

Quantitative Elucidation of the Effect of EBCT on Adsorption and Biodegradation of Biological Activated Carbon Filters

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Abstract—This research was focused on differentiating the adsorption and biodegradation quantities of a biological activated carbon (BAC) column, and on evaluating the effect of empty bed contact time (EBCT) on the performance of the two mechanisms simultaneously for the removal of dissolved organic matter from water. The performance of adsorption and biodegradation on the BAC column was studied using continuous columns tests, and four typical ozonation by-products were selected as the target compounds.

The results show that EBCT could influence the performance of both adsorption and biodegradation in extent. Increasing EBCTs could make the equilibrium more complete for adsorption, thereby improving the performance. The ratio of adsorption to biodegradation on the BAC column increased as EBCT increased, and this implied that adsorption was dominant in an equilibrium condition.

Key Words : Adsorption, Biodegradation, Biological activated carbon (BAC), Granular activated carbon (GAC), Empty bed contact time (EBCT), Ozonation by-product (OBP)

INTRODUCTION

Ozone has been widely applied in water treatment to reduce of organic matter as well as chlorinated disinfection by-product precursors (Jacangelo *et al.*, 1989; Singer, 1990; Amy *et al.*, 1991; Galapate *et al.*, 2001). Unfortunately, numerous investigators have identified ozonation by-products (OBPs) as low molecular weight aliphatic aldehydes and mixed functional and saturated carboxylic acids (Gracia *et al.*, 1996; Weinberg and Glaze, 1997), which may be related to adverse health effects (Schechter and Singer, 1995). In addition, it is well established that ozonation can enhance biodegradation (DeWaters and DiGiano, 1990; Goel *et al.*, 1995; Urfer *et al.*, 1997; Cipparone *et al.*, 1997; Griffini *et al.*, 1999) and, hence, cause microbial re-growth in the distribution system (Lehtola *et al.*, 2001). Therefore, additional treatment processes should be considered to prevent these problems from occurring.

To date, the ozonation process is usually followed by granule activated carbon (GAC) or biofilter treatments, such as biological activated carbon

(BAC). Using BAC, which includes both adsorption and biodegradation mechanisms, to reduce OBPs has the advantages of lowering the regeneration cost and prolonging the useful life of GAC (Servais *et al.*, 1991, 1996; LeChevallier *et al.*, 1992). Besides the removal of the typical OBPs, the BAC process is also applied to treat other contaminants, such as non-ionogenic synthetic surfactants (Sirotkin *et al.*, 2001), ammonia (Andersson *et al.*, 2001), and bromate as source waters contained bromide ions and reacted with ozone (Kirisits *et al.*, 2001). However, one of the major difficulties in applying BAC is identifying and quantifying the contaminant removal mechanisms on the granules. The adsorption and biodegradation mechanisms overlap in the BAC bed, and it is difficult to recognize the predominant mechanism (Servais *et al.*, 1991). Chang and Rittmann (1987) developed a mathematical model of biofilm on activated carbon, which could quantify the extent of adsorption and biodegradation; however, it could not be applied under unsteady or plug-flow conditions. In an experimental study on column breakthrough, the capacities of GAC and BAC columns were derived,

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but the interaction between the two mechanisms still remains unclear (Carlson *et al.*, 1994).

On the other hand, when designing and operating a biofilter process, *i.e.*, BAC, one of key parameters is the empty bed contact time (EBCT). Hozalski *et al.* (1995) reported that when EBCT ranged from 4 to 20 min, this did not affect removal efficiency; that is, the removal efficiency did not vary significantly under a certain EBCT range. Urfer *et al.* (1997) showed that EBCT is the key parameter for biological organic matter removal, and within the typical EBCT range, the removal efficiency is independent of hydraulic loading rate.

Another issue related to the performance of biofilters is the biomass concentration. Previous studies reported that biomass concentration profiles in biofilter beds varied markedly with the influent mass flow of biodegradable organic matter; that is, higher biodegradable organic matter mass flow rates increased the concentration of the surface biomass and the depth of penetration (Carlson and Amy, 1998).

Because both adsorption and biodegradation can be affected by EBCT, hence, the objective of this research was to differentiate the quantities of adsorption and biodegradation of a BAC column, so as to assess the effect of EBCT on the removal of OBPs.

MATERIALS AND METHODS

Lab-scale column system

The effective length of each experimental column was 50 cm, and two 5-cm-diameter glass columns were employed. Tap water (total organic carbon and ammonia nitrogen < 0.1 mg/L) was pre-aerated for dechlorination and to saturate the dissolved oxygen (DO). The aerated water was pumped (Cole-Parmer) through a water bath heater (25°C) into a mixing tank along with the feed solution. The EBCTs were controlled at 2.5, 5, 10, 15, and 25 min. A schematic diagram of the experimental procedure is shown in Fig. 1.

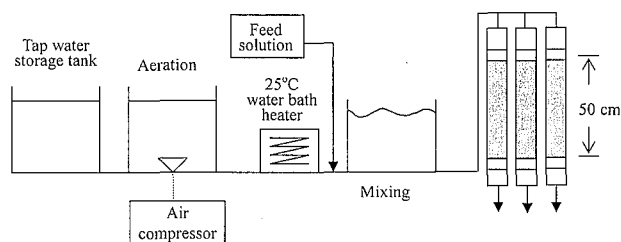


Fig. 1. Schematic flow chart of the experimental procedure.

Media and sample preparation

The packing media consisted of GAC and glass beads. The GAC used in this study was "SorboNorit 3" (Norit, crushed, and sieved, 2.4 to 2.0 mm size, with a bulk density of 0.31 g/cm³), and the glass beads were 2 mm in diameter. The media were washed in de-ionized water (Milli-Q SP) and then dried at 250°C for 24 hours. The GAC column was packed with the baked GAC with a dry weight of 304 g; the glass bead column and BAC column were packed with the glass beads and seeded GAC, respectively. For seeding, the media were immersed into natural pond water supplemented with feed solution; after 48 hours of aeration, the media were washed with de-ionized water to remove residual organics and then packed into the respective columns.

The feed solution consisted of four OBPs, including two aldehydes (formaldehyde and glyoxal, Sigma) and two ketoacids (glyoxalic and ketomalonic acid, Sigma); the initial concentrations were 200, 200, 400, and 400 µg/L, respectively. Mineral nutrients (KH₂PO₄, K₂HPO₄, Na₂HPO₄, KNO₃, MgSO₄, CaCl₂, FeCl₃, and NaHCO₃) were added in the feed solution.

Adsorption determination

The equilibrium adsorption capacity of a virgin GAC was evaluated using an isotherm test conducted at 25°C for four days. Glyoxal (with 10⁻³ M phosphate buffer, pH = 6.8 ± 0.2) was used as a target compound to represent the dissolved organic carbon (DOC). GAC granules form was crushed into powder to shorten the equilibrium time. The initial concentrations of glyoxal were 50 and 100 mg/L (as 20 and 41 mg DOC/L), and various GAC dosages (0.2 to 1.4 g) were added to yield equilibrium concentrations ranging from 0.1 to 30 mg DOC/L.

A similar procedure was used to calculate the residual adsorption capacities of the granular GAC and BAC granules: two cm³ media (with 0.62 g dry-weight) were removed from various depths of the experimental columns (0, 10, 20, and 30 cm from the top). After it was manually crushed, the sample was placed in a screw-cap bottle, with 50 mg/L glyoxal (20 mg/L as DOC), phosphate buffer and a small amount of HgCl₂. After 48 hours of shaking, the residual adsorption capacity (mg DOC/g-granules) was calculated as the difference between the initial and final solution concentrations.

Bioactivity and biomass determination

Media were removed from various depths of the testing columns and placed into 200-mL sterilized

saline buffer soaked in an ultrasonic vibration tank for the measurement of the viable biomass. The biomass was enumerated by means of heterotroph plate counting on tryptone glucose extract agar. The bioactivity of the media in terms of the removal of biodegradable organic matter was assessed using the respirometric method (biomass respiration potential, BRP) proposed by Urfer (Urfer and Huck, 2001). Briefly, a given amount of media was placed in an autoclaved BOD bottle (about 300 mL) filled with aerated tap water, and DO was measured using the feed solution and a DO-probe (Oxi330i, WTW). The DO was measured again after incubation for 5 hours on a shaker table, and the bioactivity was expressed as the difference of DO in mg O₂/L per cm³ media.

Analytical methods

Aldehydes, including formaldehyde and glyoxal, were analyzed using the method described in the Standard Methods (APHA, 1995). *O*-(2, 3, 4, 5, 6-pentafluorobenzyl)-hydroxylamine hydrochloride (PFBOA·HCl) reacted with carbonyl compounds in an aqueous solution to form corresponding oximes. Derivatives were then extracted with *n*-hexane. The extract was analyzed using GC-ECD (HP 5890) and a fused silica capillary column (DB-5, 30 m·0.25 mm ID, 1.0 m film thickness). The same procedure was modified and used to analyze the ketoacids but without buffer adding, and followed by an acidification procedure. Then 4 mL methyl tert-butyl ether (MTBE) was added to carry out extraction. The extract was then methylated using diazomethane in the MTBE solution. The excess diazomethane was quenching using silica gel, the samples were analyzed with GC-ECD. This procedure was also described in detail by Melin and Ødegaard (2000).

RESULTS and DISCUSSION

Determining the effect of EBCT on adsorption in the GAC column

The breakthrough curves of four OBPs of the GAC column are shown in Figs. 2(a) to 2(d). The results show that under low EBCT, the GAC column could not effectively remove OBPs. This was because the adsorption process was not at equilibrium, and will be proven and discussed later on. As a consequence, the effluent concentrations when EBCT = 2.5 min were higher than those in the higher EBCT case before breakthrough. Love and Eilers (1982) suggested that the performance of GAC in the removal of low molecular weight chlorinated hydrocarbons would be stable after 12 min of EBCT. Another study that used a rapid small-scale column test

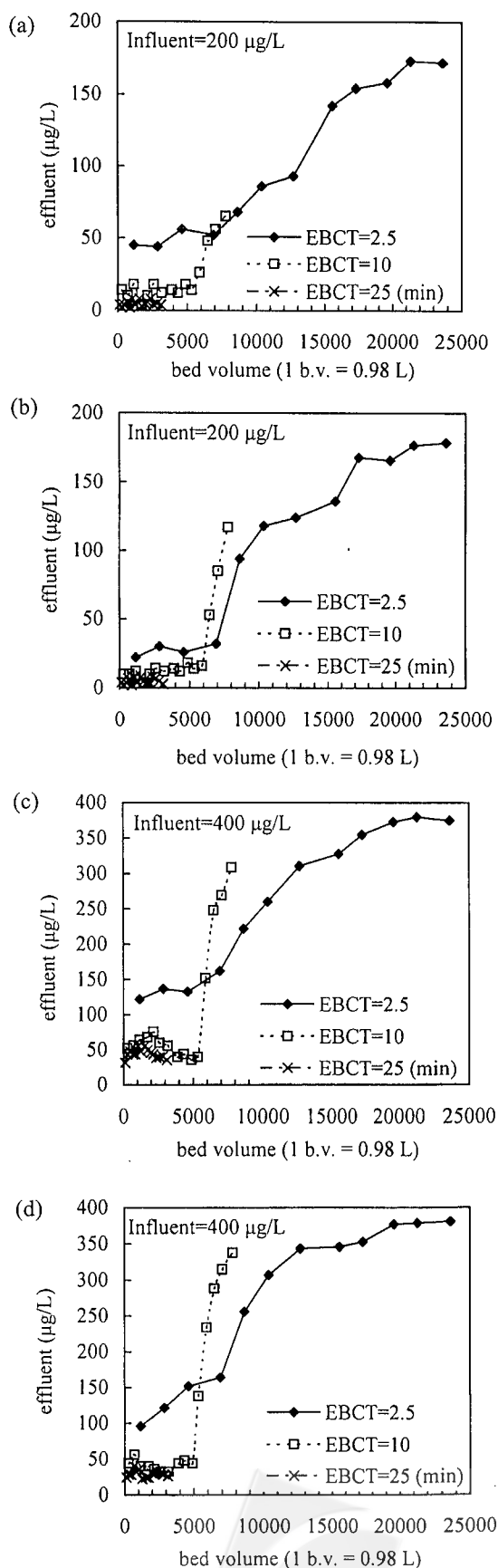


Fig. 2. Breakthrough curves of four OBPs of the GAC column. (a) Formaldehyde; (b) glyoxal; (c) glyoxalic acid; (d) ketomalonic acid.

also showed that there was no significant difference or a slight increase, in GAC in terms of either the bed life or the carbon usage rate as the EBCT ranged from 10 to 20 min (Summers *et al.*, 1995; Dvorak and Maher, 1999; Shih *et al.*, 2003).

To determine the adsorption capacities of the GAC granules, the residual adsorption capacity approach was used to calculate the amount of DOC that was adsorbed on the GAC. The quantity of DOC adsorbed on the GAC granules was defined as the difference between the residual adsorption capacity and the equilibrium adsorption capacity, which was derived from the result of the isotherm test. As for the GAC column, the amounts of DOC adsorbed on the GAC granules at various depths along the column are shown in Fig. 3. These amounts were multiplied by the total mass of the granules in the GAC column (g-GAC) to derive the total amount of DOC adsorbed. The results are shown in Table 1.

The mass balance for the adsorbed DOC in the GAC column is also shown in Table 1. In this research, the influent concentration was kept constant, so the cumulative DOC mass inflow (the third column from the left) was simply proportional to the operation time. The removed DOC from the liquid phase was determined as the difference between the influent and the effluent concentrations, and was then multiplied by the volume of treated water (the fourth column from the left). The mass balance results, represented as the recovery ratios, are shown in the last column of Table 1. In this research, the recovery ratios were in the $100 \pm 20\%$ ranges, and values more than 100% were probably due to the error of analytical process.

The profile of DOC adsorbed within the GAC column can provide information about the effect of EBCT on adsorption. Again, in Fig. 3(a), limited adsorption capacities remained after 12 days of operation (approximately 7,000 bed volume) while the GAC column was at breakthrough. This implies that at such EBCT values, the adsorption process did not reach equilibrium.

Determining the effect of EBCT on biodegradation and BRP in the glass bead column

The mechanism in a glass bead column is generally considered to be biodegradation only, and in this research, biodegradation changed from an unsteady to a steady state after approximately 2-3 weeks of operation when the effluent concentrations varied within a certain range. By then the effluent was represented as the equilibrium effluent concentration (S_{eq}), and are shown in Fig. 4. It is obvious that S_{eq} decreased as the value of EBCT increased, *i.e.*, S_{eq} changed from 46.4 to 26.7 $\mu\text{g/L}$ as EBCT increased from 2.5 to 25 min for formaldehyde. With regard to

the other three OBPs, glyoxal exhibited a trend similar to that of formaldehyde, but the average S_{eq} was greater, *i.e.*, 47.3 to 35.3 $\mu\text{g/L}$, than that of formaldehyde, which shows that the biodegradation rate of glyoxal was slower than that of formaldehyde. On the other hand, the S_{eq} values of the two ketoacids also exhibit a trend like that of the aldehydes case. That is, the S_{eq} value decreased as the value of EBCT increased (133.8 to 103.6 and 142.2 to 115.1 $\mu\text{g/L}$).

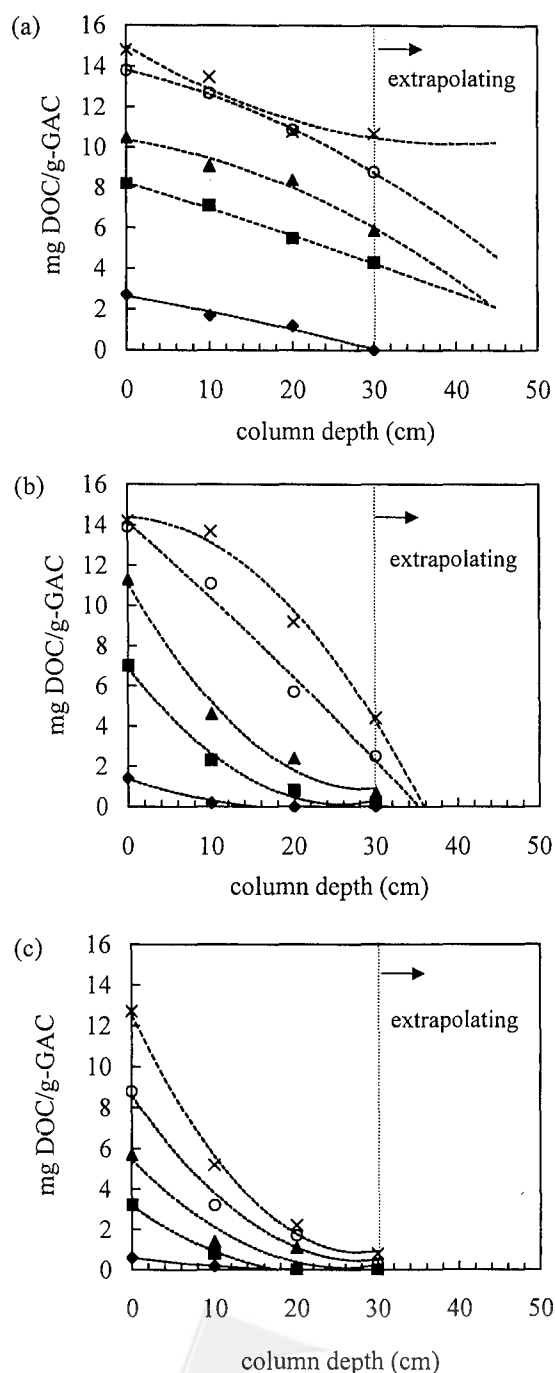


Fig. 3. Organic carbon adsorbed on the GAC granules under various EBCTs. (a) 2.5 min; (b) 10 min; (c) 25 min. (◆) 2 days; (■) 12 days; (▲) 22 days; (○) 37 days; (×) 54 days.

Table 1. Mass balance of organic carbon adsorbed on GAC.

EBCT (min)	Day (Bed volume)	DOC Influent (mg)	Measured DOC Reduction (mg) ^a	Estimated DOC Adsorption (mg)	Recovery (%) ^b
2.5	2 (1,152)	451	347	342	99
	12 (6,912)	2704	1927	1793	93
	22 (12,672)	4958	2774	2428	88
	37 (21,312)	8338	3279	3355	102
	54 (31,104)	12169	3519	3691	105
10	2 (288)	113	102	98	96
	12 (1,728)	676	609	641	105
	22 (3,168)	1239	1117	1202	108
	37 (5,328)	2084	1873	2178	116
	54 (7,776)	3042	2331	2800	120
25	2 (115)	45	43	49	114
	12 (691)	270	253	244	96
	22 (1,267)	496	463	500	108
	37 (2,131)	834	777	872	112
	54 (3,110)	1217	1137	1324	116

^a Difference between the influent and effluent concentrations times the treated volume.

^b Ratio of the estimated DOC adsorption to the measured DOC reduction.

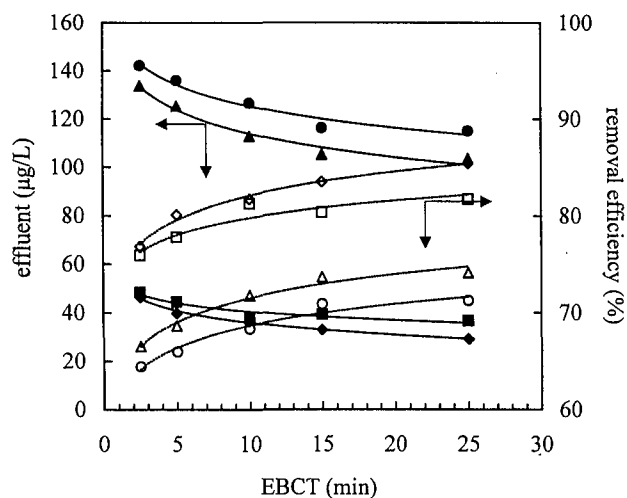


Fig. 4. Average effluent concentration (solid symbols) and removal efficiency (empty symbols) of the glass bead column. (◆) Formaldehyde; (■) glyoxal; (▲) glyoxalic acid; (●) ketomalonic acid.

The removal efficiencies of the glass bead column at steady state for the four OBPs are also shown in Fig. 4. In accordance to the trend for averaged effluents, the removal efficiencies increased as the EBCT increased. However, the increment was insignificant in comparison with the increase of the EBCT; *i.e.*, the EBCT increased from 2.5 to 25 min, whereas the removal efficiency increased only from 78 to 85%. It was reported by Rittmann and McCarty (1980) that there existed a minimum substrate concentration for maintaining the steady-state biofilm in biofilters, so that it could not reach a 100% removal efficiency under any EBCTs. The same author also reported that EBCT > 3.5 min had little effect on the

removal of dissolved organic carbon in the pilot filters treated with ozonated groundwater (Rittmann *et al.*, 2002). Melin and Ødegaard (2000) indicated that the optimum EBCT was around 20 min because a longer EBCT did not significantly increase efficiency. In this research, although increasing the EBCT could reduce the S_{eq} value and increase the removal efficiency, the improvement was insignificant when the EBCT was longer than 10 min.

The bacteria densities on the media of the glass bead column are shown in Fig. 5(a). The biomass at the top ($L = 0$ cm) of the column was much greater than that in the middle. On the other hand, the difference of the bacteria density between the top and the middle of the glass bead column increased as the EBCT increased. In studies on using natural water as the organic source, the amount of attached biomass decreased as the filter depth increased, suggesting that most of the biodegradation occurred at the top in the filter, and that the biomass concentration was related to the concentration of the organic substrate (Wang *et al.*, 1995). This result implies that higher organic loading could result in deeper microbial penetration in a biofilter.

Respirometric methods, such as measurement of the oxygen uptake rate, are commonly used ways to estimate the microbial metabolism, and most of the respirometric approaches are used in wastewater treatment or activated sludge processes. Urfer and Huck (2001) successfully employed the BRP method to determine the activity of biomass attached to drinking water biofilter media. It should be noted that the presence of anaerobic bacteria was ignored in this test because the amount of substrate degraded by potentially present anaerobic bacteria is likely to be minor compared to their aerobic counterparts.

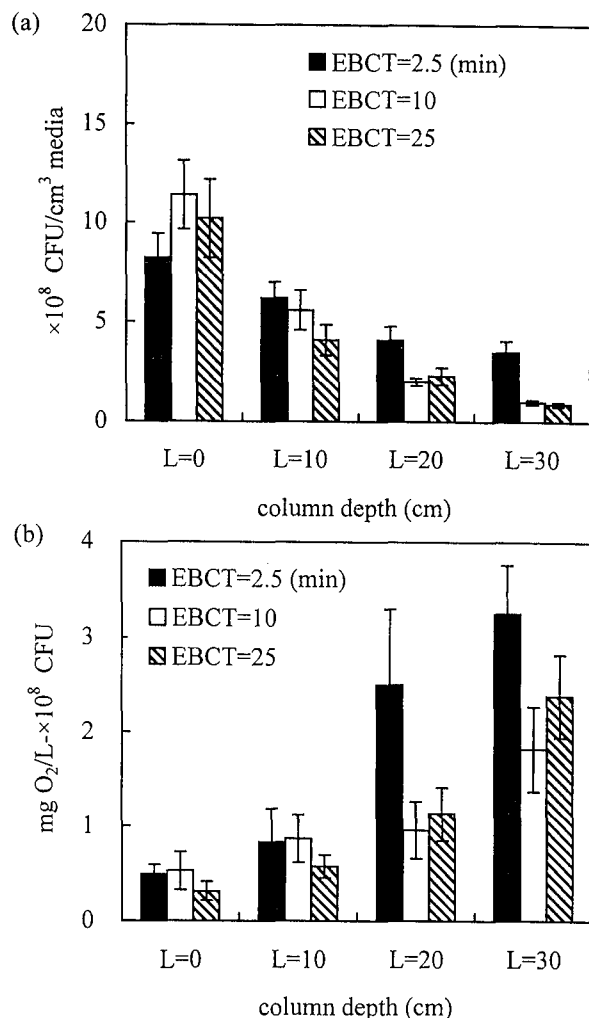


Fig. 5. The (a) bacterial densities and (b) specific BRP profiles of the glass bead column under various EBCTs (day = 37).

The specific biomass activity was calculated as the ratio of BRP to the bacterial density, and the results are shown in Fig. 5(b). The results are quite different from the profiles of the bacteria density; it is obvious that the specific BRP tended to increase with the column depth, suggesting that the bacteria located deeper in the column was more active with respect to DOC substrate removal. Generally, the magnitude of BRP depends not only on the biomass quantity, but also on the composition of the microbe itself. The structure heterogeneity of certain biofilms leading to differences in biomass activity was reported by Yang and Lewandowski (1995). Liu and Tay (2002) indicated that the hydrodynamic shear force has a significant impact on the metabolic behavior of biofilms, especially in increasing the magnitude of catabolic activity.

Effects of EBCT on the combination of adsorption/biodegradation in the BAC column

The effluents of the BAC column are shown in

Fig. 6. Even under low EBCT conditions, the effluents of the four OBPs all stayed below a certain level, *i.e.*, 24, 26, 71, and 56 $\mu\text{g/L}$, respectively, and were less than the S_{eq} values of the glass bead column. The removal efficiencies of the BAC column under various EBCTs are also shown in Fig. 6. It should be mentioned that the improvement in the removal efficiency achieved by increasing the EBCT was more significant than that achieved in the glass bead column; *e.g.*, the removal efficiency for formaldehyde improved from 88 to 98%. This improvement may be attributed to the equilibrium of adsorption achieved by increasing the contact time.

The same procedure used to determine the adsorption capacities was employed to calculate the DOC removal by means of adsorption in the BAC column, and the results are shown in Fig. 7. The adsorbed DOC on BAC granules was less than that in the GAC column. Furthermore, the liquid-phase substrate concentration in the BAC column was lower than that in the GAC column because of biological uptake; consequently, the equilibrium concentration of the BAC granules in the solid-phase was lower than that of the GAC granules. It was reported that when GAC was used as media, the adsorbed substances could enhance the bacterial activity (Saito *et al.*, 1996). In contrast, the adsorption capacity of the GAC regenerated by bacterial activity was low in comparison to that of virgin GAC, which implied that bacteria could not fully utilize the substances adsorbed on GAC (Nakano *et al.*, 2000). Either way, in this study biodegradation did reduce the organic loading of the granular.

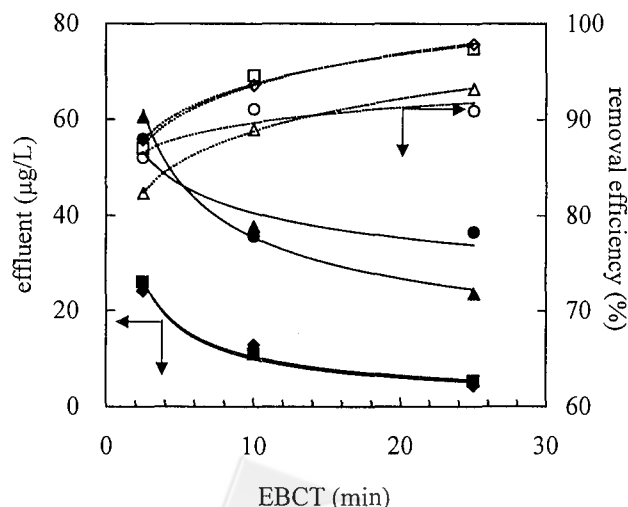


Fig. 6. Average effluent concentration (solid symbols) and removal efficiency (empty symbols) of the BAC column. (\blacklozenge) Formaldehyde; (\blacksquare) glyoxal; (\blacktriangle) glyoxalic acid; (\bullet) ketomalonic acid.

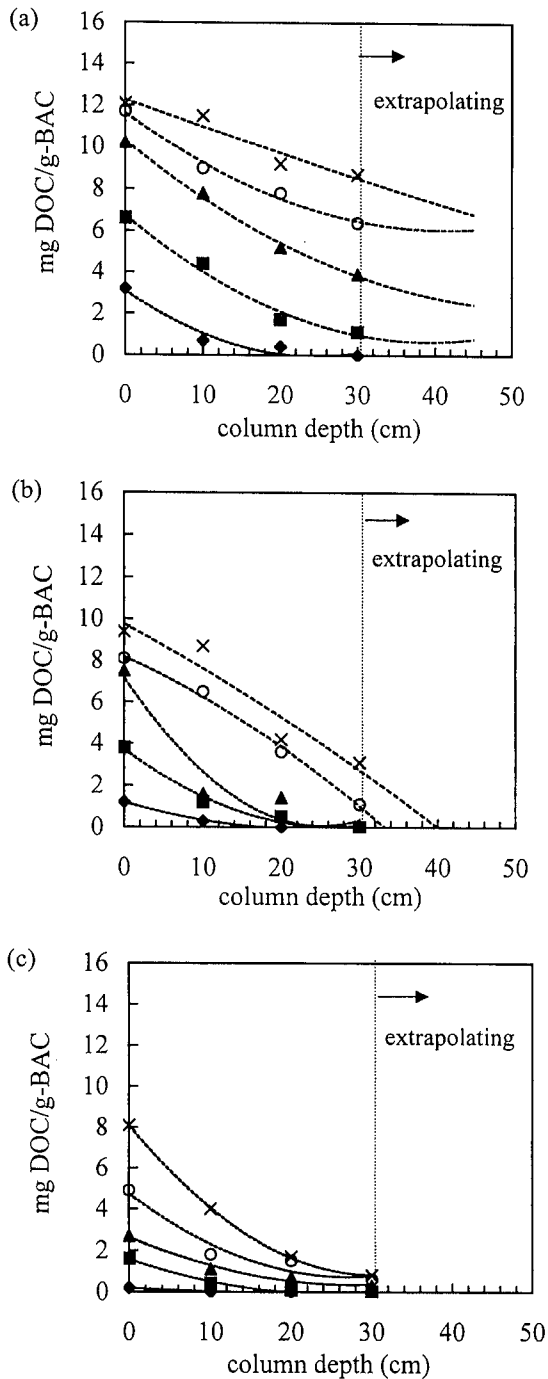


Fig. 7. Organic carbon adsorbed on the BAC granules under various EBCTs. (a) 2.5 min; (b) 10 min; (c) 25 min. (◆) 2 days; (■) 12 days; (▲) 22 days; (○) 37 days; (×) 54 days.

As the adsorption quantities were determined, the amount of biodegraded DOC could be determined. The adsorption and biodegradation quantities and their ratio are shown in Fig. 8. At low EBCT, as shown in Fig. 8(a), the amount of biodegradation exceeded that of adsorption. The fraction of biodegradation increased with time, revealing that the biodegradation mechanism began to prevail, although there remained limited adsorption capacity. How-

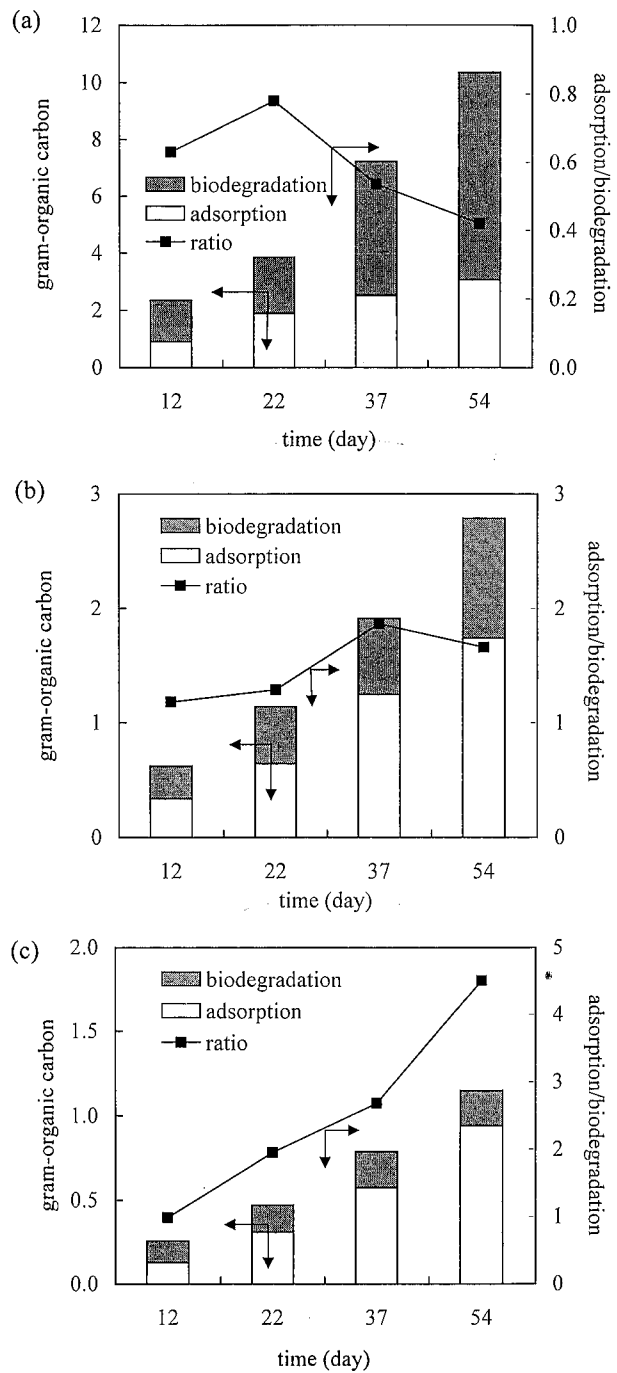


Fig. 8. Organic carbon removal through adsorption and biodegradation in the BAC column under various EBCTs. (a) 2.5 min; (b) 10 min; (c) 25 min.

ever, in this research, the dominance of biodegradation under low EBCT conditions was probably due to the adsorption disequilibrium. On the other hand, adsorption was the predominant mechanism for the removal of organic carbon at EBCT 10 and 25 min, as shown in Figs. 8(b) and 8(c). It seems that adsorption was the major mechanism by which organics were removed at high EBCT, and that biodegradation was quite insignificant in comparison with adsorption.

CONCLUSION

The results show that EBCT could influence the performance of both adsorption and biodegradation in extent. Increasing EBCT could make the equilibrium more complete for adsorption, thereby improving the performance. In addition, EBCT could also affect the quantity and the structure heterogeneity of the biofilm attaching on the media. In general, the ratio of adsorption to biodegradation on the BAC column increased as EBCT increased, and the result implies that adsorption was dominant in an equilibrium condition.

ACKNOWLEDGEMENT

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NOMENCLATURE

BAC	biological activated carbon
BRP	biomass respiration potential
DO	dissolved oxygen
DOC	dissolved organic carbon
EBCT	empty bed contact time, min
GAC	granular activated carbon
OBPs	ozonation by-products
S_{eq}	average effluent concentration at steady state ($\mu\text{g/L}$)

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生物活性碳吸附與生物降解定量之研究

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摘 要

本研究利用連續式管柱試驗，搭配四種典型臭氧副產物做為目標去除污染物，區分並定量生物活性碳上吸附與生物降解之量；並探討空床接觸時間對上述兩種機制之影響。由分別定量吸附及生物降解之結果顯示，生物活性碳上吸附對生物降解之比例，會因增加空床接觸時間而增加，此結果並暗示吸附平衡對於生物活性碳整體表現而言具有較深遠之影響。

