



SOIL FLUSHING OF RESIDUAL TRANSMISSION FLUID: APPLICATION OF COLLOIDAL GAS APHRON SUSPENSIONS AND CONVENTIONAL SURFACTANT SOLUTIONS

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(First received June 1993; accepted in revised form 29 June 1994)

Abstract—An innovative technology using Colloidal Gas Aphrons (CGAs) is applied in this study to flush residual levels of a light non-aqueous phase liquid (LNAPL) such as automatic transmission fluid (ATF) from a Superfund site soil. Performance of CGA suspensions is compared with that of conventional aqueous anionic surfactant solutions under both gravity stable (downflow) and gravity unstable (upflow) conditions. CGA suspensions were found to be more effective in washing ATF under both downflow and upflow modes. The displacement of ATF from the soil pores seems to be the mechanism of removal in the case of water floods. Increasing the surfactant concentration did not increase the removal rate correspondingly. The pressure required to pump the CGA suspension was much lower than that required for conventional surfactant solutions or water flood. Results show enough promise that more research should be directed at this potential technology for *in situ* remediation of contaminated aquifers.

Key words—NAPL, CGAs, surfactants, soil flushing, water flood, soil remediation

INTRODUCTION

The contamination of subsurface soils with non-aqueous phase organic liquids (NAPL) such as petroleum hydrocarbons is a major environmental problem. The sources of these contaminants include hazardous waste landfills, accidental spills of petroleum products, and leaks from underground storage tanks. Due to capillary forces, most of these contaminants are trapped in the soil pores in the form of non-continuous ganglia. The ganglia are formed due to the non-wetting properties of NAPL in the porous media and prevent the spreading of NAPL on the surface of the soil matrix. The immiscible organic liquid immobilized by capillary trapping acts as a continuing source of dissolved organic pollutants for the passing groundwater. Removal of these contaminants from the subsurface environment is essential in preventing the spread of contamination.

Conventional "pump and treat" methods appear to be inadequate because they require a long time period before making significant reductions in the quantity of NAPL (Mackay and Cherry, 1989). The problems associated with conventional water floods are (i) limited solubility of the contaminants in water and (ii) high interfacial tension with water. One of the methods used to increase water solubility of the NAPLs and decrease the interfacial tension is the application of surface active agents (Latil *et al.*, 1980).

Surfactants reduce the interfacial tension and alter the wetting properties of the soil matrix due to their tendency to concentrate at interfaces. Surfactants form self aggregates known as micelles at concentrations above critical micelle concentration (CMC) and dissolve appreciable quantities of non-polar solutes which are insoluble in normal aqueous solutions. Several researchers have proposed surfactant use in remediation of abandoned hazardous waste sites because of these two properties (Ellis *et al.*, 1985; Nash, 1987; Gannon *et al.*, 1989; Ang and Abdul, 1991).

Abdul *et al.* (1990) evaluated the suitability of 10 commercial surfactants for flushing residual levels of automatic transmission fluid (ATF) from a sandy material based on surface tension, soil dispersion, oil dispersivity, and batch washing tests and reported that non-ionic alcoholic ethoxylate surfactant was best suited. Subsequently, they demonstrated its use for *in situ* soil flushing of ATF (Ang and Abdul, 1991). However, anionic sulfonates and sulfates are known as oil recovery enhancers because of their tendency to decrease the interfacial tension, to create oil/surfactant aggregates of negative charge thereby repelling the soil surface, and to improve the oil wetting properties of the soil surface (Donaldson *et al.*, 1989). Anionic surfactants also have the advantage of lower adsorption on soil in comparison to non-ionic surfactants and they are easily

recovered from the effluent and reused (Gannon *et al.*, 1989).

An innovative technology that may be effective for *in situ* soil flushing is the use of microbubbles or Colloidal Gas Aphron (CGA) suspensions which are generated from surfactant solutions. CGAs were first described by Sebba (1971) as a dispersion of microbubbles (25 μm and above in diameter) in water. Chaphalkar *et al.* (1993) measured a range of 25–300 μm with a particle size analyzer. These spherical bubbles (known as aphrons) are encapsulated in a thin shell of surfactant film and have colloidal properties. The structure, fundamental properties, and possible applications of CGAs have been extensively studied (Sebba, 1971, 1982; Longe 1989; Chaphalkar *et al.*, 1993). By volume, a CGA suspension consists of approximately 65% gas and hence, is a low density liquid. Application of CGAs in soil flushing was reported by Longe (1989) and Roy *et al.* (1992). Longe (1989) reported that these suspensions were more effective in flushing a variety of hydrophobic organics from soil in comparison to conventional surfactant solutions at identical concentrations. Roy *et al.* (1992) found that CGA suspensions are more efficient than surfactant solutions on the basis of weight of contaminant removed per gram of surfactant.

In this study, a model NAPL, automatic transmission fluid (ATF) is selected as it was used in an earlier work with non-ionic surfactants by Ang and Abdul (1991). An anionic surfactant, sodium dodecylsulfate (SDS), in the form of CGA suspensions and conventional surfactant solutions was used. SDS is a commonly used surfactant that is biodegradable and considerable work has been reported on its interactions with soil. The efficiency of *in situ* flushing using CGA suspensions was compared with that of conventional surfactant solutions and water floods.

MATERIALS AND METHODS

Soil columns

An uncontaminated soil from a local Superfund site north of Baton Rouge, La, was selected for this study. The soil was air dried, homogenized, and kept in an oven overnight at 105°C. Soil passed through a 2 mm sieve was used to pack the column. Physical and chemical characterization of the soil was performed in accordance with methods of soil analysis (ASA, 1986). The results presented in Table 1 suggest that the soil is a fine silty loam.

Glass columns 10 cm long and 5.75 cm in diameter with a stainless steel top and bottom were used for all the

Table 1. Physical and chemical characteristics of the soil

Physical		Chemical	
Sand %	41	Calcium	769 mg/kg soil
Silt %	50	Calcium	211 mg/kg soil
Clay %	8.5	Potassium	35 mg/kg soil
Organic matter %	0.4	Sodium	36 mg/kg soil
pH	5.7	Phosphorus	27 mg/kg soil
		C E C	5.8 meq/100 g

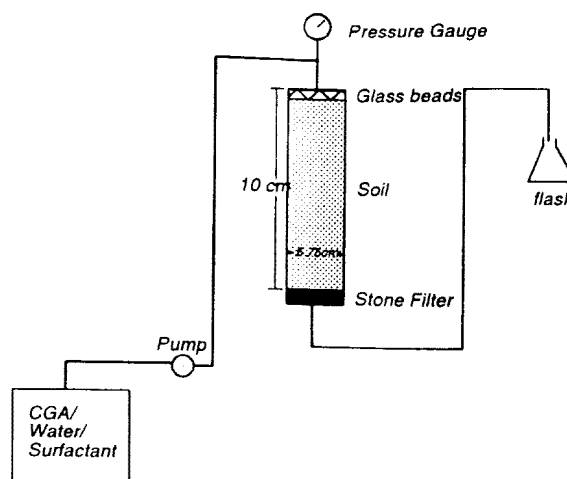


Fig. 1. Apparatus used for soil flushing experiments.

experiments (Fig. 1). To prevent soil from being washed from the column the bottom has the provision for a porous stone (fritted disc). A soil packing procedure was followed that achieves a bulk density range similar to the field soils. It was packed by dropping the soil through a funnel in four equal portions of about 100 g. Each layer was compacted by giving 25 blows with a compacting rod to obtain a bulk density of about 1.5 g/cm³ and a porosity of about 0.45, on the high side of the field bulk densities of regional soils (1.2–1.5 g/cm³). A wire mesh followed by a layer of 4 mm glass beads and a fine mesh was placed on top of the soil to distribute the flow uniformly across the soil column.

Chemicals

Lubriguard Dexron[®] II/Mercon automatic transmission fluid (Specialty Oil Company, Shreveport, La) was used as the contaminant for the experiments. The physical properties of this fluid are listed in Table 2.

An anionic surfactant, sodium dodecylsulfate (SDS) (Life Technologies Inc., Gaithersburg, Md) was used to prepare surfactant solutions and CGA suspensions. The structure and properties are tabulated in Table 2.

Column contamination

The packed column was kept in a vertical position and saturated with deionized water at a slow rate to remove the air bubbles. The saturated hydraulic conductivity was measured using a standard constant head method (ASA, 1986). The water-saturated soil column was allowed to drain under gravity for about 24 h. The drained column was then contaminated with ATF using a piston pump (FMI lab pump model QG20, Fluid Metering Inc., Oysterbay, N.Y.) until the ATF breakthrough was achieved. The column was allowed to drain under gravity for about 24 h, beyond which the drainage was negligible. The drained fluid was collected and analyzed for ATF to calculate the residual ATF remaining in the column.

Column washing

Experiments were conducted in downflow and upflow modes with water, conventional surfactant solution, and CGA suspensions. CGA suspensions were produced using a spinning disc generator fabricated in our laboratory based on the method suggested by Sebba (1985). Sebba estimated that 10,000 l of CGA suspension can be generated with less than 1 kWh of electricity. Concentrations of surfactant used were 8 mM, which is the CMC for SDS, and a higher concentration of 30 mM. CGA suspensions were generated using the same concentrations. The pressure at the influent end was monitored using an analog pressure gauge and the

Table 2. Properties of DEXRON[®] II/MERCON automatic transmission fluid and sodium dodecylsulfate

Transmission fluid		Sodium dodecylsulfate	
Viscosity (kinematic) at 99°C	7.4cSt	Structure	CH ₃ (CH ₂) ₁₀ -CH ₂ O-SO ₃ -Na
Specific gravity at 20°C	0.875	Molecular weight	288.38
Boiling range at 1 atm.	350°C	CMC at 25°C	8.08 mM
Water solubility at 20°C	<50 mg/l	Purity	≥99.5%
Surface tension at 25°C	33.7 dyne/cm	Aqueous solution pH	7-7.5
Vapor pressure at 25°C	<2 × 10 ⁻⁴ mm Hg	Biodegradable	yes

effluent collected was analyzed for ATF. The rate of pumping used for all the experiments was about 2.6 ml/min.

Analysis of chemicals

Extraction of ATF from the effluent samples was performed using the recommended Standard Method 5520 B (APHA, 1989). The procedure was verified in presence of the surfactant and a known amount of ATF, and the average recovery was 99% with a standard deviation of 5%. The procedure recommended in section 5520 E (APHA, 1989) as modified by Martin *et al.* (1991) was used for the extraction of ATF from soil. The average recovery was 96% with a standard deviation of 9% for the trial extractions using contaminated soil with known quantities of ATF.

RESULTS AND DISCUSSION

The hydraulic conductivities for the soil columns with an approximate bulk density of 1.5 g/cm³ from 0.75×10^{-4} to 2.5×10^{-4} cm/s with a mean of 1.33×10^{-4} cm/s and a standard deviation of 2×10^{-5} cm/s. These hydraulic conductivities are averages of a minimum of three measurements for each column. Columns with hydraulic conductivity values outside the above range were rejected.

All soil flushing experiments were conducted in duplicate. Typical results of selected runs showing percent ATF recovered from the column (%) with the passage of different flushing media are presented in Fig. 2(a, b). The reproducibility was considered to be satisfactory and the average of the two experimental runs was used for all comparisons.

Downflow experiments

Figure 3 shows a comparison between the CGA suspension, a conventional surfactant solution, and water flood in removing ATF from the soil in downflow operations using two concentrations of SDS, 8 and 30 mM. It is evident from the figure that both CGA suspension and conventional surfactant solution are more effective than water flood in recovering ATF from soil columns. The rate of ATF removal in all the cases was high in the first pore volume followed by a slower rate of removal for the next five pore volumes. The high removal in the first pore volume is attributed to any free phase ATF that may remain in the column after 24 h drainage.

ATF removal from soil columns was 50, 42 and 20% after 6 pore volumes of washing with CGA suspensions, surfactant solutions both at 8 mM SDS concentration and water flood, respectively. The increased removal in the case of CGA suspension and the surfactant solution may be explained by the four mechanisms suggested in the literature: displacement,

solubilization, dispersion, and electric repulsion at the surface of the soil particles (Nelson *et al.*, 1984, Donaldson *et al.*, 1989; Ang and Abdul, 1991).

In the case of a water flood, the only mechanism that is responsible for the recovery of ATF is likely to be displacement. Due to the water wetting properties of the soil matrix, at a large enough pressure head, the trapped oil is displaced from the soil. The inadequacy of water flooding is primarily due to immobilization of NAPL in the porous media as a result of snap-off and bypassing (Mercer and Cohen, 1990; Wilson *et al.*, 1990; Chatzis *et al.*, 1983). Snap-off is prevalent in a soil matrix with high aspect ratio pores. Bypassing phenomena is noted when a section of larger pores containing the NAPL are surrounded by smaller pores. It should be noted that an earlier study by Ang and Abdul (1991), which used a horizontal flow mode with a sandy soil in a 2.42×50 cm long column, reported that water removed only about 14.5% of ATF in 7 pore volumes. This is about 5.5% less than the removal rate obtained in this study in downflow mode with water in 6 pore volumes.

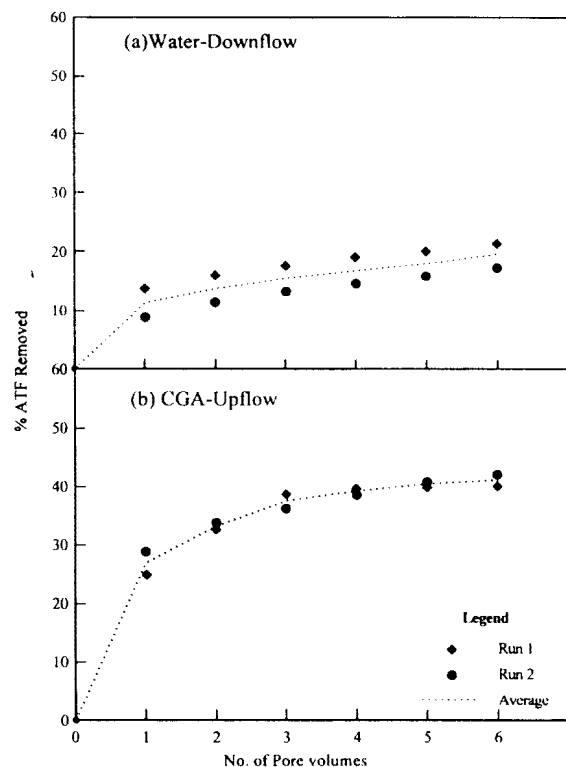


Fig. 2. Typical results showing the variation between the duplicate runs.

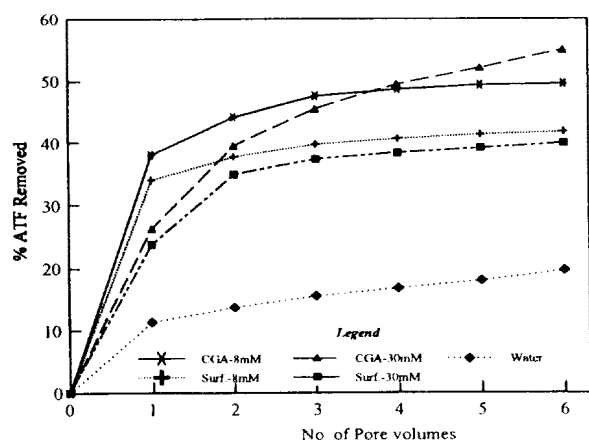


Fig. 3. ATF removal in downflow operation with different flushing media.

Adding surfactant to water should decrease oil/water interfacial tension. The reduction in interfacial tension improves the displacement and dispersion of ATF droplets, resulting in higher removal of ATF from soil. The surfactant used (SDS) has a negative charge, as do the clay particles. When ATF droplets with surfactant molecules around them approach a clay particle, the repulsive forces keep the droplets in suspension and thus are mobilized. At low surfactant concentrations (less than or equal to CMC), both emulsification and solubilization mechanisms are negligible. However, at higher surfactant concentrations (above CMC), solubilization will become significant as the increased number of micelles solubilize more droplets and thereby enhance the removal of ATF. Our results were compared with those reported by Ang and Abdul (1991). An anionic surfactant used in our experiments at a concentration of 8 mM removed about 42% of ATF in 6 pore volumes compared to 32.3, 21.8 and 29.2% in 7 pore volumes by a non-ionic surfactant (alcohol ethoxylate) at 12.75, 25.5 and 51 mM concentrations respectively. The CMC of the surfactant used by Ang and Abdul is 0.38 mM compared to 8 mM of SDS used in the present study. The non-ionic surfactant used in their study was selected from the results of a series of screening tests where they concluded that non-ionic alcohol ethoxylate surfactant was most efficient (Abdul *et al.*, 1990). However, it should be kept in mind that our experiments were done in downflow mode with a fine silty loam, whereas Ang and Abdul (1991) used sandy soil in horizontal flow mode.

Even though the CGA suspension generated from the surfactant solution is a homogeneous fluid at the time of pumping, within the porous medium separation of the gas and liquid phases will occur resulting in a two-phase flow system. The collapse of CGA bubbles in the column depends on several factors such as the flow rate, pore structure, and the type of medium. Additional research is currently under way to study this phenomenon in 1- and

2-dimensional systems. The gas and liquid phases propagate through the porous medium at different rates with the liquid phase advancing faster than the gas phase (Longe, 1989). The effluent from the soil column appears as a series of gas bubbles and liquid drops. In our case, three different phases were observed in the effluent: ATF droplets, surfactant solutions, and gas bubbles. The gas portion that exits from the column may carry some volatiles present in the soil column; however, the gas phase was not monitored for volatiles in this study. The mechanisms of removal by the liquid phase of the CGA suspension are thought to be the same as those of a conventional surfactant solution which are displacement, solubilization, dispersion, and electric repulsion at the surface of the soil particles.

CGA bubbles will have the same charge on the surface as the surfactant from which it is generated (Chaphalkar *et al.*, 1993). The surface area provided by the CGA bubbles may also play a significant role in the removal of dispersed oil droplets from pore spaces. An important feature that was noticed when soil columns were flushed with CGA suspensions was that the soil appeared to undergo significant structural changes. We observed formation of channels and rearrangement of soil particles when CGA suspensions were pumped, whereas no such channeling was observed for conventional surfactant solutions and water floods. For CGA runs channeling began by the end of the first pore volume and persisted for the duration of the experiment, but when feed was switched from CGA to water these channels disappeared gradually. When water flood was switched back to CGA, the channels reappeared, although not at the same locations. This behavior is being investigated in more detail.

Effect of surfactant concentration. The effect of surfactant concentration used to prepare CGA suspensions and conventional surfactant solutions was studied using two concentrations of surfactant: 8 mM which is the CMC for SDS and a higher concentration, 30 mM, for flushing ATF from soil columns. The CGA suspensions generated from 30 mM SDS removed about 55% as compared to 40% by conventional surfactant solution at identical concentration and to 20% by water flood. The results shown in Fig. 3 indicate that increasing the surfactant concentration does not markedly change the removal efficiency for both CGA suspensions and conventional surfactant solutions. This apparent anomaly can be explained by observations reported by Liu and Roy (1993). Liu and Roy (1993) in their recent investigation on the use of SDS in soil flushing, found that the increase in surfactant concentration changed the hydraulic conductivity and pore geometry of the soil matrix. Precipitation of divalent calcium dodecylsulfate was found to be the prevalent mechanism that reduced the hydraulic conductivity. The calcium content of the soil used in this study was high (about 750 mg/kg soil) and may have precipitated as

calcium dodecylsulfate causing pore blocking. Pore blocking prevents the wash media from reaching all pore spaces and thus decreases the removal of ATF. Close visual observation of the effluent from the soil columns flushed with CGA suspensions and surfactant solutions of higher concentration (30 mM) indicated that the effluent contained an emulsion. Approximately six to nine extractions with freon were necessary to separate ATF from such an emulsion, whereas only three or four extractions with freon were sufficient when low SDS concentration was used. Emulsification did not occur at the lower surfactant concentration of 8 mM. This indicates that at high concentrations of the surfactant the emulsification may affect the overall removal efficiency. From this discussion it appears that an optimum surfactant concentration, between the two concentrations tested here, may be more effective in maximizing solubilization and minimizing emulsification, which are required for highest recovery of ATF from soil.

Upflow experiments

To study the effects of the flow regime on the removal efficiency of ATF, identical experiments were conducted in an upflow mode. Figure 4 shows the results of upflow experiments with CGA suspension (8 mM), conventional surfactant solution (8 mM), and water flood. It is evident from the figure that both the CGA suspension and the conventional surfactant solution are more effective than water flood in washing ATF. The ATF removal for all cases was highest during the initial stages followed by a slower rate. This high removal in the initial stages may be due to the free phase ATF that is remaining in the column after 24 h drainage. Total ATF removal using CGA suspension and conventional surfactant solution was approximately 41%, while water flood removed about 33% of ATF from the column.

In comparison to conventional water floods both CGA suspension and surfactant solution are not as effective in the upflow mode as they are in the

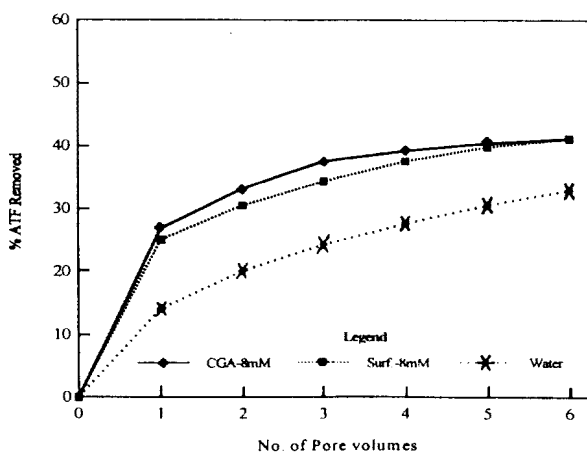


Fig. 4. ATF removal in upflow operation with different flushing media.

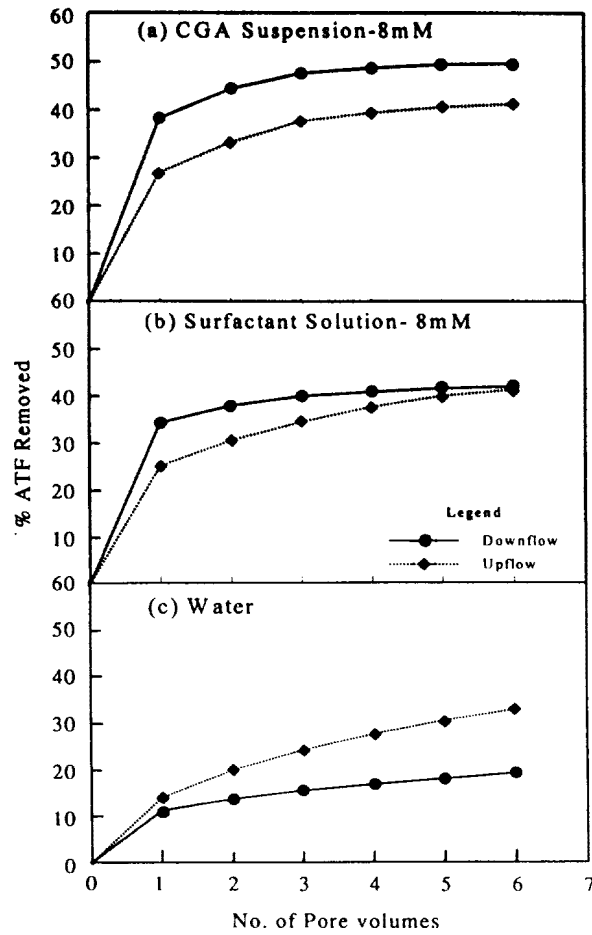


Fig. 5. Comparison of the ATF removal in downflow and upflow operations for (a) CGA—8 mM, (b) surfactant—8 mM and (c) water.

downflow mode. CGA suspensions do not appear to be beneficial over the conventional surfactant solutions. A comparison of ATF removal in downflow and upflow modes is shown in Figs 5(a-c) for CGA suspensions, surfactant solutions and water flood, respectively. The CGA suspension is more effective in the downflow mode than in the upflow mode. In the downflow mode the CGA suspension removed about 50% of ATF, while in upflow mode only 41% was removed. Conventional surfactant solution removed about 42% in the downflow mode and 41% in the upflow mode. Water flood was more effective in the upflow mode than in the downflow mode. In the upflow mode, removal was about 33%, while in the downflow mode it was only 20%. This is opposite to what was observed in the case of CGA suspensions. This can be explained by the density difference between the flushing media and ATF in each case.

The difference in density between the water and the organic liquid (ATF) can aid the mobilization of ATF. Because ATF is lighter than water, the oil will be buoyed up and tend to rise vertically. However, the vertical migration of the oil due to density difference is resisted by the capillary forces which trapped the oil in the first place (Wilson *et al.*, 1990).

When the wash solution is pumped into the column in the downflow mode, the ATF will be pushed down along with the wash solution. Even though it is lighter than the wash solution, ATF cannot stay behind the moving front since a large pressure difference exists along the column length. In the upflow mode using water flood, however, the displacement of ATF by pressure difference will be aided by the density difference. The results of the water run show the possible role of density difference in the removal of ATF. In the case of CGA runs, the removal of ATF is higher in the downflow operation than that observed for upflow runs. The specific gravity of CGA suspension is in the range of 0.3–0.4 (Sebba, 1987). ATF is denser than a CGA suspension with a tendency to sink resulting in enhanced removal in downflow mode. For conventional surfactant solutions there was no difference in ATF removal in either modes of flow.

Pressure drop across the soil columns

Figure 6(a, b) shows the variation in pressure for contaminated and uncontaminated soil column experiments using CGA suspensions, conventional surfactant solutions, and conventional water floods in downflow and upflow modes, respectively. It is evident from these figures, that the pressure drop across the column is unstable at the beginning of the run and is stabilized by the end of the first pore volume.

The pressure remained fairly low (0–12 psig) for both contaminated and uncontaminated soil columns

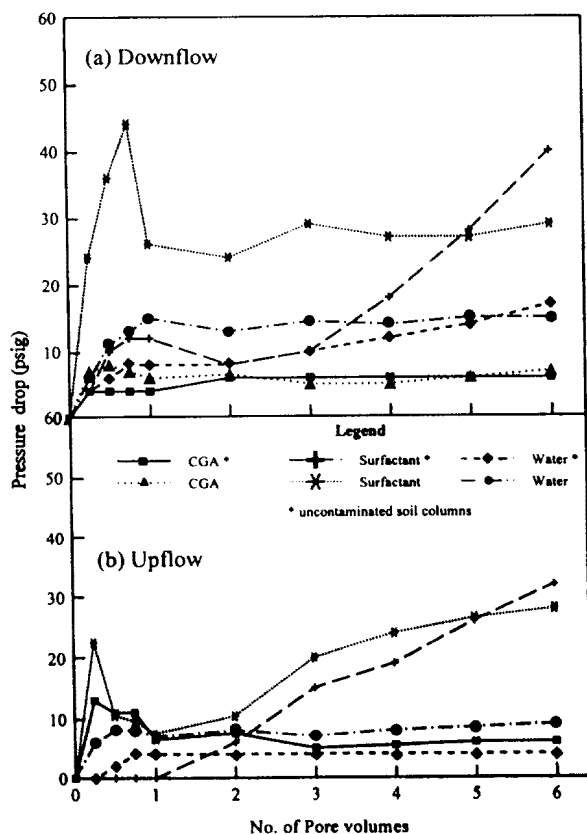


Fig. 6. Pressure drop across the column for uncontaminated soil columns and columns contaminated with ATF in (a) downflow and (b) upflow operation.

in downflow mode when CGA suspensions were pumped. The pressure trend in case of upflow operation is similar to that of downflow mode. The lower pressure in the case of CGA suspensions may be due to the formation of channels during the process as discussed in the earlier section. Earlier research showed 95% removal of 2,4-D from soil columns, suggesting that there is no extensive flow bypassing (Roy *et al.*, 1992). The lower pressure drop when compared to water and conventional surfactant solutions makes the CGA suspensions attractive for field applications.

The pressure drop across the soil columns in the case of conventional surfactant solutions is very high (25–50 psig) in comparison to CGA suspensions. Pore blocking and clay dispersion discussed earlier are thought to be responsible for the high pressure drop in the case of the conventional surfactant solutions. This behavior was also observed by Nash (1987) during the field testing of surfactant solution. It appears that the pore blocking phenomenon, which severely restricts the removal of contaminants, may be one of the limitations of the surfactant soil flushing process.

For water flood experiments, the pressure went up to about 15 psig by the second pore volume and remained constant for the rest of the experiment in downflow mode, whereas the pressure remained relatively low (0–6 psig) in upflow mode for both contaminated and uncontaminated soil columns.

The trend in pressure drop for both contaminated and uncontaminated soil columns is similar for all flushing media, thus indicating that the pressure drop phenomenon is primarily dependent on the possible interaction between the flushing fluid and the soil.

CONCLUSIONS

The following conclusions can be drawn from the results of the study on the application of CGA suspensions and conventional surfactant solutions for washing ATF from soil columns:

CGA suspension at any concentration is more effective at removing ATF than the conventional surfactant solution of the same concentration or conventional water floods.

CGA suspension generated from 8 mM SDS solution removed about 50% ATF from the soil as compared to 42% by the conventional surfactant solution of the same concentration and to 20% by a water flood in downflow operation.

In the upflow mode both CGA suspension and conventional surfactant solution recovered about 41% ATF from soil compared to 33% by water flood.

The displacement, solubilization, dispersion of ATF, and electric repulsion at the surface of the soil particles are believed to be responsible for enhanced ATF recovery in the case of CGA suspensions

and conventional surfactant solutions. However, displacement appears to be the only mechanism that removes ATF by a water flood.

Increasing the surfactant concentration from the CMC level to 30 mM does not increase the removal efficiency for either CGA suspensions or conventional surfactant solutions.

The ATF removal in the upflow mode is higher than that in the downflow mode for water floods and is about the same for conventional surfactant solutions. CGA suspensions are more effective in the downflow operation than in upflow mode. The density difference between ATF and the flushing fluid is believed to be responsible for this.

The pressure drop in the case of conventional aqueous surfactant solutions is always high (25–50 psig) in both upflow and downflow conditions. In the case of the CGA suspension, the pressure drop remained low (4–12 psig), while the pressure in the case of water flood remained intermediate (8–17 psig) between CGA suspensions and surfactant solutions in downflow. The pressure drop in upflow operation is lower than that in downflow operation for CGA suspensions and water floods.

Acknowledgements—This research was funded by the U.S. District Court, Middle District of Louisiana, through the Louisiana State University Hazardous Waste Research Center. The authors would like to express their appreciation to Mubeen Darji and Philip Lee for their help in the laboratory.

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