Aqueous Dissolution of Phenanthrene from Loess Soil using Triton X-100 and Sodium Dodecylbenzene Sulfonate

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Surfactant-enhanced remediation is a feasible technology for the clean-up of hydrophobic organic contaminants in subsurface, in which enhanced desorption of hydrophobic organic contaminants from soil medium plays a key role in remediation efficiency. In this paper, solubilization and washing of phenanthrene from loess by Triton X-100 (TX100), sodium dodecylbenzene sulfonate (SDBS) and their mixture (TX100-SDBS) solutions were tested and compared. The effects of inorganic salts (Na^+, Ca^{2+} and Mg^{2+}) on solubilization and adsorption of surfactants onto loess were investigated simultaneously. It was found that the solubilization capacities of surfactants were in the order TX100 > 3:1 TX100-SDBS > 1:1 TX100-SDBS > 1:3 TX100-SDBS > SDBS and the solubilization ratios (SR) of these surfactants were 0.021, 0.018, 0.014, 0.009 and 0.002, respectively. A synergistic solubilization was obviously found in mixed TX100-SDBS systems. An appropriate addition of inorganic salts could enhance solubilization and the mixed TX100-SDBS could endure harder water than single SDBS did. The adsorption of TX100 onto loess could be reduced by anionic surfactant. The soil washing experiment demonstrated a high desorption of phenanthrene from spiked loess soil by 3:1 and 1:1 TX100-SDBS, compared with by single TX100 or SDBS. The results showed mixed surfactant could be an attractive alternative to single ones in the remediation of the contaminated loess soils.

Key Words: Surfactant, Solubilization, Soil washing, Adsorption, Phenanthrene.

INTRODUCTION

Loess soils topographically spread the vast area expanding from central Asia to north China. Because the natural conditions of the loess soil regions at northern China are different from those in other regions, the important problems are to develop the available cleanup techniques that would be applied for the *in situ* or *ex situ* remediation of petroleum polluted soils where oil fields are widely exploited and oil resulted in the severe contamination of soils[1].

A number of reports have indicated that *in situ* flushing and *ex situ* washing by surfactant are the feasible techniques for the remediation of soils contaminated by hydrophobic organics, such as non-aqueous phase liquids (NAPLs), poly aromatic hydrocarbons (PAHs) and poly chlorinated biphenyls (PCBs), *etc.*, which are more effective than the traditional pump-and-treat method[2-4]. In general, the flushing and washing technologies involve the addition of surfactants to enhance desorption of hydrophobic organic contaminants (HOCs) from soils or displacement of NAPLs from soil pore space. The mechanisms on surfactant-enhanced flushing and washing are micellar solubilization and mobilization[3,4]. The mobilization

is the immiscible removal of NAPLs as free liquids, due to the interfacial adsorption of surfactant monomers at the interface between NAPL and water and reduction in the interfacial tension (IFT), which are derived from ternary oil recovery[3]. However, mobilizing NAPLs faces the risk of downward migration of dense non-aqueous phase liquids (DNAPLs) that are denser than the surrounding water, entering uncontaminated zones and aquifers as free liquids. Mobilization needs the formation of middle phase microemulsion (water-surfactant-oil)[3]. Thus, it is not efficient for the removal of sorbed HOCs on soils. This is limitation for the implementation of mobilizing method in remediation[5]. Moreover, reducing interfacial tension would allow the displaced free products flow into smaller pores that may be less readily accessible during remediation efforts[6]. Solubilization is usually use to the micellar surfactant solutions to increase the aqueous apparent solubilities of contaminant in a single phase microemulsion, thus, increasing desorption efficiency[3,4]. This method has been shown to be effective in numerous examples[7-13], which poses less risk of uncontrolled migration and are less complex to design.

Surfactant selection is important for remediation technology. The usual surfactants used in remediation are non-ionic and

anionic ones. The non-ionic surfactants, *e.g.*, Triton X-100 (TX100) and Tween 80, have received much attention due to their relative high solubilization capacity and their high volume of production in industry[14,15]. However, further evidence indicates that their adsorption onto subsurface mediums[16] and partitioning from water phase into NAPLs[17-19] result in large losses of surfactant and reducing the efficiency for dissolution removal of contaminants. In general, anionic surfactants, *e.g.*, sodium dodecylbenzene sulfonate (SDBS) and sodium dodecyl sulfate (SDS), neither adsorb heavily onto soils because of the repulsion force of negative charges between surfactant ions and soil surfaces[16] nor partition into NAPLs[20]. However, they can solubilize less organic compounds than non-ionic ones in unit mass[14,15]. Mixed surfactants are of practical and fundamental interest in industrial applications. They can be easily obtained because most commercial and industrial surfactants applications involve several surfactant types or several isomers of a particular surfactant type. Anionic and non-ionic surfactants usually form mixed micelles in aqueous phase, whose properties, such as solubilization capacity for organics, Krafft point, cloud point, capacity to endure salinity and hardness, are of advantage over those of the individual ones[21,22]. Moreover, our previous work showed that mixed anionic-non-ionic surfactants had advantageous solubility behaviour and low partitioning loss into DNAPLs as compared to individual ones[21,22]. Consequently, this may leads to a better flushing and washing efficiency for HOCs in soils.

To our knowledge, few studies have been conducted for loess soil remediation and dissolution of HOCs in loess soil mediums by mixed anionic-non-ionic surfactant. In this paper, TX100 and SDBS were chosen as the representative of nonionic and anionic surfactants and phenanthrene as HOCs. The objectives are (i) to compare the apparent solubilization extents for phenanthrene by single and mixed surfactant, together with the effect of inorganic salts on solubilizationin; (ii) to test adsorption of surfactant onto loess soil and (iii) to investigate the dissolution of phenanthrene from spiked loess soil by single and mixed surfactant, using batch experiment. The results may demonstrate an implication for the remediation of loess soils contaminated by HOCs.

EXPERIMENTAL

Sodium dodecylbenzene sulfonate (SDBS) and sodium dodecyl sulfate (SDS) with analytical grade were purchased from Laiyang Chemical Company, China. TX100 was obtained from Acros Organics, USA and used as received. Phenanthrene with analytical grade was obtained from Aldrich Chemical Company, USA. The physical and chemical parameters of reagents are listed in Table-1. Methanol was analytical grade and purified water was used for all tests.

A vadose zone loess soil sampled from Lanzhou, China, was air dried and sieved through a 0.28 mm sieve. The soil

has a pH of 8.14 and organic matter of 0.20 %. 250 g of soil was spiked with 0.1 g phenanthrene in acetone and mixed homogeneously. The acetone was evaporated under hood and the spiked soils with phenanthrene were mixed and homogenized. The initial concentration was 400 mg phenanthrene/kg soil.

Solubilization: The procedures were similar to those in the previous report[22,23]. Surfactants were used as single or mixed with the following concentration, 200, 500, 1000, 2000, 4000, 6000, 8000 and 10000 mg/L. The mixed surfactant solutions were prepared at the initial mass ratios of 3:1, 1:1 and 1:3 of TX100 to SDBS. A series of 20 mL of surfactant solutions were transferred into 50 mL flasks. Phenanthrene was separately added to each flask in an amount slightly more than required to saturate the solution. The samples were tapped and sealed with Parafilm (Parafilm M., USA). For each surfactant concentration, duplicate samples were prepared. Then, these samples were equilibrated on a reciprocating shaker (CHA-S Shaker, Jintan Danyang Instrumental Company, China) for 48 h at 120 rpm of speed and 25 ± 1 ℃ of temperature. The samples were subsequently centrifuged for 0.5 h at 4000 rpm of speed. An appropriate aliquot of the supernatants was then carefully transferred and diluted to 25 mL in flasks with methanol and water. The absorbance of diluted samples was tested at 248 nm on spectrophotometer (Model 752, Shanghai Spectrum Instrumental Company, China) with 1.0 cm quartz cell. The concentrations of phenanthrene were quantified from the calibration. When the effects of inorganic salts were tested, the concentrations of surfactants were kept as 2000 mg/L and various amounts of NaCl, CaCl$_2$ and MgCl$_2$ were added in the samples. Then, the procedures of equilibration, centrifugation and quantification were conducted.

Adsorption of surfactant: A total of 20 mL of TX100 or SDBS solution with 100, 200, 300, 400, 500, 800 and 1000 mg/L of initial concentration was mixed with 0.5 g of phenanthrene-free loess soil in 50 mL flask. These flasks were shaken on the reciprocating shaker for 24 h at 120 rpm of speed 25 ± 1 ℃. The soil and aqueous phase were separated by centrifugation (4000 rpm for 0.5 h). Then the concentrations of TX100 or SDBS in supernatant were determined on the spectrophotometer at 223 nm. The amounts of surfactant onto soil were calculated from initial and equilibrium concentrations in aqueous phase. When the effect of anionic surfactant on adsorption of TX100 was tested, a series of TX100 solutions with concentrations mentioned above, were mixed with 1000 mg/L of sodium dodecyl sulfate and then the adsorption experiment was carried out.

Soil washing: Batch soil washing experiments were conducted by placing a constant ratio (0.5 g to 20 mL) of soil to surfactant solution while the dissolution of phenanthrene was evaluated. A series of 0.5 g of phenanthrene-spiked loess

TABLE-1
PHYSICAL AND CHEMICAL PARAMETERS OF REAGENTS

Reagent	Molecular formula	Molar weight (g/mol)	Solubility[a] (mg/L)	CMC[b] (mg/L)
TX100	$C_8H_{17}C_6H_4(OCH_2CH_2)_{9.5}OH$	625.00	–	167.4
SDBS	$C_{12}H_{25}C_6H_4SO_3Na$	348.48	–	963.2
Phenanthrene	$C_{14}H_{10}$	178.17	1.182	–

[a]Water solubility[23], [b]The critical micellar concentration[22].

soil was added and 20 mL of surfactant solutions with 200, 500, 1000, 2000, 4000 and 6000 mg/L of initial concentrations were placed into flasks. The controls were prepared using phenanthrene-free soil. These samples and controls were shaken (120 rpm for 12 h), centrifuged (4000 rpm for 0.5 h) and quantified at 248 nm.

RESULTS AND DISCUSSION

Solubilization: Enhanced solubility of HOCs is always used as an index for evaluating the surfactant-enhanced remediation process. Fig. 1 shows the relationship of the apparent solubilities of phenanthrene (S^*) as a function of concentration of TX100, 3:1 TX100-SDBS, 1:1 TX100-SDBS, 1:3 TX100-SDBS and SDBS, respectively. The linear relation was observed between the apparent solubility and surfactant concentration. As shown in Fig. 1, given low surfactant concentration (< 1000 mg/L), single SDBS exhibited little capacity for phenanthrene solubility enhancement while single TX100 improved phenanthrene solubility linearly with its concentration. With 1000 mg/L of surfactant, the apparent solubilities of phenanthrene are 19.5 and 2.86 mg/L by TX100 and SDBS, respectively. This is attributed to the difference of CMC between of TX100 and of SDBS. Below 1000 mg/L of concentration, the molecules of SDBS can not aggregate to form micelle due to its critical micelle concentration is 963.2 mg/L. The effects of surfactant monomers on solubility enhancement of organic compounds are negligible[14]. The CMC of TX100 is 167.4 mg/L, so the obvious solubility enhancements for phenanthrene occurred at low concentration. On the basis of ideal mixing principle of mixed anionic-non-ionic surfactants[20], the CMCs of mixed TX100-SDBS are between those of single anionic and non-ionic one. Thus, the apparent solubilities of phenanthrene by mixed TX100-SDBS were between those by TX100 and SDBS alone.
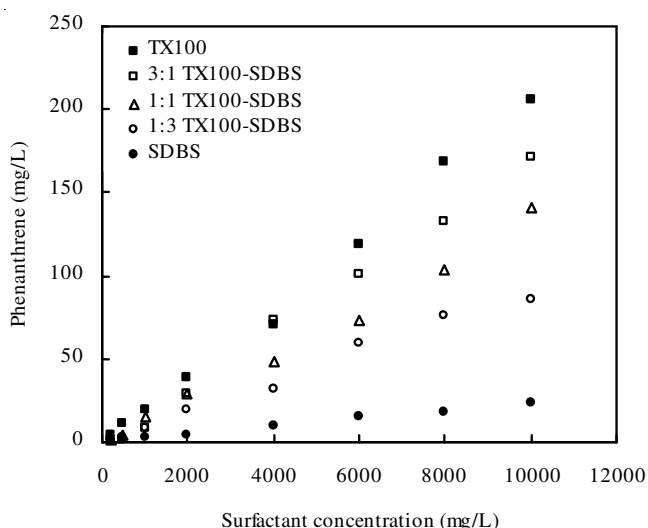

Fig. 1. Relationship between the apparent solubilities of phenanthrene and surfactant concentrations

When the surfactant addition were much more (*i.e.* 1000-10000 mg/L), the obvious solubility enhancements were observed and the difference of solubilization capacity by tested surfactants occurred, which are the results of partitioning of

hydrophobic compounds from aqueous phase into pseudo-micellar phase. Given 10000 mg/L of surfactant concentration, the apparent solubilities of phenanthrene are 206, 172, 141, 85.9 and 22.9 mg/L in the presence of TX100, 3:1 TX100-SDBS, 1:1 TX100-SDBS, 1:3 TX100-SDBS and SDBS. From the slopes of the solubilization curves, it could be also observed that the extents of solubilization by the surfactants followed the order TX100 > 3:1 TX100-SDBS > 1:1 TX100-SDBS > 1:3 TX100-SDBS > SDBS. The more the fraction of TX100 in mixed surfactant, the larger the solubilization capacity of mixed surfactant. The mass solubilization ratio (SR) is a quantitative measurement of effectiveness of a particular surfactant in solubilizing a given solute[24]. The solubilization ratio is defined as the grams of organic compounds solubilized per gram of surfactant added to the solution:

$$\text{Solubilization ratio (SR)} = (S^*_{mic} - S^*_{cmc}) / (C - CMC) \quad (1)$$

where S^*_{cmc} is the apparent solubility of organic compound at the CMC of surfactant; S^*_{mic} is the total apparent solubility of organic compound in micellar solution at a particular surfactant concentration C at which S^*_{mic} is evaluated. Solubilization ratio can be obtained from the slope of solubilization curves. The calculated values of solubilization ratio are shown in Table-2. The SR by the tested surfactants follwed the order TX100 > 3:1 TX100-SDBS > 1:1 TX100-SDBS > 1:3 TX100-SDBS > SDBS.

TABLE-2
SOLUBILIZATION RATIOS OF PHENANTHRENE BY
SINGLE AND MIXED SURFACTANTS

Surfactant	Regression equation	R^2	SR
TX100	$S^* = 0.0207\ C - 2.61$	0.9970	0.021
3:1 TX100-SDBS	$S^* = 0.0177\ C - 4.17$	0.9952	0.018
1:1 TX100-SDBS	$S^* = 0.0136\ C - 1.23$	0.9916	0.014
1:3 TX100-SDBS	$S^* = 0.0092\ C - 0.36$	0.9902	0.009
TX100	$S^* = 0.0023\ C + 0.55$	0.9902	0.002

For this binary mixed surfactant systems, S^*_{mix} is defined as the aqueous solubility of phenanthrene by TX100-SDBS in aqueous phase and S^*_{sum}, the calculated solubilities of phenanthrene in the mixed solutions based on the ideal addivity rule[20]. S^*_{sum} was calculated as:

$$S^*_{sum} = (S^*_1 + S^*_2) - S_w \quad (2)$$

where S^*_1 is the apparent solubility of phenanthrene in sole TX100 solution in which TX100 concentration is equal to that in the TX100-SDBS system; S^*_2 is the apparent solubility in sole SDBS solutions in which SDBS concentration is equal to that in the TX100-SDBS system. Both of them can be obtained from the plots of the apparent solubility of phenanthrene *versus* the concentration of single surfactant (Fig. 1) or calculated from the regression equation (Table-2). S_w is the intrinsic solubility of phenanthrene in water. Define the difference ΔS as followed:

$$\Delta S = (S^*_{mix} - S^*_{sum}) / S^*_{sum} \times 100 \quad (3)$$

If the difference ΔS are positively lager than zero, what is the synergistical solubilization is observed. Accordingly, the value of ΔS illustrates the extent of synergistic solubilization. Given the total surfactant concentration from 2000 to 10000 mg/L, the synergistical solubilization by mixed TX100-SDBS

at mass ratios of 3:1, 1:1 and 1:3 was detected (Table-3). The results suggest mixing of anionic with non-ionic surfactants could enhance the efficiency of solubilization for phenanthrene.

TABLE-3 COMPARISON OF SYNERGISTIC SOLUBILIZATION BY MIXED SURFACTANT FOR PHENANTHRENE					
Mixed surfactant	ΔS (%)				
	Total concentration of mixed surfactant (mg/L)				
	2000	4000	6000	8000	10000
3:1 TX100-SDBS	7.42	23.4	9.48	6.54	9.25
1:1 TX100-SDBS	49.7	15.3	12.8	17.7	27.7
1:3 TX100-SDBS	87.1	37.1	58.6	46.5	32.2

The performance of surfactant is often strongly affected by electrolytes in solution[20]. This is particular important in remediation application. Subsurface matrix solutions contain electrolytes such as Na^+, Ca^{2+} and Mg^{2+}, *etc.*, which can influence the solubilization capacity of surfactant. Table-4 shows the effects of common inorganic salts on solubilization behaviours by tested surfactants. The effects on solubilization changed with concentrations and types of salts. Addition of NaCl enhanced the solubilization extent of single and mixed surfactants. Increasing NaCl concentration resulted in phenanthrene solubility enhancement. Moreover, no precipitation of anionic surfactant was observed even in 10000 mg/L of NaCl. For non-ionic surfactant system, phenanthrene solubility enhancement by inorganic salts is mainly due to salting out. For anionic and anionic-non-ionic surfactant systems, this may be due to salting out and interaction of counter ions on ionic micelles. In cases of $CaCl_2$ and $MgCl_2$, obvious effects on phenanthrene solubility enhancement by SDBS and TX100-SDBS were observed even at low salt concentration. In single SDBS system, Both 150 mg/L of $CaCl_2$ and 200 mg/L of $MgCl_2$ had brought about the precipitation of SDBS, which indicates that single SDBS has low capacity to endure hard water. However, at the tested concentration, $CaCl_2$ and $MgCl_2$ did not brought bout precipitation in mixed TX100-SDBS systems, instead of phenanthrene solubility enhancement. This illustrated that the capability of anionic surfactant to endure hard water improved in the presence of non-ionic surfactant.

Adsorption of surfactant: Fig. 2 shows the adsorption of TX100 and SDBS onto loess soil when the initial surfactant concentrations are from 100 to 1000 mg/L. SDBS was less sorbed by soil, with 2.52 mg/g of adsorption quantity (Q_e) at

1000 mg/L of initial SDBS concentration. Three phases were observed on adsorption curve of TX100. When the addition of TX100 was no more than 200 mg/L, Q_e of TX100 increased linearly with its equilibrium concentration ($C_{e,s}$). When the concentration of TX100 was more than 200 mg/L, Q_e dramatically increased. A maximum Q_e 4.32 mg/g reached until the initial TX100 concentration was up to 400 mg/L. Hydrogen bonding[25] and electrostatic attraction between the negative charged soil surface and the TX100 molecule with slight positive charge[26] may be responsible for TX100 sorption. Due to the negatively charged soil surface, anionic surfactants are expected to be sorbed less than non-ionic surfactants because of electrostatic repulsion. However, precipitation of anionic surfactant ion (DBS^-) with divalent cations (*e.g.*, Ca^{2+}, Mg^{2+}) would be significant and result in SDBS loss, as shown in Table-4. In the presence of 1000 mg/L of SDS, the sorption of TX100 onto soil decreased considerably. Thibaut *et al.*[27] indicated that the free engery of mixed anionic-non-ionionic surfactant micellization was more negative than that of adsorption of non-ionic surfactant monomers on soil. Therefore, the mixed micellization is prior to adsorption of TX100, which results in reduction of sorption loss of TX100.
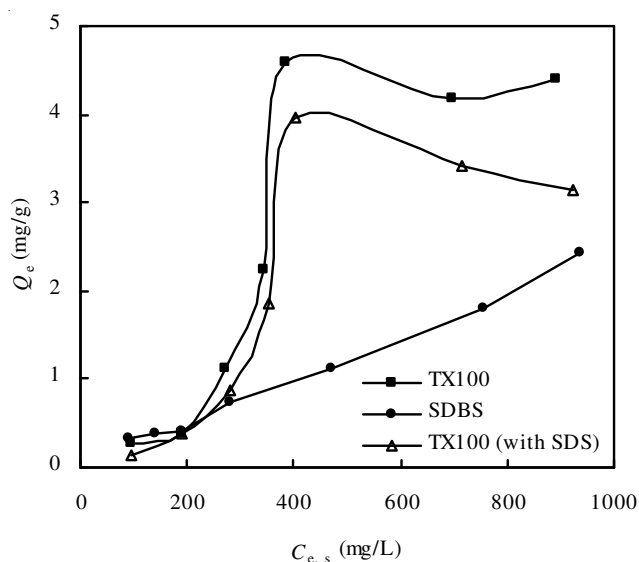

Fig. 2. Adsorption curves of surfactants onto loess soil

Soil washing: Soil washing tests were conducted to evaluate the extent of TX100, SDBS and mixed TX100-SDBS solutions to dissolve phenanthrene from loess soil. The mixed surfactant

TABLE-4 EFFECTS OF INORGANIC SALTS ON SOLUBILIZATION						
Inorganic salt	Inorganic salt concentration (mg/L)	Phenanthrene solubility (mg/L)				
		TX100	3:1 TX100-SDBS	1:1 TX100-SDBS	1:3 TX100-SDBS	SDBS
No salt	0	38.1	28.4	24.8	14.9	4.48
NaCl	500	39.3	39.8	29.2	15.5	4.99
	5000	40.8	46.8	38.3	25.1	7.99
	10000	44.4	53.8	41.2	26.8	8.18
$CaCl_2$	50	38.3	32.9	28.4	16.5	4.99
	150	39.9	36.6	29.7	17.6	Precipitation occurs
	200	39.5	37.5	30.9	17.7	Precipitation occurs
$MgCl_2$	50	39.6	32.8	28.5	17.3	4.99
	200	39.4	42.0	30.7	18.1	Precipitation occurs
	300	37.4	44.6	31.8	20.1	Precipitation occurs

solutions were employed in the washing process at the same experimental conditions as the single surfactant solutions. For each surfactant system, phenanthrene concentration in water phase was quantified as a function of the initial surfactant concentration. Fig. 3 shows the relations between phenanthrene concentration in water phase and the initial surfactant concentration from 200 to 6000 mg/L. As the plots indicated, the phenanthrene concentration in water phase increased with the surfactant addition, at large. However, the washing efficiencies by various surfactants were different. The washing extent using single SDBS was much less that using single TX100 or mixed TX100-SDBS. The removal efficiency of phenanthrene by 6000 mg/L of SDBS was only 24.3 %. The mixed TX100-SDBSs exhibited high capacity to wash phenanthrene, among which 3:1 TX100-SDBS and 1:1 TX100-SDBS were the better ones. The removal efficiencies of phenanthrene by 6000 mg/L of 3:1 TX100-SDBS and 1:1 TX100-SDBS were larger than 90 %, respectively, while by TX100 only 50.1 %. The results demonstrated that the mixed TX100-SDBS at special mass ratios could desorb more organic compound, from loess soil, which could reduce expenditure and the operation cost in remediation application.
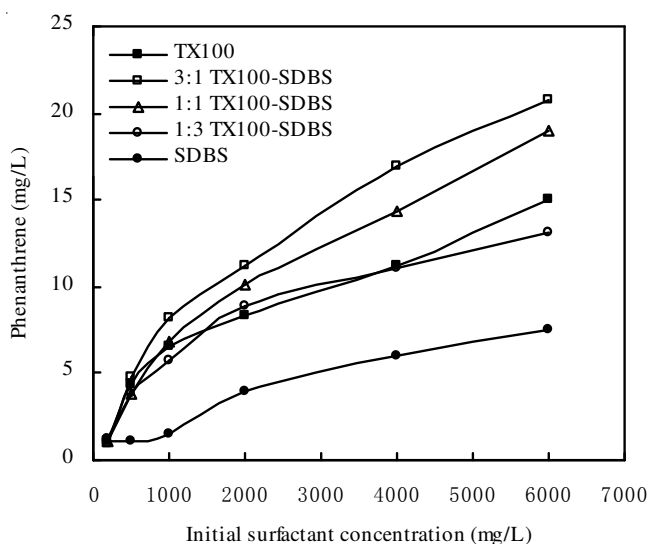

Fig. 3. Relationship between aqueous phase concentrations of phenanthrene and initial concentrations of surfactants

Generally, surfactant-enhanced soil washing involves two mechanisms[28]. Firstly, surfactant monomers are responsible for the soil roll-up mechanisms. Monomers accumulate at the soil-contaminant and soil-water interfaces and increase the contact angle (*i.e.* enhancing the wettability of the systems). Surfactant molecules adsorbed on the surface of the contaminant cause repulsion between the head group of surfactant molecule and the soil particles, thereby, promoting the separation of the contaminant from soil particles. Secondly, micellar solubilization is the main mechanism. Surfactant-enhanced solubilization results in contaminant partitioning into the hydrophobic core of surfactant micelles. Thus, concentration well above the CMC is necessary for this enhancement to be significant[28]. The effect of roll-up is much weaker than solubiliztion. However, the water soubilization capacity of surfactant in soil-water system is somewhat different from that in solution system.

Many factors such as inorganic salts (existing in soil and groundwater), adsorption of surfactant onto soil particles and precipitation of anionic surfactant by hard water (*i.e.*, containing Ca^{2+}, Mg^{2+}) could significantly affect the solubilization efficiency of surfactant. As mentioned above, single TX100, of course, is expected to removal more phenanthrene from loess soil due to its high solubilization ratio (SR). However, sorption loss could reduce the efficient concentration of TX100 in aqueous phase. Though showing lower solubilizing efficiency for phenanthrene than TX100 (Fig. 1), mixed TX100-SDBS solutions, especially at a mass ratio of 3:1 and 1:1, had higher desorption efficiency for phenanthrene than individual ones (Fig. 3). Na^+, Mg^{2+} and Ca^{2+} could increase the apparent solubility of phenanthrene in the mixed TX100-SDBS solutions (Table-4). In the presence of anionic surfactant (SDS), the adsorption of non-ionic surfactant (Triton X-100) onto soil matrix decreased while the precipitation of SDS also decreased in the presence of TX100[16]. Additionally, synergism derived from micellization improved the efficiency of mixed TX100-SDBS for solubilizing phenanthrene. Therefore, the mixing of anionic and non-ionic surfactants was conductive to solubilizing and washing organic contaminants. The results from a few researches also demonstrated this conclusion[29,30]. Laboratory experimental results showed that the sorption of TX100 onto soils was severely restricted in the presence of SDS in batch and column systems and decreased with the increasing mass fraction of SDS in mixed surfactant solutions. The enhancing solubilization of phenanthrene by SDS-TX100 was greater than that by individual surfactant[30]. The column flushing and batch washing showed that desorption efficiencies for phenanthrene-contaminated soil by SDS-TX100 was greater than that by individual surfactant[29,30].

Conclusion

The work presented here demonstrates that TX100-SDBS at mass ratios of 3:1 and 1:1 could desorb much more phenanthrene from loess soil although the solubilization capacities of surfactants were in the order TX100 > 3:1 TX100-SDBS > 1:1 TX100-SDBS > 1:3 TX100-SDBS > SDBS. This is mainly attributed to the advantageous performance of mixed anionic-non-ionic surfactant, such as synergistical solubilization, high capability to endure hardness and salinity, low sorption loss of non--ionic component, low precipitation loss of anionic component, compared with individual ones. The results showed anionic-non-ionic surfactant could be a promising alternative approach for loess soil washing and flushing remediation.

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