption of the coexisting naturally occurring dissolved organic matters (NOM), and the requirement of a very low effluent concentration (such as <1 ppb for many POPs) to meet the stringent discharge limit.[9,10] An efficient treatability study should be conducted to design a cost-effective activated carbon treatment process for each application.

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Table 1. Characteristics of the Water Samples

Parameter	Pure Water	Tap Water	Well Water
pH	7.3	7.1	7.5
TOC, mg/L	<0.4	4.5	8.8
TSS, mg/L	–*	–*	105
UV_{254}†, Abs	0	0.092	0.232

*Too low to measure.
†Using the pure water sample as the reference.

This article presents the treatability study consisting of batch adsorption equilibrium experiments and continuous flow breakthrough experiments, which were designed to compare the effectiveness of seven GACs in removing TCE from groundwater and to define the best treatment process. The objectives of the study were as follows: (1) determine the effects of the batch contact time, raw material, and initial TCE concentration on GACs' adsorptive capacities for TCE, (2) correlate the GAC's adsorptive capacity for TCE with its capacity for one of the four indicator compounds (phenol, iodine, methylene blue, and tannic acid) commonly used for selection of effective GACs,[11] (3) demonstrate the effects of NOM on TCE capacities, (4) validate the efficient microcolumn rapid breakthrough (MCRB) method for simulating full-scale adsorption treatment performance, and (5) propose a cost-effective GAC treatment process scheme for removing TCE and other POPs from groundwater.

Experimental

Materials and analytical methods

Four Chinese GACs of different raw materials (coal, coconut shell, fruit nut/shell, and bamboo of Shanghai Activated Carbon Company) and three popular US GACs (coconut-based Norit GCN830 and coal-based Calgon F300 and Calgon F400) were used in this study. The GAC samples were first pulverized, sieved to the desired mesh size fractions, washed with deionized water, dried for 12 h at 105°C, and then stored in glass jars.

Distilled and deionized (pure) water (lab prepared, TOC < 0.4 mg/L), tap water (TOC = 4.5 mg/L), and a well water sample (obtained from a well in nearby Songjiang as the groundwater, TOC = 8.8 mg/L) were used to prepare the TCE solutions used in the study. Table 1 presents important characteristics of the water samples. Some TCE solutions were prepared by dilution of the TCE-in-methanol mixture. A dilute phosphate buffer was used to maintain a constant pH in the phenol and tannic acid runs of the capacity indicator experiments.[12]

Analytical-grade TCE, iodine, phenol, methylene blue, tannic acid, methanol, potassium phosphates, and n-hexane were obtained from local suppliers. The concentrations of iodine, phenol, methylene blue, and tannic acid solutions were estimated from the standardization curves of the light absorbance measured at 600, 268, 613, and 275 nm, respectively[12]; TCE in aqueous samples was extracted by hexane, and its concentration was measured by GC/ECD.[13]

Adsorptive capacities of GAC for the indicator compounds

The GAC's pore structure and surface chemistry have been well correlated with its four capacity indicators (phenol, iodine, methylene blue, and tannic acid numbers); indeed, those indicators have been successfully used for selecting the most effective GAC in removing such organic water pollutants as chloroform, 2,4-dichlorophenol, the red dye X3B, benzene, toluene, ethylbenzene, xylenes, nitrobenzene, methyl-tert-butyl ether, 4,4-bisphenol, humic acid, and POPs of biotreated coking plant effluent.[12,14] Batch activated carbon-solution contact runs were first conducted to obtain the four adsorptive capacity indicators of the GAC samples using the established procedures.[12] Table 2 presents the four indicators of the seven GACs. A high phenol number indicates that the GAC has a large internal surface area of very small micropores (diameter < 1 nm) and a low surface acidity, which enhances the adsorption of polar organic compounds; high values of iodine number, methylene blue number, and tannic acid number indicate abundant small micropores (diameter < 1.5 nm), large micropores (diameter = 1.5–2.8 nm), and larger pores (diameter > 2.8 nm), respectively.[14]

Batch adsorptive capacity experiments

Batch equilibrium adsorptive capacity (isotherm) experiments were performed on mixtures of pulverized GAC samples (1–8 mg of 200–325 mesh) and various TCE solutions of different initial concentration and composition using the standard adsorption isotherm procedures with such improvements as smaller sample size and a head-to-bottom rotation method to provide the necessary movement for the carbon particles in a filled 42-mL glass bottle.[15] After the carbon particles and the TCE solution were mixed for a given time (2, 4, 6, 12, and 48 h), the rotation was stopped, and then the bottles were placed vertically to allow the particles to settle. TCE of the clear supernatant was extracted by hexane and then measured by GC/ECD. The adsorptive capacity was calculated by: X/M (mg/g) = $(C_0 - C_f)$ V/carbon dose (g), where V is the sample volume (L), C_0 and C_f are the initial and final TCE concentration (mg/L), respectively. Experimental data of X/M and C_f were correlated by the Freundlich adsorption isotherm model, $X/M = kC_f^{1/n}$, and plotted with the isotherm in a log–log scale graph for easy comparison of the GACs' capacities for TCE.

Table 2. Adsorptive Capacity Indicators of the GAC Samples

GAC Sample	Phenol No.*	Iodine No.†	Methylene Blue No.‡	Tannic Acid No.§
Norit GCN830	116	1204	279	12.7
Shanghai coconut	112	999	164	13.2
Shanghai fruit	102	1033	307	61.4
Calgon F400	100	1175	279	26.5
Calgon F300	98.0	1125	281	29.6
Shanghai bamboo	95.0	1163	237	20.0
Shanghai coal	62.5	998	262	31.7

*mg of phenol adsorbed by 1 g of carbon at an equilibrium phenol concentration of 20 mg/L.
†mg of iodine adsorbed by 1 g of carbon at an equilibrium iodine concentration of 0.02 N.
‡mg of MB adsorbed by 1 g of carbon at an equilibrium MB concentration of 1 mg/L.
§mg of tannic acid adsorbed by 1 g of carbon at an equilibrium tannic acid concentration of 2 mg/L.

Table 3. Design and Operating Parameters of the TCE Breakthrough Runs

	Conventional Columns	MCRB Columns
Carbon amount	5.0 g	0.5 g
GAC particle size	20–40 mesh	120–180 mesh
Column diameter	17.0 mm	5.0 mm
EBCT	2.4 min	4.0 s
Bed volume	10.4 mL	0.75 mL
Time to treat 10,000 bed volumes	16.7 days	11.1 h

Continuous flow adsorption breakthrough experiments

To confirm the relative adsorptive capacity of GACs for TCE and to obtain the capacity utilization rates of full-size treatment operations necessary for estimating the carbon consumption rate, series of continuous flow adsorption breakthrough experiments were performed using both the conventional method,[16] which used unsieved GAC particles and the same empty bed contact time (EBCT) as the full size adsorbers, and the efficient MCRB method, which was evolved from the United States Environmental Protection Agency's rapid small-scale column test techniques[17] with improvements (using smaller 120–180 mesh particles, low pressure pump, and low-cost autosampler) and simplification (no pulse dampener, pressure relief, and vent) to allow its practice in an ordinary environmental laboratory.[12] The MCRB fundamentals, the experimental apparatus, detailed test procedures, results, and discussions are covered in a related article.[18] In this study, the particle size ratio was 0.167 ($d_{120–180\ mesh} = 0.1$ mm and $d_{20–40\ mesh} = 0.6$ mm); the run time ratio was estimated by the following equation:

$$\frac{\mathrm{EBCT_{MCRB}}}{EBCT_{LC}} = \left[\frac{d_{\mathrm{p.MCRB}}}{d_{\mathrm{p.LC}}}\right]^{2-X} = \frac{t_{\mathrm{MCRB}}}{t_{\mathrm{LC}}}, \qquad (1)$$

where $d_{p,MCRB}$ and $d_{p,LC}$ are diameters of GAC particles in the MCRB column and the larger conventional column filled with 20–40 mesh GAC, respectively, and t is the run time; $X = 0$ if the intraparticle diffusivity is the same in the two systems, otherwise, $X = 1$.[19] If $X = 0$ is assumed for TCE, the MCRB run time would be about 1/36 of the conventional breakthrough method.

The MCRB column effluent samples were taken periodically for measurement of the TCE concentrations, which, in the dimensionless form of C/Cin (Cin is the influent TCE concentration), were plotted against the number of bed volume treated to show the observed breakthrough curves. Table 3 presents the design and operating parameters of the TCE breakthrough runs. Table 4 summarizes the TCE removal data of the MCRB runs.

Results and Discussion

Effects of batch contact time and initial TCE concentration on the adsorptive capacities

Figure 1 presents adsorption isotherms of TCE in tap water for the batch isotherm runs of different contact times to illustrate the contact time effect on the adsorptive capacities of the Shanghai coconut (coconut) and Shanghai bamboo (bamboo) GACs. Because of the poor mass transfer condition of the filled glass bottles, a long contact time was necessary for the GAC-TCE system to reach equilibrium. Adsorption of TCE was slower in the coconut bottles because of its far less larger pores (smaller tannic acid number) relative to bamboo (Table 2). The 48-h contact time was desirable to establish the equilibrium TCE isotherms as evidenced in Figure 2, showing that TCE isotherm of F400 of this study (the 48-h run) was nearly the same as the F400 isotherm (contact time of 6.5 days)[20] of the EPA treatability database[21] and that the shorter contact time (2 h) resulted in the lower isotherm of the EPA F300[22] relative to the 48-h isotherm of this study.

The dependence of adsorption capacity on initial concentration has been studied by past researchers with conflicting results,[23] with some reports of lower adsorption capacities obtained with higher initial concentration runs,[24] whereas others stating no initial concentration effect.[25] That a longer contact time would be necessary for the higher initial concentration system to reach equilibrium might be responsible for such differences as the failure to attain equilibrium would result in lower than true adsorption capacities. Figure

Table 4. Summary of the TCE Removal Data for the MCRB Runs

MCRB Columns	C_{in} (mg/L)	EBCT (s)	TCE Removed* (mg)		Total Available Adsorptive Capacity† (mg)	Capacity Utilization‡ (%)	
			$C/C_{in} = 0.05$	$C/C_{in} = 0.50$		$C/C_{in} = 0.05$	$C/C_{in} = 0.50$
Coconut§	2.6	4.20	4.6	23.8	38.4	12.0	62.0
GCN830§	2.8	4.13	7.7	37.8	57.1	13.5	68.4
Coal§	1.8	4.12	8.5	16.1	19.6	43.3	82.2
Fruit§	1.8	4.14	8.3	25.7	29.9	27.6	86.0
F400§	2.7	4.15	9.5	29.0	33.4	28.4	86.9
Bamboo§	2.5	4.14	7.0	23.1	28.0	25.1	82.6
Coconut¶	2.0	4.04	6.9	32.0	53.6	12.9	59.7
GCN830¶	3.6	4.02	8.6	49.8	78.6	10.9	63.4
Coal¶	1.4	4.02	4.1	18.4	23.5	16.6	78.4
Fruit¶	1.4	4.04	19.2	>36.7**	40.8	47.1	>90.0**
F400¶	3.0	4.02	5.3	32.0	38.1	13.8	83.9
Bamboo¶	1.7	4.04	12.8	23.9	28.3	45.5	84.6

*Amount removed = total amount supplied × (area above the breakthrough curve/total area).
†Total available adsorption capacity = carbon amount × adsorptive capacity at C_{in} (Figures 7 and 8).
‡Capacity utilization = (TCE removed/total available adsorptive capacity) × 100%.
§Tap water runs.
¶Well water runs.
**Estimated from the available breakthrough curve in Figure 10.

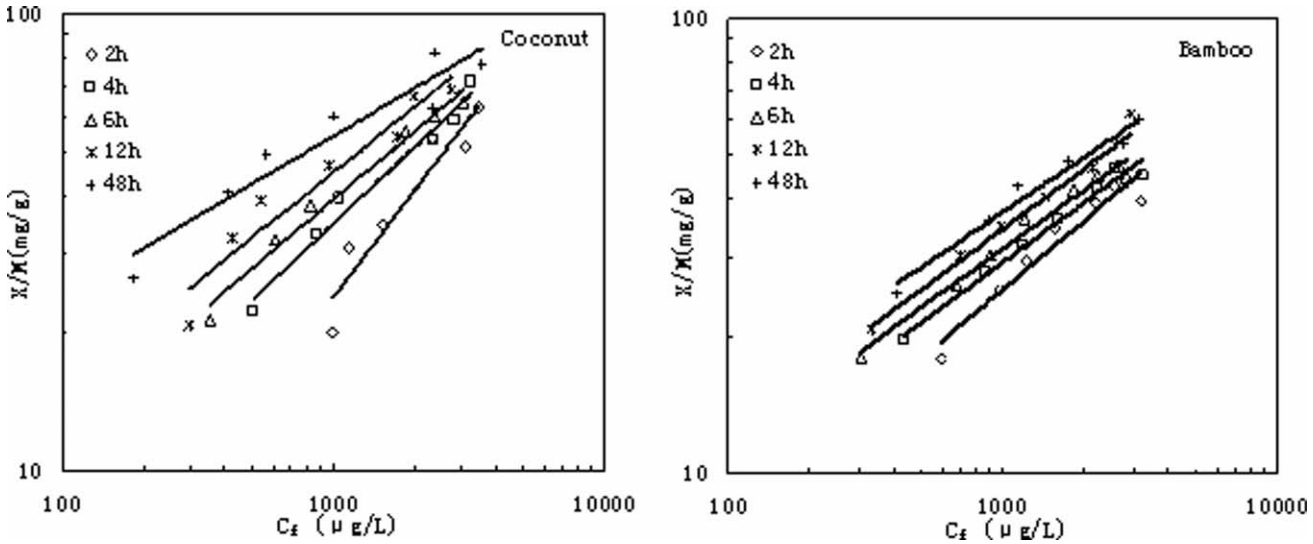

Figure 1. Effect of contact time on TCE isotherms (Co = 5 mg/L, tap water).

3 demonstrated that the TCE capacities of the GACs were not dependent on the initial concentration as the 48-h capacity test runs of both coconut and coal using 2, 5, and 10 mg/L of TCE in pure water solutions resulted in nearly identical isotherms.

Effect of a small amount of methanol on TCE capacities

As TCE is highly volatile (Henry's Law constant = 0.0012 MPa m^3/mol[21]), fresh TCE solutions should be prepared for each test series to ensure the same TCE concentration of the starting solution or influent is used in all runs of the same test series. Preparation of TCE solution on demand is easy by dilution of a TCE-in-methanol mixture. The effect of a small amount of methanol on GAC's capacity for TCE was determined by comparing the isotherms of coconut and bamboo using 2 mg/L pure water solutions with and without methanol. The nearly identical isotherms of Figure 4 show that the GACs' capacities for TCE were not affected by the presence of 0.05% (v/v) methanol, consistent with the literature report of the same for the presence of <0.1% of methanol.[26] Therefore, the TCE solutions used in the subsequent test runs were prepared by diluting a TCE-in-methanol mixture with pure water, tap water, and the well water samples as desired.

Effect of water composition on the TCE capacities

The adsorption isotherms of TCE in pure water, tap water, and the well water samples for coconut, fruit, bamboo, and coal are shown in Figure 5. The respective adsorptive capacities for TCE in pure water were reduced because of presence of the organic constituents of tap water and the well water samples; greater degrees of capacity reductions were noted for TCE in tap water than in the well water sample despite its lower background TOC (4.5 vs. 8.8 mg/L). Given the common observations of larger capacity reductions for adsorbates in the solutions of higher background TOC,[8]

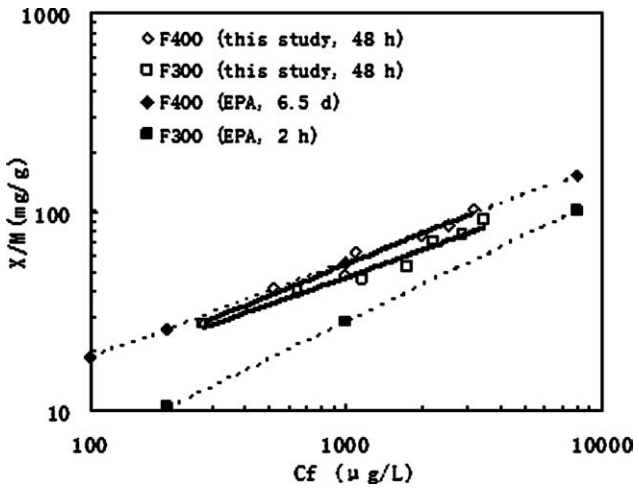

Figure 2. Confirmation of TCE isotherms of two coal GACs (Co = 5 mg/L, pure water).

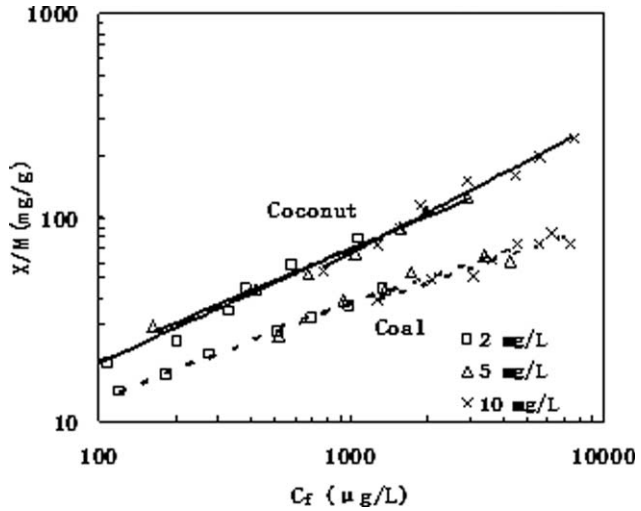

Figure 3. Effect of initial concentration on TCE isotherms (pure water).

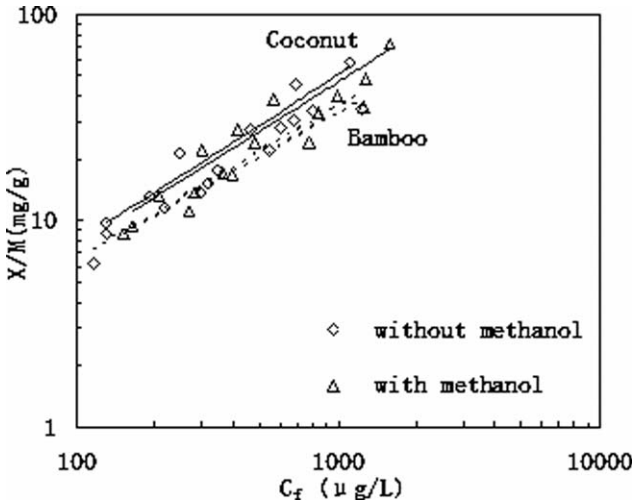

Figure 4. Effect of methanol (0.05%) on TCE isotherms (Co = 5 mg/L, pure water).

the results of this research have demonstrated the more effective competitive adsorption of smaller components of NOM on the measured adsorptive capacity as the organic constituents of tap water are much smaller than those of the well water because most large NOM components were removed by coagulation, flocculation, and sedimentation operations of the water treatment plant, as illustrated by the much smaller value of UV_{254} of the tap water sample (Table 1). The larger NOM species had a greater effect in reducing the adsorption rate because of pore blocking,[27] with little effect on the capacity given the long batch contact time (48 h), which was provided to ensure that equilibrium would be reached for water samples of varying composition.[28]

The main reason that the larger NOM constituents of well water in a higher concentration caused much less reduction in the TCE capacity than the smaller NOM in a lower concentration of the tap water is they are too large for entering the very small micropores (diameter < 1 nm) to compete with the TCE molecules for the adsorption sites. The smaller NOM of the tap water caused a significant reduction in TCE capacity because of competitive adsorption as a major fraction of the NOM species can also enter the small micropores to compete with TCE for the adsorption sites.

GAC's phenol number as the indicator of its adsorptive capacity for TCE

The carbon particle's pore structure and surface chemistry are the most important properties of activated carbon for GAC's adsorptive capacities. An organic pollutant is most strongly adsorbed on the internal surface area of pores slightly

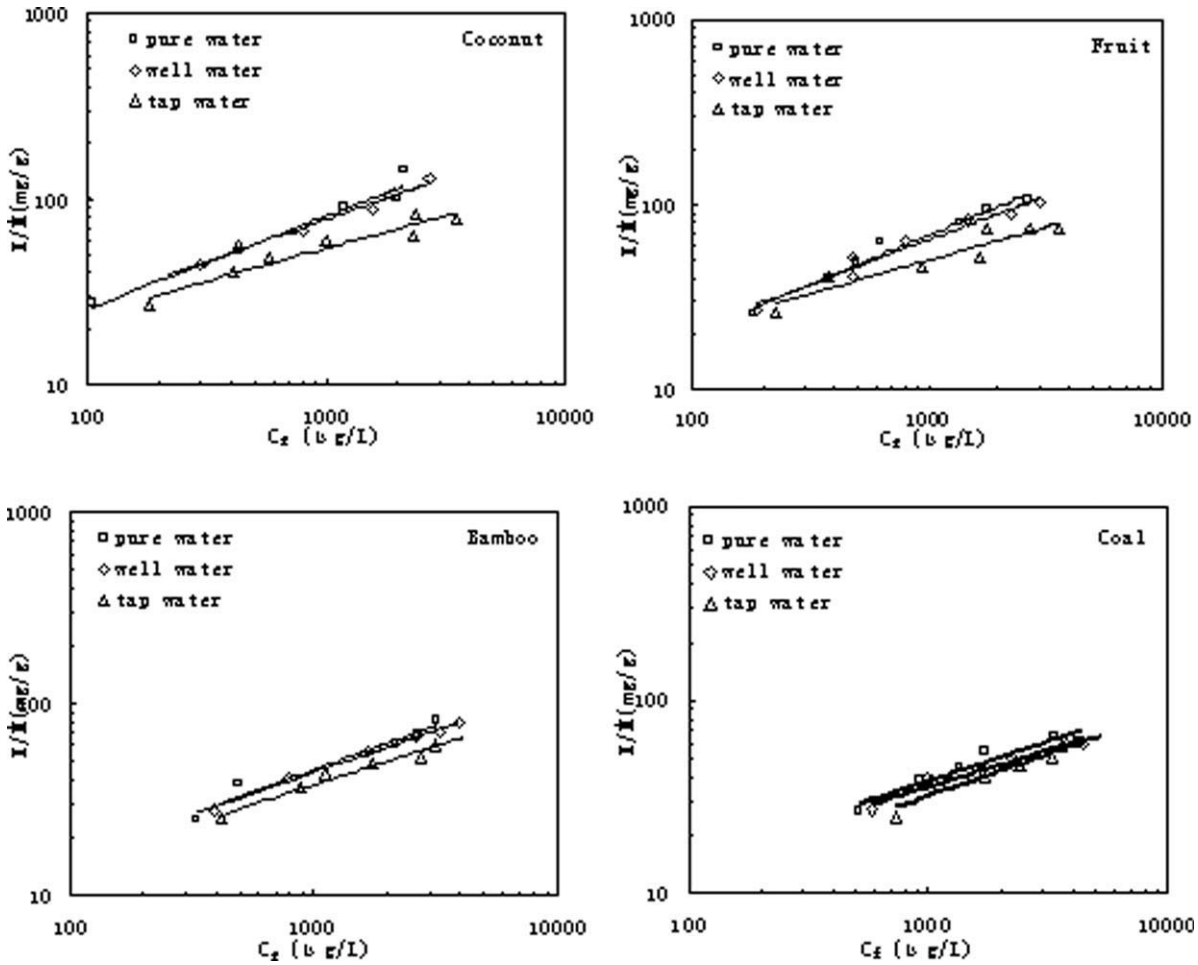

Figure 5. Effect of water composition on TCE isotherms.

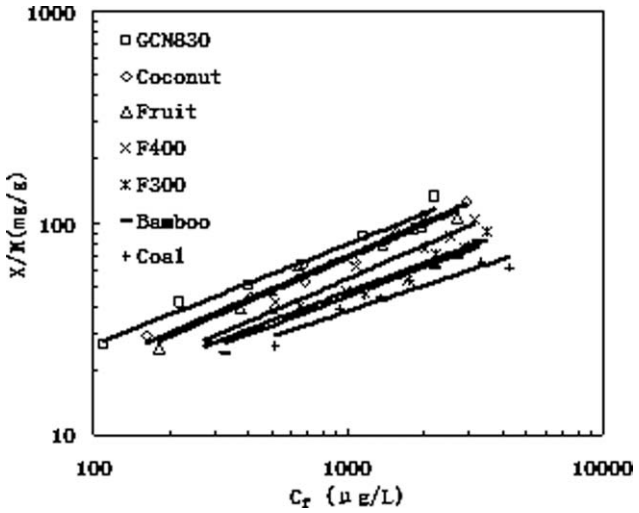

Figure 6. Adsorption isotherms of TCE in pure water.

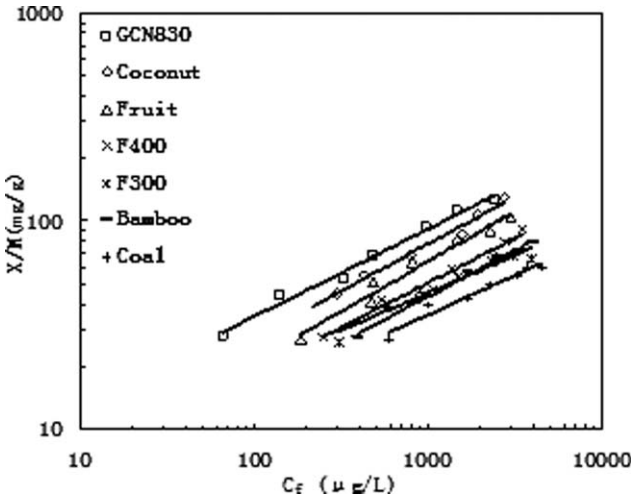

Figure 8. Adsorption isotherms of TCE in well water.

larger than its effective molecular diameter (MD) according to the concept of microporosity effect.[29] Therefore, GAC's relative capacity for TCE was expected to be best correlated with its phenol number as TCE's MD is even smaller (of about 0.56 nm[30] vs. 0.62 nm of phenol[14]) and that both are planar and polar compounds.[31] Indeed, the order of GAC's capacities for TCE in pure water (Figure 6) is the same as their relative phenol numbers (Table 2), i.e., GCN830 > coconut > fruit > F400 > F300 > bamboo > coal.

Literature reports have suggested that the relative TCE capacities of several GACs were not affected by the coexisting NOM present in the water samples.[30,31] The results of this study have confirmed such findings with the order of the adsorption isotherms of TCE in tap water (Figure 7) and well water (Figure 8) were the same as observed in pure water runs. Therefore, the GAC's phenol number is a good indicator of its adsorptive capacity for TCE with and without the coexisting NOM.

Experimental verification of the MCRB method

According to Eq. 1 and with the particle size ratio of 0.167 ($d_{120–180\ mesh}$ = 0.1 mm and $d_{20–40\ mesh}$ = 0.6 mm), the calculated run time ratio was 0.0278 (1/36), assuming X = 0 for the same intraparticle diffusivity in both situations. Given a EBCT of 2.4 min was sufficient to obtain well-defined breakthrough curves for the three small GAC columns (F400, bamboo and coal, Figure 9) operating in the conventional manner (as defined in Table 3), 4 s was selected as the EBCT of the MCRB column runs to verify that the MCRB method may indeed be used for simulating the treatment performance of a full-scale GAC adsorber. The nearly identical TCE breakthrough results of the small

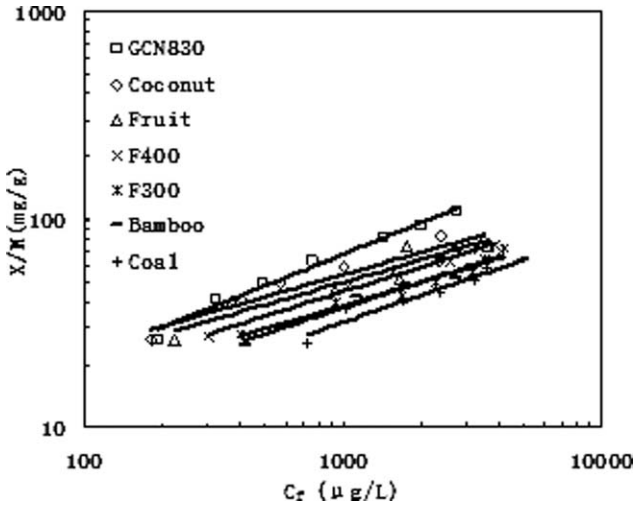

Figure 7. Adsorption isotherms of TCE in tap water.

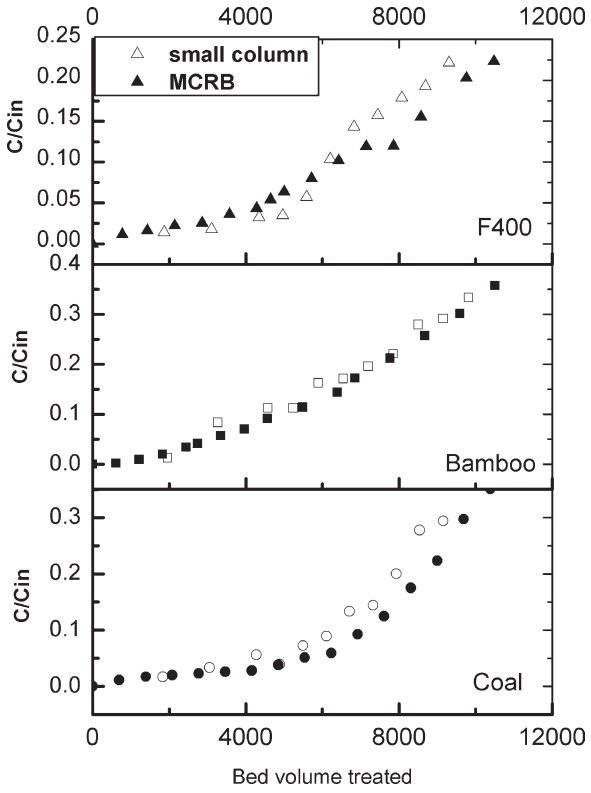

Figure 9. TCE breakthrough curves of the small conventional and MCRB column runs.

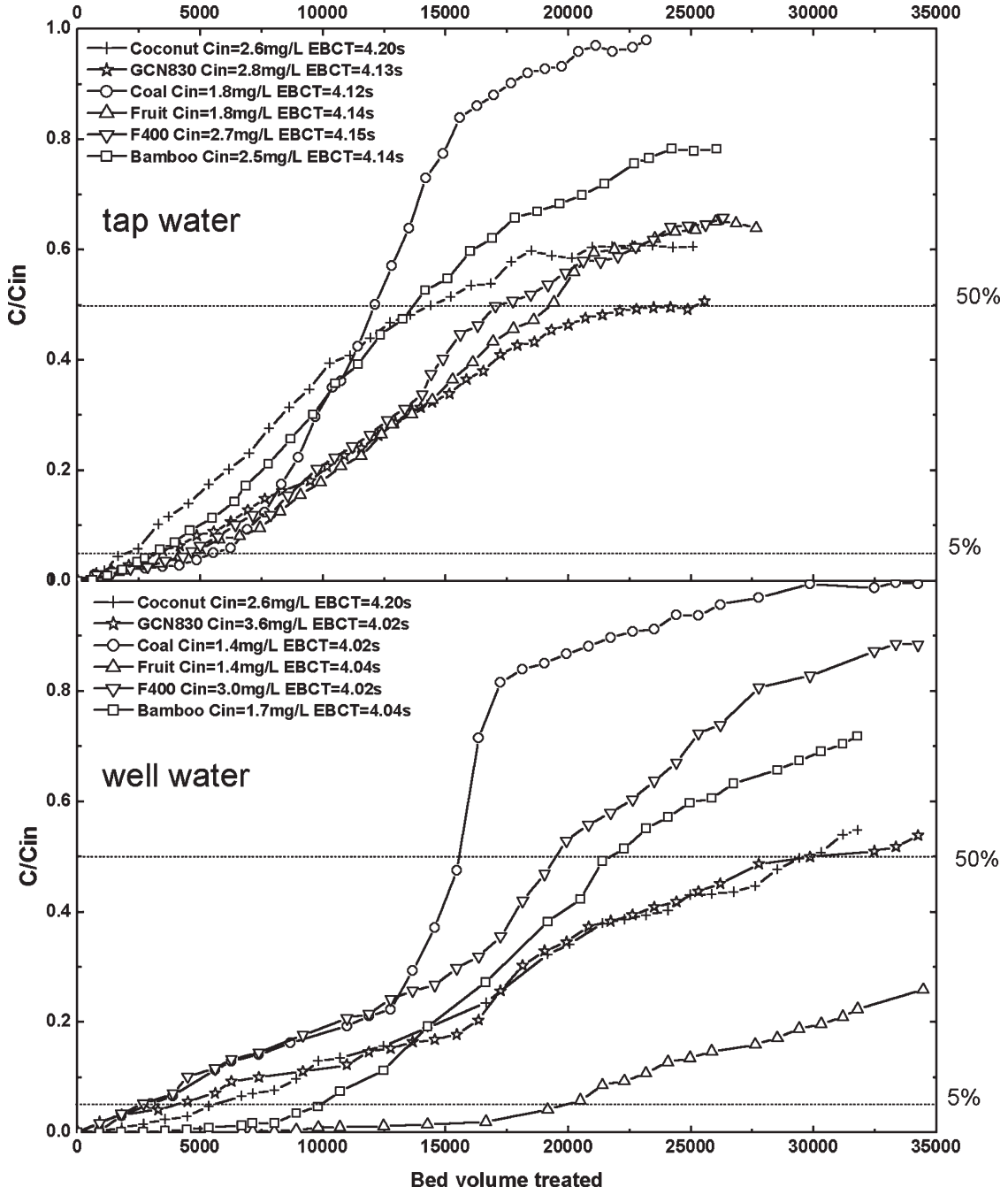

Figure 10. Adsorption breakthrough curves of TCE in tap water and well water.

conventional and MCRB columns, as illustrated in Figure 9 for the three GACs, have justified the constant intraparticle surface diffusion coefficient ($X = 0$) assumption and the use of Eq. 1 for designing the MCRB runs.[19,32]

The study results have therefore validated the MCRB as an efficient method for conducting the essential breakthrough experiments to simulate the treatment performance of a full-scale GAC adsorber for selecting the best GAC and to calculate the capacity utilization rate which is required for estimating the carbon consumption in removing TCE or other POPs.[12,18] Not only the run time was only 1/36 of those required by the conventional runs, the three MCRB curves were more consistent and smooth because of its much simpler procedure of using a single batch of the feed solution relative to the need of making up many batches of the feed for treating 10,000 bed volumes of it in the conventional columns during the 16.7 days of operation (Table 3).

TCE removal and capacity utilization of the adsorption breakthrough experiments

Figure 10 illustrates the adsorption breakthrough curves of 12 MCRB runs for removing low levels of TCE in tap water and the well water samples. Using an EBCT of 4 s, the

MCRB method enabled viable breakthrough curves to be obtained in less than 40 h instead of about 60 days that would be required using the conventional breakthrough method for treating 34,000 bed volumes of feed. Table 4 presents the important operating parameters of those column runs and the associated performance data of TCE removed and capacity utilization at two effluent limits of C/Cin of 0.05 and 0.5, chosen to represent the required high degree of pollutant removal to meet the stringent discharge limit and the more relaxed effluent limit for the first adsorber of the two-bed-in-series mode of treatment.

The GAC column loadings of TCE were lower with correspondingly faster breakthrough when it was removed from tap water than the well water sample. Such results have confirmed the greater competitive adsorption of the smaller NOM constituents of the tap water sample found in the batch capacity runs. The capacity utilization rates of all MCRB runs increased significantly when the effluent limit was raised from 5 to 50% of the influent concentration. Such results have demonstrated that the serial bed mode of treatment is essential for the cost-effective GAC treatment process because most of the GAC's adsorptive capacity would be available for removing the pollutant with the allowable high effluent concentration of the first adsorber, while excellent effluent quality can still be assured with the polishing treatment in the second adsorber.

The two coconut GACs' higher TCE capacities were less available than those of other GACs. The bamboo GAC performed well because its TCE capacity was more available than the coconut GACs because of its more favorable pore structure as indicated by its higher tannic acid number (Table 2). As bamboo (¥7500/ton; ¥6.8 = US$1.00) is less costly than coconut (¥12,000/ton) and fruit (¥11,000/ton) and has a higher capacity than the slightly less costly coal (¥7000/ton), it is the most cost-effective GACs among the seven GACs studied.

Conclusions

(1) The poor mass transfer condition of the filled bottles used for the isotherm experiments required a long batch contact time of 48 h to ensure that the measured adsorptive capacity of GAC for TCE or another volatile organic compound would approach its true equilibrium capacity.

(2) The GACs' adsorptive capacities for TCE were in the same order as their phenol numbers (the capacity indicator for very small adsorbates) and that their capacity utilization rates (availability) were indicated by their tannic acid numbers (the capacity indicator for large adsorbates). That the two coconut GACs' TCE capacities were higher but less available than other GACs are consistent with their relative high phenol numbers and low tannic acid numbers.

(3) The presence of a small amount of methanol (0.05% v/v) did not affect the TCE capacities. Dilution of a TCE-in-methanol mixture was the simple, rapid, and dependable procedure to prepare TCE solutions for the experiments.

(4) GACs' capacities for TCE in pure water were reduced by competitive adsorption of other organic constituents of the sample; small organic compounds present in tap water were more competitive than the NOM in the higher TOC well water sample.

(5) The efficient MCRB method enabled viable breakthrough curves to be obtained in a much shorter time than using the conventional breakthrough method. The MCRB data confirmed the GACs' available TCE capacities and that the serial bed mode of treatment is essential for the cost-effective GAC adsorption process because most of the capacity can be used for removal of the pollutant with the high allowable effluent concentration of the first bed.

(6) The GAC made from toxicant-free, low cost, and renewable bamboo is attractive because its relatively high TCE capacity is readily available in actual adsorption treatment.

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