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Water solubility enhancements of pyrene by single and mixed surfactant solutions

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Abstract: Water solubility enhancements of pyrene by both single-surfactant and mixed-surfactant solutions were compared and evaluated. The solubility of pyrene in water was greatly enhanced by each of Triton X-100 (TX100), Triton X-405 (TX405), Brij 35 and SDS, in which the water solubility enhancements increased with increasing surfactant concentrations. The extent of solubility enhancements at surfactant concentrations below the CMC is the order of TX100 > Brij 35 > TX405 > SDS; the sequence at surfactant concentrations above the CMC is TX100 > Brij 35 > SDS > TX405. Pyrene was solubilized synergistically by anionic-nonionic mixed surfactant solutions, especially at low surfactant concentrations. The synergistic power of the mixed surfactants is SDS-TX405 > SDS-Brij 35 > SDS-TX100. The synergism as noted is attributed to increasing K_{mc} and/or decreasing the CMC of the mixed surfactant solution. For SDS-TX405 and SDS-Brij 35 mixed surfactant solutions, an increase in K_{mc} is coupled with a decrease in the CMC; for SDS-TX100, only a decreased in the CMC value is noted. Mixed-surfactant solutions may improve the performance of the surfactant-enhanced remediation (SER) of soils by increasing the bioavailability and biodegradation of non-aqueous-phase organic pollutants and reducing the level of surfactant pollution and remediation expenses.

Keywords: pyrene; surfactants; synergistic solubilization; remediation

Introduction

Surfactants enhance the remediation of NAPL-contaminated sites by increasing the aqueous-phase concentration of the NAPL via micelle formation, microemulsification, or mobilizing the NAPL phase (Kile, 1989; Edwards, 1991; Guha, 1998a; Ko, 1998; Zimmerman, 1999; Bettahar, 1999; Willson, 1999; Park, 2000; Jeong, 2000), and hence improving the bioavailability of organic pollutants (Guha, 1998b; Boopathy, 1999; Romantschuk, 2000; Pinto, 2000; Willumsen, 1999). Zhou *et al.* (Zhou, 2000) compared and evaluated a number of commercial surfactants suitable for remediation of DNAPL by solubilization. To quantify the potential performance of surfactant-enhanced remediation (SER), a number of researchers have measured micelle solubilization capacities for various organic chemicals in surfactant solutions (Kile, 1989; Edwards, 1991; Guha, 1998; Park, 2000; Zhou, 2000; Mata-Sandoval, 2000). Observed solubility enhancements were related to organic compound and surfactant properties (Kile, 1989; Edwards, 1991; Sun, 1995; Jafvert, 1994; Diallo, 1994; Pennell, 1997). Surfactant-enhanced remediation has been suggested as a promising technology for the removal of NAPL from contaminated soil and groundwater (Abdul, 1992; Pennell, 1993). Earlier studies of the enhancements in water solubility and bioavailability of organic pollutants were usually conducted with single-surfactant solutions. In the remediation of organic-polluted environment with high surfactant concentrations, soil and groundwater may be significantly contaminated by surfactants in addition to the increased surfactant cost. Thus, an improved strategy to SER is to reduce the concentrations of surfactants while maintaining the efficiency of the surfactant solutions.

Polycyclic aromatic hydrocarbons (PAHs) are ubiquitous pollutants resulting from the incomplete combustion of fuel and organic matter. The major source of PAHs is the petroleum product, such as coal tar, creosote, and vehicle emissions. The levels of these compounds in the environment are regulated by government agencies because of the genotoxicity of some high molecular weight PAHs, i. e., the compounds containing four or more rings. Pyrene with four-rings, is usually considered to be representative of the behavior of many PAHs. Many microorganisms, including bacteria, algae, and fungi, are shown to possess degradative enzymes for the oxidative degradation of PAHs (Cerniglia, 1981), however, the in-situ microbial metabolism of PAHs is limited by low bioavailability. Surfactants have been employed with attempts to increase the aqueous phase concentrations of PAHs and thereby accelerate their biodegradation. Removal of PAHs from contaminated soil and groundwater by surfactants is governed by the concentration of surfactants in the form of micelle and microemulsion to solubilize the pollutants. It has been demonstrated that the solubility of PAH increases linearly with the surfactant concentration above the critical micelle concentration (CMC) (Edwards, 1991) and the addition of surfactant to pure cultures of

microorganisms can increase the extent of PAH metabolism (Tiehm, 1994).

There has been also a growing concern for the presence of the enormous quantity of surfactants in the environment because they could influence the fate of other pollutants in surface water and groundwater (Kile, 1990). Various kinds of surfactants usually coexist in water and soil. The sorbed surfactants may significantly affect the properties of soils/sediments and suspended particles, and thus the transport and transformation of organic pollutants (Chiou, 1991; Lee, 2000). An understanding of the water solubility enhancement of organic compounds by mixed surfactants should extend the scope of the remediation for soils and groundwater and provide information on the effect of mixed surfactants on the behavior of organic pollutants in water and soil.

Synergistic sensitization and solubilization of mixed surfactants on color reaction systems have been studied earlier for their analytical applications (Qi, 1986; 1992). To our knowledge, no studies have been conducted on the apparent water solubilities of PAHs and organic compounds in mixed surfactant solutions. The objectives of the present study are (1) to compare and evaluate the capabilities of some mixed surfactants in enhancing the water solubility of pyrene, and (2) to elucidate the cause for synergistic solubilization of pyrene in mixed-surfactant solutions. The experiments are intended to ascertain if a mixed surfactant solution with a less total surfactant amount for reducing the surfactant amount may be used in the surfactant-enhanced remediation of organic contaminants.

1 Experimental section

Surfactants were obtained either directly from the manufacturer or through a distributor and were used without further purification. Triton X-100 (TX100) and Triton X-405 (TX405) were obtained from Rohm and Hass. The purity of TX100 was greater than 98%, while the TX405 contained 70% pure material in aqueous solution. Brij 35 was obtained from Aldrich Chemical Co., with a purity of 97% and more. All Triton and Brij surfactants are molecularly nonhomogeneous because of variable ethylene oxide (EO) chain lengths in each of these surfactants. Sodium dodecyl sulfate (SDS) was obtained from Aldrich Chemical Co., with a purity of 98%. Pyrene obtained from Aldrich Chemical Co., had a purity of 99%. Methanol used for pyrene standard preparation and sample analysis was an HPLC-grade solvent by Fisher Scientific. Deionized water used for preparing solutions was obtained from a Sybron/Barnstead Nanopure II water treatment system.

Table 1 shows the molecular weights, critical micelle concentrations (CMCs) of TX100, TX405, Brij 35, and SDS. The average EO chain lengths in TX100 and TX405 are 9.5 and 40, respectively; Brij 35 has an average EO chain length of 23.

Table 1 Properties of surfactants and partition coefficients of pyrene

Surfactant	MW	CMC, mmol/L	K_{mn}	K_{mc}	$\Delta K_{mc}, \%$
Triton X-100	628	0.20	2.72×10^4	1.08×10^5	
Triton X-405	1966	0.21	1.70×10^3	2.21×10^4	
Brij 35	1200	0.047	1.88×10^4	6.85×10^4	
SDS	288.38	5.50	1.27×10^3	3.04×10^4	
SDS-Triton X-100		0.028		1.07×10^5	
SDS-Triton X-405		0.010		3.22×10^4	39.49
SDS-Brij35		0.0054		8.82×10^4	26.32

Note: K_{mn} is 1.32×10^3 for SDS solution at 5.0 mmol/L

Surface tensions of surfactant solutions were determined with a Model 20 surface tensiometer, manufactured by Fisher Scientific. The critical micelle concentration (CMC) was determined by measuring the surface tension of surfactant solutions over a wide concentration range and noting the inflection in the plot of surface tension versus surfactant concentration; the plotted surface tension value was taken when stable reading were obtained for a given surfactant concentration, as indicated by at least three consecutive measurements having nearly the same value.

The CMC values were obtained through a conventional plot of the surface tension versus the surfactant concentration. Fig. 1-3 show the surface tension versus the surfactant concentration for TX100, SDS and SDS-TX100 solutions, respectively. The CMC values of TX100, TX405, Brij 35 and SDS are 0.20 mmol/L (126 mg/L), 0.21 mmol/L (413 mg/L), 0.047 mmol/L (56 mg/L), and 5.5 mmol/L (1586 mg/L), respectively. The CMC values of surfactants in this study are somewhat lower than those reported earlier by Kile *et al.* (Kile, 1989).

Procedures for sample equilibration and solubility determination were essentially the same as described earlier by Kile *et al.* (Kile, 1989). Single surfactant/mixed surfactant solutions were placed in 25 ml Corex centrifuge tubes with Teflon cap liners, and pyrene was added to each tube in an amount slightly more than required to saturate the solution. Duplicate samples were prepared for each surfactant concentration; these samples were then equilibrated on a reciprocating shaker for 24h at $25 \pm 1^\circ\text{C}$. The samples were subsequently centrifuged at 5000 r/min (2987g) for 1h to separate the undissolved solute. Pyrene

particles adhering to the glass walls were carefully removed with a cotton swab, and the meniscus was subsequently aspirated to remove any particles suspended on the surface. This centrifugation/cleaning procedure was repeated. An appropriate aliquot of the supernatant was then carefully withdrawn with a volumetric pipet and diluted to 10 ml with surfactant-water-methanol solution. Pyrene in solution was analyzed by the UV absorbance at 333.6 nm. In order to eliminate the effect of surfactants on the analysis of pyrene, the surfactant concentration was kept constant in the standard and experimental solutions; the methanol concentration in the solution was also kept constant at 10% (V/V).

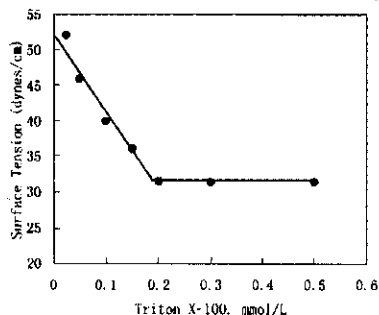


Fig.1 Plot of the surface tension versus the concentration of TX100 solution

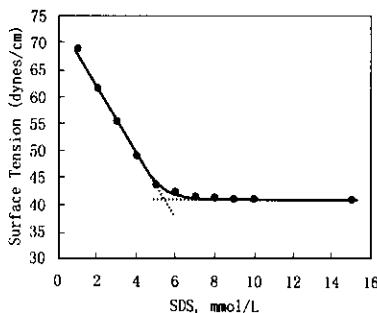


Fig.2 Plot of the surface tension versus the concentration of SDS solution

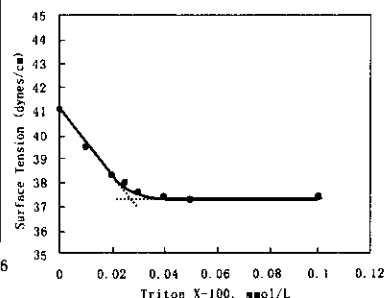


Fig.3 Plot of the surface tension versus the concentration of TX100 in SDS-TX100 mixed solution

2 Results and discussion

2.1 Water solubility enhancements of pyrene by surfactants

The solubility enhancement of a solute in a surfactant solution is related to the concentrations of monomer and micelle and the corresponding solute partition coefficients (Kile, 1989), in which

$$S_w^*/S_w = 1 + X_{mn}K_{mn} + X_{mc}K_{mc} \quad (1)$$

Where S_w^* is the apparent solute solubility at a total stoichiometric surfactant concentration of X ; S_w is the intrinsic solute solubility in "pure water"; X_{mn} is the concentration of the surfactant as monomer; and X_{mc} is the concentration of the surfactant in micelle form. K_{mn} is the partition constant of the solute between surfactant monomer and water, and K_{mc} is the partition constant of the solute between micelle and water. The separate surfactant concentration terms (X_{mn} and X_{mc}) account for differences in partition efficiency of the solute with surfactant monomer and micelle.

Enhancements of pyrene water solubility by TX100, TX405, Brij 35, SDS at surfactant concentrations ranging from below the CMC to above the CMC are shown in Fig.4 – 7. Earlier study gives the solubility of pyrene in pure water (S_w) as 0.133 mg/L at 25°C. The water solubility of pyrene was greatly enhanced by all surfactants above the CMC; the apparent aqueous solubility increases with increasing surfactant concentration. The observed difference in solubility enhancement by surfactants above and below their CMCs is a result of the different abilities of micelle and monomer to concentrate pyrene by partition interactions. The extent of solubility enhancement for pyrene by surfactants below the CMC follows the order of TX100 > Brij 35 > TX405 > SDS. The measured K_{mn} values are 2.72×10^4 for TX100, 1.88×10^4 for Brij 35, 1.70×10^4 for TX405, 1.27×10^4 for SDS, respectively. However, the sequence of the solubility enhancements by surfactants above the CMC is TX100 > Brij 35 > SDS > TX405. The corresponding K_{mc} values are 1.08×10^5 for TX100, 6.85×10^4 for Brij 35, 3.04×10^4 for SDS, 2.21×10^4 for TX405, respectively. For examples, the S_w^*/S_w values in surfactant solutions of TX100 (at 3.14 g/L), Brij 35 (at 3.6 g/L), TX405 (at 3.92 g/L), and SDS (at 3.46 g/L) are 322, 236, 57, and 50, respectively.

2.2 Water solubility enhancements of pyrene by mixed surfactants

Water solubility enhancements of pyrene by mixed surfactants of SDS-TX100, SDS-TX405 and SDS-Brij 35 were determined and compared with those by single surfactants. In these mixed-surfactant systems, the concentration of SDS was kept at 5.0 mmol/L (1.44 g/L). The apparent water solubilities of pyrene in mixed surfactant solutions are found to be generally higher than those in single-surfactant solutions at comparable surfactant concentrations. The sequence of water solubility enhancements of pyrene by mixed surfactants is SDS-TX100 > SDS-Brij 35 > SDS-TX405. With the concentrations of the nonionic surfactants in mixed-surfactant solutions at 4.0 mmol/L (2.51 g/L) for TX100, 2.0 mmol/L (2.40 g/L) for Brij 35, 2.0 mmol/L (3.98 g/L) for TX405, the S_w^*/S_w values in the mixed surfactant solutions are 346 for SDS-TX100, 275 for SDS-Brij 35, 187 for SDS-TX405, respectively. The synergistic solubilization of pyrene by a mixed-surfactant solution at a given surfactant concentration may be expressed as:

$$\Delta S\% = [(S_w^* - S_w) - (S_w^* - S_w)] / (S_w^* + S_w) \times 100 \quad (2)$$

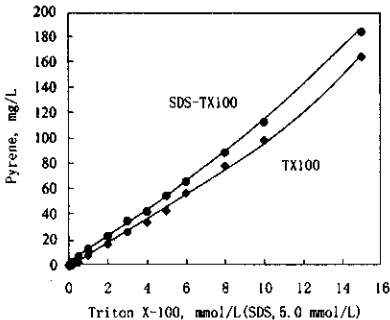


Fig.4 Water solubility enhancements of pyrene by TX100 and SD S-TX100 solutions

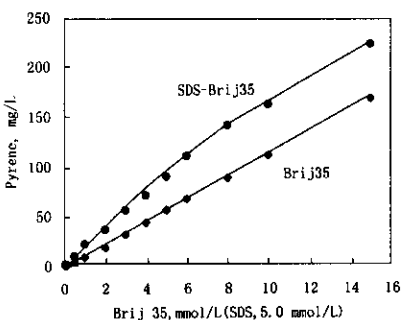


Fig.5 Water solubility enhancements of pyrene by Brij 35 and SDS-Brij 35 solutions

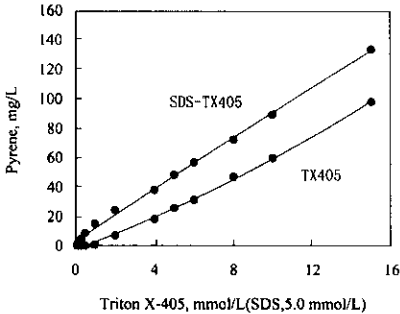


Fig.6 Water solubility enhancements of pyrene by TX405 and SDS-TX405 solutions

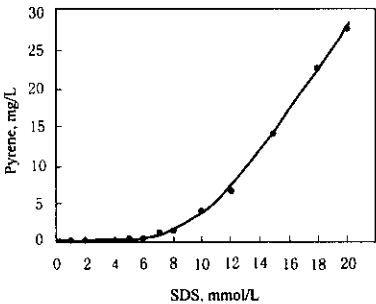


Fig.7 Water solubility enhancements of pyrene by SDS solutions

Where ΔS is the enhanced solubilization of pyrene by a SDS-nonionic surfactant mixture; S_w^* is the apparent water solubility of pyrene at a given surfactant concentration in a mixed surfactant solution; S_{w1}^* and S_{w2}^* are the apparent water solubility of pyrene in SDS solution and a nonionic surfactant solution, respectively at the same surfactant concentration.

Results in Table 2-4 show that pyrene is synergistically solubilized in each of the mixed SDS-nonionic surfactant solutions. The apparent water solubility of pyrene is synergistically enhanced with the concentration of the nonionic surfactant in the mixed-surfactant solution maintained with 5.0 mmol/L of SDS. The improved solubilization by mixed surfactants for pyrene is related to the composition and concentration of the SDS-nonionic surfactant mixture. The degree of pyrene solubility enhancement by the mixed-surfactant system follows order of SDS-TX405 > SDS-Brij 35 > SDS-TX100. The effect is more pronounced at lower concentrations than at higher concentrations of the nonionic surfactants in the surfactant mixture. For examples, with the TX405 concentration in SDS-TX405 mixtures at 0.50 mmol/L (9.83 g/L), the calculated ΔS value for pyrene is more than 9; the ΔS value decreases proportionally with increasing TX405 concentration (Table 4).

Table 2 Water solubility enhancements of pyrene by SDS-Triton X-100 solutions (SDS, 5.0 mmol/L)

Triton X-100, mmol/L	0.10	0.20	0.50	1.0	2.0	3.0	4.0	5.0
Pyrene, mg/L	1.595	2.605	6.999	13.02	23.08	35.09	46.08	55.60
ΔS , %	128.4	145.2	147.3	53.37	35.07	31.86	34.69	28.46
Triton X-100, mmol/L	6.0	8.0	10.0	15.0				
Pyrene, mg/L	65.70	89.08	113.0	184.3				
ΔS , %	15.40	13.30	13.33	12.18				

Table 3 Water solubility enhancements of pyrene by SDS-Brij 35 solutions (SDS, 5.0 mmol/L)

Brij 35, mmol/L	0.05	0.10	0.50	1.0	2.0	3.0	4.0	5.0
Pyrene, mg/L	1.803	2.012	10.08	22.18	36.59	56.03	70.56	89.49
ΔS , %	112.7	112.1	153.6	149.1	95.81	77.09	63.98	59.38
Brij35, mmol/L	6.0	8.0	10.0	15.0				
Pyrene, mg/L	110.5	140.9	161.5	221.7				
ΔS , %	66.47	59.89	45.03	32.30				

Table 4 Water solubility enhancements of pyrene by SDS-Triton X-405 solutions

	(SDS, 5.0 mmol/L)							
Triton X-405, mmol/L	0.10	0.20	0.30	0.50	1.0	2.0	3.0	4.0
Pyrene, mg/L	1.389	2.595	5.224	9.130	15.52	24.87	38.50	
ΔS , %	147.4	323.3	798.5	929.9	839.1	210.4	100.9	
TritonX-405, mmol/L	5.0	6.0	8.0	10.0	15.0			
Pyrene, mg/L	48.75	57.18	72.69	90.13	134.1			
ΔS , %	84.02	76.21	51.81	48.59	35.49			

In earlier studies, the metal-organic complexes in water solutions were sensitized and solubilized synergistically by mixed micelle of mixed surfactants (Qi, 1986; 1992; 1987). In the present investigation, with fixed SDS concentrations, the CMC values of the nonionic surfactants were found to decrease sharply in the mixed surfactant solutions. This effect appears to result from the formation of mixed micelle consisting of the two surfactants. Table 1 shows that the CMC values of TX100, TX405, Brij 35 are reduced to 0.028, 0.010, and 0.0054 mmol/L in the presence of 5.0 mmol/L of SDS. As such, the micelle concentration in the mixed-surfactant solution increases greatly over that in the single-surfactant solution.

Calculations by Eq. (1) indicated that the partition coefficient of the solute between micelle and water (K_{mc}) for SDS-TX405, SDS-Brij 35 and SDS-TX100 were 3.22×10^4 , 8.82×10^4 , 1.07×10^5 , respectively. The synergistic effect for a mixed-micelle system may be expressed alternatively in terms of K_{mc} :

$$\Delta K_{mc} = [K_{mc} - (K_{mc1} + K_{mc2})] / (K_{mc1} + K_{mc2}), \quad (3)$$

where K_{mc} , K_{mc1} and K_{mc2} are the partition coefficients of the pyrene between micelles and water in the mixed surfactant, nonionic surfactant and SDS solutions, respectively. The K_{mc2} value is 1.32×10^3 for the SDS solution at 5.0 mmol/L.

According to Eq. (3), the ΔK_{mc} values of SDS-TX405, SDS-Brij 35 are 39.49% and 26.32% higher respectively than the same of K_{mc1} and K_{mc2} . However, the K_{mc} value of SDS-TX100 was closed to that of TX100 solution. The combined data on CMCs and K_{mc} 's indicate that the synergistically enhanced water solubility of pyrene by mixed surfactants is caused by an increase in K_{mc} and/or a decrease in the CMC of the mixed-surfactant solutions. For SDS-TX405 and SDS-Brij 35 mixed solutions, an increase in K_{mc} is coupled by a decrease in the CMC value; while for SDS-TX100, only a decrease in CMC value takes place. Therefore, the sequence of synergistic solubilization of mixed surfactants on pyrene follows the order of SDS-TX405 > SDS-Brij 35 > SDS-TX100.

3 Conclusion

The experimental data from this study suggests that the use of mixed surfactants may potentially improve the performance of surfactant-enhanced remediation (SER). The observed enhanced solubilization of pyrene, especially at low surfactant levels could promote the biodegradation of non-aqueous-phase organic pollutants; meanwhile this could reduce the surfactant pollution and remediation expenses.

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