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Bioregeneration of granular activated carbon loaded with binary mixture of phenol and 4-chlorophenol

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ABSTRACT

The extent of bioregeneration of granular activated carbon (GAC) loaded with binary mixture of phenol and 4-chlorophenol (4-CP) was investigated. Batch studies were conducted at (i) different initial phenol and 4-CP loading concentrations and (ii) various GAC dosages to examine the effects of different ratios of phenol to 4-CP adsorbed (Q^f_{ph} to Q^f_{4-CP}) on bioregeneration efficiency. The bioregeneration efficiency was higher when the Q^f_{ph} to Q^f_{4-CP} ratio was higher due to lower adsorption irreversibility of phenol. Increasing the GAC dosage under saturation condition at the same initial conditions only marginally improved the bioregeneration efficiency. The characteristics of the adsorption process (i.e. the irreversibility of adsorption) of each adsorbate are an important factor influencing the extent of bioregeneration in binary component-loaded GAC.

Keywords: Bioregeneration; Activated carbon; Binary system; Phenol; 4-Chlorophenol

1. Introduction

Phenol and its derivatives are environmentally persistent and carcinogenic compounds that can exert pernicious effect on human health and aquatic life [1,2]. One of the most effective methods to remove phenolic compounds from wastewaters is by adsorption using activated carbon. However, due to the nature of the adsorption process, the active sites on the adsorbent will eventually be exhausted and regeneration of the spent adsorbents is necessary to prolong its lifetime. Various regeneration methods reported in the literature include the use of ultrasound, chemical, and biological regeneration [3–6]. Among these methods, biological regeneration is viewed as a promising and eco-friendly approach offering the prospect of a complete mineralization of the adsorbed pollutants to innocuous products at relatively low cost.

Bioregeneration involves the use of microorganisms to renew the active sites of the spent adsorbent. To date, almost all the reported bioregeneration studies involved the regeneration of adsorbents loaded with a single pollutant [7–9]. Thus, studies on the bioregeneration of activated carbon loaded with multi-pollutants are warranted as wastewaters do contain more than one type of pollutants. Previously, Ha et al. [10] quantified the extent of bioregeneration of activated carbon loaded with a binary mixture of phenol

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and 2,4-dichlorophenol and showed that longer residence time led to higher bioregeneration efficiency. The reported regeneration efficiencies for phenol and 2,4-dichlorophenol by Ha et al. were between 43 and 48%. Aktaş and Çeçen [11,12] observed that activated carbons loaded with a mixture of 2-chlorophenol and 2-nitrophenol were cometabolically regenerated in the presence of phenol. The reported regeneration efficiencies of phenol, 2-nitrophenol, and 2-chlorophenol by Aktaş and Çeçen [11,12] were between 19.5% and 89.8% depending on the activated carbon type. Nevertheless, none of these studies addressed the effect of the ratio of the amounts of adsorbed substrates on the extent of bioregeneration. As there are differences in the characteristics of adsorption and biodegradation of different substrates, different ratios of the amounts of adsorbed substrates could influence the bioregeneration efficiency.

Thus, the objective of this study is to compare and quantify the extent of bioregeneration of granular activated carbon (GAC) loaded with both phenol and 4-chlorophenol (4-CP) under the sequential adsorption and biodegradation (two-step) and simultaneous adsorption and biodegradation (one-step) approaches. The two-step approach was adopted to investigate the extent of spent GAC regeneration by biological treatment reflecting a typical adsorption–regeneration process. The one-step approach was adopted to investigate the reusability of the GAC during simultaneous adsorption and biodegradation processes which is applicable to systems such as the biological activated carbon and adsorbent-supplemented sequencing batch reactor (SBR).

2. Materials and methods

2.1. Adsorbent and chemicals

The GAC used in this study was purchased from Japan Enviro Chemicals Ltd. under the commercial name of Shirasagi X7100 Dry. The GAC was a thermally activated, wood-based GAC sieved to the size of 10–20 mesh. The GAC was dried in an oven at 103˚C to remove the moisture and kept in the desiccator prior to use. The phenol and 4-CP used were of synthesis grade with more than 98% purity, while all other chemicals used were of analytical grade. All the chemicals were used without further purification.

2.2. Culturing of biomass acclimated to both phenol and 4-CP

A SBR with a working volume of 4 L was employed to culture the biomass acclimated to both phenol and 4-CP. The seed of the biomass was obtained from a local municipal wastewater treatment plant. The operation of the SBR was conducted in five sequential periods, namely FILL, REACT (aerobic), SETTLE, DRAW, and IDLE, in the time ratio of 1:8:1.5:1:0.5, respectively, for a cycle time of 12 h. During an operational cycle, 3 L of the synthetic wastewater was introduced into the SBR during the FILL period followed by the REACT period during which aeration was applied to the SBR via an air pump. During the REACT period, the SBR was agitated using an ejector to provide homogeneous mixing of the synthetic wastewater and biomass. The average temperature and dissolved oxygen concentration were 30 ± 2˚C and >7 mg L^{-1}, respectively. Then, the sludge was allowed to settle (SETTLE period) before 3 L of the treated effluent was drawn out from the reactor during the DRAW period. Acclimation of the biomass to both phenol and 4-CP was achieved by gradually increasing the concentrations of the two compounds up to the final concentrations of 3.19 and 2.33 mmol L^{-1}, respectively. The final composition of the synthetic wastewater consisted of (in mmol L^{-1}): phenol (3.19), 4-CP (2.33), $(NH_4)_2SO_4$ (2.81), KH_2PO_4 (0.41), K_2HPO_4 (1.81), $NaHCO_3$ (7.37), $MgSO_4$ (0.71), $CaCl_2$ and $FeCl_3{\cdot}6H_2O$ (0.12).

2.3. Bioregeneration studies

The bioregeneration experiments were carried out using the one-step and two-step adsorption and biodegradation approaches. For the two-step approach, the GAC was first loaded with phenol and 4-CP before regeneration. Exactly, 0.05 g of GAC was weighed and placed into a series of amber glass reaction vessels. The GAC in each of the reaction vessels was then mixed with 50 mL of a solution containing phenol and 4-CP of various concentrations. After mixing, the reaction bottles were immediately agitated using an orbital shaker (IKA Basic 260) at 250 rpm and 25˚C for a period of 24 h. Once the adsorption equilibrium had been attained, the GAC was separated from the supernatant by decantation and the concentrations of phenol and 4-CP were determined in accordance to the method described by Wu and Yu [13] using an UV/vis spectrophotometer at the λ_{max} of 286 and 299 nm for phenol and 4-CP, respectively. The separation of GAC from the solution by decantation was feasible due to the relatively large GAC particles (10–20 mesh) and excellent settleability of the GAC by the gravity (<2 min). The amounts of phenol and 4-CP adsorbed on GAC (in mmol g^{-1}) were calculated, respectively, using Eq. (1):

$$Q = \frac{(C_0 - C_e)V}{m} \quad (1)$$

where C_0 (mmol L^{-1}) and C_e (mmol L^{-1}) are the initial and equilibrium concentrations of adsorbate, V (L) is the volume of the solution and m (g) is the weight of GAC. Then, the GAC loaded with both phenol and 4-CP was used for the regeneration study. To prepare the acclimated biomass for bioregeneration study, the mixed liquor from the SBR was collected and the mixed liquor-suspended solids (MLSS) concentration was determined. From the MLSS concentration in the SBR (average of 3,000 mg L^{-1}), a solution containing 300 mg L^{-1} of acclimated biomass with nutrients similar to the SBR composition was prepared. Exactly, 50 mL of the solution was transferred into each of a series of reaction vessels containing the phenol- and 4-CP-loaded GAC and the reaction vessels were agitated for 72 h. For the one-step approach, a solution containing phenol and 4-CP at various initial concentrations, 300 mg L^{-1} of acclimated biomass and fresh GAC were shaken in each of a series of reaction vessels for 72 h.

For both approaches, the residual concentrations of phenol and 4-CP in the bulk solution were determined at suitable time intervals by decanting the supernatant from the reaction vessel. Due to the difference in the settle ability of the biomass and GAC, most of the biomass was removed during decantation. After 72 h, the content in the reaction vessel was filtered using a GF/C filter medium, and the regenerated GAC was then autoclaved to deactivate the biomass. Similar to the previous study [14], the contribution of adsorption of phenolic compounds by the deactivated biomass was found to be negligible as most of the biomass was removed during decantation. After autoclaving, the regenerated GAC was resuspended into a solution containing the same initial concentrations of phenol and 4-CP used for the loading of fresh GAC and shaken for 24 h. Then, the residual concentrations of phenol and 4-CP were determined and the amounts of phenol (Q_{ph}) and 4-CP adsorbed ($Q_{4\text{-CP}}$) were calculated using Eq. (1). Each of the experiments was conducted in triplicate.

The effect of GAC dosage on bioregeneration efficiency was also investigated using the two-step approach. The dosages of GAC used were 1.0, 1.5, and 2.0 g L^{-1} at various Q_{ph} to $Q_{4\text{-CP}}$ ratios.

3. Results and discussion

3.1. Adsorption of GAC for phenol and 4-CP

The maximum GAC adsorption capacities for phenol and 4-CP were found to be 2.4 and 2.6 mmol g^{-1}, respectively. The adsorption mechanisms of phenol and 4-CP are very similar (e.g. π–π dispersive interactions and donor–acceptor complex formation) and both phenolic compounds compete for the same active sites for adsorption [15]. The 4-CP has a stronger affinity to be adsorbed than phenol due to its higher electronegativity and thermodynamically stronger π–π interaction strength.

3.2. Bioregeneration studies

The bioregeneration efficiency of phenol- and 4-CP-loaded GAC for either phenol or 4-CP, as calculated from the ratio between the amounts adsorbed before and after bioregeneration, is difficult to be determined by the direct reloading method. This is because the 4-CP molecule, which has a stronger affinity for GAC adsorption compared with phenol molecule, can occupy the regenerated phenol site during the reloading process. The occupancy of 4-CP into the regenerated phenol sites leads to erroneous results of having higher bioregeneration efficiency of the 4-CP than its actual value. It is more challenging to determine the regeneration efficiency in the one-step approach of either phenol or 4-CP since the adsorption affinities and biodegradation rates of phenol and 4-CP are very different. It is therefore proposed that the bioregeneration efficiency be calculated based on the ratio between the sums of the amounts of phenol and 4-CP adsorbed ($Q_{ph} + Q_{4\text{-CP}}$) (in mmol g^{-1}) in the bioregenerated and fresh GAC as shown in Eq. (2):

$$\%\,\text{Bioregeneration} = \frac{(Q_{ph} + Q_{4-CP})_{biot}}{(Q^f_{ph} + Q^f_{4-CP})_{pris}} \times 100\% \quad (2)$$

where $(Q_{ph} + Q_{4\text{-CP}})_{biot}$ and $(Q^f_{ph} + Q^f_{4-CP})_{pris}$ are the total amounts adsorbed in the bioregenerated and fresh GAC, respectively, whereas Q^f_{ph} and Q^f_{4-CP} are the amounts of phenol and 4-CP adsorbed in fresh GAC, respectively. In this regards, the bioregeneration efficiency of the spent GAC can be quantified under the (i) sequential adsorption and biodegradation and (ii) simultaneous adsorption and biodegradation approaches. Schematic illustrations of the two bioregeneration approaches are presented in Fig. 1.

3.2.1. Effect of initial phenol and 4-CP concentrations

3.2.1.1. Two-step approach. In the two-step approach, the residual phenol and 4-CP concentrations were found to be lower than 0.11 mmol L^{-1} with no obvious trend for all cases and reached below 0.01 mg L^{-1} at the end of the bioregeneration process. The calculated

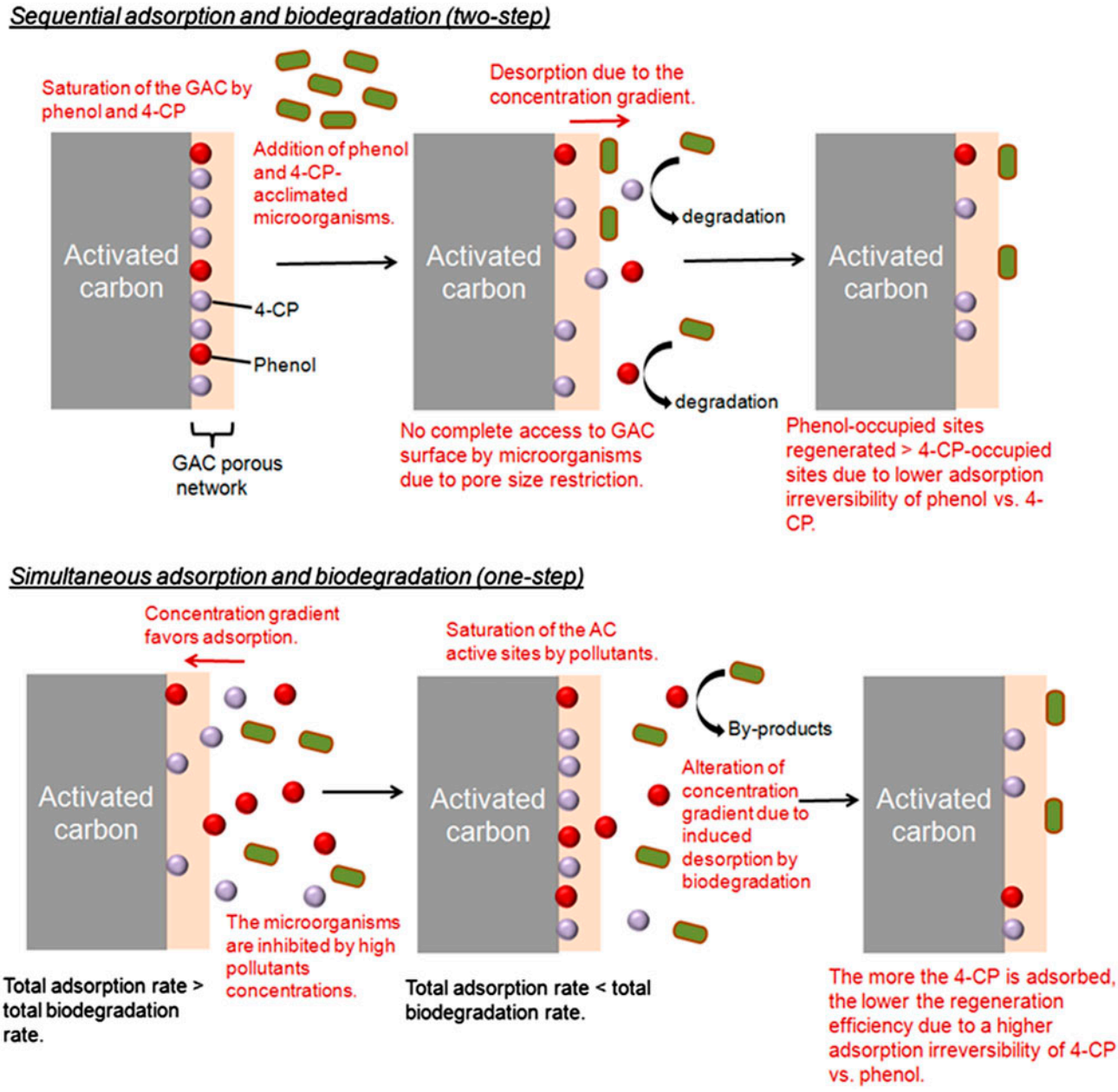

Fig. 1. Schematic illustration of the two approaches of bioregeneration of GAC loaded with phenol and 4-CP.

bioregeneration efficiencies of GAC loaded with both phenol and 4-CP based on Eq. (2) are presented in Table 1. At constant GAC dosage of 1 g L^{-1}, the initial phenol and 4-CP concentrations were the loading concentrations which determined the Q^{f}_{ph} and Q^{f}_{4-CP} values and hence their ratios. It is observed that the decrease in the Q^{f}_{ph} to Q^{f}_{4-CP} ratio from 1.8 to 0.2 led to the decrease in the bioregeneration efficiency from 65 ± 2 to 50 ± 4%. This suggests that phenol-occupied sites are relatively easier to be regenerated than 4-CP-occupied sites. The bioregeneration efficiency of GAC loaded with binary phenolic compounds is dependent on the degree of irreversibility of each adsorbate. In this case, the 4-CP molecules are more susceptible to the oxidative coupling reaction on the surface of the GAC resulting in more irreversible depletion of the active sites. Oxidative coupling reactions of phenolic compounds are recognized as the main culprit for the decrease in the adsorption capacity of bioregenerated adsorbents [9]. Since all the desorbable phenol and 4-CP will be desorbed and degraded, the final bioregeneration efficiencies of the GAC loaded with the

Table 1
Bioregeneration efficiencies of GAC loaded with different Q^f_{ph} to Q^f_{4-CP} ratios under two-step approach at constant GAC dosage of 1 g L^{-1}

Initial concentration (mmol L^{-1})		$(Q^f_{ph} + Q^f_{4-CP})_{pris}$ (mmol g^{-1})	Q^f_{ph} to Q^f_{4-CP} ratio	$(Q_{ph} + Q_{4\text{-}CP})_{biot}$ (mmol g^{-1})	Bioregeneration efficiency (%)
Phenol	4-CP				
3.19	0.78	2.21 ± 0.05	1.8	1.44 ± 0.02	65 ± 2
3.19	1.56	2.15 ± 0.12	0.7	1.24 ± 0.04	58 ± 5
3.19	2.33	2.33 ± 0.04	0.4	1.26 ± 0.12	52 ± 4
2.13	2.33	2.42 ± 0.09	0.3	1.27 ± 0.04	52 ± 3
1.06	2.33	2.29 ± 0.06	0.2	1.14 ± 0.05	50 ± 4

individual components corresponds to the adsorption irreversibility. The bioregeneration efficiencies of GAC loaded with a single component of phenol and 4-CP are 57 ± 3 and 44 ± 2%, respectively, further supporting the contention that the 4-CP is more irreversibly adsorbed than phenol. Based on this observation, it can be construed that the higher the adsorption irreversibility of the adsorbate, the lower the regeneration efficiency.

3.2.1.2. One-step approach. The time courses of residual phenol and 4-CP concentrations at various ratios of their initial concentrations in the one-step approach are shown in Fig. 2(a) and (b), respectively. Although the acclimated biomass was used for this study, the inhibition of biodegradation is expected due to the employment of a lower biomass concentration in the bioregeneration study (300 mg L^{-1}) compared with the SBR (3,000 mg L^{-1}). Comparison of the time courses of the phenol and 4-CP degradation showed that all the phenol degradation curves (Fig. 2(a)) exhibited a hump indicating the inhibitory effect of the relatively high concentration of either 4-CP or phenol on phenol degradation. In contrast, the hump is not obvious in all the 4-CP degradation curves (Fig. 2(b)) due to two reasons. First, the presence of phenol as a cosubstrate could enhance the biodegradation of 4-CP via cometabolism that could enhance the removal of toxic compounds such as 4-CP [2,16]. The second reason is the higher adsorption affinity of the GAC for 4-CP adsorption rapidly reduces the concentration of 4-CP in the solution and hence the inhibitory effect on phenol and 4-CP biodegradation.

Table 2 shows that the bioregeneration efficiencies of GAC loaded with both phenol and 4-CP under the one-step approach were generally lower than those under the two-step approach. This could be explained by the higher residual phenol and 4-CP concentrations under the former approach resulting in an increase in irreversibility due to the increase of contact time between the desorbed phenolic compounds and the GAC surface.

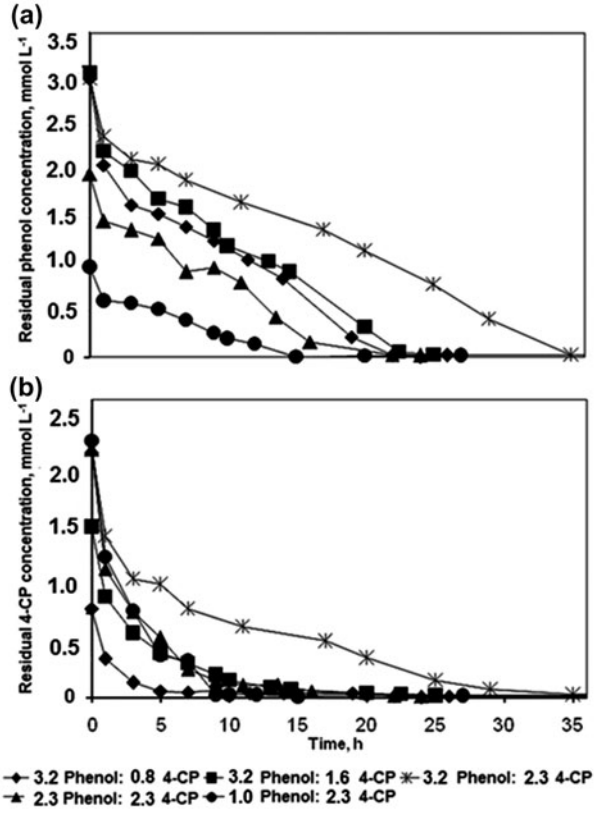

Fig. 2. Time courses of the (a) residual phenol and (b) 4-CP concentrations at various initial concentrations of phenol and 4-CP during the simultaneous adsorption and biodegradation processes.

3.2.2. Effect of GAC dosage

The effect of different GAC dosages on the bioregeneration efficiency under the two-step approach is shown in Table 3. The Q^f_{ph} to Q^f_{4-CP} ratio was varied by using different (i) initial loading concentrations of

Table 2
Bioregeneration efficiencies of GAC loaded with different Q^f_{ph} to Q^f_{4-CP} ratios under one-step approach at constant GAC dosage of 1 g L^{-1}

Initial concentration (mmol L^{-1})		Ratio of initial phenol concentration with initial 4-CP concentration	$(Q^f_{ph} + Q^f_{4-CP})_{pris}$ (mmol g^{-1})	$(Q_{ph} + Q_{4\text{-}CP})_{biot}$ (mmolg^{-1})	Bioregeneration efficiency (%)
Phenol	4-CP				
3.19	0.78	4.09	2.21 ± 0.05	0.54 ± 0.09	51 ± 5
3.19	1.56	2.04	2.15 ± 0.13	1.19 ± 0.05	55 ± 5
3.19	2.33	1.37	3.33 ± 0.04	1.03 ± 0.08	44 ± 4
2.13	2.33	0.91	2.42 ± 0.09	1.09 ± 0.12	45 ± 5
1.06	2.33	0.45	2.29 ± 0.06	1.01 ± 0.06	44 ± 4

Table 3
Bioregeneration efficiencies of GAC loaded with different initial concentrations of phenol and 4-CP at various GAC dosages under two-step approach

Initial loading concentration (mmol L^{-1})		GAC dosage (g L^{-1})	$(Q^f_{ph} + Q^f_{4-CP})_{pris}$ (mmol g^{-1})	Q^f_{ph} to Q^f_{4-CP} ratio	$(Q_{ph} + Q_{4\text{-}CP})_{biot}$ (mmol g^{-1})	Bioregeneration efficiency (%)
Phenol	4-CP					
2.13	4.67	1.0	2.78 ± 0.05	0.20	1.28 ± 0.04	46 ± 2
		1.5	2.58 ± 0.08	0.22	1.24 ± 0.06	48 ± 4
		2.0	2.44 ± 0.03	0.24	1.25 ± 0.03	52 ± 2
4.25	3.11	1.0	2.9 ± 0.05	0.49	1.43 ± 0.05	49 ± 2
		1.5	2.62 ± 0.04	0.56	1.37 ± 0.05	52 ± 3
		2.0	2.45 ± 0.05	0.73	1.31 ± 0.06	53 ± 3
6.38	1.56	1.0	2.29 ± 0.05	1.17	1.42 ± 0.08	62 ± 5
		1.5	2.25 ± 0.03	1.47	1.48 ± 0.07	66 ± 4
		2.0	2.14 ± 0.03	1.86	1.36 ± 0.06	63 ± 4

phenol and 4-CP and (ii) GAC dosages. Initial loading of GAC was conducted under excess adsorbate conditions and GAC saturation was achieved in all cases. It was observed that the bioregeneration efficiency generally increased with the increase in the Q^f_{ph} to Q^f_{4-CP} ratio following the same trend as explained in Section 3.2.1.1. At constant ratio of the loading concentrations, Table 3 shows that the increase in GAC dosage resulted in a marginal increase in bioregeneration efficiency. At lower GAC dosage, higher amount of 4-CP was removed by adsorption due to the better 4-CP adsorption affinity compared with phenol resulting in the decrease in the Q^f_{ph} to Q^f_{4-CP} ratio. This also leads to lower bioregeneration efficiency due to higher adsorption irreversibility. With increasing GAC dosage, competition for the active sites between 4-CP and phenol became less drastic since there are many active sites available for 4-CP and phenol adsorption. With more phenol occupying the active sites, the Q^f_{ph} to Q^f_{4-CP} ratio increased leading to higher bioregeneration efficiencies due to a lower degree of adsorption irreversibility.

4. Conclusions

The bioregeneration efficiencies of GAC loaded with both phenol and 4-CP under two-step and one-step approaches were determined. Under the two-step approach, the bioregeneration efficiency was found to be affected by the Q^f_{ph} to Q^f_{4-CP} ratio with higher value of the ratio leading to higher bioregeneration efficiency. Under GAC saturation condition, increasing the amount of GAC at constant initial phenol and 4-CP loading concentrations led to marginal increase in the bioregeneration efficiency. The characteristic of the adsorption process (i.e. degree of irreversibility of each component) is an important factor influencing the extent of bioregeneration of binary components-loaded GAC.

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