Processes Influencing PFAS Transport

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Managing Per- and Polyfluoroalkyl Substances (PFAS) at Your Site:
Key Technical and Regulatory Issues
1:30-2:15 pm
Thursday, September 12, 2019
Presentation Overview

- AFFF Release Scenarios
- (Fluoro)Surfactant Properties and Behavior
- Interfacial Processes Impacting PFAS Transport
- In Situ PFAS Sequestration
Surface soil, aquifer material and groundwater from 5 locations representing a range of AFFF spill scenarios at Robins Air Force Base.
Assess PFAS Concentration and Microbial Community Profiles

- Evaluate PFAS concentration and microbial communities as a function of soil properties
- Prepare microcosms to investigate precursor transformation rates and byproduct formation

<table>
<thead>
<tr>
<th>Depth (ft)</th>
<th>PFBS</th>
<th>PFOA</th>
<th>PFOS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0 - 0.5</td>
<td>265.8 ± 490.2</td>
<td>34.5 ± 39.7</td>
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<tr>
<td>2</td>
<td>17 - 18</td>
<td>5.2</td>
<td>0.66</td>
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<tr>
<td>3</td>
<td>19 - 20</td>
<td>0.66</td>
<td>0.66</td>
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<tr>
<td>4</td>
<td>26 - 27</td>
<td>37</td>
<td>17</td>
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<tr>
<td>5</td>
<td>15 - 25</td>
<td>3.4 ± 1.4</td>
<td>1.6 ± 0.2</td>
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<tr>
<td>6</td>
<td>25 - 35</td>
<td>2.4</td>
<td>1.8</td>
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2017 samples (μg/kg for soil, μg/L for water)
PFAS Release Scenario - Mixed Contaminants

Unsaturated Zone

Capillary Fringe

Water Table

Lower Confining Layer

DNAPL

LNAPL

Residual LNAPL

Water

Dissolved Contaminant Plumes

Groundwater Flow

[Diagram showing various groundwater components and contaminant plumes]
(Fluoro)Surfactant Properties and Behavior
Surface Active Agents (Surfactants)

General Properties and Nomenclature:

- Amphiphilic (polar and nonpolar moieties): Hydrophilic “head” group + Hydrophobic “tail” group
- Strong tendency to accumulate at interfaces (air-water, NAPL-water)
- Individual molecules (monomers) self assemble to form micelles as the aqueous phase concentration is increased
- Classification is based on the polar head group:
  - Anionic, Cationic, Nonionic, Amphoteric, Zwitterionic
PFAS Classified as “Fluorosurfactants”

- Low Volatility
- Recalcitrant
- Foam/Emulsion Formation
Examples of Nonionic Surfactants

Polyoxyethylene (20) Sorbitan Monooleate: (Tween 80, Polysorbate 20, Witconol 2722)

\[
\begin{align*}
\text{HO} & \left(\text{CH}_2\text{CH}_2\text{O}\right)_w\text{CH} & \text{CH} & \leftarrow \text{CH} & \left(\text{OCH}_2\text{CH}_2\right)_x\text{OH} & \text{O} \\
\text{CH}_2 & & \text{CH} & \leftarrow \text{CH} & \left(\text{OCH}_2\text{CH}_2\text{O}\right)_y\text{OC} & \left(\text{CH}_2\right)_7\text{CH} & \leftarrow \text{CH} & \left(\text{CH}_2\right)_7\text{CH}_3 \\
\end{align*}
\]

MW = 1310 g/mole
CMC = 35-45 mg/L

Dodecyl Alcohol Ethoxylate: (Witconol SN-120, Brij 35)

\[\text{C}_{12}\text{H}_{25}\left(\text{CH}_2\text{CH}_2\text{O}\right)_w\text{OH}\]

MW = 583 g/mole
CMC = 50-65 mg/L
Examples of Anionic Surfactants

Sodium dodecyl sulfate (SDS):

\[ \text{C}_{12}\text{H}_{25}\text{OSO}_3^-\text{Na}^+ \]

MW = 288 g/mole
CMC = 2,100 mg/L

Sodium dihexylsulfosuccinate (SDHSS, Aerosol MA-100):

\[ \text{O} \]
\[ \text{CH}_2\text{O} - \text{C} - \text{CH}_2(\text{CH}_2)_4\text{CH}_3 \]
\[ \text{CH} - \text{O} - \text{C} - \text{CH}_2(\text{CH}_2)_4\text{CH}_3 \]
\[ \text{SO}_3^-\text{Na}^+ \]

MW = 388 g/mole
CMC = 5,360 mg/L

Perfluorooctanesulfonic acid (PFOS)

\[ \text{C}_8\text{HF}_{17}\text{SO}_3^- \]

MW = 500 g/mole
CMC = 4,000-5,000 mg/L

...but the solubility is < 1000 mg/L???
Critical Micelle Concentration (CMC)

• At concentrations above the CMC, the number of monomers remains constant, while the number of micelles continues to increase.

• The surface tension remains constant above CMC because the air-water interface is saturated.

From dataphysics-instruments.com
Micellar Solubilization of Organic Compounds

\[ WSR = \frac{Co - Co,cmc}{C_{surf} - C_{surf,cmc}} \]
Surfactant Phase Behavior: NAPL-Water Interfacial Tension (IFT) and Emulsions

Winson Type I

LNAPL

2 Phase

3 Phase

Increasing Temp (Nonionic Surfactants) or Salinity (Ionic Surfactants)

(Middle Phase Microemulsion)

3-Phase Region

IFT at Interface

IFT\textsubscript{NAPL/W}

IFT\textsubscript{W/P}

IFT\textsubscript{NAPL/MP}

Excess NAPL (oil)

NAPL in water emulsion

Winsor Type I

Winsor Type III

Winsor Type II

Water in NAPL (oil) emulsion

Excess Water

2 Phase

3 Phase

Middle-Phase microemulsion

2 Phase
Vertical Displacement (Mobilization) of PCE
Flushed with 4% Aerosol AY/OT (IFT = 0.09 dyne/cm)
Determining Risk of NAPL Mobilization

Total Trapping Number ($N_T$)

- Entrapped NAPL
- Water
- Soil Particle

- Gravitational (Buoyancy) Force
- Advective (Viscous) Force
- Capillary Force
- Gravitational (Buoyancy) Force

Angle of flow

Direction of flow in pore

$\Delta l$

$P_A$

$P_n$

$\nu \rho \rho_g$

$2r_p$

$2r_n$

$z$

$1$

$x$
Total Trapping Number ($N_T$)

**Capillary Number:**
(viscous forces)

$$N_{Ca} = \frac{q\mu}{\sigma_{ow} \cos \theta}$$

**Bond Number:**
(gravity forces)

$$N_B = \frac{\Delta\rho g k k_{rw}}{\sigma_{ow} \cos \theta}$$

$$N_T = \sqrt{N_{Ca}^2 + 2N_{Ca}N_B \sin \alpha + N_B^2}$$

**Vertical:** $N_T = |N_{Ca} + N_B|$

**Horizontal:** $N_T = \sqrt{N_{Ca} + N_B}$

- $\mu$ = dynamic viscosity
- $\Theta$ = contact angle
- $k$ = intrinsic permeability
- $k_{rw}$ = relative permeability to water
- $\rho$ = density of fluid
- $g$ = gravity constant
- $q$ = Darcy velocity
- $\sigma_{ow}$ = interfacial tension (oil-water)
PCE Desaturation Curves for Ottawa Sands

Critical Nt Range

4% W2722
IFT=5.0 dyne/cm

4% Aerosol MA/OT
IFT=0.58 dyne/cm

4% Aerosol AY/OT
IFT=0.09 dyne/cm

Total Trapping Number ($N_T$)
Risk of Uncontrolled DNAPL Mobilization

20-30 mesh Ottawa Sand

Surfactant plume containing solubilized PCE

Uncontrolled downward flow of PCE-DNAPL

F-70 Ottawa Sand
Questions related to PFAS Transport

• How much PFAS accumulates at the air-water interface in unsaturated soils?

• How does PFAS impact soil water retention characteristics and water drainage during infiltration events?

• How does PFAS interact with NAPLs, and do these interactions result in enhanced NAPL solubility or mobilization?

• Can we modify existing mathematical models to describe PFAS fate and transport in complex systems?

• How do we account for mixtures of many surfactants, including PFAS?
Interfacial Processes Impacting PFAS Transport
Interfacial Tension Measurements

SERDP Projects ER18-1149 and ER-2714

Sigma T700 Tensiometer

Resolution of 0.01 mN/m

Note: mN/m = dyne/cm = g/s²

Surface Tension by Wilhelmy Plate

Ramé-Hart Goniometer

Interfacial Tension by Pendant Drop
Preparation of Solutions for IFT Measurements

• Stock solutions ranged from 50 to 10,000 mg/L
  • PFOA or KPFOS solids using analytical balance
  • Sonicating for 30 min and heating overnight at 40 °C
• Concentrations from 0.1 to 50 mg/L prepared by serial dilution
• Concentrations verified by LC-MS/MS
• To simulate principal aquifers in US, aqueous solutions contained MgSO$_4$, NaHCO$_3$, KCl, and CaCl$_2$
  • Low Dissolved Solids (LDS) ~40 mg/L (high purity drinking water) ~9 mM
  • Mid Dissolved Solids (MDS) ~400 mg/L (secondary drinking water standard) ~90 mM
  • High Dissolved Solids (HDS) ~1,700 mg/L (unpleasant drinking water) ~380 mM

PFOA and PFOS working solutions from 0.1 to 10,000 mg/L in 100-mL HDPE bottles
Air-Water Interfacial Tension (Surface Tension)

PFOA (Effect of Salts)

- To simulate principal aquifers in US Background solution contains MgSO₄, NaHCO₃, KCl, and CaCl₂
- Dissolved salts resulted in lower surface tension for PFOA
  - Low Dissolved Solids (LDS) ca. 40 mg/L (high purity drinking water)
  - Mid Dissolved Solids (MDS) ca. 400 mg/L (secondary drinking water standard)
  - High Dissolved Solids (HDS) ca. 1,700 mg/L (unpleasant drinking water)

Costanza et al., 2019, ES&T Letters
Air-Water Interfacial Tension (Surface Tension)

PFOS (Effect of Salts)

Similar effect, but more pronounced with PFOS

Minimal effect above 400 mg/L TDS

- **Low Dissolved Solids (LDS)** ca. 40 mg/L (high purity drinking water)
- **Mid Dissolved Solids (MDS)** ca. 400 mg/L (secondary drinking water standard)
- **High Dissolved Solids (HDS)** ca. 1,700 mg/L (unpleasant drinking water)
Gibb’s Equation $\rightarrow$ Surface Excess

Interfacial tension is a measure of surface concentration or surface “excess” ($\Gamma$) [Langmuir, 1917]

\[
\Gamma = - \frac{C}{RT} \left( \frac{\partial \gamma}{\partial C} \right)_T
\]

\[
\gamma = \gamma_0 \left[ 1 - a \times \ln \left( \frac{C}{b} + 1 \right) \right]
\]

\[
\Gamma = \frac{a\gamma_0}{RT} \frac{C}{C + b}
\]

Gibb’s Equation  \quad Szyszkowski Equation  \quad Langmuir/Szyszkowski Eq.

$\Gamma$ = surface excess  
$\gamma$ = surface tension  
$C$ = aqueous conc.  
$R$ = gas constant  
$T$ = temperature (°K)

• “a” and “b” are nonlinear fitting parameters
• This equation allows you to fit the entire surface tension vs. PFAS concentration curve
Langmuir/Szyszkowski Equation

\[ \Gamma = -\frac{C}{RT} \frac{\partial \Gamma}{\partial C} \]

Gibb’s Eq.

\[ \gamma = \gamma_0 \left[ 1 - a \ln \left( \frac{C}{b} + 1 \right) \right] \]

Szyszkowski Eq. Fit

\[ \Gamma = \frac{a \gamma_0}{RT} \frac{C}{C + b} \]

Langmuir/Szyszkowski Eq.

PFOA

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PFOS

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Surface Excess Calculations

Equation Development

\[ K_i = \text{Linear Partition Coefficient (L/m}^2) \]

\[ \Gamma = -\frac{C}{RT} \left( \frac{\partial \gamma}{\partial C} \right)_T \quad \rightarrow \quad K_i = \frac{1}{RT} \frac{\partial \gamma}{\partial C} \quad \rightarrow \quad \Gamma = K_i C_w \quad \text{Linear} \]

\[ \Gamma = -\frac{1}{RT} \left( \frac{\partial \gamma}{\partial \ln C} \right)_T \quad \rightarrow \quad K_i = \frac{1}{RT} \frac{\partial \gamma}{\partial \ln C} \quad \rightarrow \quad \Gamma = K_i \frac{C_w}{C_{\text{ref}}} \quad \text{Natural Log} \]

\[ K_i = \text{Natural Log Partition Coefficient (mg/m}^2) \]

\[ \Gamma = \frac{a \gamma_0}{RT} \frac{C}{C + b} \quad \text{Nonlinear (Langmuir/Szyszkowski)} \]

requires an arbitrary reference conc.
(e.g., Lyu et al., 2018, \(C_{\text{ref}} = 1 \text{ mg/L}\)
Surface Excess Calculations
Comparison of Different Approaches

\[
\Gamma = K_i C_w
\]
Linear

\[
\Gamma = K_i \frac{C_w}{C_{ref}}
\]
Natural Log

\[
\Gamma = \frac{a \gamma_0}{RT} \frac{C}{C + b}
\]
Langmuir/Szyszkowski
PFOS Phase Distribution in Unsaturated Soils

Nonlinear: Langmuir/Szyszkowski Equation

\[ M_{\text{Total}} = V_T(C_wS_w + C_wK_D\rho_b + S_a\frac{\alpha\gamma_0}{RT}\frac{C}{C+b}) \]

Total PFOS Mass = Mass in Water + Mass on Solids + Mass at Air-Water Interface

- \( C_w = 1 \text{ mg/L}, \ S_w = 0.26, \ K_D = 1.14 \text{ mg/kg}, \ \rho_b = 1.5 \text{ kg/L}, \ S_a = 80 \text{ cm}^{-1} \text{ sand}, \ S_a = 1000 \text{ cm}^{-1} \text{ silt} \) (Brusseau, 2018)
Surface Tension of “PFOS” Mixture

“PFOS mixture” consists of a sulfonamide and three sulfonates

- Prepared equimolar stock mixtures from solids with total concentrations of 100, 200, 400, and 600 mg/L
- Dilutions of stock mixtures in range from 0.1 to 80 mg/L
- Solutions in ultrapure water, and water with low, mid, and high dissolved solids
“PFOS mixture” contained a sulfonamide and three sulfonates in ca. 400 mg/L TDS
- PFBS, PFHxS, PFOS and FOSA (0.3:0.3:0.2:0.2 mole fractions)
- PFBS and PFHxS surface tension of 70 mN/m at 100 mg/L (not surface active)
- FOSA surface tension lowest at equivalent concentration
- PFOS mixture exhibited “ideal” surface tension behavior from 0.2 to 20 µmol/L, non-ideal at increasing concentrations
NAPL-Water Interfacial Tension

Drop of NAPL suspended in solutions containing PFAS with ca. 1700 mg/L TDS
Confirmed oleophobic nature of the perfluorocarbon chain (Moody and Field, 2000)
• Significant reduction in interfacial tension only observed for concentrated solutions (>100 mg/L)
• IFTs less than 5 mN/m (dyne/cm) typically needed for NAPL mobilization
AFFF Phase Behavior

JP4 Jet Fuel

Foam
Macroemulsion
Aqueous Phase

4 mL : 4 mL
AFFF Phase Behavior

JP4 Jet Fuel (Oil Red O)

Foam

Macroemulsion

Aqueous Phase

Collapsed Foam

JP4-NAPL
Comparison AFFF Phase Behavior

JP4 Jet Fuel

- 3% active ingredient
- 7 mL JP-4 : 7 mL surfactant solution
- After 24 hr settling
Mathematical Modeling of PFAS Transport in the Unsaturated Zone
**Modeling PFAS Adsorption at Air-Water Interface**

**Objective:** Incorporate nonlinear PFAS adsorption at air-water interface using a modified version of Hydrus 1D

**Richards Equation:**
\[
\frac{\partial \theta}{\partial t} = \nabla \cdot (k \nabla h) + \frac{\partial k}{\partial z} + S
\]

**Langmuir Isotherm:**
\[
\Gamma_i = \frac{a \gamma_0 W \alpha}{C_i + b}
\]

**Linear Isotherm:**
\[
\Gamma_i = K_i C_i
\]

Specific Interfacial Area \([L^{-1}]\), \(A_{ai} = SA \left(0.9031 - 0.9012 \frac{\theta}{\theta_s}\right)\)

SA: Geometric su rface area \([L^{-1}] = \frac{6(1 - \varphi)}{d_{50}}\) \hspace{1cm} \text{(Costanza-Robinson et al., 2008)}

\(\theta_w\): water content, \(\theta_s\): saturated water content, \(\varphi\): porosity, \(s_\alpha\): saturation of \(\alpha\) – phase

\(a\) and \(b\): Szyszkowski eq. parameters fitted using batch experimental results

\(K_i\): linear partitioning coefficient
Effect of PFAS Accumulation at Air-Water Interface on Unsaturated Zone Transport

- Pulse injection (1 PV) of PFOA or PFOS (10 mg/L)
- Medium level of total dissolved solids (400 mg/L TDS)
- F-70 Ottawa sand (40-270 mesh)
- Uniform water content, $\theta_w = 0.27$

Langmuir/Szyszkowski Isotherm

Linear Partitioning

More retention at air-water interface with linear isotherm assumption
PFAS Vertical Concentration Profiles in Unsaturated Soil

- Pulse injection (1 PV) of PFOA or PFOS (10 mg/L)
- Medium level of total dissolved solids (400 mg/L TDS)
- F-70 Ottawa sand (40-270 mesh)
- Uniform water content, $\theta_w = 0.27$
Effect of Input Concentration on Unsaturated Zone Transport of PFOS

- Pulse injection (1 PV) of PFOS (10 mg/L or 50 mg/L)
- Medium level of total dissolved solids (400 mg/L TDS)
- F-70 Ottawa sand (40-270 mesh)
- Uniform water content, $\theta_w = 0.27$

Greater proportion retained at lower concentrations (influence of non-linearity)
Effect of Soil Water Content and TDS on PFOS Transport

Interfacial Area \([ \text{L}^{-1} ]\), \( A_{ai} = SA \left( 0.9031 - 0.9012 \frac{\theta_w}{\theta_s} \right) \)

\( \theta_w = 0.20 \) Langmuir/Szyszkowski Isotherm \( \theta_w = 0.27 \)

- Pulse injection (1 PV) of PFOS (10 mg/L)
- F-70 Ottawa sand (40-270 mesh)
- Uniform water content, \( \theta_w = 0.20 \) or 27
In Situ Sequestration of PFAS
Coagulant polymers (cationic surfactants)

SERDP Project ER-2425

Poly-DADMAC (PDM)

- Accepta 4351
- ~ 28% OC
- Quaternary Amine
- diallyl dimethylamine
- MW ~ 350,000

Polyamine (PA)

- Accepta 4350
- ~ 26% OC
- Quaternary Amine
- epichlorohydrine and dimethylamine
- MW ~ 240,000
To improve performance,...combine Powdered Activated Carbon (PAC) with polyDADMAC (PDM)

- PDM acts to stabilize PAC in suspension, facilitates delivery
- Both PDM and PAC can serve as sorbents (wide range of effectiveness)

<table>
<thead>
<tr>
<th></th>
<th>1 g/L PAC</th>
<th>1 g/L PAC + 5 g/L PDM</th>
<th>1 g/L PAC</th>
<th>1 g/L PAC + 5 g/L PDM</th>
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</thead>
<tbody>
<tr>
<td>Immediately after Sonication</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
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<td>48 hrs after Sonication</td>
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DARCO® 100 mesh (150 µm)
Powdered Activated Carbon (Sigma Aldrich)

Commerciaally Available (Proprietary) PFAS Sorbents

- **RemBind™-Tersus**
  Activated carbon, aluminum hydroxide, organic matter and other additives, intended for near surface soil mixing

- **PlumeStop® Liquid Activated Carbon™—Regenesis**
  Activated carbon (1-2μm) suspended in water dispersed with organic polymer

- Limited independent verification
- Limited data (e.g., mass balance)
- In situ delivery issues rarely addressed
PFOA and PFOS Batch Adsorption Studies With Darco® PAC (100-mesh)

Langmuir Isotherm

\[
C_s = \frac{C_s, max \times b \times C_{w, eq}}{1 + b \times C_{w, eq}}
\]

PFOA

- \(C_s, max = 323 \text{ mg/g}\)
- \(b = 0.0413 \text{ L/mg}\)

PFOS

- \(C_s, max = 316 \text{ mg/g}\)
- \(b = 0.0673 \text{ L/mg}\)
(1) Non-reactive tracer test (pulse injection), (2) Inject PDM+PAC suspension, (3) Inject background electrolyte, (4) Inject PFAS solution (e.g., 100 ug/L PFOS)
Injection of PDM+PAC Suspension

40-50 mesh Ottawa Sand \((d_{50} = 358 \text{ um})\), \(k_i = 7.37 \times 10^{-11} \text{ m}^2\), \(n = 0.37\), SSA = 0.0125 \text{ m}^2/\text{g}, PV = 22 \text{ mL}

PDM+PAC Suspension: 1,000 mg/L PAC + 5,000 mg/L PDM, viscosity = 1.18 cP
Flow rate: 0.12 mL/min; pore-water velocity \(\sim 1.0 \text{ m/day}\)
Images of PDM+PAC Treated Ottawa Sand
Based on the measured $C_{s,\text{max}} = 316$ mg/g and mass of retained PAC (~27 mg), the capacity of the column should be ~ 8.65 mg PFOS, consistent with the observed retention of ~10.04 mg PFOS.

For a 100 µg/L injection; ~2 ug PFOS retained/PV, capacity would be reached after ~5,020 PV.
Based on the measured $C_{s,\text{max}} = 323 \text{ mg/g}$ and mass of retained PAC ($\sim 14.8 \text{ mg}$), the capacity of the column should be $\sim 4.78 \text{ mg PFOS}$, consistent with the observed retention of $\sim 6.57 \text{ mg PFOA}$

For a 100 µg/L injection; $\sim 2 \text{ ug PFOA retained/PV}$, capacity would be reached after $\sim 3,600 \text{ PV}$
Configuration of Aquifer Flow Cell

PFAS Injection Ports

PDM+PAC Injection Ports

Downgradient Sampling Ports

PV=1.45L
Tracer Test Before PDM+PAC Injection
Side-port Injection of 1 g/L PAC + 5 g/L PDM

40 mL (0.08 mL/min) with background flow (2.4 mL/min)

80 mL (0.08 mL/min) with no background flow
Tracer Test After PDM+PAC Injection
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BROWN School of Engineering

Tufts University School of Engineering

University of Minnesota

Jacobs