Selective adsorption of phenanthrene in nonionic–anionic surfactant mixtures using activated carbon

Chi Kyu Ahn[a], Seung Han Woo[b,*], Jong Moon Park[a,**]

[a] *Advanced Environmental Biotechnology Research Center, School of Environmental Science and Engineering, Department of Chemical Engineering,*
Pohang University of Science and Technology, San 31, Hyoja-Dong, Pohang 790-784, Republic of Korea
[b] *Department of Chemical Engineering, Hanbat National University, San 16-1, Deokmyeong-Dong, Yusung-Gu, Daejeon 305-719, Republic of Korea*

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ABSTRACT

Surfactant enhanced soil washing is an efficient remediation process for sites contaminated with hazardous hydrophobic organic compound (HOC). To reduce the cost of the process, the used surfactant should be recovered. This paper presents investigation of selective adsorption of HOC in nonionic–anionic surfactant mixtures by activated carbon as a means of recovering surfactants, using phenanthrene (PHE) as an HOC, Triton X-100 (TX100) as a nonionic surfactant, and sodium dodecyl sulfate (SDS) as an anionic surfactant. The sorbed amount of TX100 on activated carbon decreased from 0.433 to 0.227 mmol/g as SDS dose increased. However the sorbed amount of PHE increased from 0.125 to 0.178 mmol/g as SDS dose increased because molar solubilization ratio of the surfactant mixtures decreased. As a result, selectivity for PHE sorption that represents the ratio of PHE partitioning to surfactant partitioning increased with increase in the proportion of SDS in the surfactant mixture. Selectivity for PHE to surfactant was much higher than 1 over a wide range of surfactant composition. The highest selectivity, 95.97 ± 49.94 at 5.61×10^{-2} mmol-PHE/L, was obtained in the solution containing only SDS. These results suggest that addition of anionic surfactant may improve surfactant recovery when selective adsorption for surfactant recovery is included in soil remediation by surfactant enhanced soil washing.

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1. Introduction

Hazardous hydrophobic organic compounds (HOCs) cause environmental concern due to their toxic and carcinogenic properties [1,2]. Moreover HOCs have low water solubility; thus they are strongly sorbed into soils and sediments [3,4]. Surfactant enhanced soil washing has been frequently used for rapid removal of HOCs from contaminated soils [3,5–8]. Soil washing efficiency significantly depends on types and doses of surfactant [7,9]. Particularly, nonionic surfactants have been widely used for soil remediation in both physical and biological remediation processes because they have low toxicity and lower critical micelle concentration (CMC) than ionic surfactants [3,7].

Mixtures of nonionic and anionic surfactant improve the soil washing process because of increased solubility of HOC and lower partitioning losses to non aqueous phase liquids compared to individual surfactants [8,10,11]. Furthermore, the addition of nonionic surfactants to anionic surfactant solutions can decrease

the precipitation between anionic surfactants and divalent electrolytes (e.g., Ca^{2+}) [12–14]. Therefore surfactant losses due to precipitation or adsorption onto soil can be reduced by using a surfactant mixture. Consequently, in soil washing for HOC-contaminated soils, a mixture of nonionic–anionic surfactant may be more efficient than single surfactants, cationic–anionic surfactant mixtures, or cationic-nonionic surfactant mixtures [15–17].

Use of surfactants significantly improves the performance of soil washing, but operational costs increase as surfactant dosages increase [18]. Costs can be reduced if the used surfactant from soil-washed solutions can be recovered effectively and economically. Selective adsorption of contaminants by activated carbons was previously proposed as a means of reusing surfactants [19] and enhanced sorption of HOC was demonstrated by mathematical partitioning modeling [20]. In order to reuse surfactant, the contaminants in the washed solution should be removed in a greater ratio than the surfactant. Activated carbon would be an excellent selective adsorbent because HOCs are highly hydrophobic compared to most nonionic surfactants used in soil washing process. HOCs would be directly adsorbed on activated carbon surfaces, or incorporated into surfactant micelles or hemimicelles sorbed on activated carbon [20]. Thus the surfactant recovery process using activated carbon could be a potentially effective method because

Table 1
Selected physico-chemical properties of surfactants and PHE.

	Molecular formula	MW (g/mol)	Ionic property	K_{ow}	CMC[a] (mM)
PHE	$C_{14}H_{10}$	178.23	–	$10^{4.57}$	–
TX100	$C_8H_{17}C_6H_4OE^b_{9.5}H$	628	Nonionic	–	0.385
SDS	$CH_3(CH_2)_{11}OSO_3Na$	288.38	Anionic	–	3.919

[a] The CMC values were obtained in this study.
[b] E: OCH_2CH_2.

the contaminants in the washed solution should be removed in a greater ratio than the surfactant.

Nonionic–anionic surfactant mixtures have been studied extensively [8,10,11,21], but the effectiveness of HOC removal by surfactant mixtures using activated carbon has not been reported. In our previous studies, various nonionic surfactants were used for evaluate the efficiency of soil washing and surfactant recovery [22], but nonionic–anionic surfactant mixtures has not been studied yet. Herein, we investigated the selective adsorption of an HOC, phenanthrene (PHE), by activated carbon in nonionic–anionic surfactant mixtures. We used Triton X-100 (TX100) as a nonionic surfactant and sodium dodecyl sulfate (SDS) as an anionic surfactant because they are widely used in industrial applications and their application to soil washing has been often studied [10,11,21].

2. Materials and methods

2.1. Materials

Sodium dodecyl sulfate (SDS), Triton X-100 (TX100) and phenanthrene (PHE: >98%) (Table 1) and granular-type activated carbon (GAC; Darco 20–40 mesh) were purchased from Sigma. Methylene blue ($C_{16}H_{18}ClN_3S$) and analytical grade chloroform ($CHCl_3$) for determining the SDS concentration were purchased from Sigma and JT Baker, respectively. The activated carbon was washed with deionized water several times, then oven-dried at 60 °C for several days.

2.2. Surface tension measurement

Surface tensions of each surfactant solution were determined using a Du Noüy Tensionmeter (Itoh Seisakusho, Japan). Surface tension values for a given surfactant concentration were taken after a stable reading had been obtained. CMC was determined from a plot of the surface tension vs. logarithmic scale of the surfactant concentrations.

2.3. Phenanthrene solubilization

Batch tests for solubilization of PHE were performed in nonionic–anionic surfactant (TX100-SDS) mixtures having a range of total concentrations (2.0–10 mmol/L) and for solutions containing only SDS (4.0–10 mmol/L). Surfactant mixtures used (TX100:SDS) molar ratios of 1:0, 2:1, 1:1, 1:2 and 0:1. For each surfactant mixture, 10 mL of solution with excess PHE was placed in a 20-mL screw-cap vial equipped with a Teflon-lined septum to prevent loss of PHE by volatilization. The vials of each mixture were shaken at 200 rpm for 76 h. The liquid was filtered using a 0.45 μm PTFE syringe filter (Whatman, USA), then PHE concentration was determined using a high performance liquid chromatograph (HPLC; Dionex, USA). The experiments were performed with quadruplicates.

2.4. Adsorption experiments

Selective adsorption experiments were performed at a fixed total concentration of surfactant mixtures (10 mmol/L) and PHE.

PHE concentration was adjusted to 2.81×10^{-1} mmol/L in the surfactant mixtures, but to 5.61×10^{-2} mmol/L in the solution containing only SDS, because SDS has a much lower molar solubilization ratio (MSR) for PHE than the mixtures. Activated carbon (0.1 g) was added to 100 mL of each surfactant solution. Experiments were conducted in 250-mL Erlenmeyer flasks with a working volume of 100 mL. The flasks were shaken at 100 rpm at 20 °C for 24 h. All adsorption experiments were performed with duplicates. The amount of PHE adsorbed on activated carbon was quantified by comparing the initial and final concentrations of each compound in a batch system.

2.5. Analytical methods

PHE and TX100 concentrations in the surfactant mixtures were quantified using the HPLC with an ultraviolet detector at 250 nm for PHE and at 230 nm for TX100. The analytical column was a reversed-phase SUPELCOSIL LC-PAH column (150 mm × 4.6 mm). The mobile phase (85% acetonitrile and 15% de-ionized water) was eluted at a flow rate of 1.5 mL/min [19].

SDS concentrations in the surfactant mixture were quantified using modified methylene blue colorimetry [23]. The assay mixture consisted of 4 mL of methylene blue solution (0.25 g/L), 6 mL of chloroform and 1 mL of the target sample. The mixture was vigorously mixed for 1 min and allowed to stabilize at room temperature for 2 h. The optical absorbance of the chloroform phase was measured at 650 nm using a spectrophotometer (DR-2010, HACH).

3. Results and discussion

3.1. Critical micelle concentration

Surface tension was measured for various compositions of TX100 and SDS in their mixture (Fig. 1). In each solution, the surface tension decreased rapidly, and then either remained relatively con-
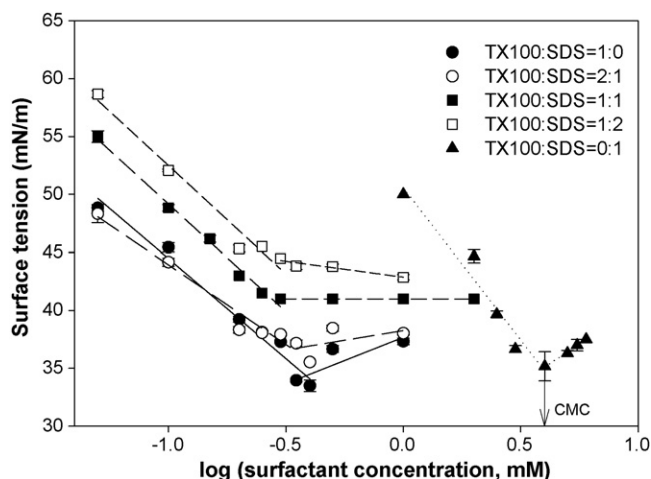

Fig. 1. The surface tensions and CMC determination in the surfactant mixtures with various ratios of TX100:SDS ratios.

Table 2
The CMC values and MSR for PHE of the nonionic–anionic surfactant mixtures.

TX100:SDS	CMC (mM)	MSR (PHE)
1:0	0.385	0.104
2:1	0.336	0.066
1:1	0.276	0.058
1:2	0.270	0.045
0:1	3.919	0.022

stant or began to increase or decrease slightly. It has been reported that surface tension decrements at supra-CMC often observed particularly in surfactant mixtures due to mixed micelle formation [24]. The CMC values for each surfactant mixture (Table 2) were determined from plots of surface tension on log_{10}(surfactant concentration) by fitting straight lines separately to the rapidly decreasing and the stable or increasing portions of the curves, then calculating the surfactant concentration at which the lines intersected. The CMC values in the mixtures decreased with increasing molar proportion of SDS, from 0.385 mM (TX100:SDS = 1:0) to 0.270 mM (TX100:SDS = 1:2). However, CMC in the solution containing only SDS was 3.919 mM, i.e., more than 10 times greater than in the mixtures. The CMC of the surfactant mixture decreased significantly and was similar to the CMC of TX100 solution when a small amount of TX100 was added into the SDS solution. This decrease was likely because during the formation of mixed micelles, TX100 was predominantly present in the mixed surfactant solution. Otherwise, nonionic surfactant molecules (TX100) were inserted into the anionic surfactant (SDS) micelles and the repulsion among the ionic heads of anionic surfactant might be reduced [21].

3.2. Phenanthrene solubilization

The apparent solubility of PHE increased linearly with increasing total surfactant concentration (Fig. 2). MSR was calculated from the solubility slopes (i.e., mole PHE solubilized per mole surfactant added) at supra-CMC [25]. MSR increased as the proportion of TX100 in the mixture increased (Table 2). This increase occurs mainly because PHE is much less soluble in SDS solution than in TX100 solution. Therefore, because the total mole concentration of surfactant in the surfactant mixture was fixed, addition of SDS to the mixtures decreased the concentration of TX100, and therefore reduced PHE solubility. In case that the total amount of surfactant mixture was 10 mmol/L, the PHE solubility in TX100-SDS mixtures with the ratio of 2:1, 1:1, 1:2 and 0:1 for TX100:SDS was reduced to 61.1, 53.7, 41.7 and 20.3% of the PHE solubility in the pure TX100
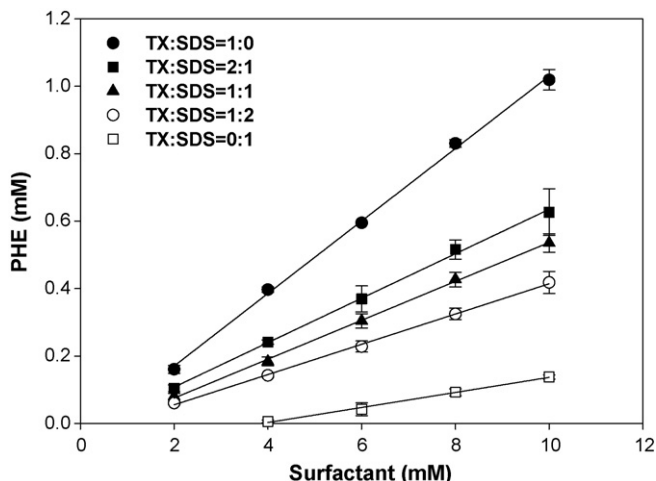
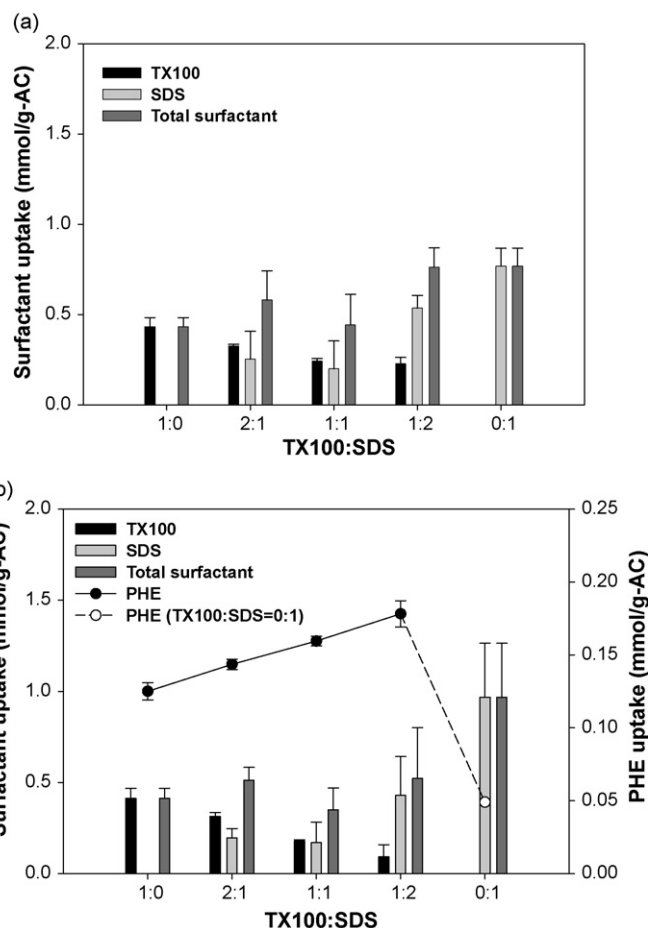**Fig. 2.** The solubility of PHE in various TX100-SDS mixtures.

Fig. 3. Adsorption of TX100, SDS, and PHE on activated carbon in various TX100-SDS mixtures in the absence (a) or presence (b) of PHE.

solution (TX100:SDS = 1:0), respectively. It is noticeable that the PHE solubility decreased nonlinearly with increasing SDS proportion in the mixtures. At the low TX100 portion (TX100:SDS = 1:2), the synergistic solubilization was observed, but it was opposite at the high TX100 portion (TX100:SDS = 2:1). It has been reported that the synergism of solubilization in mixed micelle solutions was decreased with increasing the mole fraction of nonionic surfactant [21]. The nonlinearity would be caused by the mixed micelles with different structure formed with different mole fractions of TX100 and SDS.

3.3. Adsorption of PHE and surfactant in surfactant mixtures

Adsorption of PHE or surfactants onto activated carbon in the surfactant mixtures was quantified for various TX100:SDS ratios at a fixed total surfactant concentration (10 mmol/L). As the proportion of SDS increased, the amount of TX100 sorbed onto activated carbon gradually decreased from 0.433 ± 0.050 to 0.227 ± 0.036 mmol/g (Fig. 3a), whereas the amount of SDS sorbed onto activated carbon increased. This result indicated that adsorption of TX100 in the presence of SDS may be significantly restricted by competitive adsorption between TX100 and SDS. For example, in TX100:SDS = 2:1, the percentage of TX100 sorbed was 56.4% of total sorbed surfactant. Competitive adsorption on activated carbon can occur when the molecular sizes of adsorbates are similar or when they compete for the same adsorption sites [26]. The hydrophobic moieties of TX100 and SDS are sorbed mainly onto the hydrophobic surface of activated carbon [27,28], so in this study, competition for sorption sites was likely the main reason for the reduced adsorp-

Table 3
Overall performance of PHE removal and surfactant recovery in the various surfactant mixtures.

Surfactant mixture	PHE removal (%)	Surfactant loss (%)		Selectivity	Overall surfactant recovery (%)
TX100:SDS = 1:0[a]	46.59	TX100	5.18 ± 0.55	16.01	94.82
	± 2.18	SDS	–	± 0.38	± 0.55
TX100:SDS = 2:1[a]	55.27	TX100	4.23 ± 0.28	23.77	94.99
	± 1.36	SDS	7.12 ± 1.80	± 4.75	± 0.69
TX100:SDS = 1:2[a]	63.97	TX100	3.90 ± 0.14	47.84	96.21
	± 1.32	SDS	3.67 ± 2.42	± 17.59	± 1.14
TX100:SDS = 1:2[a]	67.63	TX100	3.77 ± 2.67	41.54	94.61
	± 3.41	SDS	5.95 ± 2.94	± 17.48	± 2.87
TX100:SDS = 0:1[b]	90.74	TX100	–	95.97	89.81
	± 1.68	SDS	10.19 ± 3.11	± 49.94	± 3.11

"±" represents standard deviation obtained from duplicate experiments.
 [a] The initial phenanthrene concentration is 2.81×10^{-1} mmol/L.
 [b] The initial phenanthrene concentration is 5.61×10^{-2} mmol/L.

tion of TX100 in the presence of SDS. In the solution containing only SDS, 1.8 times more surfactant (0.768 mmol/g) was sorbed than only TX100 solution (0.433 mmol/g) on a molar basis.

The amount of PHE sorbed increased as the TX100 portion in the surfactant mixtures decreased (Fig. 3b). The sorbed amount of TX100 and SDS was almost the same as in the absence of PHE (Fig. 3a), indicating that the presence of PHE did not significantly affect surfactant sorption. This property is desirable, because surfactants will remain in aqueous solutions with low losses by adsorption onto activated carbon, and thereby a large proportion of surfactant can be recovered.

To measure PHE adsorption, the initial PHE concentration was appropriately adjusted to consider the PHE solubility of each surfactant mixture (Fig. 2); these concentrations were 2.81×10^{-1} mmol-PHE/L for TX100:SDS mixtures and 5.61×10^{-2} mmol-PHE/L for the solution containing only SDS. The sorbed amount of PHE gradually increased from 0.125 ± 0.006 to 0.178 ± 0.009 mmol/g as the proportion of SDS increased except in the SDS-only solution, in which the much lower initial PHE concentration reduced the sorption of PHE. The increased adsorption of PHE (or decreased concentration in liquid) with increasing SDS proportion in the mixtures occurs because the MSR value decreased from 0.128 (TX100:SDS = 1:0) to 0.048 (TX100:SDS = 1:2), with increasing SDS proportion.

3.4. Selective adsorption by activated carbon

Surfactant recovery is considered theoretically possible only if the adsorption process removes relatively more contaminants than surfactant. For effective surfactant recovery, a high sorbed proportion of PHE and a high liquid proportion of surfactant are preferred. In the various surfactant mixtures, the proportion of surfactant remaining after adsorption onto activated carbon was quite high, ranging from 89.81 to 96.21%, and was not very different among surfactant ratios except for the solution containing only SDS solution (Table 3). The amount of PHE sorbed onto activated carbon ranged from 0.049 to 0.178 mmol/g. The highest PHE removal efficiency ($90.74 \pm 1.68\%$) was obtained in the solution containing only SDS solution, even though a low initial concentration of PHE was used. The lowest removal efficiency was obtained in the solution containing only TX100, due to its high MSR value, which caused a greater partitioning of PHE into the liquid phase than occurred in other mixtures.

In the surfactant mixtures, PHE was successfully removed using activated carbon, and surfactant recovery efficiency was good. The efficiency of the selective adsorption process can be evaluated by selectivity (S) expressed as [19]:

$$S = \frac{C_{AC,j}}{C_{l,j}} \times \frac{C_{l,\text{surf}}}{C_{AC,\text{surf}}} \tag{1}$$

where $C_{AC,j}$ is the concentration of PHE sorbed onto activated carbon (mmol/g), $C_{l,j}$ is the concentration of PHE in the liquid phase after adsorption (mmol/L), $C_{l,\text{surf}}$ is the concentration of surfactant in the liquid phase after adsorption (mmol/L), and $C_{AC,\text{surf}}$ is the concentration of surfactant sorbed onto activated carbon (mmol/g).

If $S > 1$, more contaminants than surfactants are adsorbed to the activated carbon, and surfactant recovery is theoretically possible [19]. Values of S for the surfactant mixtures ranged from 16.01 to 95.97. This result suggests that nonionic–anionic surfactant mixtures as well as pure nonionic surfactant can be effectively recovered when using activated carbon in surfactant enhanced soil washing.

As the proportion of SDS in the mixtures increased, the MSR decrease, so S increased. The increasing trend seems clear even though the values of S varied greatly due mainly to the low value of $C_{l,j}$, i.e., the concentration of PHE in the liquid phase after adsorption (mmol/L), in Eq. (1). S was highest (95.97) when the solution contained only SDS. This is understandable because SDS with low solubilizing power or a low MSR value increases the PHE removal by activated carbon adsorption. Even though more SDS was sorbed than TX100, this difference did not significantly affect S because most of the surfactants occur in the liquid phase. Thus, S was determined more by the surfactant's MSR than by its adsorption capacity.

As SDS concentration increased, the PHE removal efficiencies were increased, however, when the surfactant mixture as larger anionic surfactant fraction used in soil washing process, the soil washing efficiency would be reduced due to lower PHE solubility (MSR). For this reason, a larger ratio of nonionic surfactant to anionic surfactant may be desirable for effective soil washing. In an actual soil washing process, the use of an optimized mixture of nonionic and anionic surfactants has been recommended to reduce loss of surfactant by adsorption onto soil and to enhance the soil washing efficiency [29]. The results of the present study suggest that the addition of anionic surfactant may increase surfactant recovery when selective adsorption for surfactant recovery process is included in the process of soil remediation by washing.

The application of this process in actual soil washed solution the removal of HOC is more important than recovery of surfactant in view of environmental control. The HOC remaining in washed solution after a selective adsorption should be minimized below a regulatory level by the addition of greater amounts of activated carbon or use of higher efficient activated carbon. Therefore optimization of the type and amount of activated carbon for selective adsorption should be investigated. Another thing is that dissolved organic matters in actual soil-washed solution might decrease the efficiency of surfactant recovery. In addition, the effect of various chemicals or particles in washed solution such as ionic compounds, dissolved organic matters, and soil particles on the effectiveness of surfactant recovery should be further studied.

4. Conclusions

Use of activated carbon for selective adsorption of PHE and surfactant recovery was investigated in surfactant mixtures of non-ionic (TX100) and anionic (SDS) surfactants. The sorbed amount of TX100 on activated carbon was reduced by addition of SDS. The removal efficiency (46.59–90.74%) of PHE gradually increased with increasing SDS proportion due to low the MSR value of SDS, and over 90% of surfactant remained in the solution after selective adsorption. Moreover, the selectivity which measures the efficiency of the selective adsorption process was much higher than 1 in all cases. Thus, activated carbon adsorption can be a good method for surfactant recovery in nonionic–anionic surfactant mixtures as well as in pure nonionic surfactant solutions. Furthermore, the selectivity increased with increasing SDS proportion, indicating that the addition of anionic surfactant into the nonionic surfactant solution can increase the efficiency of surfactant recovery.

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