Boundary Condition and Soil Attribute Impacts on Anionic Surfactant Mobility in Unsaturated Soil

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Abstract

Surfactant mobility in unsaturated soil will impact the effectiveness and efficiency of using these compounds for in situ environmental remediation above the water table. For this reason, transient unsaturated column tests were used to study the influence of boundary conditions and soil attributes on anionic surfactant transport. In these tests, aqueous surfactant solutions were injected into the inlet of horizontally mounted soil columns. Two commercial anionic surfactants were used, an alkyl ether sulfate (AES) and a linear alkylbenzene sulfonate (LAS).

The overall study was divided into two parts. First, boundary condition effects including injected surfactant solution concentration, initial moisture content, and surfactant application rate were investigated. Increasing the injection solution concentration increased anionic surfactant mobility in the column while changes in the initial soil moisture content and surfactant application rate had no significant impact. Second, the impacts of soil attributes such as texture, dominant exchangeable cation, and resident organic matter were measured. With respect to texture, mobility was found to be greater in a sandy soil as compared with two loamy soils. Both surfactants, especially LAS, were found to be more mobile in a Na^+ dominated soil rather than one dominated by Ca^{+2}. The absence of soil organic matter increased LAS mobility.

Introduction

Surfactants are presently being considered for use in environmental cleanup. The effectiveness and efficiency of using surfactants for in situ removal of contaminants located above the water table will depend on several factors. Some of the more important surfactant properties which need to be considered include biodegradation potential, ability to solubilize/emulsify contaminants, and mobility under unsaturated flow conditions. If surfactants become quickly immobilized when introduced into an unsaturated soil, they will not be useful for remediation. To address this concern, transient unsaturated column experiments were conducted under a variety of conditions. To date, laboratory work (Mustafa and Letey, 1971; Miller and Letey, 1975) and field pilot studies (Abdul et al., 1992; Abdul and Ang, 1994) have focused on nonionic surfactants with respect to use in unsaturated soil. More study is needed on anionic surfactants. Anionic surfactants are the most widespread type of surfactant in terms of commercial availability and would in all likelihood be the most economical for utilization in environmental remediation. For these reasons, two common anionic surfactants were chosen for testing.

Investigation of anionic surfactant mobility under unsaturated flow conditions was accomplished using experimental procedures described by Brown and Allred (1992). These tests were maintained under strict boundary conditions and involved the injection of an aqueous surfactant solution into the inlet of a horizontally mounted soil column. This study focued on the influence of the test boundary conditions along with soil attributes on anionic surfactant mobility. The test boundary conditions include injected surfactant solution concentration, initial soil moisture content, and application rate. For soil attributes, the effects of texture, dominant exchangeable cation, and the presence of organic matter were tested. Application concentration and rate are important parameters to consider before using surfactants in the field. Also, any detrimental impacts that soil conditions (moisture content) or attributes have on surfactant remediation capability need to be taken into account. Consequently, an understanding of boundary condition and soil attribute impacts on surfactant mobility under unsaturated flow conditions will provide insight into better techniques which can be used in the removal of contaminants located above the water table.

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Table 1. Surfactant Characteristics

Chemical name	*Trade name*[1]	*Abbreviation*	*Chemical formula*	*pH*[2]	*Viscosity*[2] *gm/(cm-s)*	*Critical micelle conc. mole/kg*	*Surface tension*[2] *dynes/cm*
Sodium alkyl ether sulfate	Witcolate 7093	AES	$C_{6-10}H_{13-21}(OCH_2CH_2)_3OSO_3Na$	6.9	0.0104	0.005	34.4
Sodium linear alkylbenzene sulfonate	Witconate 90F	LAS	$C_{10-13}H_{21-27}C_6H_4SO_3Na$	6.6	0.0104	0.001	33.8

[1] Both surfactants were obtained from the Witco Corp.

[2] Properties were obtained for 0.025 mole/kg surfactant solutions. Temperature = 22° C. For water at T = 22° C, surface tension is 72.4 dynes/cm and viscosity is 0.00956 gm/(cm-s).

Materials

Surfactants

Surfactants are organic compounds that on the molecular level are comprised of both hydrophobic and hydrophilic groups. This amphipathic structure causes surfactant molecules to concentrate at boundaries between phases, thereby altering interfacial properties such as surface tension. Above a certain solution concentration, called the critical micelle concentration (CMC), surfactant molecules form aggregates called micelles. Surface tension usually reaches a minimum value at or above the CMC. Surfactants are classified according to charge of the hydrophilic group as being anionic, cationic, zwitterionic (positive and negative molecular charge), or nonionic. For those interested, Rosen (1989) provides a complete overview on surfactants and their properties.

Anionics were the only surfactant type used in this study for reasons discussed in the introduction. The two commercial anionic surfactants tested were a sodium alkyl ether sulfate (AES) and a sodium linear alkyl benzene sulfonate (LAS). These two surfactants were chosen based on their properties along with also having widespread commercial availability. Table 1 lists some of the characteristics of the two surfactants. Both surfactants are comprised of a series of homologs. The individual members of the homologous series differ from one another only in the number of carbon atoms present in the hydrocarbon (alkyl) chain portion of the surfactant molecule. The active ingredient weight percents of the AES and LAS surfactant products were 39% and 91%, respectively. Typical surfactant product impurities include 1% sulfate and 1% free oil for AES, while LAS had 3% sulfate and 2% free oil. Surface tension and CMC values were measured with a Fisher Scientific Model 21 Tensiomat tensiometer. Viscosities were obtained with a Cannon Instrument Co. size 50 viscometer. The specific gravity was essentially equal to 1 for the 0.025 mole/kg solutions listed in Table 1.

Soils

Teller loam (Thermic Udic Argiustoll), Slaughterville loam (Thermic Udic Haplustoll), and Dougherty sand (Thermic Arenic Haplustalf) were obtained from field locations in Payne County, Oklahoma. The Teller and Dougherty are soils formed from weathering of alluvial sediments, while the Slaughterville is a weathered eolian deposit. Soil characteristics are presented in Table 2. These characteristics were determined using the procedures described in *Methods of Soil Analysis, Part 1 & 2* (ASA and SSSA, 1982 and 1986). Specific surface area was calculated from nitrogen gas (N_2) adsorption isotherms by use of the B.E.T. equation (Brunauer et al., 1938). Teller loam was used in the

Table 2. Soil Characteristics

Soil	*USDA classification*	*Extractable bases meq/100 g*	*Cation exchange capacity*[1] *meq/100 g*	*pH*	*Specific surface area* m^2/g	*Organic carbon content weight %*
Teller	"Loam" 52% sand 31% silt 17% clay	$Na^+ = 0.84$ $K^+ = 0.99$ $Ca^{+2} = 6.28$ $Mg^{+2} = 2.39$	~14	6.0	16.2	1.2
Dougherty	"Sand" 98% sand 2% silt and clay	$Na^+ = 1.40$ $K^+ = 0.14$ $Ca^{+2} = 2.40$ $Mg^{+2} \sim 0.00$	~5	5.9	1.9	0.1
Slaughterville	"Loam" 47% sand 35% silt 18% clay	$Na^+ = 0.22$ $K^+ = 0.26$ $Ca^{+2} = 8.05$ $Mg^{+2} = 1.62$	~10	8.0	13.4	0.5

[1] Cation exchange capacities for both the Teller and Dougherty were calculated assuming a base saturation of 75 percent, which is average for Payne County, Oklahoma soils in this pH range. With a pH of 8.0, total extractable bases are assumed to equal the cation exchange capacity for the Slaughterville.

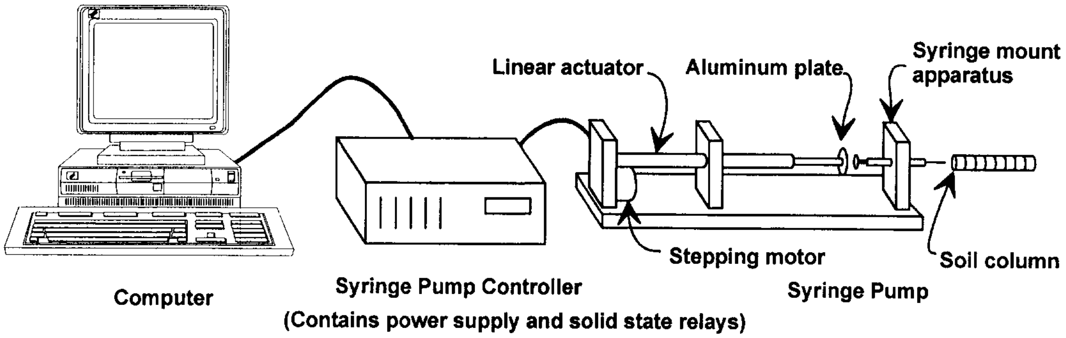

Fig. 1. Testing apparatus.

majority of the tests. This particular soil was chosen because its high adsorption/precipitation potential made it a good candidate for studying anionic surfactant transport. The Teller loam adsorption/precipitation potential for anionic surfactants was expected to be significant due to a high level of exchangeable Ca^{+2} (6.28 meq/100 g) and a relatively large specific surface area (16.2 m^2/g).

Experimental Procedures

Testing procedures described by Brown and Allred (1992) were used to conduct transient unsaturated anionic surfactant transport experiments. Figure 1 depicts the computer controlled syringe pump apparatus which was used to inject surfactant solution into the inlet of a horizontally mounted soil column. The boundary conditions which govern these experiments are injection concentration, initial soil moisture content, and injection rate which is a function of test duration time and the volume of solution applied. For the majority of experiments conducted, 20 ml of 0.025 mole/kg surfactant solution were injected over a period of 24 hours (h). A 0.025 mole/kg solution corresponds to a weight percent value of approximately 0.9% for both surfactants and is approximately 5x and 25x CMC for AES and LAS, respectively.

The inlet moisture content, and hence inlet soil moisture potential, were held constant during testing. The computer controlled syringe pump accomplished this by regulating the injected flow at a rate inversely proportional to the square root of elapsed time. The proportionality constant used for determining the injection rate was based on total test duration time and the volume of solution injected. Besides injection rate, the inlet moisture content value also depends on properties of the soil and the injection solution.

The column itself was comprised of individual acrylic rings and packed with soil to an average dry bulk density of 1.60 g/cm^3, which corresponds to a porosity of 0.40. The individual rings had a diameter of 3.5 cm and a length of 0.5, 1.0, or 2.0 cm. The shorter rings were placed adjacent to the column inlet to provide better resolution of the surfactant concentration and moisture content profiles within this region. Column lengths ranging from 18 to 35 cm insured that the wetting front and hence surfactant concentration front did not advance close (< 8 cm) to the column outlet. Upon test completion, the soil column was broken apart and the soil from within each ring divided into two parts for analysis of volumetric moisture content and anionic surfactant concentration.

Volumetric moisture content along the column was determined by oven drying the soil sample at 105°C for 24 h. The mean of the injected moisture percent accounted for by oven drying was 95%. Prior to chemical analysis, an extraction process was required to separate the anionic surfactant from the moist soil. The extraction process found to be the most successful required multiple steps. First, 10 ml of a 0.1 mole/kg NaCl solution was added to an Erlenmeyer flask containing a 5 g sample of moist soil. The flask was then hand-shaken and allowed to equilibrate for 0.5 h. After this, 90 ml of reagent grade acetone was added, and the flask placed in a shaker bath for 1 h at 300 rpm. Next, a 1 ml quantity of the extraction solution was taken from the flask and diluted to 100 ml with deionized water. The 100 ml sample was chemically analyzed for anionic surfactant concentration using the standard methylene blue method (APHA et al., 1985). The analyzed concentration value was in turn used to determine the amount of surfactant present in the soil from within each ring of the column. The mean of the percent surfactant recovered from each test was 90%.

The NaCl solution enhances surfactant extraction efficiency by reducing electrostatic attractions and/or precipitation. Multivalent cations can coadsorb anionic surfactants onto soil particles (Gaudin and Chang, 1952). Sodium ions will prevent this by competing with divalent calcium (Ca^{+2}) and magnesium (Mg^{+2}) ions for soil cation exchange sites. The presence of an electrolyte such as NaCl will also reduce the anionic surfactant CMC (Rosen, 1989). Surfactant precipitates will dissolve if the CMC is reduced to a level below the solubility limit for a Ca-surfactant or Mg-surfactant salt.

The addition of acetone enhances extraction by reducing hydrophobic adsorption of anionic surfactants onto soil particles. Surfactant molecules can dissolve in some polar solvents without distorting the liquid structure to a significant extent. As a result, surfactants present in such solvents will have little tendency to concentrate at phase interfaces such as soil particle surfaces (Rosen, 1989).

Boundary Condition Tests

All tests conducted during this part of the study used Teller loam soil. Baseline test conditions include a surfactant solution injection concentration of 0.025 mole/kg, an initial soil volumetric moisture content of 0.01, a time duration of 24 h, and an injection volume of 20 ml. Boundary condition effects were investigated by altering one of these test conditions while keeping the others constant. For each boundary condition studied, two sets of tests were conducted. One set was with the AES surfactant and the other with LAS. Injected surfactant concentration effects were tested with 0.01, 0.025, and 0.1 mole/kg solutions. Initial soil moisture contents were varied from 0.01 to 0.08 to 0.16. To quantify the effects of application rate on surfactant mobility, tests were conducted having different time durations (24 h and 96 h) but the same injection volume (20 ml).

Soil Attribute Tests

The attributes tested with respect to both AES and LAS were texture, dominant exchangeable cation, and resident organic matter. For all soil attribute tests, 20 ml of 0.025 mole/kg surfactant solution were injected over a period of 24 h into soil having an initial moisture content of 0.01. Soil texture effects on surfactant mobility were studied using the Teller loam, Slaughterville loam, and Dougherty sand. Surfactant mobility as affected by the dominant soil cation was investigated by replacing the resident soil cations in the Teller loam with either Na^+ or Ca^{+2}. Cation replacement was accomplished through equilibration of 1 kg of Teller loam with 2 kg of 0.2 mole/kg NaCl or $CaCl_2$ solution followed by soil drainage. This cycle was then repeated a second time. Results of the AES and LAS tests conducted with the Na-Teller and Ca-Teller were then compared with those from tests run on the unaltered soil. The effects of resident soil organic matter on AES and LAS mobility were studied by testing these surfactants on Teller loam in which organic matter had been removed and then comparing the results with those obtained using unaltered Teller loam which contained 1.2% organic carbon. Organic matter was removed from the Teller using a hydrogen peroxide oxidation procedure (ASA and SSSA, 1986).

Experimental Results

Test results are presented in plots of surfactant concentration versus distance. The concentration values are given in mole/kg of the total porous media. Here, total porous media includes both soil solids and soil solution. Distance refers to the length in cm from the column inlet. The evaluation of surfactant mobility was based on the distance the concentration front advanced within the soil column. Moisture content profiles from the boundary condition tests are also provided.

Boundary Condition Tests

Figure 2 depicts the results of the test series in which surfactant injection concentrations were varied. The AES data are shown in Figures 2a (surfactant concentration) and 2c (moisture content). LAS surfactant concentration and moisture content profiles are presented in Figures 2b and 2d, respectively. For both surfactants, the column distance penetrated by the concentration front increased with an increase in the injected solution concentration. The AES surfactant had roughly twice the mobility of LAS in tests having similar conditions. The ratio of peak concentration to inlet concentration for the 0.01, 0.025, and 0.1 mole/kg tests was 1.11, 1.33, and 1.33 for AES and 1.24, 2.21, and 3.58 for LAS. Allred and Brown (1996) have proposed two likely mechanisms, which include chromatographic separation of surfactant homologs and/or displacement of divalent cations at the column inlet, to explain why inlet concentrations are significantly less than peak profile values.

Inlet moisture contents and inlet surfactant concentrations were also observed to increase with increasing AES or LAS

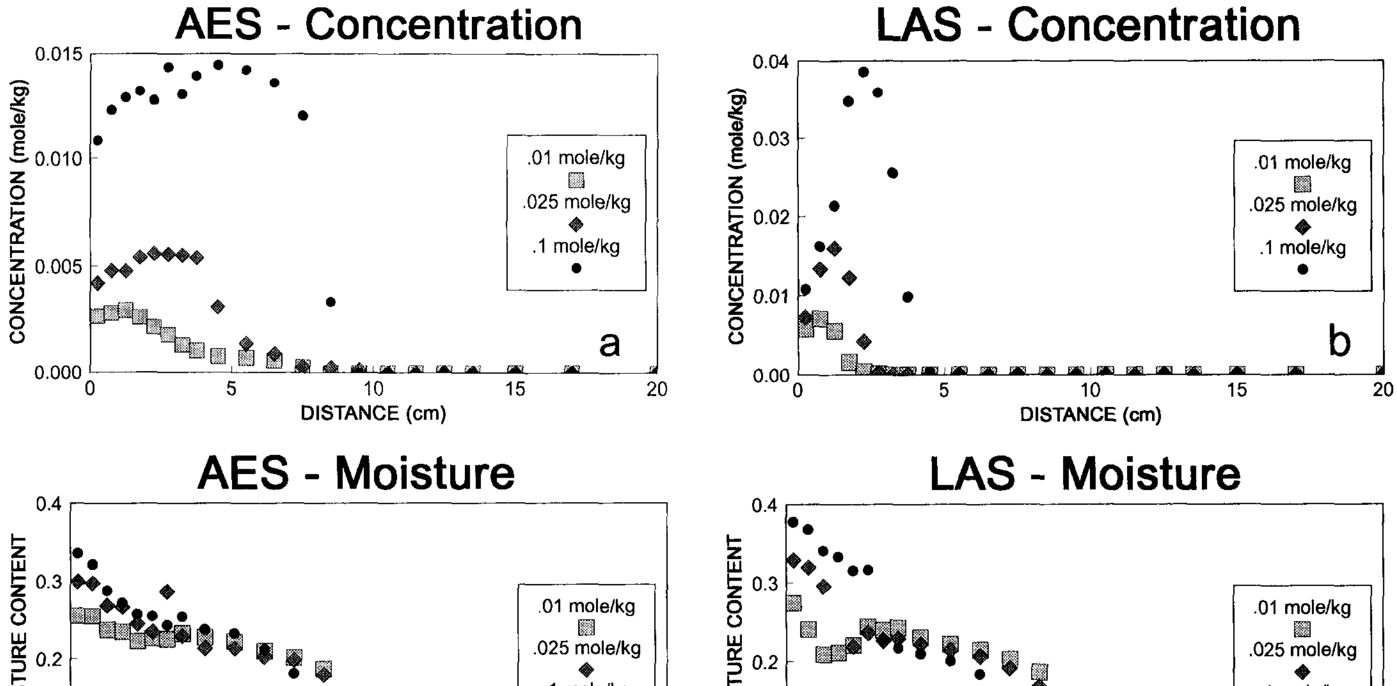

Fig. 2. Injected surfactant concentration boundary conditions test results. (a) AES concentration profiles. (b) LAS concentration profiles. (c) AES moisture content profiles. (d) LAS moisture content profiles.

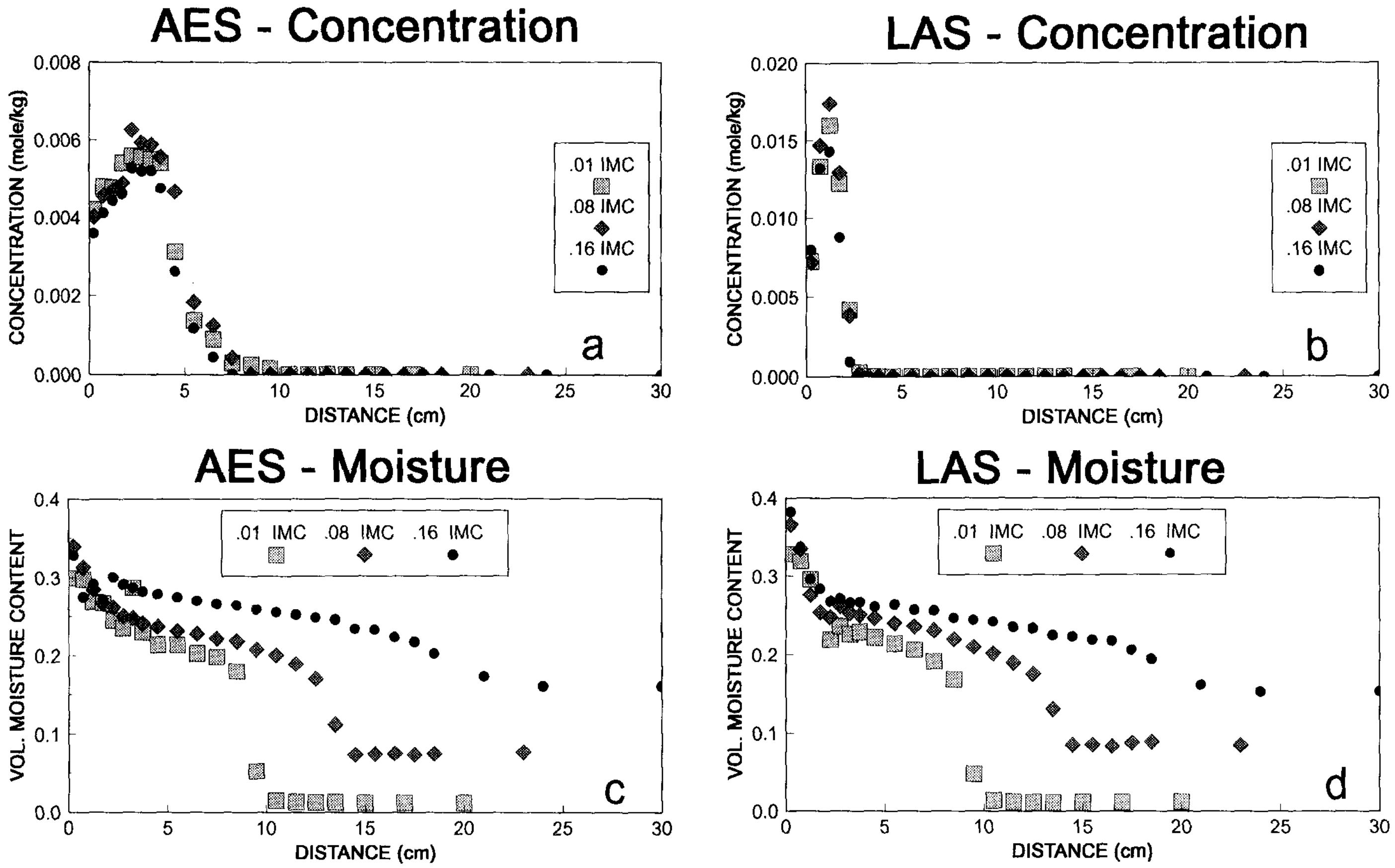

Fig. 3. Initial moisture content (IMC) boundary condition test results. (a) AES concentration profiles. (b) LAS concentration profiles. (c) AES moisture content profiles. (d) LAS moisture content profiles.

injection concentrations. For LAS, the moisture content profiles (Figure 2d) from the 0.01 and 0.025 mole/kg tests exhibit notches which coincide with the surfactant concentration fronts (Figure 2b). The notch from the 0.01 mole/kg test is readily apparent while the one from the 0.025 mole/kg test is more subtle. These notches represent a reversal in the usual trend of a continuous decrease in moisture content from inlet to wetting front edge. The notches, which are discussed in detail by Allred and Brown (1996), result from the pressure head versus moisture content relationship in the transition zone of the soil column between the region of high surfactant concentration and the region where no surfactant is present. Although there is no notch present in the moisture content profile from the 0.1 mol/kg LAS test, a steep drop-off corresponding to the surfactant concentration front does occur.

Results from the test series in which initial moisture contents were varied are provided in Figures 3a and 3c for AES and 3b and 3d for LAS. As shown, increasing the initial moisture content causes an increase in the distance penetrated by the wetting front. However, this has no significant impact on surfactant mobility.

Figure 4 illustrates how the surfactant application rate affects AES and LAS transport. A decrease in the injection rate, which is reflected by a reduction in the inlet moisture content and a slight increase in the wetting front penetration, affected at best a very small increase in AES and LAS surfactant mobility. A large notch is quite prevalent in the moisture content profile of the LAS test having the lowest injection rate (Figure 4d).

Soil Attribute Tests

AES and LAS surfactant mobility in three different soils are shown in Figures 5a and 5b, respectively. When compared with the two loamy soils, surfactant mobility as determined by concentration front position was greater in the Dougherty sand by a factor of two for AES and by a factor of four for LAS. Concentration profiles for the Teller and Slaughterville soils were similar with respect to each surfactant tested. Unlike profile configurations for the Teller and Slaughterville, the Dougherty concentration curves were relatively flat from the inlet to the front.

Figure 6 shows the effects due to the dominant exchangeable soil cation on surfactant mobility. AES results are given in Figure 6a while those for LAS are provided in Figure 6b. As determined from concentration front positions, both surfactants exhibited the greatest mobility in the Na^+ dominated Teller. The least mobility was found in the Ca^{+2} dominated soil. The difference in surfactant mobility between the Na^+ and Ca^{+2} dominated Teller was greatest with LAS.

As depicted in Figure 7, resident soil organic matter affected the mobility of LAS but not AES. LAS mobility (Figure 7b) increased when organic matter was removed from the Teller loam.

Discussion

Surfactant mobility will depend on the amount adsorption/precipitation losses which take place. There are a number of different mechanisms which can reduce anionic surfactant

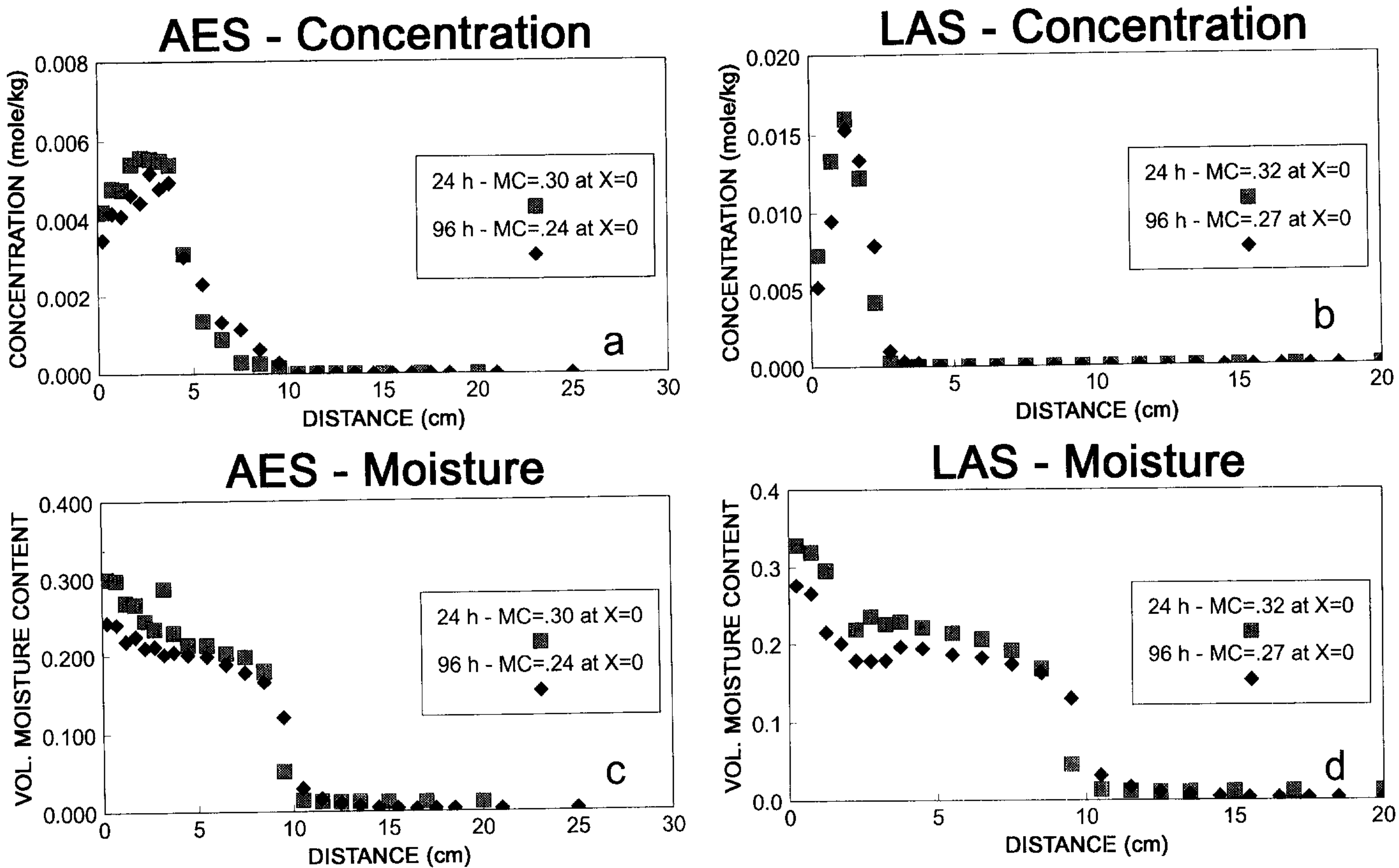

Fig. 4. Surfactant solution application rate boundary condition test results. The overall injection rate for the 24 h tests were four times that of the 96 h tests. (a) AES concentration profiles. (b) LAS concentration profiles. (c) AES moisture content profiles. (d) LAS moisture content profiles.

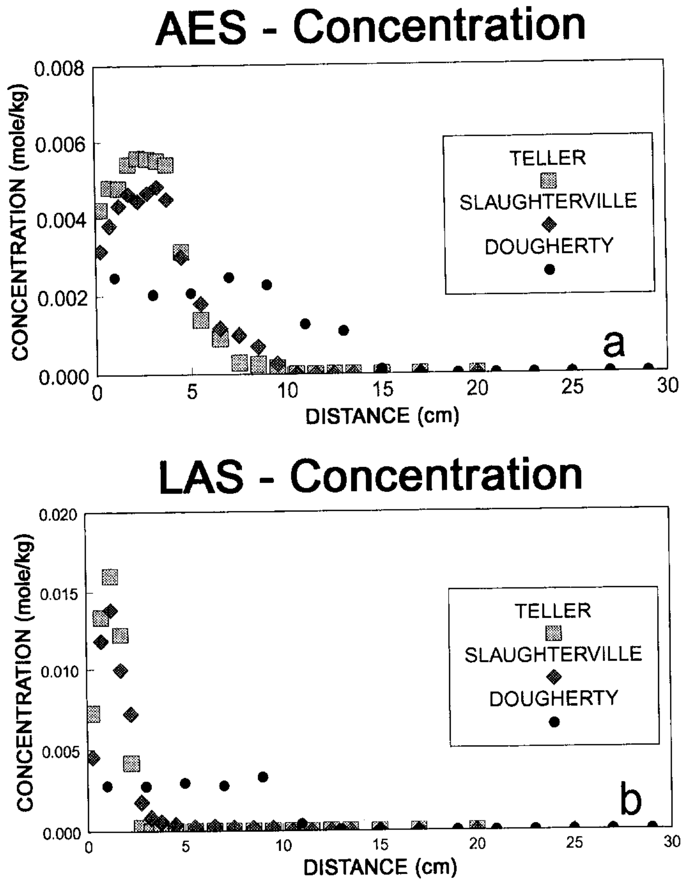

Fig. 5. Results of surfactant testing on different soils. (a) AES concentration profiles. (b) LAS concentration profiles.

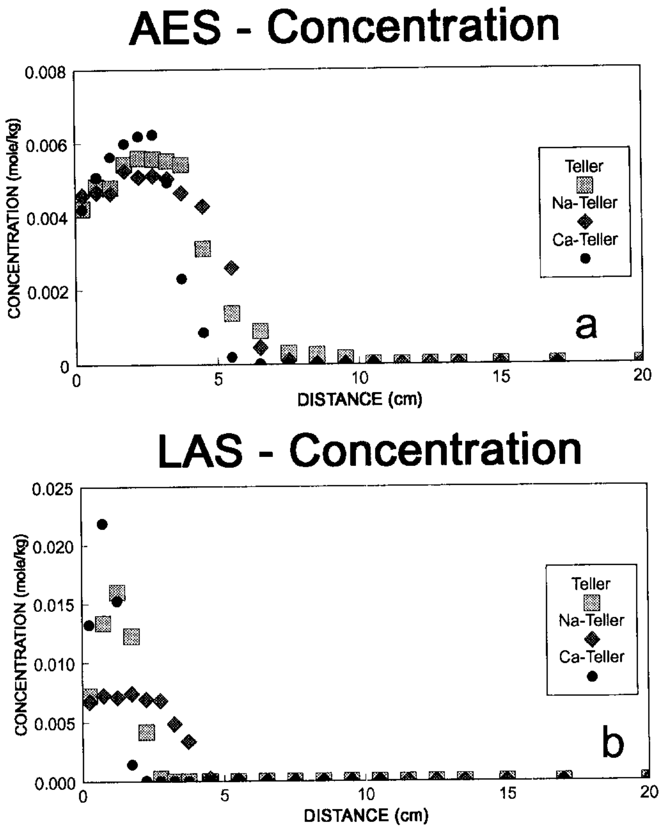

Fig. 6. Dominant exchangeable cation effects. (a) AES concentration profiles. (b) LAS concentration profiles.

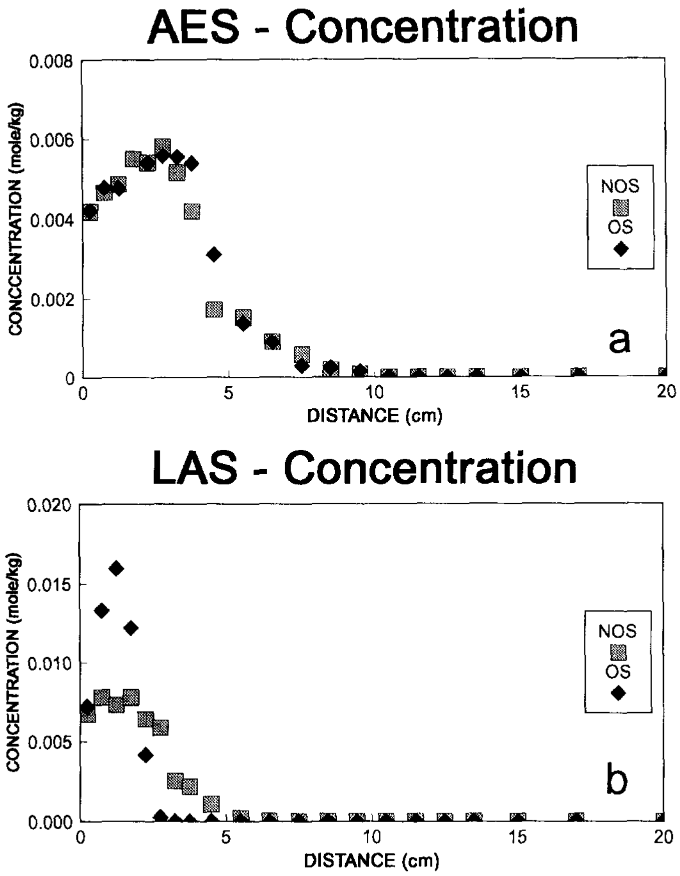

Fig. 7. Soil organic matter effects. NOS—Teller loam with organic matter removed. OS—Teller loam with organic matter present. (a) AES concentration profiles. (b) LAS concentration profiles.

mobility in unsaturated soils. These include anion exchange, coadsorption, hydrophobic adsorption, and precipitation. At low pH, surface sites on soil particles and resident organic matter become protonated and assume positive charge (Bohn et al., 1985). This in turn gives rise to an anion exchange capacity which allows negatively charged surfactant molecules to become electrostatically adsorbed. Testing by Clementz and Robbins (1976), indicated that small amounts of dodecylbenzene sulfonate were adsorbed at positively charged crystal edge sites on montmorillonite. Because injection solution and soil pH values were at most only slightly acidic (Tables 1 and 2), it is unlikely that anion exchange played a major role in governing surfactant mobility during the tests conducted in this study. However, electrostatic attraction between soil and anionic surfactants may be possible due to coadsorption (Gaudin and Chang, 1952). Essentially, coadsorption involves multivalent cations such as Ca^{+2} and Mg^{+2} which bridge surfactant anions to the clay minerals or resident soil organic matter which usually have negative charge. Hydrophobic adsorption also affects surfactant mobility. It results from the surfactant tendency to escape from its aqueous environment by concentrating at phase boundaries or by interacting with surfactant which has been previously adsorbed at an interface (Rosen, 1989). Additionally, precipitation can immobilize anionic surfactants (West and Harwell, 1992). The most likely precipitates in soils are Ca-surfactant and Mg-surfactant salts. Therefore, as with coadsorption, soils having high levels of Ca^{+2} and Mg^{+2} ions pose a threat with respect to anionic surfactant precipitation.

AES and LAS test results can be explained for the most part by the various adsorption/precipitation mechanisms previously described. Surfactant mobility was noticed to increase with an increase in injection concentration (Figure 2). At the highest injection level (0.1 mole/kg), the AES surfactant concentration profile is relatively flat with a front position coinciding with the leading edge of the moisture content curve. These are indications that AES overwhelms the adsorption/precipitation capacity of the Teller loam when injected at this concentration (4% by weight). For LAS on the other hand, this capacity is not met with 0.1 mole/kg injected solution. This is evident from the concentration profile which penetrates only half the distance of the wetting front edge. Consequently, even for soils having a high adsorption/precipitation capacity, sufficient mobility can be obtained with the application of the proper surfactant in moderately high concentrations.

The initial soil moisture content and solution injection rate did not greatly impact anionic surfactant mobility (Figures 3 and 4). For the Teller loam at least, this suggests that mobility depends much less on hydraulic conditions and more on the adsorption/precipitation capacity of the soil with respect to a particular surfactant.

Considering mobility only, soils such as the Dougherty sand are better candidates for surfactant enhanced remediation than those such as the Teller and Slaughterville loams (Figure 5). There are two explanations which can account for this. First, the lower specific surface area of the sand reduces the amount of solid/liquid interface available for surfactant adsorption. Second, the Dougherty has lower amounts of Ca^{+2} and Mg^{+2} than the loamy soils (Table 2). Because of this, anionic surfactant mobility in the sand was less likely to be reduced due to coadsorption and/or precipitation.

Both surfactants had greater mobility in the Na^{+} saturated Teller and less where the soil was Ca^{+2} dominated (Figure 6). Mobility in the unaltered Teller, which contained a mixture of monovalent and divalent cations, was between these two extremes. These results indicate the influence of coadsorption and/or precipitation processes which in turn depend on the amount of multivalent cations which are present. One implication from this set of tests and those conducted by Ducreux et al. (1990) is that NaCl preflushing prior to environmental remediation can enhance anionic surfactant mobility. Saturated core flood tests done by Lau and O'Brien (1988) show that anionic surfactant mobility can also be increased through addition of NaCl (4% by weight) to the injection solution.

LAS mobility was observed to increase when the resident soil organic matter was removed from the Teller loam (Figure 7). Removal of the organic matter reduces the cation exchange capacity along with making the soil less hydrophobic. This could have the effect of limiting both coadsorption and hydrophobic adsorption, thereby increasing LAS mobility. More study is needed to determine why AES mobility is not affected by the resident organic matter.

AES was significantly more mobile than LAS under all conditions tested. Schwuger (1984) noted that surfactants similar to AES, which contain oxyethylene structural groups, exhibit resistance to precipitation. AES may be more resistant than LAS with respect to other mechanisms affecting transport as well. Consequently, surfactant molecular structure needs to be addressed in order to properly assess anionic surfactant mobility in unsaturated soil.

Summary and Conclusions

The following is a list of the results obtained from the tests conducted in this study on anionic surfactant mobility in unsaturated soil.

Boundary Condition Effects

1. Increasing the concentration of the applied surfactant solution can substantially increase anionic surfactant mobility.

2. Mobility is not affected by the initial moisture content of the soil.

3. The surfactant solution application rate has only a slight impact on mobility.

Soil Attribute Effects

4. Anionic surfactants show good mobility in a coarse-grained soil having low Ca^{+2}/Mg^{+2} concentrations and specific surface area.

5. Anionic surfactants are significantly more mobile in a soil dominated by exchangeable Na^+ rather than Ca^{+2}.

6. Resident soil organic matter can affect mobility for certain surfactants.

The most important implication from the test results is that adsorption/precipitation processes govern the mobility of anionic surfactants in unsaturated soil while hydraulic boundary conditions have little or no affect. Consequently, based on the criteria of mobility only, the effectiveness and/or efficiency of in situ surfactant enhanced environmental remediation above the water table will depend on soil attributes and the applied concentration of the anionic surfactant.

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Authors' Note

Use of trade names is for informational purposes only and does not imply endorsement by the authors, Oklahoma State University, or the New Mexico Bureau of Mines.

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