The Effect of Humic and Fulvic Acids on Arsenic Solubility in Drinking Water Supplies

Basic Information

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Publication

The Effect of Humic and Fulvic Acids on Arsenic Solubility in Drinking Water Supplies

Final Report

Statement of Critical Regional or State Water Problem
Arsenic in ground water results primarily from natural geochemical interactions that occur between water and As-containing rocks and minerals (Fleischer 1983). Due to its known toxic effects on humans, arsenic in drinking water is a threat to public health and is regulated in the United States by the Safe Drinking Water Act. On January 22, 2001 the United States Environmental Protection Agency (USEPA) published a Final Rule in the Federal Register (40 CFR 141.62(b)(16)) establishing a new maximum contaminant level (MCL) for arsenic of 10 µg/L, down from 50 µg/L. This revision reflects an improved understanding of the toxic effects of arsenic on humans, and is expected to substantially decrease annual deaths from cancer (USEPA 2002). On February 22, 2002 the new arsenic drinking water limit became effective. Enforcement of the Rule begins on January 23, 2006 and it is expected to increase annual treatment costs by approximately $181 million (USEPA 2002).

Ground water is crucial to meeting the industrial and domestic needs of the residents of Ohio, with 79% of the community water systems, over 99% of the non-community water systems, and nearly 1 million rural homes utilizing ground water (OhioEPA 2000). Arsenic concentrations in ground water above the MCL of 10 µg/L occur throughout Ohio, particularly in areas with sand and gravel aquifers (OhioEPA 2000). In many instances treating As-containing ground water to meet regulatory needs and protect public health will require costly modifications to optimize existing treatment processes or the addition of point-of-use treatment techniques. The United States Environmental Protection Agency has issued guidance in selecting treatment methods for removing arsenic, and emerging technologies utilizing membranes (e.g., reverse osmosis), adsorptive processes (e.g., activated alumina), and precipitative processes (e.g., enhanced coagulation/filtration) show particular promise (Hering et al. 1997; Brandhuber and Amy 2001). Common inorganic and organic ground water constituents reduce removal efficiency, and natural organic matter (NOM) decreases removal by 20 - 50 % (Hering et al. 1997; Brandhuber and Amy 2001). Recent evidence suggests that NOM can complex arsenic to form stable solution complexes (Redman et al. 2002), and the increase in arsenic solubility resulting from the presence of such stable As-NOM complexes could be responsible for reduced removal efficiency. Details of these interactions are limited, however, and their importance is currently unknown. Knowledge of the fundamental processes that control As solubility, transport, and treatment, including interactions between arsenic and NOM, is crucial to maintaining the quality of ground water that approximately five million people in Ohio depend upon for their daily needs.

Nature and Scope of the Research
Arsenic (As) is a naturally occurring trace element in the earth’s crust and is a common constituent in many igneous and sedimentary rocks. Arsenic is readily mobilized into solution through the combined effects of geochemical interactions and biological activity, and is normally present in surface and ground water at low concentrations (≤ 1 µg/L) (Welch et al. 2000;
Smedley and Kinniburgh 2002). Although high As concentrations are associated with anthropogenic sources, the majority of environmental As problems are the consequence of natural processes (Welch et al. 2000; Smedley and Kinniburgh 2002). Arsenic toxicity to humans is well documented (Council 1999), and its presence at elevated concentrations in the public water supply is of great contemporary concern (Nickson et al. 1998; Council 1999; Welch et al. 2000; Berg et al. 2001; Smedley and Kinniburgh 2002).

In natural water systems, arsenic predominantly exists in the inorganic form as oxyanions of trivalent arsenite, As(III), or pentavalent arsenate, As(V). Oxidizing conditions favor the formation of arsenate species (H$_3$AsO$_4$, H$_2$AsO$_4^-$, and HAsO$_4^{2-}$), whereas reducing conditions favor arsenite species (H$_3$AsO$_3$ and H$_2$AsO$_3^-$). The species H$_2$AsO$_4^-$, HAsO$_4^{2-}$, and H$_3$AsO$_3^-$ prevail under environmental conditions, where the pH spans 4 to 9 (Baes and Mesmer 1976). Although the redox state of a system is important, arsenic solubility and transport is dominated by adsorption reactions that occur at the surface of reactive iron and aluminum oxide minerals. Adsorption of arsenic oxyanions by mineral surfaces is favored at low pH, and adsorption decreases in magnitude with increasing pH in a manner consistent with other anions (Sigg and Stumm 1981). In general, arsenate is adsorbed to a greater extent than arsenite, except at elevated pH (≥ 9) where the opposite occurs (Xu et al. 1988; Wilkie and Hering 1996; Raven et al. 1998). Consequently, in most environmental systems arsenite is more mobile and bioavailable, hence more toxic than arsenate (Council 1999; Smedley and Kinniburgh 2002).

Co-occurring anionic solutes alter the adsorption and thus the solubility of arsenic. Sulfate and phosphate directly compete with arsenic for surface sites on reactive metal oxides, particularly at low pH, and increase arsenic solubility (Xu et al. 1988; Manning and Goldberg 1996; Wilkie and Hering 1996); molybdate, however, has little net effect on As adsorption or mobility (Manning and Goldberg 1996). Surface complexation models suggest that dissolved carbonate should interfere with arsenic adsorption on mineral surfaces at carbonate concentrations typically measured in ground and soil waters (Appelo et al. 2002). Experimental evidence in support of these calculations is still lacking because carbonate adsorption reactions are difficult to study (Wilkie and Hering 1996).

The formation of solution complexes between arsenic oxyanions and other elements is limited (Cullen and Reimer 1989), however, even such limited interactions still influence arsenic speciation (Lowenthal et al. 1977; Wilkie and Hering 1996; Redman et al. 2002). For example, in artificial seawater arsenate forms ion pairs with magnesium and calcium (Lowenthal et al. 1977). These ion pairs result from charge screening that is induced by the high solution ionic strength and their presence increases the concentration of arsenic (Lowenthal et al. 1977). A similar process decreases arsenic solubility by enhancing As (V) adsorption at elevated pH (Wilkie and Hering 1996) where the adsorption of calcium reduces unfavorable coulombic interactions that otherwise would limit the adsorption of arsenate oxyanions.

The adsorption of arsenate and arsenite to mineral surfaces is reduced in the presence of natural organic matter (NOM) (Xu et al. 1988; Xu et al. 1991; Bowell 1994; Grafe et al. 2001; Grafe et al. 2002; Redman et al. 2002). NOM is ubiquitous in aquatic systems and consists of a heterogeneous mixture of polyfunctional molecules of varying size and reactivity. The ability of NOM to bind contaminants and mineral surfaces can markedly alter contaminant mobility and
has resulted in extensive research (e.g., Davis 1984; Pignatello and Xing 1996; McCarthy et al. 1998; Lenhart and Honeyman 1999). The effects of NOM on As adsorption differ depending upon the NOM source, as well as the charging characteristics and surface area of the adsorbent mineral (Xu et al. 1988; Xu et al. 1991; Bowell 1994; Grafe et al. 2001; Grafe et al. 2002). Like sulfate and phosphate, the reduction in arsenic adsorption is presumed to result from competition between As and NOM for surface sites (Xu et al. 1988; Xu et al. 1991; Bowell 1994; Grafe et al. 2001; Grafe et al. 2002). Redman et al. (Redman et al. 2002), however, present evidence that supports the formation of stable As-NOM solution complexes, which could be the reason for the reduced As adsorption. The complexion of As by NOM depended upon the NOM source and increased with NOM-bound cationic metals, particularly Fe (Redman et al. 2002).

A comprehensive framework for understanding the extent and importance of arsenic complexion by NOM in natural waters awaits development. Scant evidence, other than that presented by Redman et al. (Redman et al. 2002), exists examining the formation of solution complexes between NOM and As (Tanizaki et al. 1985; Thanabalasingam and Pickering 1986). Thanabalasingam and Pickering (Thanabalasingam and Pickering 1986) find that the association of As(V) and As(III) with two commercial humic acids followed a Langmuir relationship, and that NOM binds arsenate more strongly than arsenite. Tanizaki et al. (Tanizaki et al. 1985) sampled river water in Japan and report that approx. 60% of the As was associated with colloidal matter that consisted primarily of organic carbon. These results provide little additional insight into the complexion of As by NOM, and many questions remain, including the role of coexisting cationic solutes, the impact of solution pH, and the dependence of As complexation on the physicochemical properties of NOM.

**Research Objective**

The objective of this research is to investigate the association of inorganic arsenic with different sources of NOM in the presence of metal cations (e.g., Ca$^{2+}$). Arsenic is highly toxic and readily mobilized in significant concentrations by natural processes that occur in ground water. The EPA considers arsenic to be a priority pollutant and recently lowered the MCL to 10 µg/L from 50 µg/L. Results focused on examining interactions between As, metal cations, and NOM using potentiometric titration, dialysis and for speciation, capillary electrophoresis.

**Materials and Methods**

*Materials.* Stock solutions of arsenate (As(V)) and arsenite (As(III)) were prepared using sodium hydrogenarsenate heptahydrate, Na$_2$HAsO$_4$·7H$_2$O, and sodium metaarsenite, Na$_3$AsO$_3$, respectively (both purchased from Aldrich). Water for all experiments was supplied from a Milli-Q water system (>18 MΩ × cm resistance, Millipore). Three samples of NOM were purchased from the International Humic Substances Society, IHSS (Table 1); Suwannee River NOM (SRNOM), Nordic Lake NOM (NLNOM) and Pahokee Peat. The Suwannee River and Nordic Lake NOM were used as supplied. DOM from Pahokee peat was isolated following IHSS standard methods for soil, followed by dialysis using a 1000MW cutoff membrane. The Pahokee Peat NOM extracted in this manner was then stored in the dark at 4°C after being freeze dried. The elemental compositions of the NOM samples were analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES; Varian) and graphite furnace atomic
absorption spectrometry (GFAA; Varian) and confirmed the arsenic content in each sample was negligible (e.g., results for SRNOM in Table 2).

<table>
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<tr>
<th>NOM Type</th>
<th>H₂O</th>
<th>Ash</th>
<th>C</th>
<th>H</th>
<th>O</th>
<th>N</th>
<th>S</th>
<th>P</th>
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<tr>
<td>Suwannee River</td>
<td>8.15</td>
<td>7.0</td>
<td>52.47</td>
<td>4.19</td>
<td>42.69</td>
<td>1.10</td>
<td>0.65</td>
<td>0.02</td>
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<tr>
<td>Nordic Lake</td>
<td>nd</td>
<td>41.4</td>
<td>53.17</td>
<td>5.67</td>
<td>nd</td>
<td>1.10</td>
<td>nd</td>
<td>nd</td>
</tr>
<tr>
<td>Pahokee Peat*</td>
<td>6.2</td>
<td>12.7</td>
<td>46.90</td>
<td>3.90</td>
<td>30.3</td>
<td>3.42</td>
<td>0.58</td>
<td>Nd</td>
</tr>
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*Typical results, not necessarily representative of those for the substance examined in this study.

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<th>Al</th>
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<th>Ba</th>
<th>Ca</th>
<th>Cu</th>
<th>Fe</th>
<th>K</th>
<th>Mg</th>
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<th>Si</th>
<th>Sr</th>
<th>Zn</th>
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<tr>
<td>0.85</td>
<td>nd*</td>
<td>&lt;0.01</td>
<td>0.35</td>
<td>0.01</td>
<td>2.19</td>
<td>0.28</td>
<td>0.09</td>
<td>nd</td>
<td>2.90</td>
<td>nd</td>
<td>4.20</td>
<td>&lt;0.01</td>
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* Limit of detection by GFAA for As is 0.9 µg/L

**Potentiometric Titrations.** Titrations were performed on three solutions: (1) 20 mg/L As(V) in 0.1 M NaCl, (2) 170 mg/L PPNOM in 0.1 M NaCl, and (3) 20 mg/L As(V) and 170 mg/L PPNOM using a computer controlled automatic titrator (Mettler-Toledo DL77). Experiments were performed at a constant temperature of 25 °C in a nitrogen atmosphere. Prior to titration, appropriate aliquots of PPNOM, As(V), and NOM + As were prepared in CO₂-free Milli-Q water and allowed to equilibrate overnight. The ionic strength was adjusted to 0.1 M using NaCl. Titrations were run from the initial starting pH to a pH of 10 – 10.5 using standardized 0.1 or 0.01 N NaOH. Equilibrium between additions was defined to be when the pH drift was less than 0.01 pH/min.

**Dialysis Experiments.** Experiments were performed to examine interactions between As(V) and PPNOM using a batch dialysis experimental approach. Solutions of variable As and 10 mg/L PPNOM were equilibrated in the dark at pH 6.0 in solutions comprised of 0.01 M NaCl with and without 0.0005 mM CaCl₂. 100 mL aliquots of these solutions were transferred to dialysis tubing with a nominal molecular weight cutoff of 1 kDa. The dialysis tubing was placed within 2 L beakers and the As-PPNOM solutions were dialyzed against 2000 mL of the electrolyte solution. Solutions within the beaker were stirred continuously using a Teflon coated magnetic stir bar and samples were periodically extracted to determine As and TOC concentrations. To complete the mass balance, at the conclusion of the experiment the final solution concentrations of As and TOC within the tubing and the dialysis solutions were determined.
Arsenic Speciation using Capillary Electrophoresis. An HP3D capillary electrophoresis (CE) system (Agilent Technologies, Inc.) with direct UV detection and normal electroosmotic flow (EOF) was used as the CE device to separate arsenate and arsenite. A fused-silica capillary of 50 µm i.d. × 48.5 cm was used in all experiments. The effective length of the capillary to the detector is 40 cm and the capillary temperature was maintained at 20°C. The separation voltage was set to +15 kV, although a range in values between +10 kV and + 25 kV was investigated. Hydrodynamic injection at a pressure of 50 mbar was used for sample introduction and on-capillary UV diode-array was used for detection at a wavelength of 192 nm. (The minimum wavelength is 191 nm for the Agilent, diode-array with a deuterium lamp). The total injection time was 5 s or 10 s. To ensure uniform capillary surface conditions, the capillary was washed with a 0.1 M NaOH solution for 10 min at the beginning of each workday. Prior to each injection, the fused-silica capillary was flushed with 1 M NaOH for 1 min, Milli-Q water for 1 min and electrolyte buffer for 2 min.

Computational Platform. All model calculations and parameter optimizations presented in this report were performed using the nonlinear least-squares optimization program FITEQL, Version 3.2 (Herbelin and Westall 1996). Specific details of this program are discussed by Westall et al. (Westall and Hohl 1980; Westall et al. 1995; Herbelin and Westall 1996). The determination of the model’s goodness of fit is provided using a weighted sum of squares term (WSOS/DF), which is the sum of the squares of residuals divided by the degrees of freedom. The WSOS/DF term is a function of the absolute (s_abs) and relative errors (s_rel) of the data. In general, lower values of WSOS/DF indicate a better fit of the model to the data. Appropriate model fits are indicated when 0.1 < WSOS/DF < 20.

Results
Potentiometric Titration Studies: Potentiometric titration results for PPNOM, As(V) and PPNOM + As(V) in 0.1 M NaCl are shown in Figures 1A, 1B, and 1C, respectively. The titration curves for PPNOM and PPNOM+As(V) demonstrate the characteristic broad, featureless, “smeared” titration curve associated with natural organic substances (Bartschat et al. 1992). Titrations of arsenate solutions are characterized by buffering associated with the second dissociation of arsenic acid at pH 6.8. The first and third dissociation reactions are obscured by the hydrolysis of water at low and high pH, respectively.

Model fits to the PPNOM were accomplished using the discrete-ligand approach described by Westall et al. (Westall et al. 1995). This approach allows for the estimation of NOM acid-base chemistry by depicting NOM as a suite of monoprotic acidic ligands, HL_i, with defined pK_a values (e.g., pK_a,1 = 2; pK_a,2 = 4, etc.). No direct physical relationship of the acid groups to the NOM ‘molecule’ is implied; instead the acids are treated as a suite of discrete ligands that act independently of each other. Total concentrations of each acid site (T_HL_i) result from simulating the potentiometric titration data. To limit the number of model variables, no explicit corrections are employed to account for activity or electrostatics.

The data are simulated within FITEQL using a set of protolysis reactions and mass-action expressions for each acid site, HL_i:

\[ HL_i = H^+ + L_i^- \]

\[ K_{a(i)} = \frac{[L_i^-][H^+]}{[HL_i]} \]
where \( i \) refers to the specific monoprotic ligand. Model results using a 4-ligand model with pKa 4, 6, 8 and 10 provide excellent fit to the data (solid line in Figure 1A). We next applied this model, amended with the constants for arsenate protolysis, to predict the As(V)+NOM data. As shown in Figure 1C, the model fit predicts the behavior very closely indicating that for the conditions studied that interactions between PPNOM molecules and arsenate if present are very weak and do not perturb the protonation state of either reactant.

![Figure 1 - Acid-base titration of (a) 170 mg/l PPNOM, (b) 20 mg/L As(V) and (c) 170 mg/L PPNOM and 20 mg/L As(V). Solid lines represent model fits.](image)

**Dialysis of As(V)-PPNOM Solutions:** Dialysis experiments also did not provide evidence for sodium or calcium mediated interactions between PPNOM and arsenate as the concentration of As(V), corrected for dilution, approached the starting values of 20 mg/L, 100 mg/L and 200 mg/L (e.g., see Figure 2 for results for systems with calcium). Experiments were conducted for over 100 hrs, although steady-state conditions appear to have been reached in each system at approximately 24 hours. Concentrations of carbon in the solution outside the dialysis bags remained at background levels and at the conclusion of the experiment the carbon content was essentially the same as at the beginning of the experiment (data not shown). These results confirm that all of the PPNOM was retained within the dialysis tubing and that the arsenate distributed uniformly throughout the hydraulically connected solutions inside and outside the dialysis tubing to the same concentration.
CE studies with Arsenite and Arsenate: The absorption of ultraviolet light by inorganic arsenic species increases as the wavelength is decreased below 250 nm and appears strongest near 190 nm. Sun et al. 2002 applied CE at a wavelength of 192 nm to measure arsenate and arsenite solutions down to approximately 1 to 6 mg/L. Under similar experimental conditions we measured arsenite at 5.2 min., but seemed unable to detect arsenate (Figure 3). Investigating further using a UV-VIS spectrophotometer (Shimadzu Co., Kyoto, Japan), we scanned arsenite and arsenate samples from a wavelength of 190 to 300 nm. For a 20 mg/L arsenite sample at pH 6.58 the maximum absorbance at a wavelength of 192.8 nm was 0.73; however, at the same wavelength the maximum absorbance of a 200 mg/L arsenate sample at a similar pH of 6.84 was only 0.12. This suggests that the low absorptivity of arsenate (0.6 L cm\(^{-1}\) g\(^{-1}\)) compared with arsenite (36.5 L cm\(^{-1}\) g\(^{-1}\)) might be responsible for our inability to detect arsenate and thus in the remainder of this report we focus on results obtained with arsenite.

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Figure 2 – Measured As(V) concentrations outside dialysis tubing, corrected for dilution, as a function of time in the presence of 0.0005 M Ca\(^{2+}\).

Figure 3. Electropherogram of arsenite (44.11 mg/L) in the borate buffer (20 mM, pH 10).
The ability of CE to separate analyte species is dependent upon a proper buffer selection. We tested four common buffer solutions; carbonate (20 mM, pH 10), borate (20 mM, pH 10), phosphate (20 mM, pH 5, 7, 9, 10, and 11), and acetate (20 mM, pH 7.3). Optimum separation of arsenite occurs at elevated pH where both borate and carbonate are the most effective buffers, but due to its lower UV background borate is a better choice than carbonate.

Studies examining the pH effect on arsenic and NOM complexation were performed in a phosphate buffer at pH values of 5, 7, 9, 10, and 11 to investigate the influence of buffer pH on arsenite detection. At pH values of 5 and 7, the arsenite peak was very close to the EOF peak, making it difficult to accurately analyze peak areas. The pH of the electrolyte has a significant influence on the migration time of analytes and potentially the separation efficiency as well. Furthermore, it is possible that the electrophoretic mobility of arsenic is a weighted average of individual arsenic species.

**Evaluation of two-component interactions.** Results examining interactions between arsenite and SRNOM were collected by mixing 5-mL aliquots of arsenite and SRNOM solutions in polyethylene tubes. The initial arsenite and SRNOM concentrations were 44 mg/L and 57 mg/L, respectively. The pH value was not recorded. Tubes were rotated in the dark for 24 hours at room temperature (25 °C). Blanks run in parallel ensured that adsorption of arsenite to the surface of the polyethylene tubes was negligible. After equilibration, the mixture of arsenite and SRNOM solution was analyzed using CE. The detection wavelength was fixed at 192 nm although the maximum absorbance of SRNOM was measured at 195.5 nm.

The peak in the electropherogram associated with As(III) exhibits a slight decrease (~ 3%) in the mixed arsenite-SRNOM sample (Figure 4a) compared with the peak in an NOM-free electropherogram at the same concentration (Figure 3). This we attribute to a decrease in the concentration of free arsenite anions due to the formation of a small amount of SRNOM-As(III) complexes. The arsenite peak in Figure 4a occurs simultaneously with an “NOM hump” in the arsenite-free SRNOM sample (Figure 5). Changes in the peaks associated with NOM also suggest the formation of As(III)-NOM complexes (Figure 4b). For example, the height of the NOM peak at 9 minutes, NOM C, decreases and splits in the presence of arsenite (Figure 4b) which occurs, according to Nordén and Dabek-Zlotorzynska (1996), when complexes are formed with NOM. Note that the sharp peaks in the SRNOM electropherogram (NOM A, NOM B, and NOM C) and the broad NOM hump occur as a result of the heterogeneity and polydispersity of NOM. NOM macromolecules span a range in size and functional group content, and thus exhibit nonuniform charge-to-mass ratios and electrophoretic mobilities. In general, during normal EOF molecules having more negative charge-to-mass ratios also have greater electrophoretic mobilities and longer migration times.
Figure 4a. Electropherogram of arsenite and SR-NOM (20 mM borate, pH 10).

Figure 4b. Exaggeration of lower absorbance region in the electropherogram of arsenite and SR-NOM mixture in the borate buffer (Figure 2a).

Figure 5. Electropherogram of SR-NOM (57.27 mg/L) in the borate buffer (20 mM, pH 10).
Conclusions
Results collected using potentiometric titration and dialysis procedures indicate that under the conditions studied that arsenate does not form complexes with PP NOM, even in the presence of a weakly binding ion (Ca$^{2+}$). Our results examining As(III) interactions with SRNOM, however, suggest attractive interactions are a possibility. The content of iron and other cations within SRNOM could be the root cause for this observation and thus the system merits further study. Experimental work further characterizing interactions between NOM and As(III), which could change oxidation state to As(V), are contingent on method development to simultaneously analyze As(III) and As(V). For example, by coupling capillary electrophoresis with a technique that better detects arsenate (e.g., ICP-AES or ICP-MS).

References


