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Influence of Wettability on the Recovery of NAPLs from Alluvium

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The physicochemical characteristics of five nonaqueous phase liquids (NAPLs) recovered from contaminated alluvial aquifers are presented. The five include two chlorinated degreasing solvents, one chlorinated dry-cleaning solvent and two weathered fuel hydrocarbons. In addition to density, viscosity, and interfacial tensions, the equivalent alkane carbon number (EACN), spreading coefficients and Amott-Harvey and USBM wettability indices with respect to alluvial aquifer materials are used as a means to characterize three of these NAPLs. Experimentally measured spreading coefficients of four of these NAPLs illustrate that field NAPLs can have positive initial spreading coefficients. Furthermore, capillary desaturation curves for two NAPLs with alluvial aquifer material collected from the NAPL zone are presented as an additional and important means to infer the practical implications of the wetting characteristics on the efficacy of NAPL recovery. The results from the wettability and capillary desaturation experiments show that these NAPLs are mixed-wet to oil-wet when measured in the alluvium from their respective field sites. Furthermore, these results indicate that the displacement of NAPLs from soils by water is more difficult for mixed-wet or oil-wet soils than it is for water-wet or weakly water-wet soils. Finally experimental data indicate that adding anionic surfactants to the water shifts the wettability toward water-wet and makes the NAPL easier to displace and recover.

Introduction

The recovery of NAPLs from alluvium is an essential step in the prevention and control of groundwater contamination in the large alluvial aquifers that provide most of the nation's produced groundwater supply. NAPLs of particular importance include coal tar, creosote, and chlorinated degreasing solvents, which are dense nonaqueous phase liquids or DNAPLs, and jet fuel, diesel, and gasoline, which are light nonaqueous phase liquids or LNAPLs. NAPL distribution at the pore scale is controlled primarily by its wettability (*1*, *2*). Although water has a natural tendency to wet mineral surfaces, some NAPLs also have a tendency to wet mineral surfaces, so a competition between the two fluids arises when they are both present for a sufficiently long time. NAPL distribution in the subsurface is also strongly affected by its interfacial properties, especially the spreading coefficient (*3*). The migration of NAPL in alluvium in two-phase or saturated conditions depends on its capillary pressure and relative permeability, and both of these are strongly dependent upon the wettability as well as the interfacial tension between the NAPL and water (*1*, *4*). Under three-phase or vadose-zone conditions, the spreading coefficient is more likely to dominate in determining the subsurface NAPL distribution (*3*).

An understanding of the physics of NAPL migration and parameters that control NAPL distribution in both the vadose and water-saturated zones is essential in selecting appropriate remedial technologies. Such an understanding requires that the spreading coefficients as well as wettability be adequately characterized. With respect to wettability, a few subsurface environmental researchers (*5*, *6*) have assumed that all soils and rocks are water-wet. While some researchers, such as Feenstra et al. (*7*, p 58), have stated that "most DNAPLs of interest will be nonwetting on geologic solids with respect to water but wetting with respect to air," other environmental researchers have reported oil-wet conditions (*2*, *8*) or pH-dependent wettability (*9*). The misconception that NAPLs are water-wet can be attributed to the use of laboratory grade contaminants as surrogate NAPLs in typical experimental studies. These laboratory grade contaminants do not have the impurities and surface-active agents typically present in field NAPLs and generally have high interfacial tension (*10*). The work conducted by Tuck et al. (*10*) has shown that with increasing time DNAPLs can potentially become less water-wet due to their complex multicomponent nature and the potential for some DNAPL constituents to reduce interfacial tension and adsorb on mineral surfaces.

The above discussions and other research indicate that conditions other than water-wet frequently exist in the subsurface (*1*, *11*–*13*). The mixed-wet nature may be attributed to the presence of surface-active agents in NAPLs or high molecular weight oils that coat mineral surfaces that alter wettability (*14*). In strongly water-wet minerals, the NAPL will reside in the large pores, and the water will reside in the small pores and in the form of thin films (*1*). This behavior is reversed in oil-wet environments, where NAPL resides in small pores and in the form of thin films. Under these conditions the NAPL is usually poorly accessible and therefore not easily recoverable.

The impact of wettability on waterflooding of oil reservoirs has been studied for decades. It has been found that the efficiency of a waterflood in a mixed-wet or oil-wet rock is less than in a water-wet or weakly water-wet rock (*1*). Even though previous work by Powers and her colleagues (*2*) have demonstrated mixed-wet conditions and also demonstrated the use of bottle tests to estimate NAPL–alluvium wettability, the practical significance of wettability on remediation of NAPL, particularly chlorinated and fuel hydrocarbons, has not been fully investigated.

The contact angle between the fluid and the mineral surface is a universal measure of wettability (*1*). A surface is considered water wet if the contact angle measured through the water is less than 75 degrees; intermediate wet if 75 to 105 degrees and oil wet if 105 to 180 degrees. However, measuring the contact angle under in-situ conditions is not practicable, and the contact angle of a NAPL in the presence of a polished silica surface in a vacuum can hardly be representative of in-situ conditions or mixed wet conditions typical of many media with multiple minerals with differing

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affinities for the NAPL. Therefore, other techniques, such as Amott-Harvey and USBM wettability tests, provide a more practical estimate of wettability.

In this paper, we present evidence that the wettability varies significantly with the physicochemical properties of the NAPL. Results show that NAPLs with a combination of high equivalent alkane carbon number (*15*, *16*) values and high viscosities are more likely to be mixed-wet to oil-wet. This will typically be the case for creosote, coal tar, and fuel hydrocarbons (*2*). NAPLs having low viscosity and low EACN but with a high fraction of solubilized grease are likely to be mixed-wet (*14*). Furthermore, we present experimental data that indicate that interfacial tensions of field NAPLs are significantly lower than that of pure liquid hydrocarbons as reported by Demond and Lindner (*17*).

Interfacial tension measurements further indicate that two degreasing solvents and two fuel hydrocarbons studied in this paper have positive initial spreading coefficients. Finally, we demonstrate the practical effect of wettability on recovering NAPL via pumping or waterflooding by using capillary desaturation experiments as an additional measure for quantifying wettability. The capillary desaturation relationship is the relationship between a residual saturation trapped in a porous medium by capillary forces and the viscous and gravity forces acting on the fluid to displace it. As discussed below, this relationship can be expressed as the residual saturation as a function of a nondimensional trapping number.

We verify our hypotheses by presenting results from capillary desaturation experiments for two NAPLs and their associated aquifer materials. One of these NAPLs is a typical degreasing solvent, while the other is a burnt and disposed aviation fuel. We illustrate that under mixed- to oil-wet conditions much higher advective or viscous force is required to displace NAPL compared to water-wet conditions, which makes NAPL recovery technically impracticable unless the wettability of the geosystem is returned to water-wet conditions. Finally, we discuss the potential effects of wettability and positive spreading coefficients on designing remedial systems.

Experimental Section

Materials and Methods. All samples of NAPL were obtained by pumping them from wells installed in alluvium. One chlorinated degreasing solvent DNAPL was obtained from Operable Unit 2 (OU2) at Hill Air Force Base, UT, while the second degreasing DNAPL was obtained from Kelly AFB, San Antonio, TX. A dry cleaning solvent was obtained from the U.S. Marine Corp Base, Camp Lejeune, NC. The weathered jet fuel LNAPL was obtained from a fire-training area (OU1) at Hill Air Force Base, UT. The weathered gasoline LNAPL was obtained from a former refinery site in Ohio. The NAPLs were recovered from alluvium containing >90% alumino-silicate minerals at Hill and a mixture of alumino-silicate and 25% carbonate minerals in the Ohio aquifer. The soil columns used for the capillary desaturation experiments as well as the surfactant flood experiments were either 30.5 cm long and 2.21 cm diameter stainless steel columns or 4.8 cm diameter and 30 cm long jacketed glass columns.

The experimental procedures to pack and saturate these soil columns are described by Dwarakanath et al. (*18*). The capillary desaturation experiments were conducted with the soil column oriented in a vertical position. LNAPL was injected into the column from the top downward until no water production was observed. In the case of DNAPL, it was injected from the bottom upward to ensure gravity-stable displacement. In both instances the flow direction was reversed for waterflooding. The inlet pressure was increased to achieve a higher flow rate, and the additional volume of NAPL produced was collected and measured to quantify a change in the saturation.

The pressures across the soil columns were measured using Cole Parmer industrial transducers capable of measuring differential pressures between 0 and 35 kPa (0−5 psid) and 0−350 kPa (0−50 psid). Viscosities were measured using a Contraves Low-Shear 30, which is a Couette-type viscometer and has a precision of ±0.1 cp. The interfacial tensions were measured using a DuNouy ring tensiometer, which has an experimental precision of ±0.1 mN/m. The density measurements are precise to within ±0.002 g/mL. The experimental procedures described in Dwarakanath and Pope (*19*) were used to estimate the NAPL's equivalent alkane carbon number. The Amott (*20*) and USBM (*21*) wettability experiments for the OU1 LNAPL and OU2 DNAPL were conducted by Terratek Inc., Salt Lake City, UT. The Amott-Harvey wettability index of the weathered gasoline LNAPL was measured at the Center for Petroleum and Geosystems Engineering at the University of Texas at Austin.

Theory

The mobilization and recovery of oil or NAPL either by pumping or by waterflooding can be predicted using capillary desaturation curves. The mechanisms of capillary trapping and mobilization are well discussed in the literature (*22*). Under two-phase, water-saturated conditions, NAPL is mobile at higher than residual saturations and can be recovered by pumping. However, if pumping continues and NAPL saturation is decreased, some NAPL is trapped in the form of immobile ganglia due to snap-off mechanisms (*4*, *22*). Hence residual NAPL saturation is defined as the fraction of the pore space occupied by NAPL that is trapped in the form of immobile ganglia by capillary forces and therefore immobile. Mobilizing residual NAPL from these immobile ganglia requires that the capillary forces trapping the NAPL be exceeded by externally imposed forces. The normalized residual saturation is defined as the NAPL saturation divided by the maximum residual NAPL saturation. At residual NAPL saturation, the normalized residual NAPL saturation is one and decreases when NAPL is mobilized as in capillary desaturation experiments.

The advective force due to the viscosity of the flowing groundwater and the gravity force due to NAPL density are the displacing forces on NAPL ganglia. Mobilization will occur when the vector sum of these forces exceeds the capillary forces that trap NAPL (*23*). The ratio of viscous to capillary force on the NAPL is termed the capillary number, and the ratio of gravity to capillary force on the NAPL is termed the Bond number. An increase in advective forces or hydraulic gradient will result in an increase in the capillary number, which may be induced by increasing the viscosity of the water at a given pump rate or by increasing the pumping rate. Similarly, an increase in the difference in density between the NAPL and water will increase the gravity force or the Bond number. Alternatively both the capillary and Bond number can be increased by the reduction in the NAPL−water interfacial tensions induced by the application of surfactants.

Therefore, capillary desaturation experiments can be conducted by increasing the capillary number by increasing the hydraulic gradient across the column. Alternatively, a surfactant that lowers the interfacial tension can be injected to increase both the capillary and Bond numbers. In either case, if the vector sum of the capillary and Bond numbers exceeds the capillary forces trapping the NAPL, it will be mobilized in the direction of the effective force, described by a dimensionless number called the trapping number. The minimum trapping number at which residual NAPL begins to flow (decreasing the residual saturation) is defined as the

TABLE 1. Physicochemical Properties of Several NAPLs[a]

NAPL	solvent type	density, g/mL	viscosity, mPa-s	γ_{ow}, mN/M	γ_{oa}, mN/M	S, mN/m	EACN
weathered gasoline, sample 1	fuel hydrocarbon	0.78	1.2	18.3	26.2	27.5	7.15
weathered gasoline, sample 2	fuel hydrocarbon	0.88	6.9	12.4	27.7	31.9	6.12
Camp Lejeune DNAPL	dry cleaning solvent	1.59	1.1	10.4	nm		2.9
Kelly AFB DNAPL	degreasing solvent	1.60	0.9	14.6	22.4	35.0	nm
Hill OU2 DNAPL	degreasing solvent	1.37	0.8	8.6	19.8	43.6	−5.41
Hill OU1 LNAPL	weathered fuel hydrocarbon	0.88	36	8.0	28.0	36.0	6.01

[a] Density, viscosity, and interfacial tensions measured at 23°C.

TABLE 2. Wettability of NAPLs with Alluvium[a]

NAPL	soil	Amott wettability index	Amott-Harvey wettability index	USBM wettability index
weathered gasoline, sample 1	Ottawa sand	nm	0.68	nm
weathered gasoline, sample 1	alluvium from refinery site	nm	0.07	nm
Hill OU2 DNAPL	OU2 alluvium	0	nm	−0.138
Hill OU1 LNAPL	OU2 alluvium	0	nm	−0.195

[a] Note: air−water interfacial tension = 72 mN/m; nm = not measured.

critical trapping number (*23*). The capillary, Bond, and trapping numbers were calculated using the following equations:

$$N_{Ca} = \frac{k}{\sigma}\frac{\Delta\Phi}{L} \quad (1)$$

$$N_B = \frac{kg\Delta\rho}{\sigma} \quad (2)$$

$$N_T = \sqrt{N_{Ca}^2 + N_B^2 - 2N_{Ca}N_B \mathrm{Sin}\alpha} \quad (3)$$

The driving potential $\Delta\Phi$ can be determined by measuring the pressure across the inlets and outlets of the soil column using the following equations:

Downward flow:

$$\Delta\Phi_j = \Delta p_T + (\rho_j - \rho_x)gh \quad (4)$$

where ρ_j and ρ_x are densities of the flowing phase and of the liquid in the pressure lines connecting the column to the transducer, respectively, and Δp_T is the pressure drop across the column as measured by the pressure transducers.

Upward flow:

$$\Delta\Phi_j = \Delta p_T - (\rho_j - \rho_x)gh \quad (5)$$

In water-wet sediments, the NAPL is trapped in large pores and is accessible to displacement by water. However in oil-wet pores, the NAPL resides in smaller pores and cannot be easily displaced. The critical trapping number in water-wet sediments is on the order of 10^{-5}–10^{-4} (*23*). However, in oil-wet sediments a higher trapping number will be required to displace NAPL. Therefore, capillary desaturation experiments provide a measure of the practical implication of wettability. A high critical trapping number indicates a higher tendency toward oil-wet conditions and vice versa.

The spreading pressure or spreading coefficient (*S*) is a measure of a NAPL's tendency to move across an air−water interface and is defined by the following equation (*3, 24*).

$$S = \gamma_{aw} - \gamma_{ow} - \gamma_{oa} \quad (6)$$

A positive spreading coefficient indicates that the NAPL will spread in the form of a thin film under three-phase conditions. The significance of a positive spreading coefficient condition is that NAPL can both vertically and laterally spread across a site (*25*).

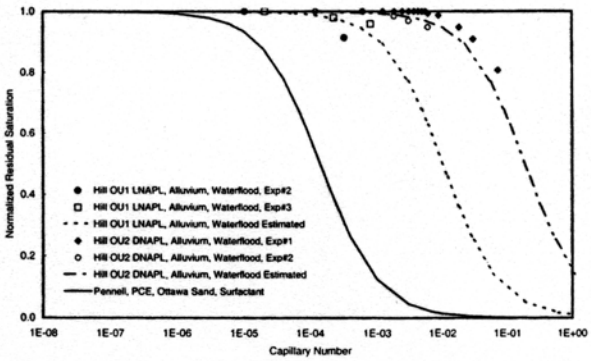

FIGURE 1. Comparison of capillary desaturation curves with various NAPLs and alluvium.

Experimental Results

The density, viscosity, interfacial properties, spreading coefficient, and EACN of five NAPLs are summarized in Table 1. The Hill OU1 LNAPL, the weathered gasoline-rich LNAPL from Ohio, the Hill OU2 TCE-rich DNAPL, and the Kelly AFB DNAPL have positive spreading coefficients. The wettability of the NAPLs with respect to their respective aquifer material is summarized in Table 2. In general, the field NAPLs in the alluvial material from the field are mixed to oil-wet. The wettability of the weathered gasoline in Ottawa sand is presented as an illustration of water-wet behavior. Figure 1 presents results from various capillary desaturation experiments. Capillary desaturation data using laboratory grade tetrachloroethene (PCE) in Ottawa sand (*23*) measured from surfactant flood experiments are plotted for comparison with waterflood capillary desaturation data for Hill OU2 DNAPL in OU2 alluvium and OU1 LNAPL in OU1 alluvium. Figure 2 illustrates the effect of surfactant on the capillary desaturation characteristics of the Hill OU2 DNAPL. The corresponding critical trapping numbers from experiments with PCE in Ottawa sand, Hill OU2 DNAPL in OU2 alluvium, and Hill OU1 LNAPL in OU1 alluvium are summarized in Table 3.

The critical trapping number of Hill OU2 DNAPL in OU2 alluvium in the absence of surfactant is on the order of 10^{-2}–

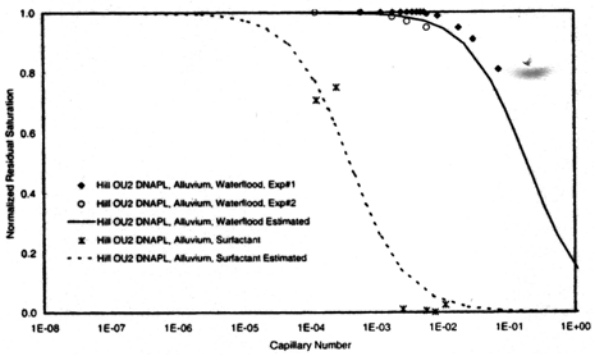

FIGURE 2. Effect of surfactant on capillary desaturation characteristics of the OU2 DNAPL and OU2 alluvium.

TABLE 3. Critical Trapping Numbers of Various NAPLs with Alluvium

NAPL	alluvium	critical trapping no.
PCE (*23*)	Ottawa	10^{-5}–10^{-4}
OU2 DNAPL	OU2 alluvium	10^{-2}–10^{-1}
OU2 DNAPL, surfactant flood	OU2 alluvium	10^{-5}–10^{-4}
OU1 LNAPL	OU1 alluvium	10^{-3}–10^{-2}

10^{-1} and is much higher compared to the critical trapping number of pure PCE in the water-wet Ottawa sand. Furthermore, the critical trapping number for this DNAPL and its alluvium measured by surfactant flood experiments is on the order of 10^{-5}–10^{-4} and indicates water-wet behavior. These results indicate that the Hill alluvium is water-wet with respect to the Hill DNAPL in the presence of surfactant.

Discussion

The above results have profound implications for NAPL characterization and removal from the subsurface. The interfacial tensions of field NAPLs are between 8 and 18 mN/m, which is significantly lower than that of the corresponding laboratory grade hydrocarbons and halogenated hydrocarbons. As an illustration, the interfacial tension of pure PCE with deionized water is 47.48 mN/m (*17*) compared to the measured value of 10.4 mN/m for the PCE-rich Camp Lejeune DNAPL and 14.6 mN/m for the PCE-rich Kelly AFB DNAPL. This suggests that the DNAPL contains dissolved surface-active agents, which bring about a lowering in the interfacial tension. Such agents can also potentially alter wettability (*10*).

The initial spreading coefficient for the two LNAPL samples and two degreasing solvents is positive, which is in contrast to Charbeneau (*24*, p 473), who states that halogenated organic solvents are likely to have negative spreading coefficients. However, the halogenated solvents being referred to by Charbeneau are probably laboratory grade halogenated hydrocarbons and are therefore likely to have high NAPL–water interfacial tensions and consequently negative spreading coefficients.

The positive initial spreading coefficients for degreasing DNAPLs are consistent with results reported by Tuck (*10*). Since the degreasing DNAPLs are likely to contain a mixture of surface active agents or components that alter wettability, they are likely to have much lower interfacial tensions and therefore positive spreading coefficients. The implications of positive spreading coefficients are that NAPL will not exist in the form of discrete ganglia under three-phase conditions but rather will spread across the air–water interface and move distances much larger than would be predicted by Darcy's law with relative permeabilities and residual saturations typical of nonspreading systems (*24*).

With regard to wettability, the weathered gasoline from Ohio and the Hill OU1 LNAPL have high EACNs and therefore can be expected to contain hydrocarbon components that alter wettability through sorption (*2*). In contrast to the fuel-hydrocarbon LNAPLs, the experimentally measured EACN of the Hill OU2 DNAPL is −5.41, which is similar to that of pure TCE (*19*). The viscosity of the OU2 DNAPL is 0.8 cp, which is also similar to pure TCE. Such results might suggest to an unwary reader that the Hill OU2 DNAPL is water-wet in the alluvium at Hill OU2. However the USBM and Amott indices indicate that the Hill OU2 DNAPL is mixed-wet. It is therefore hypothesized that the surface-active agents in the DNAPL that reduced its interfacial tension also altered its wettability. Under such conditions, this DNAPL will be more difficult to displace and recover by pumping or waterflooding.

The capillary desaturation experiments indicate that the critical trapping number for the Hill OU2 DNAPL when no surfactant is added is high, between 10^{-2} and 10^{-1}. Such a high critical trapping number means that the DNAPL cannot be easily displaced by water even though a high advective force or hydraulic gradient is imposed. This suggests that the DNAPL resides in poorly accessible or small pores and cannot be easily displaced by waterflooding alone, thereby confirming our hypothesis that NAPL will be more difficult to recover from mixed to oil-wet material. The addition of an anionic surfactant however reduces the critical trapping number strongly as shown in Figure 2. The anionic surfactant has the potential to make the system preferentially water-wet (*26*) and therefore moves the NAPL to the more accessible pores where it can be readily recovered. From these observations it is evident, as Donaldson (*21*) noted over 30 years ago, wettability is as important as viscosity and permeability in NAPL recovery.

A high critical trapping number and a negative USBM index are observed for the Hill OU1 LNAPL. Both these results indicate that this LNAPL is mixed-wet to oil-wet. The average Amott-Harvey index of the weathered gasoline from Ohio is 0.07, which indicates mixed- to water-wet conditions. However, the Amott-Harvey index of the same weathered gasoline with water-wet Ottawa sand is 0.68, i.e., strongly water-wet behavior. These results indicate that it is necessary to use the field NAPL and soil to determine the in-situ wettability and critical trapping number. One should not assume water-wet conditions. Also, one should not use pure hydrocarbon or solvent nor clean silica sand in laboratory experiments done for the purpose of designing remedial operations.

Another implication of the wettability results discussed in this paper is that more attention should be given to the impact of wettablity in flow and transport models. Capillary pressure, relative permeability, and capillary desaturation relationships all need to be carefully modeled as a function of NAPL wettability, and the appropriate relationships based upon laboratory data using field materials should be used in numerical modeling.

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Nomenclature

α	angle of dip
$\Delta\Phi$	driving potential to induce flow across the soil column (Pa)
k	intrinsic permeability (μm^2)
L	length of column (m)
N_B	bond number
N_{Ca}	capillary number
N_T	trapping number
Δp_T	pressure drop across the column as measured by the pressure transducers (Pa)
S	spreading coefficient (mN/m)
γ_{aw}	air–water interfacial tension (mN/m)
γ_{ow}	NAPL–water interfacial tension (mN/m)
γ_{oa}	NAPL–air interfacial tension (mN/m)
σ	interfacial tension (mN/m)
$\Delta\rho$	difference in density between NAPL and water (Kg/m^3)
ρ_j	density of the flowing phase (Kg/m^3)
ρ_x	fluid density in the pressure lines connecting the column to the transducer (Kg/m^3)

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