

Gaseous TCE and PCE Degradation with or without a Nonionic Surfactant

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Abstract

This study was conducted to investigate the biodegradation of gaseous trichloroethylene (TCE) and tetrachloroethylene (PCE) in an activated carbon biofilter inoculated with phenol-oxidizing microorganisms and to study the effect of surfactant concentration below its critical micelle concentration (CMC) on the re-moval efficiency of TCE or PCE. The investigation was conducted using two specially built stainless steel biofilters, one for TCE and the other for PCE, at residence times of 1.5~7 min.

The removal efficiency of gaseous TCE was 100% at a residence time of 7 min and its average inlet concentration of 85 ppm. For gaseous PCE, 100% removal efficiency was obtained at residence times of 4~7 min and its average concentrations of 47~84 ppm. It was found that adsorption by GAC was a minor mechanism for TCE and PCE removal in the activated carbon biofilters. Transformation yields of gaseous TCE and PCE were about 8~48 g of TCE/g of phenol and 6~25 g of PCE/g of phenol, according to residence times. This values showed one or two orders of magnitude less than aqueous TCE degradation. The TCE and PCE activated carbon biofilter performances were observed to be a little enhanced but not significantly, when the surfactant was introduced at concentrations of 5~50 mg/L.

Key words : activated carbon biofilter, trichloroethylene, tetrachloroethylene, cometabolism, surfactant, phenol-oxidizing microorganisms

1. Introduction

Many chlorinated aliphatic hydrocarbons (CAHs) have been detected in contaminated groundwaters and waste disposal sites in the United States (Opatken *et al.*, 1989; Turner, 1989; Westrick *et al.*, 1984; Symons *et al.*, 1975). As a result of treatment of such hydrocarbons, off-gases are generated from air strippers for groundwater decontamination and from vapor extraction systems for soil decontamination. Most of the CAHs are detrimental to hu-

man health and hence needed to be remediated.

Since the early 1980s, biofiltration has been widely applied to the treatment of organic off-gases containing biodegradable volatile organic compounds (VOCs) (Ottengraf, 1986). Biofiltration method employing microorganisms on a filter material has been known to be a reliable and cost-effective technology for the treatment of odor-causing VOCs. Various types of biofilters have been developed using different filter materials. The filter materials for the biofilters were made from either natural or syn-

thetic materials (Prokop and Bohn, 1985). Because of the limitations of biofilters using natural materials such as soil or compost, a new and improved approach is needed to remove trichloroethylene (TCE) and tetrachloroethylene (PCE).

Although TCE is the most frequently detected compound in contaminated groundwaters and soils along with PCE, few biofiltration studies have been conducted for gaseous TCE and PCE removals due to their recalcitrant characteristics. Many researchers have reported that cometabolism is responsible for the degradation of TCE and PCE (Oldenhuis *et al.*, 1991; Folsom *et al.*, 1990; Winter *et al.*, 1989; Wackett and Gibson, 1988). Microorganisms utilizing primary substrates such as methane, phenol, or toluene produce non-specific enzymes that can degrade the compounds.

Surfactants are frequently used for the remediation of hydrophobic organic compounds in contaminated soils and sediments (Churchill *et al.*, 1993; Aronstein and Alexander, 1992; Oberbremer *et al.*, 1990). Aronstein and Alexander (1992) reported that nonionic surfactants stimulate the solubilization or desorption of the compounds and that the solubilized molecules are degraded by microorganisms in the aqueous phase. Edwards *et al.* (1994) reported that a surfactant, when applied to soil, increased the equivalent fractional organic contents, which tended to increase the amount of hydrophobic organic compound sorbed. Surfactant molecules sorbed onto the activated carbon may stimulate biodegradation. Fountain *et al.* (1991) found the optimal hydrophilic/lipophilic balance (HLB) number for TCE and PCE were 14 and 15 in order to achieve the maximum solubilization.

The objectives of this study were to investigate the biodegradation of gaseous TCE and PCE in an activated carbon biofilter inoculated with phenol-oxidizing microorganisms and to study the effect of surfactant concentrations below its critical micelle concentration (CMC)

on the removal efficiency of TCE or PCE. For the enhanced biofiltration process, granular activated carbon (GAC), phenol-oxidizing microorganisms, or a surfactant were introduced in order to increase the biodegradation of gaseous TCE and PCE. Activated carbon biofilter was introduced, because GAC was capable of providing several advantages such as sufficient surface area and high porosity (Leson and Winer, 1991). In addition, the activated carbon biofilters have low pressure drops due to no sloughing and do not require the replacement of the filter material (Liu *et al.*, 1993). A biodegradable nonionic surfactant, Alfonic^R 810~60 ethoxylate, below its CMC, was added to the biofilters in order to investigate the enhanced degradation of TCE and PCE. The TCE and PCE activated carbon biofilter performances were studied when the surfactant was introduced at concentrations of 5~50 mg/L. The below CMC of the surfactant was selected due to its toxicity and low biodegradability at above CMC. The CMC is defined as a surfactant concentration at which monomers begin to create colloidal aggregates. The Alfonic^R 810~60 ethoxylate has a HLB number of 12, that means below optimal HLB number of the maximum solubilization.

2. Materials and Methods

2.1 Materials

TCE (99.997%) and PCE (reagent grade) were obtained from Fisher Scientific (Pittsburgh, PA, USA). Phenol (>99.5%) was obtained from Sigma Chemical Co. (St. Louis, MO, USA). Alfonic^R 810~60 ethoxylate [$\text{CH}_3(\text{CH}_2)_x\text{CH}_2(\text{OCH}_2\text{CH}_2)_n\text{OH}$], {average molecular weight (MW)=356, $x=4, 6$, or 8 , and $n=1$ and 5 } was obtained from Vista Chemical Co. (Houston, TX, USA). HLB number, average moles of ethylene oxide per mole of ethylene, and average weight percentage of ethylene oxide for Alfonic^R 810~60 ethoxylate are 12, 4.8, and

60%, respectively. The specific gravity of the surfactant at 22°C is 0.983. Granular activated carbon (GAC), type BPL 4×6, was obtained from Calgon Carbon Corp. (Pittsburgh, PA, USA).

Microorganisms were obtained from raw wastewater at the Rolla Wastewater Treatment Plant in the City of Rolla, Missouri, USA. The microorganisms were continuously acclimated to phenol as the primary substrate with a nutrient solution in a 1000 mL erlenmeyer flask sealed with a rubber stopper under aerobic conditions. Table 1 shows the composition and concentration of the nutrition solution. To verify growth of phenol-oxidizing microorganisms, phenol concentration was measured daily. The phenol-oxidizing microorganisms was aerobically cultivated by placing GAC using phenol and the nutrient solution in a 4-Lamber glass bottle to acclimate in the GAC. The degradation of phenol was monitored for 35 hr by analyzing the supernatant from the bottle. A rapid degradation of phenol was observed within 18 hr. The phenol was completely degraded by 35 hr after the initial startup.

2.2 Biofilter Study

The biofilter setup consisted of two identical lab-scale 316 stainless steel biofilters having 2 1/16 inch ID and 2 ft long, VOC source, a flow meter, and a phenol and nutrient supply

Table 1. Composition and concentration of a nutrient solution.

Composition	Concentration (mg/L)
NaH ₂ PO ₄	50
KH ₂ PO ₄	85
K ₂ HPO ₄	165.6
NH ₄ Cl	100
MgSO ₄ · 7H ₂ O	0.1
FeSO ₄ · 7H ₂ O	0.12
MnSO ₄ · H ₂ O	0.036
ZnSO ₄ · 7H ₂ O	0.03
CoCl ₂ · 6H ₂ O	0.01
CaCl ₂ · 2H ₂ O	0.1
Yeast Extract	0.5

pump. Fig. 1 illustrates the schematic diagram of the biofilter setup. The VOC gas stream was fed to the biofilter using a 1/4 inch OD teflon tubing and teflon or kynar fittings. The phenol, nutrient, and surfactant solutions were supplied through a 1/4 inch OD Tygon tubing. Air as oxygen source for feeding each biofilter was supplied from the laboratory air spigots. Before entering the biofilters, the air was passed through an activated carbon filter to remove organic contaminants. The inlet vapor concentration was controlled by a teflon needle valve. Stainless needle valves were used for the inlet and outlet sampling ports. Depth and weight of the GAC in biofilters was 15.5 inch and 388 g, respectively. Gaseous TCE or PCE were fed to each biofilter from the bottom to be operated in a upflow mode. A solution containing nutrients was introduced from the top of the biofilter using a Masterflex pump (Cole-Parmer, Niles, IL, USA) through a teflon tubing. A

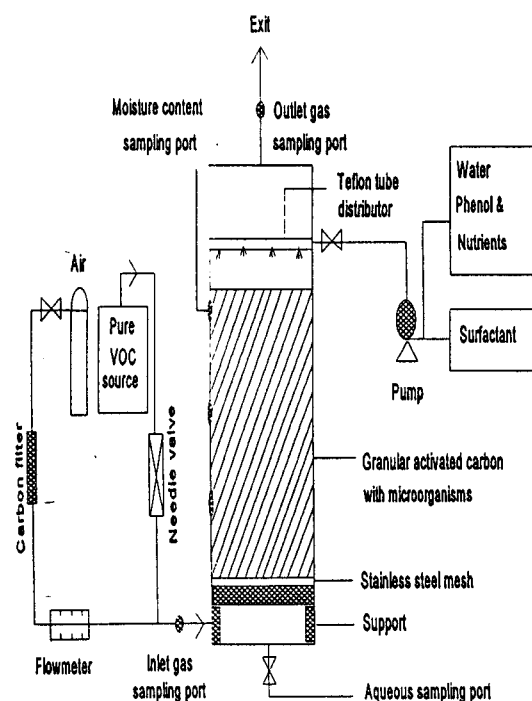


Fig. 1. Schematic diagram of a biofilter.

200 mL and 100 mL of influent solution at a phenol concentration of 6.4 or 32.4 mg/L was fed at 12 hr intervals to provide the primary substrates and to maintain optimal moisture content in the range of 40~47%.

2.3 Sampling and Analytical Methods

Gaseous TCE or PCE samples were collected using 1.6 L tedlar gas sampling bags with the on/off and septum valves. The on/off valve was used for collecting gaseous sample and the septum valve for needle injection. Before each sampling, the bags were filled with nitrogen gas and cleaned out by an air sampling pump four times. Aqueous effluents were collected in a 1000 mL erlenmeyer flask directly connected to the biofilter through a teflon tubing.

Gaseous and aqueous TCE, PCE, and phenol samples were analyzed with a SRI 8610B gas chromatograph (GC) fitted with a J & W Scientific DB-624 megabore column (30 m) or a Restek MXT-1 column (15 m) by FID. Nitrogen was used at a flowrate of 16 mL/min as a carrier gas. The column temperature for TCE and PCE analysis was first maintained at 40°C for 3 min, and then heated to 120°C at a rate of 8°C/min. The column temperature for phenol analysis was cooled to 80°C for 3 min, and then heated to 106°C at a rate of 8°C/min. Gaseous samples were directly injected into the injection port of the GC using a 1 mL Pressure-Lok gas syringe (Series A-2) with a push-button valve. Gaseous standards were prepared by considering the dimensionless Henry's law constants (H_a) for both TCE (0.392) and PCE (0.723) at 24.8°C (Gossett, 1987). Two measurements for the sample analysis were made and the readings were averaged.

The surface tension of the surfactant was measured using a tensiometer (Fisher Scientific Model 30, Pittsburgh, PA, USA). Fig. 2 illustrates the relationship between surfactant concentration and the surface tension to determine

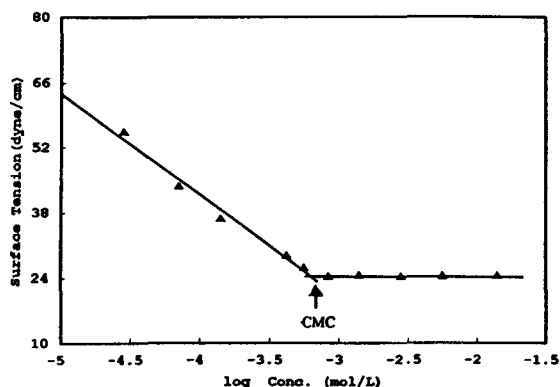


Fig. 2. Relationship between surfactant concentration and surface tension.

the CMC. The CMC of the surfactant is 225 ± 50 mg/L. The pH of aqueous samples was measured by a pH meter (Corning Model 610 A).

3. Results and Discussion

3.1 Biofilter Performance

Biofilter performance was evaluated in terms of TCE and PCE removal efficiency in the absence of surfactant is presented below. Average inlet TCE and PCE concentrations were determined by the moving-average method, on the basis of three sampling times. The average inlet TCE concentration ranged from 71 to 109 ppm in the gaseous phase, whereas the average inlet PCE concentration ranged between 47 and 84 ppm depending on gas residence times of 1.5 to 7 min. The PCE biofilter was operated at lower inlet concentrations than the TCE biofilter due to lower degradation rate. The moisture content ranged from 44 to 47% during the periods of biofilter operation. The removal efficiency for each compound was determined by the concentration of gaseous TCE or PCE removed by the biofilter, and expressed as its percentage relative to the inlet concentration.

3.1.1 TCE and PCE Removal

Fig. 3 shows variations of inlet and outlet concentrations of gaseous TCE as a function of different residence times during 188 days of operation. Table 2 summarizes the operational conditions and removal efficiencies for the TCE biofilter according to various residence times. The average gas flow rates were 560, 240 and 120 mL/min through the biofilter, resulting in average gas residence times of 1.5, 3.5, and, 7 min, respectively. The average inlet TCE concentration ranged from 71 to 109 ppm in the gaseous phase, at 95% confidence limits of the inlet concentrations.

As shown in Fig. 3, a complete 100% TCE removal (at a residence time of 7 min) occurred as a result of the adsorption and degradation of the TCE on GAC by the phenol-oxidizing microorganisms. However, the removal efficiency of the biofilter decreased when residence time was reduced. TCE removal efficiency of 88~100% was obtained at a residence time of 3.5 min. The TCE biofilter was operated two times at the residence time of 3.5 min. A 100% removal was observed at first, and an 88~91% removal was observed at second. This inconsistency removal efficiency might have been

due to higher inlet concentration by 40 ppm and the associated mass transfer limitation at the second time. The TCE removal efficiency decreased to 16% in 32 days after changing the residence time from 3.5 to 1.5 min. The TCE removal efficiency ranged from 16 to 21% at a residence time of 1.5 min. At the high loading rate (low residence time), TCE was not degraded quickly because its degradation rate in the biofilter was relatively low.

Fig. 4 shows variations of average inlet and outlet concentrations in PCE biofilter as a function of different residence times: 2, 4, and 7 min, for 208 days. Table 2 presents the operational conditions and removal efficiencies of the PCE biofilter at various residence times. Gaseous PCE was removed up to 100% at residence times ranging from 4 to 7 min, but its removal efficiency decreased to 79% in 89 days after lowering the residence time from 4 to 2 min.

As a result of the phenol degradation, the following effects were expected: (1) a biofilm would have grown, (2) the biofilm thickness might have increased, and (3) the diffused oxygen was probably consumed by phenol-oxidizing microorganisms before it could penetrate into the full depth of the biofilm. Thus,

Table 2. Removal efficiency of biofilters for TCE and PCE.

Operational Factor	TCE			PCE		
Avg. Gas Flow Rate (mL/min)	560	240	120	435	210	120
Avg. Gas Loading Rate (mL/m ² min)	260	112	56	202	98	56
Residence Time (min)	1.5	3.5	7.0	2.0	4.0	7.0
Average Inlet Conc. (ppm)	109	71	85	57	84	47
Inlet Conc. at 95% Confidence Limit (ppm)						
Lower Limit	92	56	76	45	77	39
Upper Limit	126	86	94	69	91	55
Removal Efficiency (%)	16~21	88~100	100	77~80	100	100
Removal Efficiency at 95% Confidence Limit (%)						
Lower Limit	10	88	100	77	100	100
Upper Limit	28	100	100	80	100	100

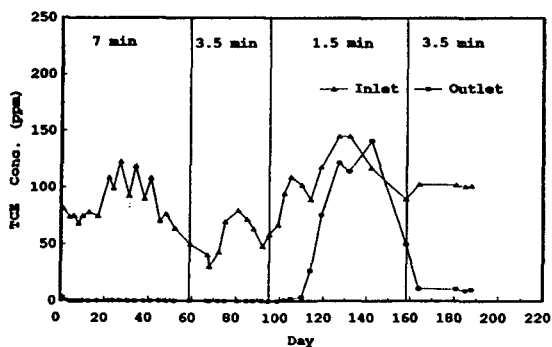


Fig. 3. Performance of TCE biofilter without surfactant.

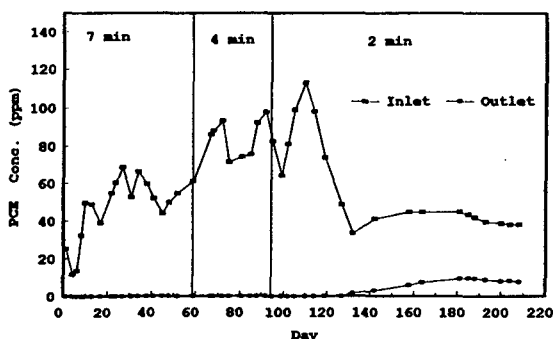


Fig. 4. Performance of PCE biofilter without surfactant.

an anaerobic condition might have occurred at near the surface of GAC. It is likely that adsorbed and dissolved PCE onto biomass near the GAC surface was degraded by the microorganisms. At present, it is not possible to know the thickness of anaerobic zone, if there are any within the biomass. High TCE and PCE removals were obtained at residence times of 3.5~7 min due to the lower gas loading rate as well as the greater microbial activity that could have been inferred from the increase of influent phenol concentration.

In this study, transformation yield of TCE and PCE in the gaseous phase was calculated. Maximal transformation yields were about 8~48 g of TCE/g of phenol and 6~25 g of PCE/g of phenol. Hopkins *et al.* (1993) reported the

highest transformation yield to be 0.11 g of TCE/g of phenol in the aqueous phase. One or two orders of magnitude higher transformation yield of TCE was observed in the gaseous phase than in the aqueous phase.

Biomass was frequently observed in both effluents from TCE and PCE biofilters. This indicated the growth of phenol-oxidizing microorganisms and sloughing of the biomass from the biofilters throughout the periods of operation.

3. 1. 2 Adsorption of TCE and PCE

In order to investigate the adsorbed TCE or PCE on the GAC in each biofilter, about 2~5g of GAC samples were taken at the end of the biofilter operation and extracted with 99.9% methanol for 64 hr. Table 3 presents the amount of TCE and PCE remaining on GAC after biofilter operation. About 2.1% of total amount of TCE and 1.1% of total amount of PCE applied to the respective biofilter was adsorbed probably by GAC. Most gaseous TCE and PCE were adsorbed by GAC before the biofilm thickness might have increased for the biodegradation process. From this investigation, it can be concluded that the adsorption was a minor mechanism for the removal of gaseous TCE and PCE in the biofiltration process.

3. 1. 3 Phenol Removal

The phenol concentration in the effluent was 0 mg/L, and was completely degraded by the phenol-oxidizing microorganisms throughout the periods of operation. To prove the degradation of phenol in each biofilter, phenol adsorption on GAC was performed as a column study. Saturation of the carbon with phenol was noticed when 2.0g of phenol was treated. However, even after 2.0g of phenol were treated, no residual phenol was found in the effluents of either biofilter. The phenol was clearly utilized by the phenol-oxidizing microorganisms in the biofilters. If the primary substrate disappeared without adsorption, the microbial population must grow. Therefore, the presence and growth

Table 3. Amount of TCE and PCE remaining on GAC after biofilter operation.

Compound	Amount Adsorbed by GAC (g)	Total Amount Applied to Biofilter (g)	Percent due to Adsorption (%)
TCE	2.02	95.5	2.1
PCE	0.62	59.1	1.1

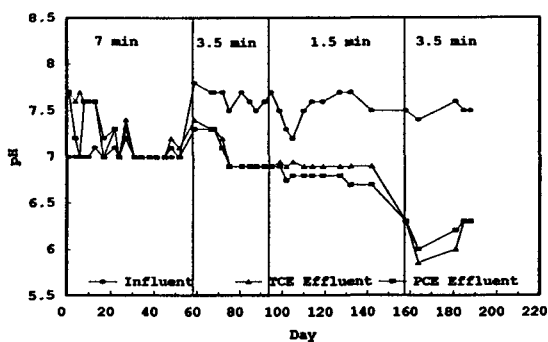


Fig. 5. Influent and effluent pH in TCE and PCE biofilters.

of microorganisms on the GAC indicated indirectly the degradation of substrate.

3.1.4 pH

The effluent pH from each biofilter was measured to study the mineralization of TCE and PCE. Fig. 5 illustrates the pH variations of the influent and effluent from the TCE and PCE biofilter as a function of different residence times. The pH of the influent solution was between 7 and 7.7. The pH of the effluent from the TCE biofilter decreased to 6.8 after 75 days, at a residence time of 3.5 min, due to the formation of mineral acids by the TCE degradation. The pH decreased continuously to 5.8 after 164 days at a residence time of 1.5 min.

The pH of the effluent from the PCE biofilter was similar to that from the TCE biofilter. The pH of the effluent from the PCE biofilter decreased to 6.7 after 103 days at a residence time of 4 min due to the formation of mineral acids by the PCE degradation.

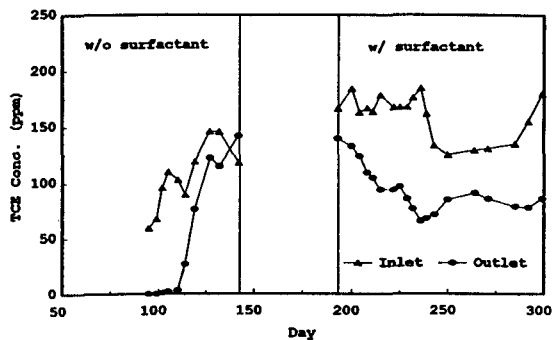


Fig. 6. TCE biofilter with and without surfactant at a residence time of 1.5 min.

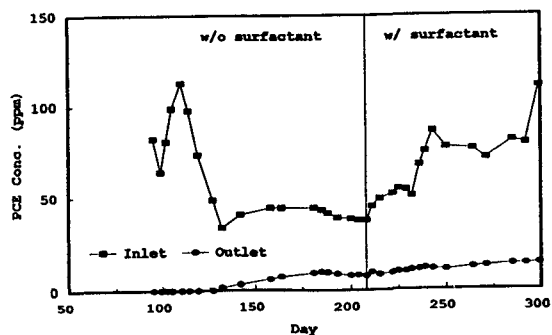


Fig. 7. PCE biofilter with and without surfactant at a residence time of 2 min.

3.2 Effect of Surfactant on Biofilter Performance

The performance of the biofilters was investigated by introducing into GAC a biodegradable nonionic surfactant below its CMC. The low residence times (1.5 min for the TCE biofilter and 2 min for the PCE biofilter) were selected because removal efficiency of each compound showed the lowest level in the absence of surfactant.

Figs 6 and 7 show the performance of the TCE and PCE biofilters at three different surfactant concentrations (5, 15, and 50 mg/L) as below CMC. The average inlet concentrations for TCE and PCE were 160 ± 9 and 69 ± 9 mg/L, respectively (at 95% confidence limits). The average inlet TCE concentration was about two times higher than the concentration used in the absence of surfactant. As shown in Fig. 6, an initial surfactant concentration of 50 mg/L was fed to each biofilter. The TCE removal efficiency increased to 41% within 20 days after the addition of the surfactant. This was twice the TCE removal efficiency, when the surfactant was added to the biofilter. The explanations and discussions of the results will be presented later. An enhanced performance of the TCE biofilter was observed over the range of the surfactant concentrations studied. About $168 \pm 55\%$ (at 95% confidence limit) higher removal efficiency was obtained at a surfactant concentration of 15 mg/L.

The average inlet PCE concentration was approximately 21% higher than that used in the absence of the surfactant. About $6.5 \pm 0.3\%$ (95% confidence limit) higher PCE removal efficiency was obtained at surfactant concentrations ranging from 5 to 15 mg/L, whereas $9 \pm 0.5\%$ (95% confidence limit) higher removal efficiency occurred at a surfactant concentration of 50 mg/L.

In this study, TCE or PCE biofilter performance was observed to be little high, but not significantly, in the presence of surfactant probably due to more free gaseous TCE and higher inlet concentration. It may be difficult to evaluate the surfactant effects on TCE biofilter performance due to high variations at 95% confidence limits. PCE removal as biofilter performance was about the same with and without the addition of surfactant.

Little enhancement of TCE degradation in the biofilter might have occurred because more free gaseous TCE was solubilized, and the

TCE attached to the sorbed or dissolved surfactant molecules into a biofilm on the surface of GAC. Edwards *et al.* (1994) reported soil sorption of surfactant increased the equivalent fractional organic contents, which tended to increase the amount of hydrophobic organic compound sorbed. It is likely that the surfactant was sorbed or dissolved on the surface of GAC and biofilm, and stimulated degradation of TCE. Because surfactant reduces the surface tension of water, perhaps more gaseous TCE was sorbed onto the biofilm. Churchill *et al.* (1993) found that lowering the surface tension increased organic compound uptake by microorganisms. A relatively smaller amount of free PCE due to higher removal efficiency did not increase PCE removal in the presence of surfactant. Further work is needed to elucidate the removal mechanisms of TCE or PCE in the presence of the surfactant in the biofilters. From the present study, no inhibitory effect on the degradation of TCE or PCE was found below the CMC. One of the interesting characteristics of surfactants is the desorption of organic compounds that were sorbed originally onto solids. However, no desorption of phenol in the biofilters was found in the presence of the surfactant due to its complete degradation.

4. Conclusions

The following conclusions can be drawn based on the findings of this study:

1. Gaseous TCE and PCE were removed by activated carbon biofilters inoculated with phenol-oxidizing microorganisms.
2. The activated carbon biofilter was capable of achieving the TCE removal efficiency as much as 100% at a residence time of 7 min and an average inlet concentration of 85 ppm. However, the removal efficiency decreased to about 18% when the residence time was reduced to 1.5 min.
3. The activated carbon biofilter was capable

of achieving the PCE removal efficiency as much as 100% at residence times of 4 ~7 min and average inlet concentrations of 47~84 ppm. The PCE removal efficiency decreased to about 79% when the residence time was reduced to 2 min.

4. It was found that adsorption by the GAC was a minor removal mechanism of TCE and PCE in the biofiltration process.
5. When fed to biofilters, phenol was completely utilized by the phenol-oxidizing microorganisms throughout the period of the biofilter operation.
6. No significant improvement in removal efficiency of gaseous TCE or PCE was observed when a nonionic surfactant was introduced into biofilter at a residence time of 1.5 or 2 min.

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비이온 계면활성제의 주입과 비주입 할 경우 기체 상태의 TCE와 PCE 분해

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요 약 문

본 연구는 페놀산화미생물을 주입한 입상활성탄 biofilter를 이용하여, 기체 상태의 트리클로로에틸렌 (TCE)과 테트라클로로에틸렌 (PCE)을 생분해시키는 것과 임계미셀농도 (CMC)값 이하에서의 계면활성제가 TCE와 PCE의 처리효율에 미치는 영향을 조사하기 위해 수행하였다. 기체 상태의 TCE와 PCE를 처리하기 위하여, 두개의 개별적 biofilter를 체류시간이 1.5~7분이 되게 운전하였다.

기체 TCE는 체류시간 7분과 평균 유입농도 85 ppm에서 100% 처리되는 것으로 조사되었다. 그리고, 기체 PCE는 체류시간 4~7분과 평균 유입농도 47~84 ppm에서 100% 처리되었다. 활성탄에 의한 흡착은 TCE와 PCE 처리에 영향을 적게 준 것으로 나타났다. 기체 상태의 TCE와 PCE의 transformation yield값은 체류시간에 따라 각각 8~48 g of TCE/g of phenol과 6~25 g of PCE/g of phenol으로 조사되었으며, 액체 상태의 TCE 값과 비교하면 1~2차수 작게 나타났다. Biofilter에 계면활성제의 농도를 5~50 mg/L 이하로 주입한 결과, 기체 상태의 TCE와 PCE의 처리효율은 계면활성제를 주입하지 않을 때보다 약간 증가하였으나, 큰 차이를 발견할 수는 없었다.