



Effects of pure and dyed PCE on physical and interfacial properties of remedial solutions[☆]

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Abstract

Hydrophobic dyes have been used to visually distinguish dense non-aqueous phase liquid (DNAPL) contaminants from background aqueous phases and soils. The objective of this study was to evaluate the effects of a dyed DNAPL, 0.5 g Oil-Red-O/1 of PCE, on the physical properties of remedial solutions: water, co-solvents (50, 70, and 90% (v/v) ethanol), and surfactants (4% (w) sodium dihexyl sulfosuccinate). This study compared the densities, viscosities, and interfacial tensions (IFTs) of the remedial solutions in contact with both dyed and undyed PCE. The presence of the dye in PCE substantially alters the IFTs of water and ethanol solutions, while there is no apparent difference in IFTs of surfactant solutions. The remedial solutions saturated with PCE showed higher viscosities and densities than pure remedial solutions. Solutions with high ethanol content exhibited the largest increases in liquid density. Because physical properties affect the flow of the remedial solutions in porous media, experiments using dyed DNAPLs should assess the influence of dyes on fluid and interfacial properties prior to remediation process analysis.

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1. Introduction

Release of non-aqueous phase liquids (NAPLs) to the subsurface may result in soil and groundwater contamination. Dense NAPLs (DNAPLs) have been problematic as recalcitrant contaminants. Therefore, much effort is being expended in developing remediation techniques that remove DNAPLs trapped in porous media. For these studies, the most widely used target compounds are trichloroethylene (TCE) and tetrachloroethylene (PCE). Since TCE and PCE are colorless liquids, many researchers have used hydrophobic dyes to visually distinguish DNAPL contaminants from background aqueous phases [1–6]. Many of the DNAPL studies assume that hydrophobic dyes have negligible effect on the interfacial tension (IFT) between the DNAPL and aqueous phase. The physical properties of pure remedial solutions are sometimes used for remediation system analysis and modeling, but these properties may change as DNAPL dissolves into the solutions.

The objective of this study was to evaluate the effects of dissolved DNAPL on the physical properties of remedial solutions: water, co-solvents (50, 70, and 90% (v/v) ethanol), and surfactants (4% (w) sodium dihexyl sulfosuccinate). Physical and interfacial properties (density, viscosity, and IFT) of the remedial solutions saturated with dyed (0.5 g Oil-Red-O/l of PCE) and clean PCE were measured and compared with physical properties of pure remedial solutions. The physical properties of remedial solutions will affect the transport of these solutions in porous media, and should be evaluated when designing and modeling remediation activities.

This study was designed to mimic fluid and interfacial conditions used during lab-scale investigations of DNAPL dissolution and mobilization. During these investigations the porous medium containing PCE was flooded with remedial solutions that either had been previously equilibrated with PCE or had not been equilibrated. The residual PCE had not been exposed to the remedial solutions prior to flooding. Thus, for this study, IFTs were measured using PCE which had not been equilibrated with remedial fluid.

2. Materials and methods

2.1. Chemicals

PCE and Oil-Red-O were chosen as the DNAPL and hydrophobic dye for this study, respectively. PCE (>99.9% pure, Sigma) was dyed with Oil-Red-O (Fisher Scientific Inc.) at a concentration of 500 mg/l. The dye has been widely used in several DNAPL studies. Three types of remedial fluids were evaluated: (1) water; (2) co-solvent solution (ethanol/water); and (3) a surfactant (sodium dihexyl sulfosuccinate, aerosol MA-80I (AMA-80), Cytec Inc.). Three volume-basis co-solvent solutions, 50% ethanol + 50% water, 70% ethanol + 30% water and 90% ethanol + 10% water, were prepared by mixing ethanol and water. Surfactant solutions contained 4% weight basis AMA-80 and 0, 500, or 5000 mg/l of calcium chloride (CaCl_2). Water was obtained from a Millipore purification system.

2.2. Preparation of PCE saturated solutions

Dyed and clean PCE were placed in separate 40 ml vials. About 30 ml of selected remedial solutions were placed in the vials. The vials were then placed on a shaker for 10 days. PCE was continuously added to the vials during the period as necessary to maintain a separate PCE phase. The vials were placed on a table for 10 days to allow complete separation of the liquid phases. Forced separation methods were not used. No milky phase was observed after separation. The supernatant was carefully taken by an eppendorff pipetter and used for measurements. Three replicate vials for each equilibrated condition were prepared. Properties of equilibrated PCE were not measured in this study.

2.3. Measurements

Densities of pure liquids were determined by using a pycnometer (25 ml gravity pycnometer, Kimble Glass Inc.). Because the volumes of remedial solutions equilibrated with PCE were insufficient for all measurements, densities of these solutions and pure liquids were also determined by placing a known volume of the liquid (by using a eppendorff pipetter) on an analytical balance (AG 285, Mettler Toledo Inc.) and weighing it to an accuracy of 10^{-4} g. For the pure liquids, differences in densities between the two methods were less than 1.0% of the values obtained using the pycnometer. All measured densities are shown in Table 1. Measurements were made at room temperature (22 ± 0.5 °C).

Viscosity was measured by a Cannon[®]-Ubbelohde viscometer and determined according to the procedures of ASTM D 445. Surface tension (liquid–air) was measured by a tensionmeter (Surface tensionmeter model 20, Fisher Scientific Inc.) using the Du Nouy ring method [7].

IFTs (liquid–liquid) between the PCE and aqueous solutions were measured by the drop volume method [8]. The drop volume measuring system for IFT consisted of a capillary tube assembly (parts of DVT-10, Kruss Inc.), a syringe pump (100DM, ISCO Inc.), microscope (SV 11, Carl Zeiss Microimaging Inc.) and video camera (ZVS-47E, Carl Zeiss Microimaging Inc.). PCE was injected at a rate of 1 ml/h, based on the results of Hool and Schuchardt [8]. The number of PCE drops detached from the capillary tip were counted and the elapsed time measured. The IFT (γ) is calculated by the following equation [8]:

$$\gamma = \frac{V_d \Delta \rho g}{\pi d_c} \quad (1)$$

where V_d is the volume of the drop, $\Delta \rho$ is the density difference between the dense and the light phase, g is the gravity constant, and d_c is the capillary diameter (254 μm). In order to evaluate the accuracy and reproducibility of IFT values obtained with the drop volume method, the measured IFT between PCE (clean) and water (saturated with PCE) was compared with those reported in the literature (Table 1). The measured value of 47.02 ± 0.10 dyn/cm was within 2.5% of the average value, 45.83 dyn/cm, of the reported IFTs.

Because the drop volume method is not reliable for low IFT (<0.1 dyn/cm) [8], a sessile drop method was also employed for low range IFT measurements [9]. The method was used to measure IFTs between 4% AMA-80 + 5000 mg/l CaCl_2 and PCE. Accuracy of the sessile drop method was evaluated by comparing IFTs measured using this method with

Table 1
Reported IFTs of DNAPL with water

Reference	IFTs (dyn/cm)	DNAPL	Dyed	Equilibrated	Measurement methods	Solubility (mg/l)
[15]	51.0	PCE	No	No	Drop volume	NA
[1]	47.8	PCE	NA Oil-Red-O (10 ⁻⁴ M) ^a	Yes	Spinning drop	200 ^b
[17]	47–48	PCE	NA Oil-Red-O (0.04 g/l) ^a	NA	Pendant drop	NA
[18]	47.48	PCE	No	NA	NA	1.62 × 10 ⁻³ (mol%) ^b
[19]	47	PCE	No	NA	Spinning drop	NA
[20]	47.0 ± 2.9	PCE	No	NA	Du Nouy ring	150
	40.0 ± 1.1	PCE	Oil-Red-O (1.0 g/l)			
[21]	45.7	PCE	No	Yes	Drop volume	NA
[22]	45.0	PCE	NA [23]	Yes	Spinning drop [23]	240
[24]	44.5	PCE	NA Oil-Red-O (1.6 mg/l) ^a	Yes	Du Nouy ring	225
[25]	43.71 ± 0.0375	PCE	No	Yes	Drop volume	
[26]	43.0	PCE	No	Yes	Pendant drop	165
[15]	43.2	PCE	Sudan IV (0.5 g/l)	No	Drop volume	NA
[2]	38	PCE	Sudan IV (0.1 g/l)	NA	Du Nouy ring	150–200 ^b
[4]	34.5	PCE	No	NA	Spinning drop	221 ^b
[27]	24	TCE	Oil-Red-O (0.5 g/l)	NA	Du Nouy ring	1270
[28]	27.3	TCE	Oil-Red-O (0.5 g/l)	No	Pendant drop	NA
[29]	34.5	TCE	NA Oil-Red-O (0.05 g/l) ^a	NA	NA	1100

NA: not available (not shown in the reference).

^a Dyed PCE used for experiments (but, NA for IFT measurements).

^b Cited from references.

IFTs determined using the drop volume method over the reliable range for both methods. The IFTs of 4% AMA-80 solution and dyed PCE measured by the drop volume and sessile drop methods were 1.92 ± 0.03 and 1.98 ± 0.04 dyn/cm, respectively.

Solubilities of PCE in the remedial solutions were determined by gas chromatography (HP 6890 Plus). Supernatant from equilibrated mixtures were diluted and injected into the GC by a headspace auto-sampler (Tekmar 7000).

3. Results and discussion

3.1. Remedial solution properties at PCE-saturated and unsaturated conditions

Table 2 shows the physical and interfacial properties of the pure liquids. Table 3 shows the properties of remedial solutions saturated with clean PCE, while Table 4 shows the properties of solutions saturated with dyed PCE. Comparing data in Table 2 to those in Tables 3 and 4 reveals the influence of PCE saturation on properties of remedial fluids.

Table 2
Solution properties (mean \pm S.D.)

Solution	Concentration	Density ^a (g/cm ³)	$\pm 95\%$ CL	Viscosity ^a (cP)	$\pm 95\%$ CL	Surface tension ^b (dyn/cm)	IFT with dyed PCE ^{a, c, d} (dyn/cm), not saturated	$\pm 95\%$ CL
PCE		1.622 \pm 0.007 ^e	0.009	0.84 ^f		31.3 \pm 0.2 ^e		0.249
		1.627 \pm 0.008 ^g	0.010			29.0 \pm 0.1 ^g		0.124
Water		1.001 \pm 0.004	0.004	0.91 \pm 0.002	0.002		48.85 \pm 0.76	0.945
Ethanol	50% (v/v)	0.935 \pm 0.001	0.001	2.38 \pm 0.01	0.012		7.32 \pm 0.05	0.062
	70% (v/v)	0.895 \pm 0.007	0.007	2.33 \pm 0.01	0.012		3.14 \pm 0.10	0.124
	90% (v/v)	0.853 \pm 0.007	0.007	1.86 \pm 0.01	0.012		1.09 \pm 0.04	0.050
AMA-80	4% (w)	1.010 \pm 0.002	0.002	1.16 \pm 0.01	0.012		1.98 \pm 0.03	0.037
	4% (w) +500 mg/l CaCl ₂	1.011 \pm 0.002	0.002	1.15 \pm 0.01	0.012		1.28 \pm 0.04	0.050
	4% (w) +5000 mg/l CaCl ₂	1.015 \pm 0.001	0.001	1.17 \pm 0.01	0.012		0.11 \pm 0.02 ^h	0.025

^a Room temperature (22 °C).

^b Du Nouy ring method.

^c Not saturated with PCE.

^d Drop volume method.

^e Clean PCE.

^f [30].

^g Dyed PCE (0.5 g Oil-Red-O/l PCE).

^h Sessile drop method.

Table 3
Solution properties measured after saturated with pure PCE (mean \pm S.D.): after complete separation of the liquid phases, supernatant was taken and measured

Solution	Concentration	Density (g/cm ³)	$\pm 95\%$ CL	Viscosity (cP)	$\pm 95\%$ CL	IFT with pure PCE (dyn/cm)	$\pm 95\%$ CL	PCE solubility (mg/l)
Water		1.007 \pm 0.002	0.005	0.97 \pm 0.01	0.025	47.02 \pm 0.10	0.248	119
Ethanol	50% (v/v)	0.939 \pm 0.001	0.002	2.64 \pm 0.03	0.075	7.10 \pm 0.13	0.323	6460
	70% (v/v)	0.922 \pm 0.001	0.002	2.39 \pm 0.05	0.124	3.17 \pm 0.04	0.099	47500
	90% (v/v)	0.981 \pm 0.008	0.020	2.05 \pm 0.08	0.199	0.97 \pm 0.02	0.050	283000
AMA-80	4% (w)	1.014		1.19 \pm 0.01	0.025	1.96 \pm 0.04	0.099	
	4% (w) +500 mg/l CaCl ₂	1.020		1.19 \pm 0.02	0.050	1.34 \pm 0.02	0.050	18400
	4% (w) +5000 mg/l CaCl ₂	1.021		1.34 \pm 0.04	0.099	0.11 \pm 0.01	0.025	69400

Table 4

Solution properties after saturated with red-dyed PCE (mean \pm S.D.): after complete separation of the liquid phases, supernatant was taken and measured

Solution	Concentration	Density (g/cm ³)	$\pm 95\%$ CL <i>t</i> -test ^a	Viscosity (cP)	$\pm 95\%$ CL <i>t</i> -test ^a	IFT with dyed PCE (dyn/cm)	$\pm 95\%$ CL <i>t</i> -test ^a	PCE solubility (mg/l)
Water		1.006 \pm 0.001	0.002 1.73 < <i>t</i> ₉₅	0.97 \pm 0.01	0.025 0 < <i>t</i> ₉₅	39.10 \pm 0.40	0.994 34.3 > <i>t</i> ₉₅	150
Ethanol	50% (v/v)	0.944 \pm 0.001	0.002 8.66 > <i>t</i> ₉₅	2.62 \pm 0.02	0.050 1.73 < <i>t</i> ₉₅	5.86 \pm 0.06	0.149 35.8 > <i>t</i> ₉₅	4970
	70% (v/v)	0.915 \pm 0.004	0.010 3.03 < <i>t</i> ₉₅	2.47 \pm 0.09	0.224 1.54 < <i>t</i> ₉₅	2.71 \pm 0.06	0.149 13.28 > <i>t</i> ₉₅	49100
	90% (v/v)	0.987 \pm 0.005	0.012 2.08 < <i>t</i> ₉₅	2.16 \pm 0.03	0.075 6.35 > <i>t</i> ₉₅	0.77 \pm 0.03	0.075 11.55 > <i>t</i> ₉₅	214000
AMA-80	4% (w)	1.016				1.92 \pm 0.03	0.075 2.30 < <i>t</i> ₉₅	
	4% (w)+500 mg/l CaCl ₂	1.018		1.20 \pm 0.02	0.050 0.87 < <i>t</i> ₉₅	1.32 \pm 0.02	0.050 1.70 < <i>t</i> ₉₅	37700
	4% (w)+5000 mg/l CaCl ₂	1.027		1.32 \pm 0.04	0.099 0.87 < <i>t</i> ₉₅	0.13 \pm 0.01	0.025 1.73 < <i>t</i> ₉₅	74300

^a *t*-test between results of Tables 2 and 3; The null hypothesis: there is no difference in the means; Reject the null hypothesis if $t > t_{95}$ (t value at $\alpha = 0.05$); The false rejection error rate was set at 5%.

Comparison of the results of Table 3 to those of Table 4 shows the effects of the dye on these properties. Relationships between remedial fluid properties and ethanol and CaCl₂ content for co-solvent and surfactant solutions are shown in Fig. 1.

Generally, remedial solutions saturated with PCE showed higher viscosities and densities than pure remedial solutions. The solution with the highest ethanol content (90%) exhibits the greatest increases in liquid density. Note that the PCE solubility of the 90% ethanol solution was highest of the remedial solutions evaluated and this remedial solution had the lowest initial density (see Tables 3 and 4). The increases in the densities of surfactant solutions saturated with PCE were insignificant.

The viscosities of all ethanol solutions were increased after saturation with PCE, while only the surfactant solution with high salt content (5 g/l) showed a significant increase in viscosity as a result of PCE saturation. The ionized surfactant and electrolyte compounds exhibit electroviscous effects (“increase in viscosity due to presence of electric charge on the surface of droplets” [10]) and reduce the IFT between PCE an aqueous phase, resulting in a high PCE concentration in the remedial fluid. The viscosity of the emulsions is proportional to the dispersed-phase concentration [10]. There were no significant differences in IFTs of surfactant solutions with and without PCE. However, PCE saturation significantly decreased IFT for water and co-solvent solutions (see Tables 2–4).

These results imply that properties of remedial fluids could change while flowing through DNAPL contaminated zones. The magnitude of these changes will likely be a function of the concentration of contaminant in the remedial fluid with the maximum impact expected when the remedial fluid is saturated with DNAPL. Altered properties of the remedial solutions affect their transport through porous media [11]. Hydraulic conductivity of a fluid in porous media is inversely related to viscosity. For surfactant solubilization, increased density of the microemulsion phase could promote vertical migration of a remedial fluid [12]. However, the surfactant solutions studied here would not be expected to exhibit vertical migration because no significant differences in densities were observed. The surfactant solution with the highest salt concentration (4% surfactant with 5 g/l of CaCl₂) may reduce hydraulic conductivity of a porous medium as DNAPL dissolves into the surfactant solution and the viscosity is increased.

Because of buoyancy effects, displacing ethanol solutions tend to override resident water during horizontal flooding [13]. This override results in poor mixing between DNAPL and the remedial solution. Densities of co-solvent flooding solutions will increase as DNAPL is dissolved, resulting in diminished override.

3.2. Dye effects on properties of remedial solutions saturated with PCE

A statistical test, *t*-test, was employed to compare the means of the dyed results with those of undyed results and the results of the *t*-test are shown in Table 4. The null hypothesis assumed no dye effect (i.e. There is no statistically significant difference in the two means). The false rejection error rate was set at 5% and the null hypothesis was rejected if a *t* value is greater than a critical statistic value, *t*₉₅ [14]. Because the *t*-test values for the IFTs of water and ethanol solutions are much greater than their *t*₉₅ values, the differences of the means are statistically significant and it would appear that the dye affects the IFT between the aqueous phases and PCE. Based on the *t*-test results, the dye significantly affects the IFTs

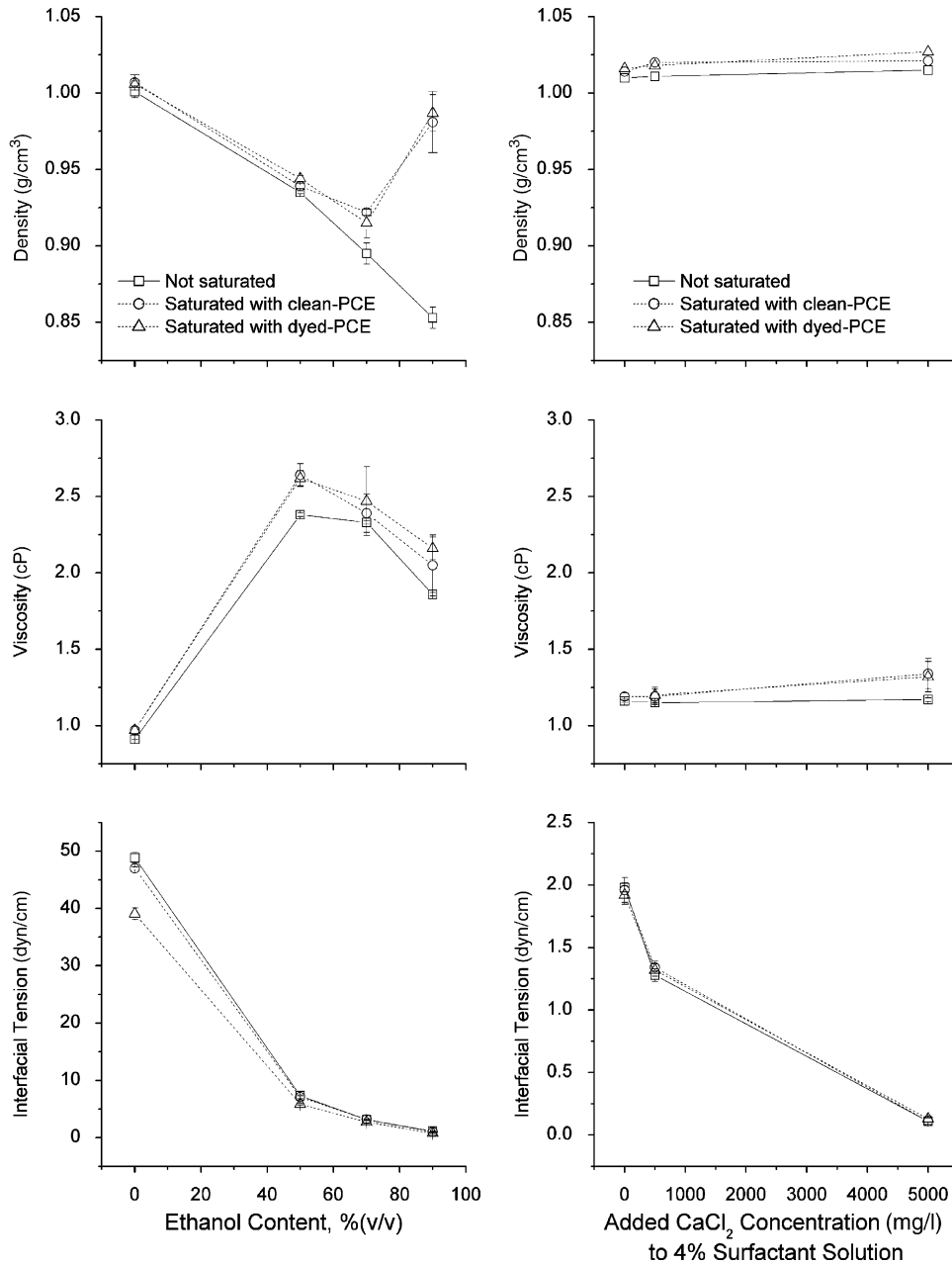


Fig. 1. Solution properties of remedial solutions (water, ethanol solutions and surfactant solutions) at three conditions: pure solutions, saturated with clean PCE, and saturated with dyed PCE. An ethanol content is volume basis. Surfactant solutions were prepared by adding CaCl₂ to 4% (w) aerosol AMA-80 solutions. The 95% confidential intervals are shown with error bars.

of water and ethanol solutions, while there is no apparent difference in IFTs of surfactant solutions.

The presence of the Oil-Red-O dye at a concentration of 500 mg/l had a substantial impact on IFTs between water and PCE. Also, as shown in Table 2, use of dye reduced the surface tension of PCE from 31.3 to 29.0 dyn/cm. Based on the Oil-Red-O dye effects shown in this study and the Sudan IV dye effects observed by Tuck [15] and Tuck et al. [16], dyes can measurably alter the IFTs between PCE and water. Further study is needed to examine the effects of Oil-Red-O concentration on IFT of water and PCE.

Dyed PCE may more readily invade pore spaces in a porous medium than pure PCE because the entry capillary pressure (P_c) decreases in proportion to surface and IFTs (σ); for cylindrical and spherical capillaries, the capillary pressure is described as Eq. (2):

$$P_c = \frac{2\sigma \cos \theta}{r} \quad (2)$$

where r is the radius of a cylindrical pore and θ is the contact angle that two fluids create. Approximately, 17% less entry capillary pressure is required for dyed PCE to invade water-saturated pores (because of the reduced IFT). These results suggest that multiphase displacement and remediation studies using dyes need to consider dye effects on IFTs. Table 1 shows a list of the IFTs of water to DNAPL reported in the literature. The IFTs of water to PCE range from 34.5 to 51.0 dyn/cm. An average value of the IFTs is 45.8 dyn/cm. The wide range of the IFT can be attributed to differences in measurement conditions (e.g. dyes, equilibration, temperature, and measurement methods).

4. Conclusions

The densities and viscosities of remedial solutions were increased after remedial solutions were saturated with PCE. The results imply that the properties of the remedial solutions may change as they flow through a porous medium, containing DNAPL. These altered properties of the remedial solution can affect their transport through porous media.

The presence of a dye (500 mg Oil-Red-O/l of PCE) alters the IFTs between PCE and water or PCE and ethanol solutions. This was most apparent in the case of water and PCE. The lower IFT could substantially alter DNAPL migration in porous media. The dye also affects the surface tension of PCE. If dyed DNAPL is used for investigation of DNAPL migration under saturated and unsaturated conditions, this effect should be taken into consideration.

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