

Kinetics of *in situ* Surfactant Soil Flushing at Moderate Washing Conditions

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Abstract—Economic *in situ* soil flushing using common surfactants may be a good substitute for exhaustive, pressurized soil washing or bioremediation requiring high energy consumption or laborious technique. Two model surfactants, sodium dodecyl sulfate (SDS) and polyoxyethylene (20) sorbitan monooleate (Tween-80), were chosen as flushing agents. Those surfactant solutions were applied to clean hydrocarbon (motor oil) contaminated sand soil. A kinetic investigation such as order of reaction of pollutant compounds, flushing mode (semi-continuous and continuous), change of soil pore matrix, etc. comprised a main part of this work. We found that the hydrocarbon elution curves were dropping in an exponential way, which was stiffer with higher surfactant concentrations. Higher surfactant concentration, higher flow rate, and lower porosity guaranteed higher removal efficiency as well as higher removal rate. Strong initial lag phases were found for Tween-80 solutions. A modified Monod-type reaction model describing the removal kinetics was proposed to be the first-order reaction, which agreed well with most of the experimental results. The curve-fitted parameters, n , k_0 and K_1 were linear functions of surfactant concentration and reciprocal of soil porosity.

Key words: Kinetics, *in-situ* Soil Washing, Hydrocarbon Oil, Surfactant

INTRODUCTION

A large volume of hydrocarbon oil discarded or leaked from various sources is reported to have heavily contaminated subsurface soil for decades worldwide [West and Harwell, 1992; Cho, 1997; Park et al., 2001]. In the United States, about 10% of 10 year old storage tanks are said to have leakage due to corrosion and physical damage [Cho, 1997]. On the similar basis, the oil spill/leakage beneath the subsurface around industrial and commercial (mainly gas stations) areas could be estimated as 12 million tons in Korea. The contaminant might be transferred to the subsurface or below and ground water wells in the end.

Surfactant flushing is regarded as a faster, more economic method for soil clean-up than others requiring high energy consumption or laborious techniques [Lee et al., 1999]. Especially, *in situ* flushing has been shown as promising for soil remediation (targeted to aquifer area contaminated by nonaqueous phase compounds (NAPC)) in that it needs smaller time frames than traditional pump and treat methods [Fountain et al., 1996; Rao et al., 1997]. Surfactants have been used to isolate lipophilic compounds attached to the soil by forming micelles or admicelles that would contain lipophilic molecules inside. Two classes of surfactant, ionic and nonionic, have been applied to soil flushing in combination or independently. The two kinds are known to be mutually compensatory by forming larger micelles, provided with larger internal voidity in a way and by giving electrostatic stability to the micelles in another [Choi et al., 1998; Myers, 1985]. Effectively structured or economically competent surfactants are preferably chosen for successful flushing. Like enhanced oil recovery [Taylor and Hakins, 1992; Holm, 1977], flushing technologies have evolved to addition of various chemical agents to a

basic fluidization fluid or mixture in order to improve the solubilization and/or mobilization (migration) of NAPCs from contaminated aquifers. Traditional chemical agents, surfactants [West and Harwell, 1992; Pennell et al., 1994] and cosolvents [Imhoff et al., 1995], have been used to accelerate the removal of trapped residual NAPCs from porous media through either enhanced solubilization or mobilization. Mobilization of NAPC trapped within the media is facilitated by a reduction in the interfacial tension between the NAPC solution and the flushing solution. Solubilization technique, on the other hand, is much simpler and more practical in the sense of design and operating parameter optimization. Many researchers have contributed to the solubilization strategies for remediation [Ouyang et al., 1996; Rhue et al., 1997] and to the field-scale application of solubilization using surfactants and cosolvents [Fountain et al., 1996; Peters and Luthy, 1993].

As for operating cost, operating conditions such as mild concentration of flushing agent, ambient pressure and temperature without any mechanical aid could provide an economically feasible approach to the soil flushing. In the other aspect, an *in situ* method, practically useful for wide contaminated areas, is more exploitable. Yet few significant, successful attempts for *in situ* flushing oil-contaminated soil at plain and normal conditions were reported [Jeon et al., 2001]. Ellis et al. [1985], Kile and Chiou [1989] and Vignon and Rubin [1989] just investigated the possibility for recovery of hydrocarbon or removal of hydrophobic organic matter using somehow costly, highly concentrated feeding. In this report, in order to evaluate economics of surfactant flushing, kinetics of hydrocarbon removal was considered *a priori* rather than agent formula for high performance, lower CMC, and addition of cosolvents [GWRTAC, 1996]; it includes hydrocarbon removal rate, time variation of residual hydrocarbon, removal efficiency, influential factors including soil bed porosity (water permeability), flushing model, surfactant percentage in flushing fluid, etc. In our experiments, captured hydro-

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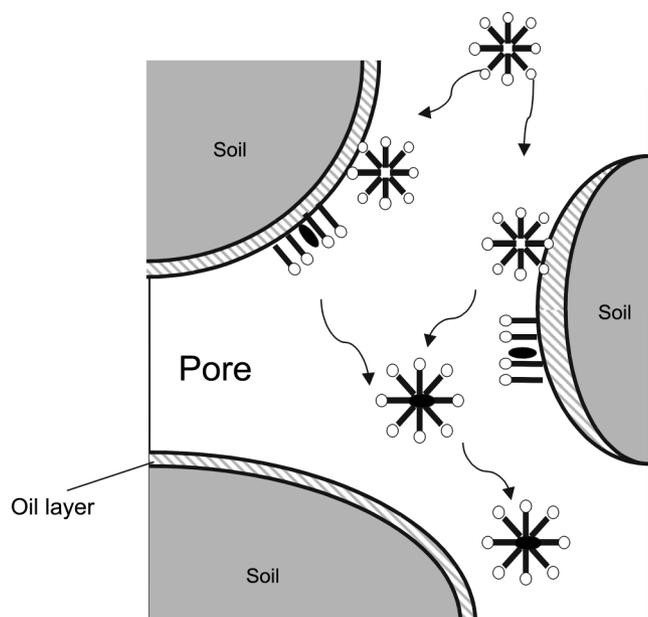


Fig. 1. Postulative removal of hydrocarbon inside soil by surfactant flushing.

carbon portion with flushing would be “dissolved” in the micellar cavity, which is followed by formation of emulsion with a certain degree of mechanical perturbation. And then, the emulsive clumps can move down along the void space in the soil bed by gravity or external force (Fig. 1).

MATERIALS AND METHOD

1. Materials and Apparatus

Sodium dodecyl sulfate (Sigma Chemical Co.; 99%) and polyoxyethylene (20) sorbitan monooleate (Tween-80; Duk-San Pure Chemicals; +98%) were used as flushing agent (water soluble for easy application to soil) without further purification. Motor oil (commercially available from SK Chem. Co.; viscosity, 75 cP; density, 0.87 g/cm^3) was used as a contaminating agent. Fine sand with 0.29 mm of effective particle diameter after sieving was prepared with repetitive washing and drying at 110°C . The equilibrium salt conductivity of distilled water including purified sand (considering the same volume as flushing solution per gram of packed sand) was about $50 \mu\text{S/cm}$. All flushing solutions had pH 5 to 6, similar to distilled water.

A cylindrical glass column ($D \times L = 29 \text{ mm} \times 167 \text{ mm}$) with a sintered glass filtering plate was placed as shown in Fig. 2. An oil-contaminated soil sample ($0.084 \text{ ml-oil/g-soil}$), 10.1 g was put in the column in pre-mixed state. Three kinds of soil beds with porosities

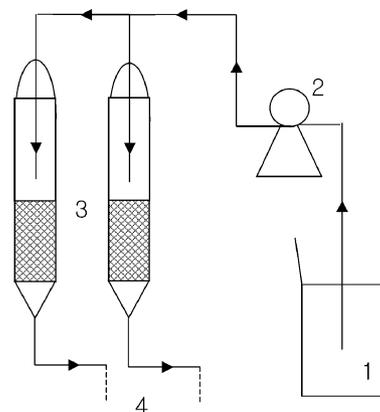


Fig. 2. Schematic of the lab-scale soil flushing unit.

1. Surfactant solution
2. Peristaltic pump
3. Soil column
4. Volumetric analysis of hydrocarbon

of 0.29, 0.2 and 0.16 were prepared with stamping the top of the beds. The hydraulic conductivity at each flushing condition is shown in Table 1.

2. Methods

Five flushing solutions for each surfactant were prepared at near or above its CMC (0.24 wt% for SDS and 0.0013 wt% for Tween-80 at 20°C): 0.12, 0.25, 0.5, 1.0, and 2.0 wt% for SDS while 0.00065, 0.0013, 0.0026, 0.0065, and 0.013 wt% for Tween-80. For gravity-driven flushing, 40 ml of SDS or Tween-80 solution was cautiously poured into a prepared column, letting the solution flow through the column and be collected in a measuring bottle for analysis. Oil eluted from the bottom of the bed was measured by volume in a finely graduated mass cylinder after ‘oil-water’ phase separation. Another volume of washing solution was injected and so on until the analysis precision limit (at which no significant amount of oil eluted), $0.25 \pm 0.1\%$ was attained. In continuous down flow mode, the glass column with soil bed (the same as in the batch experiments) was filled with a surfactant solution at first. And then, the solution flowed downward through the soil bed by a peristaltic pump operating at 0.757, 1.742 or 2.575 cm/min (superficial velocity). Eluted amount of oil was recorded at each empty soil bed volume. All experiments were carried out at room temperature.

MODIFIED MONOD-TYPE REACTION MODEL

Assuming that hydrocarbon is being removed by a surfactant in n -th reaction and its removal is limited to a certain level (saturation), we can simply draw a model satisfying this description as follows:

Table 1. Hydraulic conductivity measured at various conditions (unit: 10^{-2} cm/s)

ϵ	SDS concentration (wt%)					Tween-80 concentration (10^{-3} wt%)				
	0	0.12	0.25	0.5	2.0	0.65	1.3	2.6	6.5	13.0
0.29	1.79	1.07	1.17	0.64	0.44	4.58	3.8	0.9	1.3	0.87
0.20			0.84	0.43						
0.16			0.67	0.33						

$$-\frac{dS}{dt} = k_0 \frac{S^n}{K_1 + S^n} \quad (1)$$

where S is concentration of hydrocarbon in soil and n , k_0 and K_1 are reactivity coefficient, saturation coefficient and order of reaction, respectively.

The proposed equation, Eq. (1), is phenomenological in that a washing agent elutes out hydrocarbon moieties spottily embedded in the soil matrix with time or number of washing in case of gravity-driven feeding.

Integrating Eq. (1) over time (total elapsed time needed for exhaustive washing) gives

$$\frac{K_1}{n-1} (S^{1-n} - S_0^{1-n}) - (S - S_0) = k_0(t - t_L) \quad \text{when } n \neq 1 \quad (2a)$$

$$K_1 \ln \frac{S_0}{S} + S_0 - S = k_0(t - t_L) \quad \text{when } n = 1 \quad (2b)$$

Where $t_L > 0$ if Tween-80 is used

$t_L = 0$ otherwise

and t_L denotes lag time

Letting $Y = \text{LHS of Eq. (2)}$ and plotting Y vs t , we have a straight line with n , k_0 and K_1 determined.

RESULTS AND DISCUSSION

Soil could be washed *in situ* by intermittent flushing (gravity driven) or by continuous pumping. Non-continuous mode would take advantage of cost if applicable while continuous flushing would probably be better in operability and stability. Next, the results and discussion included the experiment of two modes and a removal kinetics model that supports the experiments.

1. Hydrocarbon Removal by Continuous Flushing

Figs. 3 and 4 show that the hydrocarbon embedded in the sand was reduced exponentially with flushing agents' concentration. Note the significant initial drops of residual hydrocarbon fraction in both

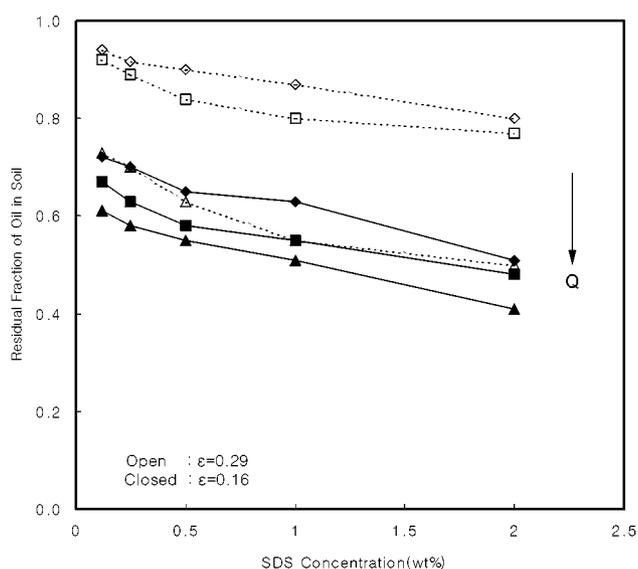


Fig. 3. Residual oil fraction in soil after exhaustive washing at different SDS concentrations. \blacklozenge : $V_s = 0.757$ cm/min; \blacksquare : $V_s = 1.742$ cm/min; \blacktriangle : $V_s = 2.575$ cm/min.

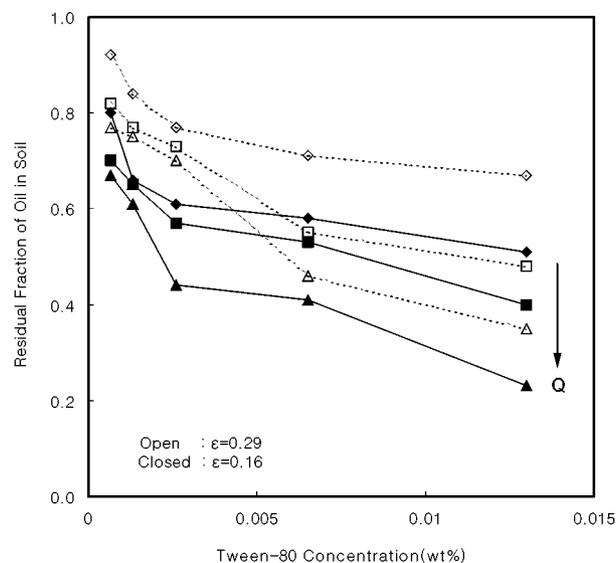


Fig. 4. Residual oil fraction in soil after exhaustive washing at different Tween-80 concentrations. \blacklozenge : $V_s = 0.757$ cm/min; \blacksquare : $V_s = 1.742$ cm/min; \blacktriangle : $V_s = 2.575$ cm/min.

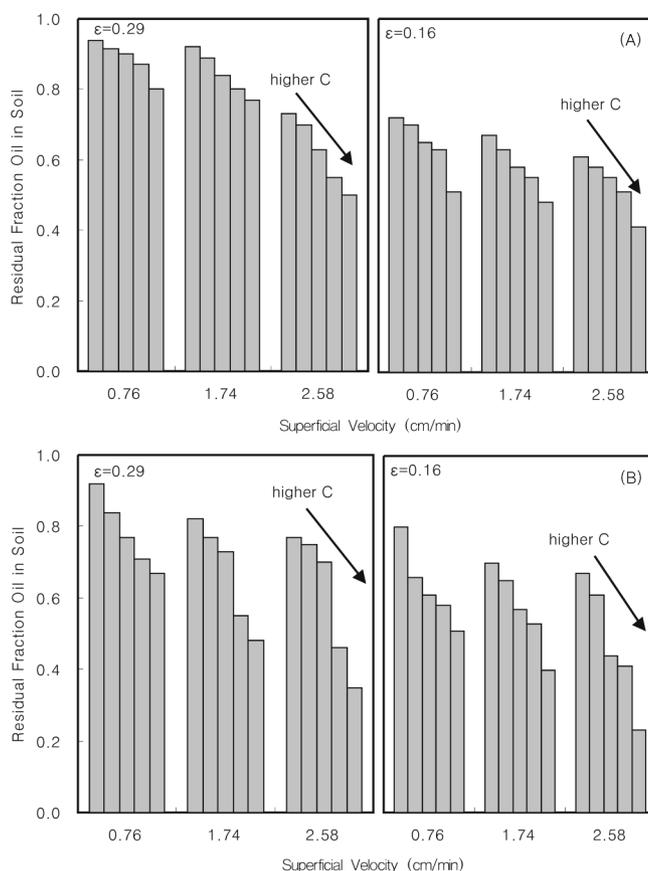


Fig. 5. Residual oil fraction in soil after exhaustive washing at different surfactant solution flow rates (a) for SDS and (b) for Tween-80. Surfactant concentrations are manifest in Figs. 3 and 4.

figures. And the increase of flow rates of flushing agents resulted in higher hydrocarbon removal. Also, adjusting the degree of soil

packing (porosity) brought in drastic change in the removal efficiency (maximum efficiency, 73% in Fig. 4). In fact, this has been already found in some gravity-driven washing experiments elsewhere [Cho and Kwon, 2001]. It was understood that the shorter residence time at the larger porosity did not form as much amount of solubilized micelles or admicelles as the longer retention time at the smaller porosity did. In contrast, for the sake of higher flow rates or shorter retention times of the continuous mode, the hydrocarbon removal was found to be faster and larger. That is because the oil was removed mostly by mobilization or forced migration mechanism, not just by solubilization. In this case, "channeling effect" leading less flowing paths through the soil bed seemed to be blocked by dense soil packing and it appeared to be minor as verified with a pigment tracer method in the lab (blue dye was intruded into the solidified soil bed where the void space was stripped out by the surfactant solution; not shown here). And more interestingly, the larger void volume at $\epsilon=0.29$ decreased hydrocarbon elution by more than 100% in SDS flushing; meanwhile it decreased the elution only by 10-20% in Tween-80 flushing. That is, the non-ionic surfactant was more effective with high doses regardless of porosity (compare Fig. 5a with 5b). As pointed out elsewhere [Cho and Kwon, 2001], it is believed to be probably due to the characteristics of Tween-80 micelles or admicelles-larger size and/or abnormal shape - in solubilization process [Evans and Wennerstrom, 1999].

2. Kinetics on Hydrocarbon Elution

Figs. 6 and 7 plot "Residual hydrocarbon fraction over Pore vol-

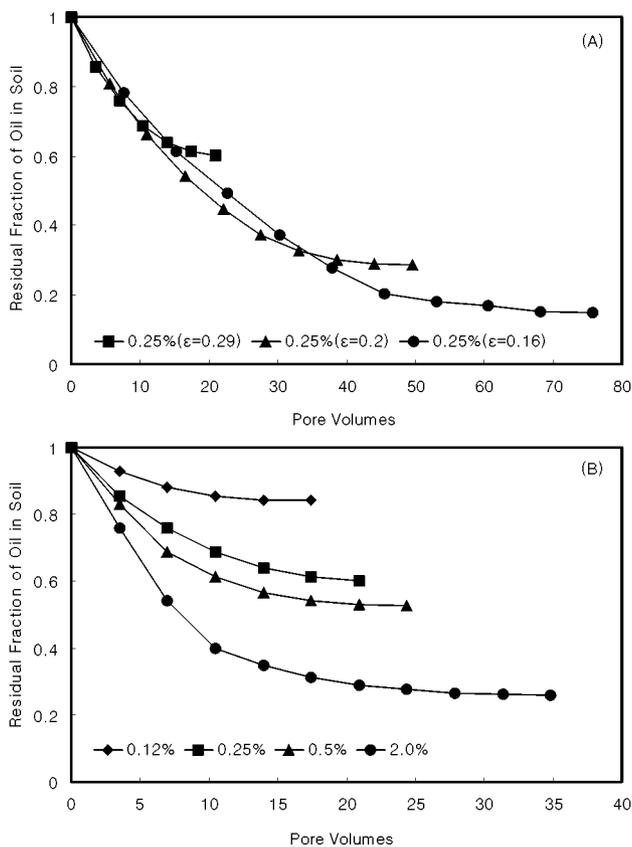


Fig. 6. Residual oil fraction in soil against pore volumes (a) at different pore densities (0.25 wt% of SDS) (b) at different SDS concentrations ($\epsilon=0.29$) when intermittently flushed.

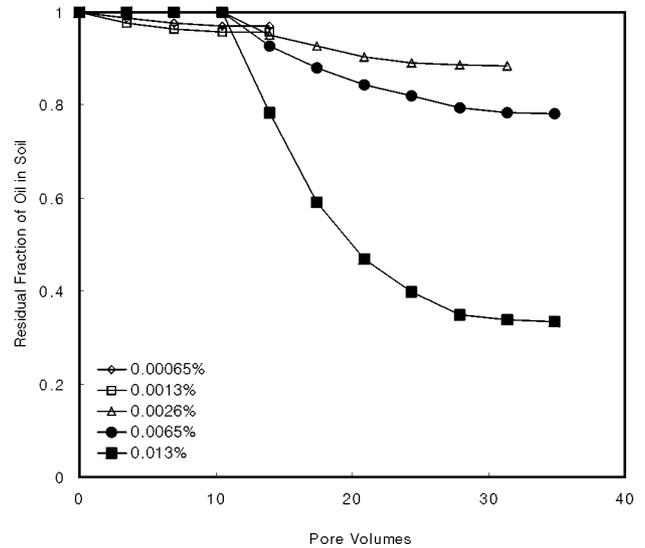


Fig. 7. Residual oil fraction in soil against pore volumes at different Tween-80 concentrations ($\epsilon=0.29$) when intermittently flushed.

umes of flushing solutions" in intermittent flushing by gravity. Those figures depict typical "kinetic data on eluted hydrocarbon through the soil" since the pore volumes are linearly related to flushing time as well as accumulated volumes of surfactant solution applied. With sufficient flushing of SDS solution, final removal efficiency ended up to maximum 83%, which strongly depended on porosity and

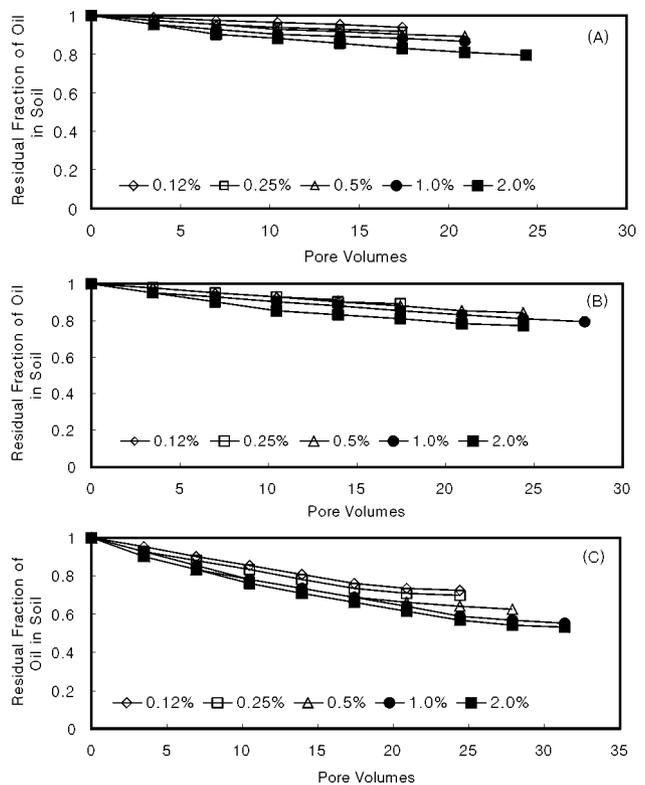


Fig. 8. Residual oil fraction in soil against pore volumes (a) at $V_s=0.757$ cm/min, (b) at $V_s=1.742$ cm/min and (c) at $V_s=2.575$ cm/min for SDS when $\epsilon=0.29$.

surfactant concentration (Fig. 6a and 6b). In Fig. 7, we can see the retarded micelle formation for a while in Tween-80 flushing. Once micelles formed (after a certain lag time), the hydrocarbon could start to elute out of the column dependently under different conditions as expected in SDS flushing. For both of the surfactants, the hydrocarbon removal pattern curved exponentially. In other words,

the removal occurred in deep at first few washings and was followed by sluggish saturation.

On the other hand, in continuous mode, hydrocarbon removal efficiency over time was greatly reduced, showing a linear-like pattern (Figs. 8 and 9). The linear-like removal was more distinct with mild or lower surfactant concentrations and larger porosities, which

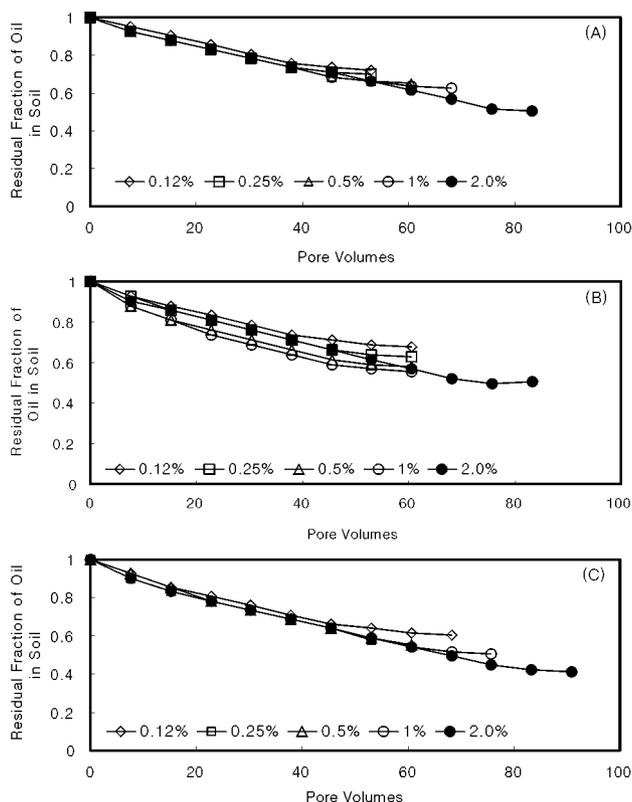


Fig. 9. Residual oil fraction in soil against pore volumes (a) at $V_s=0.757$ cm/min, (b) at $V_s=1.742$ cm/min and (c) at $V_s=2.575$ cm/min for SDS when $\epsilon=0.16$.

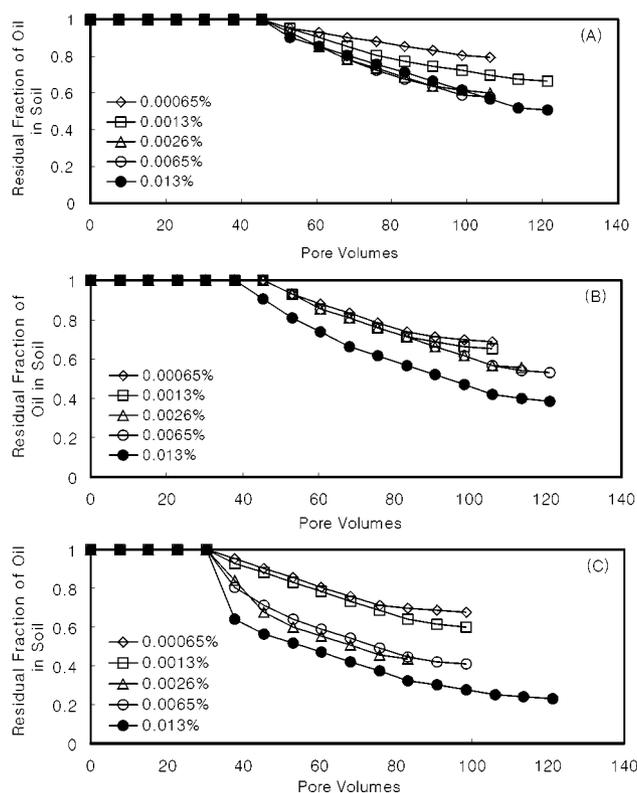


Fig. 10. Residual oil fraction in soil against pore volumes (a) at $V_s=0.757$ cm/min, (b) at $V_s=1.742$ cm/min and (c) at $V_s=2.575$ cm/min for Tween-80 when $\epsilon=0.29$.

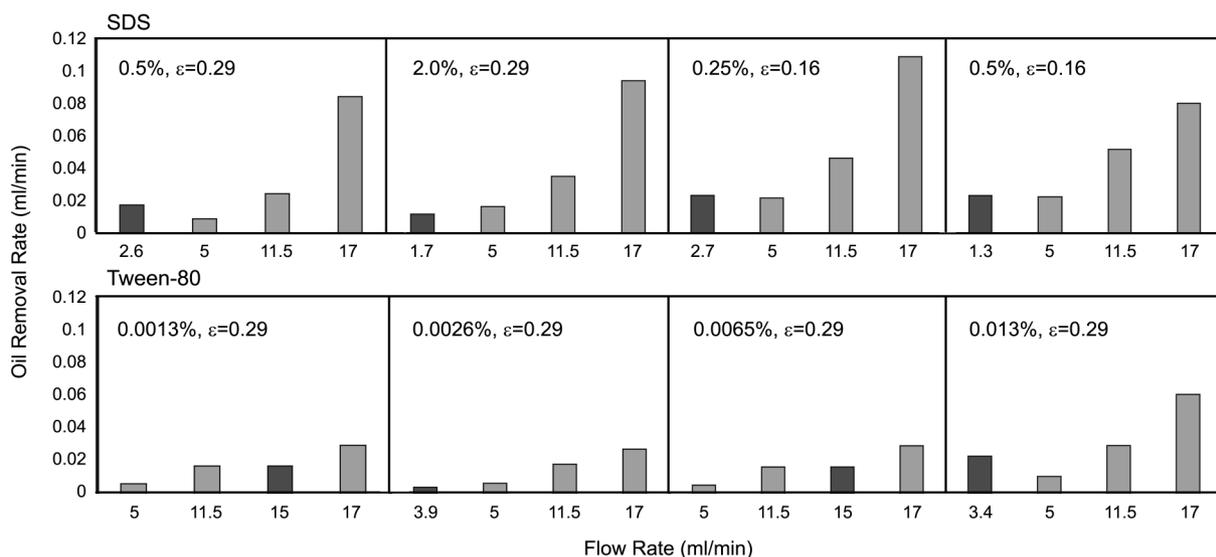


Fig. 11. Comparison of hydrocarbon removal rates against surfactant flow rates under various conditions. Dark bar stands for gravity-driven flushing; others for continuous flushing.

means that, for early flushing, the pressurized flow did not allow sufficient time for solubilization as well as amplified “channeling

Table 2. Kinetic parameters determined at each flushing experiment

$\langle V_s \rangle = 0.757 \text{ cm/min}$						
SDS	$\epsilon = 0.29$			$\epsilon = 0.16$		
	n	k_0	K_1	n	k_0	K_1
0.12%	1	0.000056	3.4	1	0.00026	3.4
0.25%	1	0.000088	3.4	1	0.00030	3.4
0.5%	1	0.00010	3.4	1	0.00033	3.4
1.0%	1	0.00013	3.4	1	0.00033	3.4
2.0%	1	0.00018	3.4	1	0.00042	3.4
Tween 80						
Tween 80	$\epsilon = 0.29$			$\epsilon = 0.16$		
	n	k_0	K_1	n	k_0	K_1
0.00065%	1	0.00010	4.0	1	0.00013	2.0
0.0013%	1	0.00013	4.0	1	0.00022	2.0
0.0026%	1	0.00015	4.0	1	0.00038	2.0
0.0065%	1	0.00020	4.0	1	0.00043	2.0
0.013%	1	0.00030	4.0	1	0.00047	2.0
$\langle V_s \rangle = 1.742 \text{ cm/min}$						
SDS	$\epsilon = 0.29$			$\epsilon = 0.16$		
	n	k_0	K_1	n	k_0	K_1
0.12%	1	0.00023	3.4	1	0.00057	3.4
0.25%	1	0.00024	3.4	1	0.00071	3.4
0.5%	1	0.00025	3.4	1	0.00091	3.4
1.0%	1	0.00032	3.4	1	0.00100	3.4
2.0%	1	0.00044	3.4	1	0.00086	3.4
Tween 80						
Tween 80	$\epsilon = 0.29$			$\epsilon = 0.16$		
	n	k_0	K_1	n	k_0	K_1
0.00065%	1	0.00036	3.0	1	0.00040	1.9
0.0013%	1	0.00029	3.0	1	0.00046	1.9
0.0026%	1	0.00032	3.0	1	0.00053	1.9
0.0065%	1	0.00059	3.0	1	0.00061	1.9
0.013%	1	0.00096	3.0	1	0.00091	1.9
$\langle V_s \rangle = 2.575 \text{ cm/min}$						
SDS	$\epsilon = 0.29$			$\epsilon = 0.16$		
	n	k_0	K_1	n	k_0	K_1
0.12%	1	0.0009	3.4	1	0.0013	3.4
0.25%	1	0.0010	3.4	1	0.0016	3.4
0.5%	1	0.0013	3.4	1	0.0016	3.4
1.0%	1	0.0016	3.4	1	0.0018	3.4
2.0%	1	0.0018	3.4	1	0.0023	3.4
Tween 80						
Tween 80	$\epsilon = 0.29$			$\epsilon = 0.16$		
	n	k_0	K_1	n	k_0	K_1
0.00065%	1	0.00041	3.0	1	0.00068	2.0
0.0013%	1	0.00045	3.0	1	0.00089	2.0
0.0026%	1	0.00059	3.0	1	0.0019	2.0
0.0065%	1	0.0011	3.0	1	0.0016	2.0
0.013%	1	0.0018	3.0	1	0.0027	2.0

effect.” Flow rates and porosities were predominant factors; meanwhile the effect of surfactant concentration was less for effective flushing. As found in gravity-driven flushing, a time lag was found in all continuous operations when Tween-80 was applied (Fig. 10). The lag was shorter with flow rates as expected. The lag was followed by linear reduction in residual oil fraction at most of mild surfactant concentrations. Fig. 11 summarizes how rapid the hydrocarbon removal process is depending on experimental conditions. Overall, the figure stresses the criticality of “migration effect” enforced by continuous flow; whatever mode is taken, hydrocarbon removal rate would proportionally increase with flow rate. Note that SDS cannot ensure higher removal rate than Tween-80 because of their substantially different dose bases and time lags.

In this study, we found that removal efficiency and removal rate did not come along in parallel. In fact, high flow rates were sure to be related to high removal rates, but not necessarily to high efficiency. As Chan et al. [1976] assumed the desorption of oil-included micelles from the particle surfaces to be the most critical step for oil removal and elution, adsorbed oil-included micelles could not be simply washed out by fluid shear if they were not located in the vicinity of fluid flow. And we know that fast fluid flow could be seriously affected by “channeling.” Consequently, the slow desorption process might occur for the micelles to be expelled as much at timely moments, not at high flow conditions as shown in Fig. 2 (we cannot verify what components and how they were working inside with our standard analysis; probably, more sophisticated and extensive analyses are required in the future work).

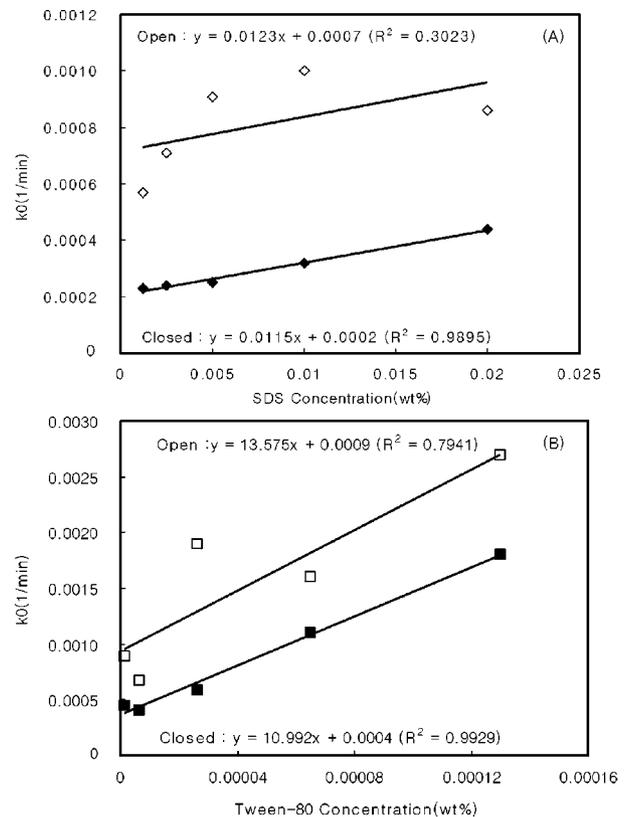


Fig. 12. Dependence of the model parameter, k_0 , on the surfactant concentrations of (a) SDS and (b) Tween-80. Open symbol: $\epsilon=0.16$; closed one: $\epsilon=0.29$.

3. Flushing Kinetic Model Fitted to Experimental

As mentioned earlier, we proposed a simple, reactive kinetic model with three unknown parameters, n , k_0 and K_1 . All the values of the parameters were listed on Table 2. From the table, we see the following:

$$k_0 \sim C \text{ (surfactant concentration)} \quad (3a)$$

$$k_0 \sim \frac{1}{\varepsilon} \quad (3b)$$

$$K_1 \sim \frac{1}{\varepsilon} \text{ at initial removal} \quad (3c)$$

$$n \approx 1 \quad (3d)$$

The reaction order, $n=1$ implies that *in situ* surfactant flushing is basically mild, linearly dependent on how much pollutant is. The most influential coefficient, k_0 is a function of surfactant concentration and porosity. Its strong, positive dependence on smaller porosity provides a good promise to *in situ* flushing on real soil of which particle size is finer even if very dense soil blocks the flow in root. Fig. 12a and 12b are two representations of Eq. (3a). Most of “ k_0 vs C ” plots show good linearity except for some plots at $\varepsilon=0.16$, shown in Fig. 12b. Opposite to the gravity-driven flushing, the highly packed bed in continuous mode has shown a fluctuating or unstable removal pattern. That is because forced flow might create more changeable flow paths at higher soil density than at loosened soil, which possibly leads to highly restricted flow paths or “chan-

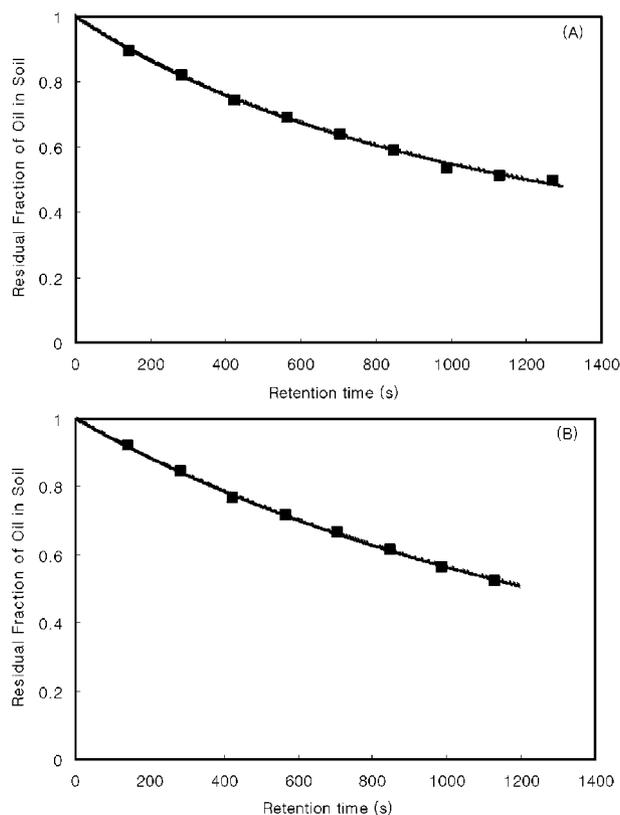


Fig. 13. Two examples of model calculation compared to the corresponding experimental data for (a) an SDS solution ($C=2.0$ wt%, $\varepsilon=0.29$ and $V_s=2.575$ cm/min) and (b) another SDS solution ($C=0.5$ wt%, $\varepsilon=0.16$ and $V_s=2.575$ cm/min). Solid lines denote model calculation results.

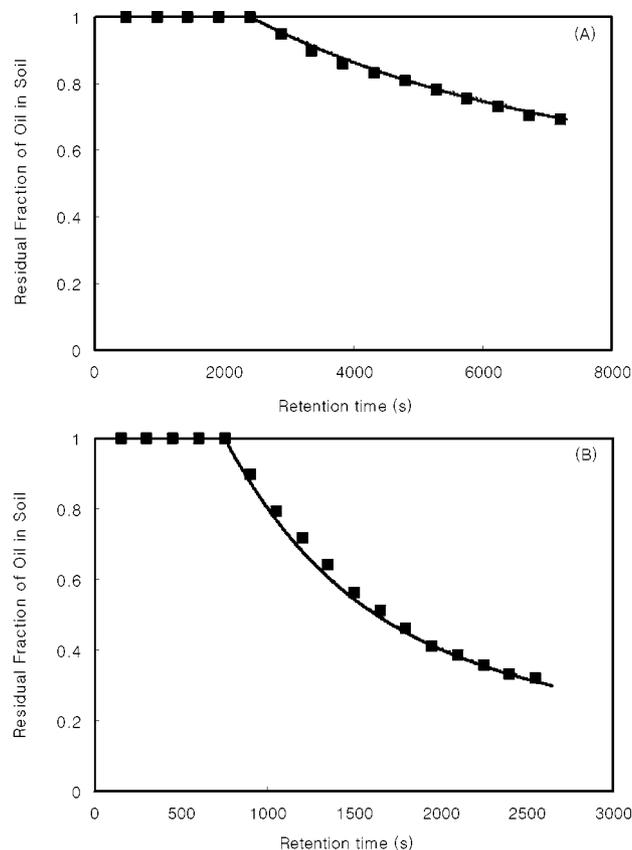


Fig. 14. Two examples of model calculation compared to the corresponding experimental data for (a) a Tween-80 solution ($S=0.0065$ wt%, $\varepsilon=0.29$ and $V_s=0.757$ cm/min) and (b) another Tween-80 solution ($S=0.013$ wt%, $\varepsilon=0.29$ and $V_s=2.575$ cm/min). Solid lines denote model calculation results.

neling.” Good prediction examples are shown in Figs. 13 and 14 with determined values of the parameters. The parameters were obtained by using the Marquardt Optimization Routine [Press et al., 1986].

CONCLUSION

The hydrocarbon removal kinetics, focused on this study, showed that 1) hydrocarbon buried in soil would be eluted in an exponential form, depending on surfactant concentrations. 2) Gravity-driven washing gave stiffer removal rates than continuous feeding where channeling might be more influential in initial stages. 3) However, overall removal rates were higher with greater flow rates regardless of operation mode. 4) Surfactant concentration, flow rate, and reciprocal of porosity increased both removal efficiency and rate. 5) The non-ionic surfactant, Tween-80, showed clear lags for the first few flushes, which occurred probably due to retarded formation of admicelles containing the hydrocarbon moieties. 6) According to the modified Monod reaction model, removal kinetics followed the first-order reaction with two parameters, strongly dependent on surfactant concentration and soil porosity.

ACKNOWLEDGMENT

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NOMENCLATURE

C	: surfactant concentration [wt%]
k_0	: reactivity constant [min^{-1}]
K_1	: saturation constant (dimensionless)
n	: reaction order
S	: concentration of hydrocarbon residue (dimensionless)
S_0	: initial concentration of hydrocarbon contaminant
t	: time used for flushing [min]
t_L	: time lag [min]
V_s	: superficial velocity

Greek Letter

ε	: soil porosity (dimensionless)
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