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MODELING THE TWO STAGES OF SURFACTANT-AIDED SOIL WASHING

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Abstract—This paper provides new insights into modelling the distribution of hydrophobic compounds between soil and water phases in the presence of nonionic surfactant micelles. Experimental measurements were made of various systems comprising a non-ionic surfactant, five soils of different fractional organic carbon contents, and a hydrophobic (disperse) dye. Soil-washing performance was quantified using reciprocal surfactant-soil solubilization coefficients ($1/K_d$). Two stages of partitioning were identified. In stage 1, the dye concentration increased slightly with increasing surfactant dose until surfactant monomers saturated the bulk solution at the critical micelle concentration (cmc). The washing performance was 1 : 1 proportional to the surfactant monomer concentration. Most of the surfactant in this stage is sorbed. In stage 2, above the cmc, soil-washing performance increased linearly with increasing available surfactant micelles in the bulk solution. Reciprocal surfactant-soil solubilization coefficients ($1/K_d$), octanol–water partition coefficients (K_{ow}), fractional organic carbon content of the soil (f_{oc}), and surfactant concentration were correlated for each stage in the soil-washing process using two simple equations.
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Key words—hydrophobic dye, micelle, modeling, partition, surfactant, soil-washing

NOMENCLATURE

a	a constant for correlating K_m and K_{ow} (l/mol)
b	a constant for correlating K_{oc} and K_{ow} (l/kg)
C_0	total initial concentration in solution (mg/ml)
C_e	equilibrium concentration in solution (mg/ml)
$[D]$	total surfactant dosed to the system (mol/l)
f_{oc}	fractional organic carbon content of the sediment
HOCs	hydrophobic organic compounds
K_d	overall distribution coefficient (l/kg)
K_m	micelle–water partition coefficient (l/mol)
K_{oc}	organic carbon normalized distribution coefficient at equilibrium (l/kg)
K_{ow}	octanol–water partition coefficient
$[P]_{mic}$	concentration of HOCs in the surfactant micelles (mol/l)
$[P]_s$	concentration of HOCs in the soil or sediment (mol/kg)
$[P]_{surf}$	concentration of HOCs solubilized by both monomeric and micellar phases (mol/l)
$[P]_w$	concentration of HOCs in the water (mol/l)
$[S]$	concentration of surfactant in both monomeric and micellar forms (mol/l)
$[S]_{mic}$	concentration of surfactant in micellar form (mol/l)
$[S]_{mon}$	concentration of surfactant in monomer form (mol/l)
$[S]_{sorb}$	concentration of surfactant sorbed to the soil (mol/l)
v	solution volume (ml)

w	weight of the soil (g)
x/m	concentration in soil (mg/g)

INTRODUCTION

The removal of hydrophobic organic compounds (HOCs) from contaminated soil or sediment is becoming a major concern as their water-insoluble characteristics may retain those compounds in solid phase and eventually impair human health through the bio-accumulation, bio-concentration and food-chain systems (Vigon, 1989). Much attention has been paid to the performances of surfactant aided soil-washing of HOCs contaminated soil or sediment (Jafvert *et al.*, 1995; Chu *et al.*, 1998) and to the investigation of the chemical remediation of surfactant/HOCs mixtures extracted from soils and sediments (Chu and Jafvert, 1994; Chu, 1999). Surfactant micelles can strongly enhance the apparent solubility of HOCs and assist the remediation of contaminated soils and sediments, especially for anionic and nonionic surfactants (Kile and Chiou, 1989; Edwards *et al.*, 1991). West and Harwell (1992) have studied surfactants for their ability to act as soil-washing agents and as facilitators of subsurface remediation of hydrocarbon spills. The utilization of surfactants as a sediment-washing agent appears to be a promising strategy for site remediation.

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The extent to which surfactants influence HOC distribution in environmental systems depends on HOC sorption to solid phases (Chiou *et al.*, 1998; Karickhoff, 1984), HOC solubilization by micellar or monomeric surfactant (Almgren *et al.*, 1979; Valsaraj and Thibodeaux, 1989), and surfactant interactions with sediment or soil components (Liu *et al.*, 1992). However, there is relatively little information (Edwards *et al.*, 1994a, b) about how to account for HOC characteristics, soil properties, and surfactant concentrations in a simple model, which can be used to predict the partitioning of HOC between a solid phase (i.e., soils and/or sediments) and a liquid phase (i.e., aqueous solution and surfactant micelles). In particular, effects of surfactant sorption and HOC solubility enhancement associated with surfactant monomers have been neglected in most research. This neglect could cause significant errors in predicting an appropriate surfactant dose for use in soil-washing application. The purpose of this work is to study and model the partitioning of HOC between liquid and solid phases in the presence of soils and surfactant. A practical model is proposed for the solubilization process to facilitate system prediction and operational design. Experimental results support the proposed model. These results include quantifying the distribution of a probe HOC (a typical hydrophobic dye) between solid and liquid phases in a system containing nonionic surfactant monomers and micelles.

MATERIALS AND METHODS

Soil

The soil samples of this study were undistributed, subhumid soil collected from various locations in Hong Kong territory (Shing Mun, Tai Mo Shan, Aberdeen, Tai Mei Tuk and Tai Hang Tun). Five soil samples were air-dried and screened through a US standard No. 10 mesh (2 mm) sieve to remove coarse fragments. The fractions of organic carbon in the soil samples were determined to be 0.197% (Shing Mun), 0.457% (Tai Mo Shan), 0.753% (Aberdeen), 1.039% (Tai Mei Tuk) and 1.477% (Tai Hang Tun) by Shimadzu Total Organic Carbon Analyzer (model TOC-5000A). Organic carbon was determined by total carbon (dry combustion) less inorganic carbonates (gas purge of acidified suspensions).

Chemicals

The hydrophobic dye selected for this study was Disperse Orange 11, an anthraquinone dye. It was obtained from Aldrich Chemical Co., Inc. with a purity greater than 95%. The surfactant was Tween 80 (Polyoxyethylene (20) sorbitan monooleate), obtained from China National Chemicals Import & Export Corporation with 3% water content. It was used as received from the supplier without further purification. A nonionic surfactant was used in this study because cationic surfactant sorb strongly to most soils, and nonionic surfactants typically have less toxicity, greater biodegradability (Attwood and Florence, 1983), and lower cmc values (1.0×10^{-5} mol/l for Tween 80) (Fendler and Fendler, 1975).

Partitioning of hydrophobic dye in soil-surfactant-water systems

A series of batch experiments was performed to examine the partitioning of the dye compound between solid and liquid phases. In each batch tests, 1.0 ml of 600 mg/l dye solution (prepared in isooctane) was placed in a 25 ml Pyrex glass tube, spread on surfaces and air-dried so that the dye was evenly coated on the tube walls. Various amounts of stock surfactant solution were added to each tube and diluted with distilled/deionized water to create a predetermined bulk solution concentration of surfactant with a final fluid volume in each tube of 20 ml. A known amount of air-dried soil (particle size <2 mm) was then mixed with the solution in each tube, and the tube was sealed with a Teflon-lined screw cap. Samples were shaken in a mechanical rotary shaker at 20°C for 24 h. After equilibrium partitioning was attained, the suspensions were centrifuged for 1.5 h at 2500 rpm to separate solid-phase materials from aqueous-phase materials. Dye in the supernatant was then quantified by HPLC using a ISCO model 2350 pump, a WatersTM 486 tunable absorbance detector, and a reverse-phase column (Restek pinnacle octyl amine 5 μ m, 0.46×25 cm). Elution solvent used was acetonitrile/water at a ratio of 70/30. Its flowrate was 1 ml/min. All batch tests were carried out in duplicate. Dye was detected using the maximum wavelength of the dye in the visible region of the spectrum. The equilibrium solute concentration in the solid phase was calculated as the difference between the total initial dye concentration and the equilibrium aqueous-phase concentration, according to the following expression:

$$\frac{x}{m} = (C_0 - C_e) \frac{v}{w} \quad (1)$$

where x/m is the dye concentration in soil (mg/g), C_0 is the total initial concentration of dye in solution (mg/ml), C_e is the equilibrium concentration of dye in solution (mg/ml), v is the solution volume (ml), and w is the weight of the soil (g).

Surface tension of soil-water systems containing nonionic surfactant

Surface tension was used as an indicator to determine the cmc for systems with and without soil (Liu *et al.*, 1992). Samples were prepared by mixing 3.75 g of soil with various concentrations of surfactant solution, and diluted by deionized water to 50 ml in Iwaki glass tubes. These samples were placed in a rotary shaker at 20°C for 24 h and then centrifuged at 2500 rpm for 1.5 h. Supernatant was separated from each tube and placed in a beaker. The surface tensions of solutions at different surfactant concentrations were determined using a calibrated Fisher Surface Tensiometer Model 21 tensiometer, which employed a Du Nuoy platinum-iridium ring suspended in surfactant solution from a torsion balance. The average of a series of consistent readings for each sample was then corrected to account for the tensiometer configuration, yielding a corrected surface tension value.

RESULTS AND DISCUSSION

Proposed Model

Solubilization of HOCs by nonionic surfactant micelles

Solubilization of HOC by surfactant may be analogous to similar reactions of HOC with aqueous organic matter. Almgren *et al.* (1979) have shown that for HOCs solubilized within micelles, solubilization of one molecule does not affect the solubilization of another. Solubilization of HOCs generally is initiated at the cmc and is proportional to the bulk

solution surfactant concentration beyond this point, which can be described by a micelle–water partition coefficient, K_m (l/mol), see Equation (2):

$$K_m = \frac{[P]_{mic}}{[P]_w[S]_{mic}} \quad (2)$$

where $[P]_w$ is the concentration of a pollutant (HOC) in the water (mol/l), $[P]_{mic}$ is the concentration of solubilized pollutant in micelles (mol/l), and $[S]_{mic}$ is the concentration of micellar surfactant (mol/l). The partitioning of pollutant into a surfactant solution involves both the micellar phase and the monomeric phase (Kile and Chiou, 1989). Equation (2) can be reformulated in practice in a more general form as Equation (3) without causing any significant errors, due to the fact that most cmc values of non-ionic surfactants are low compared to the applied surfactant dose:

$$K_m = \frac{[P]_{surf}}{[P]_w[S]} \quad (3)$$

where $[P]_{surf}$ is the concentration of pollutant solubilized by both monomers and micelles, and $[S]$ is the concentration of surfactant in both monomeric and micellar form (mol/l).

Jafvert (1991) correlates values of K_m with octanol–water partition coefficients (K_{ow}) for a series of PAH compounds in dodecylsulfate micelles, and shows a linear slope and near-zero intercept, suggesting the similarity of solvation energies for these HOCs in water-saturated octanol and aqueous micelles,

$$K_m = aK_{ow} \quad (4)$$

Sorption of HOC on soils

Many researchers such as Karickhoff *et al.* (1979) and Hassett *et al.* (1980) reported the sorption of HOCs on soil components. Their investigations suggest that as sorption processes for those compounds approach equilibrium, the organic-carbon normalized distribution coefficient (K_{oc}) correlates well with the octanol–water partition coefficients (K_{ow}) of the respective HOCs through a linear free-energy relationship and water solubilities. The K_{oc} is as

$$K_{oc} = \frac{[P]_s}{[P]_w f_{oc}} \quad (5)$$

where $[P]_s$ is the HOC (pollutant) concentration (mol/kg) in the solid phase (soil or sediment), and f_{oc} is the fractional organic carbon content of the soil or sediment. For similar classes of HOCs, values of K_{oc} can be correlated to the respective compounds' octanol–water partition constants, K_{ow} , through a nearly constant slope (Karickhoff 1981).

$$K_{oc} = bK_{ow} \quad (6)$$

Hence, the sorption of hydrophobic compounds to soils can be estimated with the knowledge of K_{ow} and f_{oc} . It was noted that the equation (6) is limited by kinetic constraints, soil carbon content, clay content, and solute properties as noted by Karickhoff (1984).

Partitioning of HOCs among soil, water and surfactant phases

When surfactant is added to a saturated soil or sediment system, the partitioning of a HOC between different phases becomes more complicated. Theoretically, the distribution of a HOC between the solid phase (soil or sediment) and the liquid phase ($[P]_w + [P]_{surf}$) may be described by an overall distribution coefficient, K_d :

$$K_d = \frac{[P]_s}{[P]_w + [P]_{surf}} \quad (7)$$

A K_d value can be easily measured in a laboratory and can be used as an index to quantify the efficiency of a surfactant-aided soil-washing system. However, in equation (7), no terms characterizing inherent surfactant, soils, or HOC properties are used, therefore such an equation has little predictive value pertaining to the application of surfactant in soil-washing. A linear model is proposed so that the K_d may be expressed as a function of K_{ow} , f_{oc} , and $[S]$. The derivation of the model is initiated by combining equations (3) and (4), which gives

$$\frac{[P]_{surf}}{[P]_w} = aK_{ow}[S] \quad (8)$$

The combination of equations (5) and (6) gives

$$\frac{[P]_s}{[P]_w} = bK_{ow}f_{oc} \quad (9)$$

Equation (9) divided by equation (8) and rearrangement gives

$$[P]_s = \frac{bf_{oc}}{a[S]}[P]_{surf} \quad (10)$$

The substitution of the term on the right hand side of equation (7) by equation (10) gives

$$K_d = \frac{(b/a)f_{oc}[P]_{surf}}{([P]_w + [P]_{surf})[S]} \quad (11)$$

Dividing both numerator and denominator in equation (11) by $[P]_{surf}$ gives

$$K_d = \frac{(b/a)f_{oc}}{([P]_w/[P]_{surf} + 1)[S]} \quad (12)$$

Substituting for the first term of the denominator by equation (8) gives

$$K_d = \frac{(b/a)f_{oc}}{1/aK_{ow} + [S]} \quad (13)$$

Equation (13) can be linearized with respect to $[S]$ by taking the equation reciprocal. This gives a governing equation to predict the efficiency of sediment washing

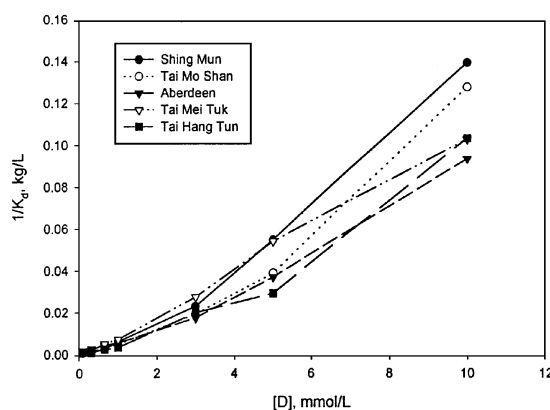


Fig. 1. Soil-washing of disperse orange 11 by surfactant Tween 80 in systems with five different soils. Dye and soil concentration are 30 mg/l and 3.75 g/50 ml, respectively.

in terms of HOC property K_{ow} , soil property f_{oc} , and surfactant concentration $[S]$, as

$$\frac{1}{K_d} = \frac{1}{bK_{ow}f_{oc}} + \frac{a}{bf_{oc}}[S] \quad (\text{or}) \quad \frac{f_{oc}}{K_d} = \frac{1}{bK_{ow}} + \frac{a}{b}[S] \quad (14)$$

Theoretically, according to equation (14), a plot of $1/K_d$ vs. $[S]$ will give a straight line with a slope of a/bf_{oc} and an intercept of $1/bK_{ow}f_{oc}$. Since K_{ow} values for HOCs are available, and f_{oc} for soil and/or sediment are measurable, K_d as well as $[S]$ can be determined in a laboratory. This equation may be useful in designing *ex situ* soil-washing systems or an *in situ* pump-and-treat operation.

Model verification

Partitioning of the dye (Disperse Orange 11) as a function of nonionic surfactant Tween 80 dose is shown for five different soil systems in Fig. 1. The reciprocal of the overall distribution coefficient, $1/K_d$, is plotted against the total surfactant dose added to the system, $[D]$. For low $[D]$ values, good linear correlation between $1/K_d$ and surfactant concentration is observed. Intercepts and slopes are summarized in Table 1. As the surfactant concentration is increased to 1×10^{-3} mol/l and above, however, the slopes double or triple those for low surfactant ranges, and all intercepts become negative (see also Table 1). Soil-washing performance ($1/K_d$)

is minimal until the total surfactant concentration reaches 1.00×10^{-3} M, a value considerably higher than its cmc in pure water (around 10^{-5} M). Significant dye solubilization does not commence until a surfactant concentration is reached that is approximately two orders of magnitude greater than its cmc in water. This is likely due to the sorption of surfactant on the soil. Consequently, the effective concentration of surfactant in the solution is reduced, thus increasing the dose of surfactant required for soil-washing.

Surfactant sorption can be quantified for soils using surface-tension measurements. In Fig. 2 the surface tension of Tween 80 surfactant solution is plotted against the logarithm of the total surfactant concentration in the presence of two different soils and water only. The breakpoints in the surface tension curves indicate the micelle formation, since at this concentration the bulk solution is saturated with surfactant monomers. The cmc of Tween 80 in pure water obtained from this test is 2.54×10^{-5} M. This approximates literature data of 1.00×10^{-5} M. In the presence of soils, however, the breakpoints occur at about a surfactant dose of 10^{-3} M, which coincides with attainment of the cmc in bulk solution, and which concurs with our observation in Fig. 1. After the cmc is attained in the bulk solution, the surface tension remains relatively constant, suggesting surfactant monomer is saturated in the aqueous phase and surfactant transfer between the aqueous phase and the soil phase is at or nearly at equilibrium. For surfactant-aided soil-washing systems, it is important to quantify the amount of surfactant sorbed, which then permits prediction of soil-washing performance through examination of effective surfactant concentration in both monomer and micellar forms.

An arbitrary selection of a point on the abscissa in Fig. 2 gives a monomer surfactant concentration (section A) and a corresponding total surfactant dose required in the soil/aqueous system to obtain the same surfactant monomer concentration in bulk solution (i.e., the same surface tension). The difference between the two concentrations (section B) therefore can be used to quantify the surfactant sorbed to soil. Once surface tension is lower than a critical point (about 35–40 dyn/cm), sorption sites in soil apparently are likely saturated by surfactant and no more loss is observed. It is also possible to

Table 1. Correlation of soil-washing performance by equation (14) at high and low surfactant doses

Soil sources	f_{oc}	At low $[D]$ ($< 1 \times 10^{-3}$ mol/l)			At high $[D]$ ($> 1 \times 10^{-3}$ mol/l)		
		Intercept	Slope	r^2	Intercept	Slope	r^2
Shing Mun	0.00197	0.00033	5.18260	0.89321	-0.01647	15.22866	0.98773
Tai Mo Shan	0.00457	0.00037	4.52996	0.99551	-0.01896	14.02518	0.96658
Aberdeen	0.00753	0.00146	4.25389	0.97875	-0.00921	10.01619	0.98755
Tai Mei Tuk	0.01039	0.00101	6.25851	0.96264	-0.00281	10.67934	0.99671
Tai Hang Tun	0.01477	0.00080	2.57274	0.88957	-0.01431	11.20854	0.96057

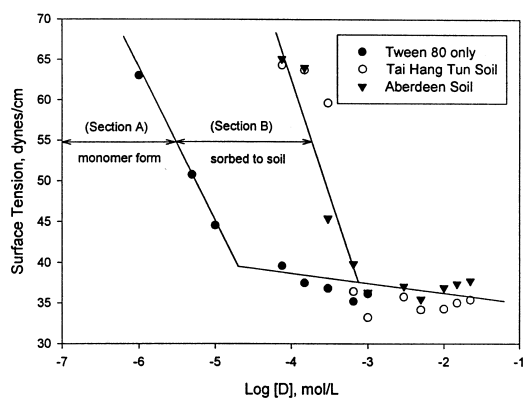


Fig. 2. Surface-tension change in Tween 80 solution as a function of surfactant dose for systems with and without soil.

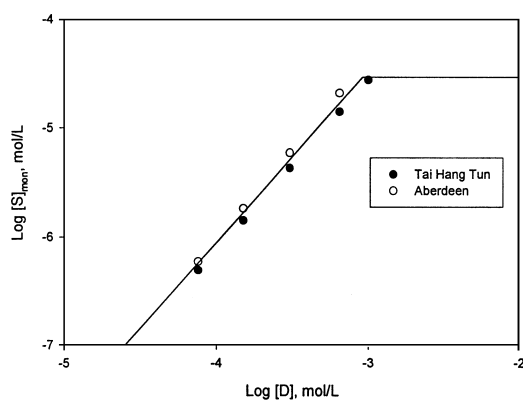


Fig. 3. A linear correlation between surfactant monomer ($\log [S]_{\text{mon}}$) and surfactant dose ($\log [D]$) in two different soil/water systems containing Tween 80.

estimate the monomer concentration in a soil/aqueous system at doses for which sorption process is still active, by plotting surfactant dose $[D]$ against its corresponding monomer surfactant concentration $[S]_{\text{mon}}$ (section A), as shown in Fig. 3. A linear relationship is found between $\log [S]_{\text{mon}}$ and $\log [D]$ for systems with two different soils and Tween 80. The slope of the rising section in Fig. 3 is 0.030 (in terms of $[S]_{\text{mon}}$ vs. $[D]$ with a r^2 of 0.98), indicating that nearly 97% of the initial surfactant dose is sorbed by the soil before the soil becomes saturated, and only a small fraction (about 3%) of $[D]$, in the monomer form, becomes effective as a soil-washing agent. Due to the fact that there is no surfactant loss after the soil is saturated by the surfactant, the partitioning of HOC in surfactant-aided soil-washing system should be discussed as occurring in two stages.

In stage 1, when $[D]$ is less than the effective cmc (10^{-3} M), only the free surfactant monomers ($[S]_{\text{mon}}$)

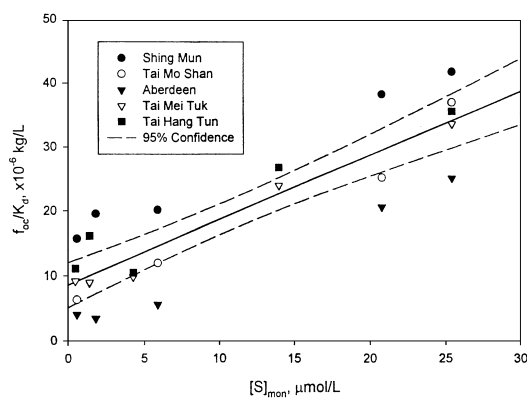


Fig. 4. The stage 1 soil-washing performance in five different soil systems by Tween 80 monomer.

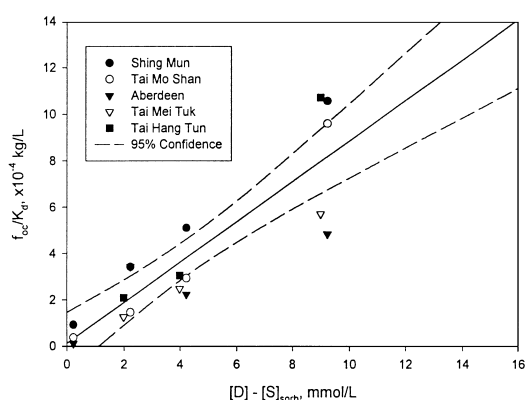


Fig. 5. The stage 2 soil-washing performance in five different soil systems by Tween 80 micelles.

contribute to the soil-washing, so equation (14) can be modified and rearranged as:

$$\frac{f_{\text{oc}}}{K_d} = \frac{1}{bK_{\text{ow}}} + \frac{a}{b}[S]_{\text{mon}} \quad (15)$$

The $[S]_{\text{mon}}$ can be calculated from Fig. 3 at different surfactant doses $[D]$ (below the effective cmc as a result of surfactant sorbed to soil). So a plot of f_{oc}/K_d versus $[S]_{\text{mon}}$, according to the proposed equation, should give a straight line with a constant slope and intercept. The results obtained by using five different soils are shown in Fig. 4, and the linear equation with a correlation r^2 of 0.770 for all of them is

$$\frac{f_{\text{oc}}}{K_d} = 8.531 \times 10^{-6} + 1.008[S]_{\text{mon}} \quad (16)$$

Since the slope is quite close to unity, the soil-washing performance ($1/K_d$) by surfactant monomer in stage 1 is 1:1 proportional to the $[S]_{\text{mon}}$, and the

higher the product of f_{oc} and K_{ow} (in the denominator of the intercept), the lower is the performance. The physical meaning of the intercept is the equilibrium concentration of HOC in the aqueous phase (likely due to molecular diffusion) when no surfactant is added. Both the K_{ow} of the pollutant and the f_{oc} of the soil are responsible for the determination of the equilibrium concentration as seen in equation (15).

In stage 2, when $[D]$ is higher than that needed for cmc to be attained, surfactant in the bulk solution is dominated by the micellar form, and the surfactant available for soil-washing is essentially the difference between the total surfactant dose and that lost to the soil. The term $[S]$ in equation (14), therefore, should be substituted by $[D] - [S]_{sorb}$:

$$\frac{f_{oc}}{K_d} = \frac{1}{bK_{ow}} + \frac{a}{b}([D] - [S]_{sorb}) \quad (17)$$

The $[S]_{sorb}$ in stage 2 is close to a constant (Liu *et al.*, 1992) and is measurable from Fig. 3. In our test, the $[S]_{sorb}$ is around 7.69×10^{-4} mol/l, resulting from simple expression of $10^{-3.1} - 10^{-4.6}$. The correlations of f_{oc}/K_d against $[D] - [S]_{sorb}$ for solubilization of the hydrophobic dye in five different soils by Tween 80 are shown in Fig. 5, and the linear equation with a correlation r^2 of 0.809 is

$$\frac{f_{oc}}{K_d} = 2.789 \times 10^{-5} + 0.083([D] - [S]_{sorb}) \quad (18)$$

In a real application, the surfactant dose applied for soil-washing is generally high, so the small intercept in equation (18) becomes trivial and can be ignored. Therefore, soil-washing performance, measured by f_{oc}/K_d , mainly relies on the surfactant available in the system ($[D] - [S]_{sorb}$) and on the soil characteristics (f_{oc}).

CONCLUSIONS

The results of these experiments show that the performance of HOC removal from soil by surfactant micelles is different from that of surfactant monomers. Soil-washing in the presence of surfactant may occur in one of two distinct stages. In stage 1, HOCs can be extracted from the soil by surfactant monomer, but most of the surfactant (97% in this study) is lost to the soil due to sorption. Under these circumstances, washing performance is 1:1 proportional to the available monomer concentration in the liquid phase. In stage 2, sorption sites on the soil are saturated by surfactant, so surfactant micelles exist in bulk solution. The micelles can greatly increase HOC apparent solubility. Soil-washing performance is mainly dominated by the surfactant micelles available in the system and the organic content of soil.

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