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Mixing effect of NaCl and surfactant on the remediation of TCB contaminated soil

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ABSTRACT: In order to evaluate the mixing effect of NaCl and surfactant on the remediation of TCB contaminated soil, column experiments were performed. Selected soils for this study were two Iowa field soils (Fruitfield, and Webster) and one Korea field soil (Pyeongtaek). We used four nontoxic, water-soluble anionic (DOSL, SLS) and nonionic surfactants (POE20, POE4). 1, 2, 4-trichlorobenzene (TCB) was chosen as the model contaminant. Selected electrolyte for this study was NaCl. The TCB contaminated soils were leached with surfactant solution of 4% (v/v) or 4% (w/v) with or without 5% (w/v) and 10% (w/v) NaCl. As controls, TCB contaminated soils were also leached with deionized water. For the nonionic surfactants (POE20, POE4), the mixing effect of NaCl on the solubilization of contaminant was insignificant. However, much greater mixing effectiveness was observed using anionic surfactant (SLS, single head structure) solutions containing electrolyte (NaCl) from two Iowa soils and one Korea soil. In contrast, another anionic surfactant (DOSL, double head structure) had little mixing effectiveness on the solubilization of TCB from three soils. However, the NaCl mixing effect of DOSL anionic surfactant was found after leaching of 2500 ml solution with Pyeongtaek soil. The effect of NaCl in changing effectiveness was due to Na^+ (counterion) effects. The maximum recovery of added TCB in column tests was 97% for 4% (w/v) aqueous SLS anionic surfactant + 10% (w/v) NaCl. A little effectiveness of 4% (v/v) DOSL (anionic surfactant) with electrolytes may be due to small adsorption of the double-head sulfate polar heads onto soil particles. Therefore, these results showed that the key factor in the variation of TCB removal effect due to surfactant + NaCl leaching was not surfactant types but surfactant structure. Also, results of this study suggest that the anionic surfactant (SLS) solutions containing electrolyte (NaCl) is a good candidate for surfactant-assisted remediation of TCB contaminated soil.

Key words: mixing effect, surfactant, remediation, field soils, trichlorobenzene

1. INTRODUCTION

Hydrophobic organic compounds such as 1, 2, 4 - trichlorobenzene (TCB) frequently enter the subsurface as a result of industrial accidents (spills, leaks, and leaking underground storage tank), and these chemicals represent a long-term source of soil and aquifer contamination (Martel and Gelinas, 1996). Removal of hydrophobic organic substances from contaminated soils is difficult because they possess low solubilities and high interfacial tensions (Cort et al., 2002; Lee et al., 2002; Zhong et al., 2003). Pump-and-treat methods are neither effective nor economical in recovering residual hydrophobic organic compounds from contaminated aquifers (Lee et al., 2001). Extensive research on soil and groundwater remediation has demonstrated that surfactant washing and/or flushing is a viable alternative for improving the efficiency of pump-and-treat remediation (Deshpande et al., 2000; Chu and Kwan, 2003).

Surfactants (surface active agents) are amphiphilic molecules with a hydrophobic portion and a polar hydrophilic head group. Surfactants have the ability both to solubilize hydrophobic substances and to increase mobility of the contaminants. Even though surfactant solutions may aid in the washing of hydrophobic organic compounds from soils, many surfactants are not suitable for soil remediation due to potential toxicity of surfactant (Allred and Brown, 1994; Cort et al., 2002). Further, aqueous surfactant solutions are affected by environmental conditions such as electrolyte (Rosen, 1989; Lee et al., 2001). The addition of an electrolyte such as NaCl to an anionic surfactant solution can both increase the micelle aggregation number (size of micelle) and decrease the CMC (critical micelle concentration) (Hiemenz, 1986). Rosen (1989) and Harwell et al. (1999) showed that micelle formation of surfactant is enhanced by control of electrolyte conditions, but they did not offer an explanation for the effect. Lee (1999) showed that aqueous surfactant solutions containing NaOH enhanced the removal of hydrophobic contaminants from soil. Changes in effectiveness for remediation could be caused either by pH changes or by changes on the counter-ion Na^+. Also, the effectiveness of surfactant-based remediation can be limited by soil adsorption of surfactants to clay, silt, and by high organic soil contents. Surfactants could alter the natural solubility equilibria for clays by ion exchange and complexation mechanisms (Edwards et al., 1994). More laboratory

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tests are needed to understand the modifying surfactant effectiveness by electrolytes. Therefore, the objective of this study was to evaluate the effect of NaCl on surfactant-assisted soil remediation for removal of TCB from soils.

2. MATERIALS AND METHODS

2.1. Materials

Two Iowa soils (Fruitfield, and Webster) and one Korea soil (Pyeongtaek) were selected for this study. Prior to use they were air dried and passed through a 2-mm sieve. Soil properties, presented in Table 1, were determined on < 2-mm soil samples except for organic carbon, which was measured on < 180-μm samples. Organic carbon content was determined by the dichromate oxidation method (Mebius, 1960), pH by a combination electrode at a 1:2.5 soil/water ratio, surface area by ethylene glycol monoethyl ether method (Cihacek and Bremner, 1979), and particle-size distribution by the pipette method (Gee and Bauder, 1986). They were also oven-dried for 24 hours at 100 °C to decrease the potential for microbial activity during the experiments.

1, 2, 4-trichlorobenzene (TCB) was chosen as the model contaminant because it has been frequently detected in soil and groundwater contaminated at industrial sites (Jafvert and Heath, 1991). Spectrophotometric grade TCB ($C_6H_3Cl_3$) was obtained from Aldrich Chemical (St. Louis, MO, USA).

The food-grade surfactants used in this study were anionic sodium diphenyl oxide disulfonate (DOSL, trade name Dowfax 8390), anionic sodium lauryl sulfate (SLS, trade name SLS), nonionic polyoxyethylene (20) sorbitan monostearate (POE 20, trade name T-Maz 60) and nonionic polyoxyethylene (4) sorbitan monostearate (POE 4, trade name Tween 61). DOSL is a twin-head disulfonate surfactant. Previous studies have shown that sodium diphenyl oxide disulfonate (DOSL, trade name Dowfax 8390) is a good candidate for surfactant-aided remediation of soils contaminated with hydrophobic organic contaminants (Deshpande et al., 2000; Lee, 1999; Rouse et al., 1993; Sabatini et al., 1997). Disulfonates are less susceptible to precipitation and sorption than monosulfates, such as SLS, and are also less prone to sorption than nonionic surfactants (Rouse et al., 1993). DOSL meets criteria as an indirect food additive under FDA Regulation 21CFR178.3400 (Rouse et al., 1993) and SLS

Table 1. Characteristics of the soils used in this study

Soil Series	Texture	Clay Content (%); Dominant Clay	Organic Carbon Content (%)	pH	Surface Area (m^2/g)	CEC[a] (cmol/kg)
Pyeongtaek	Sand	0.8; kaolinite	<0.03	6.4	33	17
Fruitfield	Sand	4; kaolinite	0.22	6.2	15	4.4
Webster	Clay Loam	35; Smectite	0.61	8.2	81.4	27

[a] = Cation-Exchange Capacity

Table 2. Characteristics of surfactant used in this study

Trade Name / Abbreviation Chemical Name Generic[GAS]	HLB[a]	CMC[b] (mM)	Molecular Formula	Type Manufacturer
Dowfax 8390 / DOSL Disodium n-hexadecyl Diphenyloxide disulfonate [65143-89-7], [70191-76-3]	N/A	0.5	$C_{16}H_{33}C_{12}H_7O(SO_3Na)_2$	Anionic Dow Chemical
SLS / SLS Sodium lauryl sulfate [151-21-3]	40	8	$CH_3(CH_2)_{11}OSO_3Na$	Anionic Fisher Scientific
T-Maz60 / POE20 POE[c](20) sorbitan Monostearate [9005-67-8]	14	0.023	$C_{18}H_{34}O_2C_6H_{10}O_4(CH_2CH_{20})_{20}$	Nonionic PPG/Mazer Chemicals
Tween61 / POE4 POE(4) sorbitan Monostearate [9005-67-8]	9.6	0.03	$C_{18}H_{34}O_2C_6H_{10}O_4(CH_2CH_2O)_4$	Nonionic ICI Chemicals

[a] = Hydrophilic-lipophilic balance

[b] = Critical micelle concentration. Data provided by the surfactant manufacturers

[c] = Polyoxyethylene

N/A =Not Available

is approved for direct addition to foods (21CFR172.822). SLS had been previously tested and its data showed the superior recovery rates from an Iowa soil (Lee, 1999). SLS is also considered to be one of the most readily biodegradable surfactants. POE 20, a combination of fatty acids and sugars, is used for food formulations and household products. POE 20 has shown minimum surfactant losses in remediation research (Shiau et al., 1995; Chu and Kwan, 2003). POE 4 is used as an emulsifier, solubilizer, and wetting agent (Rosen, 1989). Properties of the four surfactants are listed in Table 2 and surfactants were used without further purification. All solutions were made using deionized water at room temperature. Selected electrolytes for this study were NaCl. These were obtained from Aldrich Chemical (St. Louis, MO, USA).

2.2. Column Experiment

The column setup was placed in a constant temperature laboratory. The column remained in a vertical orientation during the course of experiments (Lee, 1999). These columns were obtained from the soil physics laboratory of Iowa State University, USA. Column experimental procedures were the same as those used in a previous study (Lee, 1999; Lee et al., 2001). A porous ceramic plate beneath the soil prevented loss of soil during leaching. Approximately 350 g of soil was incrementally packed to a height of 15.4 cm in glass columns (5 cm O.D., 30 cm high). Compaction of the dry soil 0.5 cm layers was standardized by tapping the side of the column 25 times; this degree of compaction minimizes preferential liquid channeling (Martel and Gelinas, 1996). After a column was packed, deionized water was pumped at a rate of 3 mL/min into the column for 3 hours to saturate the soil. The contaminant (5 mL of TCB) was then injected by syringe into the middle of the column, which is a method of contamination analogous to a point source of contamination at the field scale. The column soils were leached with 4% (v/v) or 4% (w/v) aqueous surfactant solution with or without electrolyte. Used electrolytes were 5% (w/v) and 10% (w/v) NaCl. As controls, soils were leached with deionized water. Effluent was collected in 15 individual 250 mL fractions to give a total volume of 3750 mL.

2.3. Chemical Analysis

TCB in aqueous samples was extracted by solvent extraction with hexane using standard separatory funnel method 3510 and analysed by gas chromatography with split/splitless injection system (Hewlett Packard Model 5890 series II) (Lee, 1999; Lee et al., 2001). The system was temperature programmable and has a flame ionization detector (FID). The solid samples were extracted using a soxhlet extraction method. Extractants were concentrated using Kuderna-Danish evaporators and analyzed by gas chromatography. The soil sample contaminated with TCB was mixed with anhydrous sodium sulfate, placed in an extraction thimble and extracted using an appropriate solvent (methylene chloride) in a soxhlet extractor. The main criteria for selecting an internal standard are similar water solubility, chromatographic similarity, and a similar structure as the model contaminants. 1, 2 -dichlorobenzene (purity: 99.9%) was used as internal reference standard. Prior to the analysis of the extracted samples, the response factor and linearity of detection for the internal standard and contaminant were determined. After calculating the response factor, a calibration graph was prepared. New standard curves were prepared after approximately 15 to 20 injections. All sample extracts were analyzed within this calibration curve range. If an extract was too concentrated or dilute, it was adjusted to a concentration within the linear detector response range.

3. RESULTS AND DISCUSSION

3.1. Mixing Effect of NaCl and Surfactant for Removal of TCB from Soil

Table 3a, 3b, 3c show the mixing effect of electrolyte (NaCl) and surfactant in removal effectiveness of TCB from three soils. As expected, TCB removal efficiencies increased with increasing sand percentage (Table 3a, 3b, 3c). For the nonionic surfactants (POE4, POE20), the effect of electrolyte on the recovery of TCB was insignificant (Table 3a, 3b, 3c). However, the highest effectiveness was observed using anionic surfactant (SLS) solution containing NaCl (Table 3a, 3b, 3c). The greatest recovery of TCB in column tests was 97%, which was obtained with 4% (w/v) SLS surfactant + 10% (w/v) NaCl in Pyeongtaek Korea soils. The effect of electrolytes (NaCl) in changing effectiveness of anionic surfactants was due to Na^+ (counter-ion) effects (Rosen, 1989). Increased binding of the counter-ion causes a decrease the CMC of the surfactant and corresponding increase in aggregation number, with stabilization of micelles (Harwell, 1999). The NaCl mixing effect of DOSL anionic surfactant was also found after leaching of 2500 ml solution with Pyeongtaek soil (Table 3a). The increased removal effect of DOSL with NaCl shows the little lesser than that of SLS with NaCl. These results (Table 3a, 3b, 3c) show that the factor of TCB removal by surfactant was not surfactant types but surfactant structure. The double-head hexadecyl disulfonate surfactant, sodium diphenyl oxide disulfonate (DOSL), caused much less pore clogging during surfactant leaching than the single-head sulfate surfactant, sodium lauryl sulfate (SLS) (Lee et al., 2002). As shown in Table 3c, attempts at surfactant-enhanced remediation in clay-rich soils must consider the possibility of the removal effect losses due to the interactions between soil and surfactant and/or electrolytes. Rosen (1989) also showed that “The degree of binding of the counter ion to the micelle also depends on the surface charge density of the micelle.” In another column experiments (Lee, 1999), electrolytes (NaOH) increased the effectiveness of a nonionic surfactant (Pluronic L44) by 11 %, an anionic surfactant (Sandopan JA36) by 26 %, and water by 3 % with Ottawa sand. The model contaminant was toluene in that study (Lee, 1999). These results suggest that surfactant structure and added electrolytes should be considered in field remediation work.

In contrast, Na^+ as 10 % NaCl (wt NaCl per volume solution) had almost no effect on DOSL effectiveness compared to surfactant solutions without the added NaCl. We should also point out that DOSL is considered as a low Na surfactant containing about 0.1 % - 0.3 % NaCl (Rouse et al., 1993) so that counter-ion effects of Na^+ from the DOSL itself should be very minor. Effectiveness of DOSL without the added NaCl may be due to small adsorption of the double-head sulfate polar heads onto soil particles, or to small amounts of surfactant loss by precipitation with soil components (Rouse et al., 1993; Shiau et al., 1995; Sabatini et al., 1997; Lee et al., 2002). The cause of measured effectiveness with NaOH is not clear. The effect of OH^- itself is not well understood, especially its effect in nonionic surfactant effectiveness. However, some research papers showed that hydrogen ion concentration in nonionic surfactant solution was an important factor in surfactant effectiveness (Rosen, 1989; Rajput et al., 1994; Park and Bielefeldt, 2003).

3.2. Effect of Electrolytes in Surfactant-Based Soil Remediation

Among the factors known to affect the CMC of surfactants in aqueous media are structure, HLB number, temper-

Table 3. (a) Removal efficiency(%) of TCB with surfactant in Pyeongtaek soil column

L.V.(mL)	DOSL	DOSL*	DOSL**	SLS	SLS*	SLS**	POE20	POE20**	POE40	POE40**	Water
250	71	71	78	73	75	77	60	63	57	62	12
500	72	72	78	73	75	78	67	67	60	62	12
750	72	75	78	77	79	78	72	70	68	66	13
1000	76	79	78	82	79	80	77	75	70	70	15
1250	76	80	79	85	80	86	80	81	75	70	15
1500	76	80	80	85	84	87	80	81	76	72	15
1750	83	83	85	85	84	87	82	81	80	79	16
2000	83	83	85	85	84	87	82	81	80	81	16
2250	85	83	84	86	86	90	85	86	80	81	16
2500	87	89	95	86	88	92	85	86	84	85	16
2750	90	92	95	86	90	94	85	86	84	85	16
3000	91	92	95	86	90	97	85	86	84	85	16
3250	91	92	95	86	90	97	85	86	84	85	16
3500	91	92	95	86	90	97	85	86	84	85	16
3750	91	92	95	86	90	97	85	86	84	85	16

(b) Removal efficiency(%) of TCB in Fruitfield soil column

L.V.(mL)	DOSL	DOSL*	DOSL**	SLS	SLS*	SLS**	POE20	POE20**	POE40	POE40**	Water
250	65	67	67	75	75	78	56	63	55	62	11
500	73	70	70	76	75	78	70	69	69	65	11
750	75	73	75	80	79	80	73	70	69	70	12
1000	77	77	75	80	80	80	73	80	70	80	14
1250	77	77	77	83	85	85	75	80	70	80	14
1500	77	80	82	83	90	91	75	80	70	80	14
1750	84	86	84	84	90	91	75	82	74	83	14
2000	85	86	84	84	90	95	80	83	74	83	15
2250	85	86	84	85	94	95	81	83	77	83	15
2500	86	86	86	86	95	95	84	85	80	83	15
2750	86	86	90	86	95	95	84	88	80	88	15
3000	88	89	90	86	95	95	85	88	84	88	15
3250	88	89	90	86	95	95	85	88	84	88	15
3500	88	89	90	86	95	95	85	88	84	88	15
3750	88	89	90	86	95	95	85	88	84	88	15

Table 3. (continued) (c) Removal efficiency(%) of TCB in Webster soil column

L.V.(mL)	DOSL	DOSL*	DOSL**	SLS	SLS*	SLS**	POE20	POE20**	POE40	POE40**	Water
250	38	44	44	40	41	41	38	45	40	40	9
500	39	48	48	44	45	45	38	45	40	44	9
750	44	49	49	44	46	46	43	48	42	45	9
1000	55	50	50	47	48	48	45	48	42	47	9
1250	55	54	55	47	51	51	45	50	46	50	9
1500	57	56	56	50	51	53	46	53	49	52	10
1750	59	60	60	53	56	56	53	53	55	55	10
2000	59	60	60	54	57	57	53	55	55	55	10
2250	60	60	60	56	60	60	56	55	55	56	10
2500	64	64	64	62	62	62	60	60	60	60	10
2750	64	66	69	62	65	65	60	65	60	61	10
3000	65	66	69	62	69	69	63	65	60	61	10
3250	65	66	69	62	70	75	64	65	61	61	10
3500	65	66	69	62	70	75	64	65	61	61	10
3750	65	66	69	62	70	75	64	65	61	61	10

Note: 95% confidence interval provided when 2 or more data points available
L.V.=Leaching Volume
* = Mixing with 5% NaCl(wt NaCl per volume solution)
* = Mixing with 10% NaCl(wt NaCl per volume solution)

ature, and the presence of added electrolyte in the solution. The additional of small amounts of pH neutral electrolyte such as NaCl to an anionic surfactant solution can both increase the micelle aggregation number (size of micelle) and decrease the CMC, thereby modifying surfactant effectiveness for remediation (Hiemenz, 1986; Harwell et al., 1999; Henry and Smith, 2002). Also Rosen (1989) showed that the addition of small amounts of pH neutral electrolyte to solution of ionic surfactants appears to increase the extent of solubilization of hydrocarbons that are solubilized in the inner core of the micelle. Increased binding of cations such as Na^+ should cause the CMC of the surfactant to decrease and the aggregation number to increase with the stabilization of micelles. Based on these experimental results, SLS with electrolytes (especially NaCl) can be a good candidate on surfactant-assisted remediation for removal of TCB. DOSL also has good solubilizing abilities for 1,2,4-trichlorobenzene, phenanthrene, naphthalene and related substances (Chen and Knox, 1997; Lee et al, 2001). DOSL should show minimal loss during surfactant flushing because of low sorption to soil particle and resistance to precipitation with cations (Na^+).

The hydrophilic ions of anionic surfactants were converted into anionic hydrated ions. Therefore, suitable quantity of inorganic cations (ex. Na^+) in aqueous phase could improve the solubilization capacities of anionic surfactants. In addition, recovery rates of anionic surfactants considered counter-ion effects. SLS had a Na^+ ion in hydrophilic moiety. Na^+ ion affected the counter-ion that dissociated into the aqueous solution. Bystryak et al. (1999) reported an unusual increase in conductivity upon binding of SLS to weak polyelectrolytes, which was attributed to an enhanced mobility of the Na^+ counter-ions. The rise of cationic concentration was related to the addition of Na^+ ions from SLS and it showed the highest recovery rates from two Iowa soil and one Korea soil for removal of TCB.

4. CONCLUSIONS

Study results show that increase in clay reduces surfactant effectiveness for removal of TCB. The structure of surfactant and addition of electrolytes to surfactant solution should be considered in surfactant-assisted remediation. Much greater effectiveness was observed using anionic surfactant (SLS, single head structure) solutions containing NaCl in column tests. The maximum recovery of added TCB in column tests was 97 % for 4 % (w/v) aqueous SLS anionic surfactant + 10 % (w/v) NaCl. A little effectiveness of 4 % (v/v) DOSL (anionic surfactant, double head structure) with electrolytes (NaCl) was also observed. These results showed that the key factor in the variation of TCB removal effect due to surfactant leaching was not surfactant types but surfactant structure. Counter-ion (Na^+) effect in surfactant-based remediation provides an opportunity to increase the knowledge in soil and groundwater remediation. Also, the results of this study suggest that the anionic surfactant (SLS) solutions containing NaCl is a good candidate for removal of TCB from two Iowa soils and one Korea soil.

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