

POSSIBLE GEOLOGICAL-GEOCHEMICAL SOURCES OF AMMONIUM IN GROUNDWATER: PRELIMINARY RESULTS

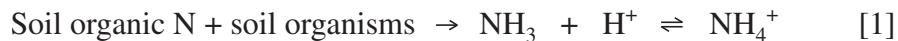
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Abstract

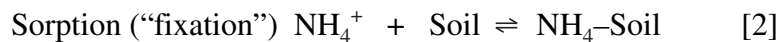
Ammonium has been detected in water wells in relatively large (>2 mg/L) concentrations in east-central Illinois and in central Iowa. Ammonium can interfere with drinking-water disinfection, and can be converted into nitrate and nitrite. The possible sources of ammonium in groundwater include fertilizers, landfill leachate, and wastewater disposal. However, the hypothesis of this study is that ammonium in groundwater can occur naturally from subsurface organic zones such as buried soils. Historical data indicated that ammonium has been detected in groundwater samples in relatively large concentrations before the current, voluminous use of nitrogen-containing fertilizers. The approach being used in this study is to conduct laboratory and field work to provide data on the possible link between buried organic material in Quaternary-age deposits and ammonium in water-wells. The results of the first year of this project indicate that the Robein silt is a potential source of ammonium in groundwater via dissolution, ion exchange, and mineralization reactions. The Robein silt can also retain ammonium via sorption. Enhanced (>2 mg/L) concentrations of ammonium were detected in water samples collected from wells located in two study areas (Champaign and Piatt Counties). However, there was insufficient evidence to link the ammonium in the well samples specifically with the Robein silt. On-going research will include the collection of additional groundwater samples, and the soil physical chemical characterization of recently collected soil samples.

Introduction

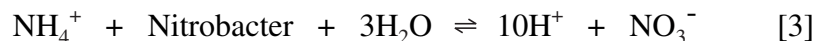
Based on 1,885 analyses, the state-wide median concentration of ammonium in groundwater in Illinois is 0.41 mg/L (Warner, 2000). Ammonium, however, has been detected in water wells in relatively large (>2 mg/L) concentrations in east-central Illinois (Larson and Roy, 2000). Enhanced concentrations of ammonium have also been detected in water-supply wells in Iowa (Schilling, 2002). Ammonium can interfere with the disinfection of water supplies by chlorine or chloroamine. Excess ammonium can also result in an increase in bacteria and coliform populations in water distribution systems (Schilling, 2002). Ammonium can also be oxidized into nitrite and nitrate by biotic and abiotic reactions. The chemical fate of nitrogen as ammonium is complex. More than 90% of the nitrogen in soil occurs in organic forms, many of which have not been identified. Known compounds include amino acids, amino sugars, peptides, and other proteins. The remaining nitrogen is in the form of ammonium. Soil nitrogen can be converted to ammonium by complex biotic and abiotic reactions. For example, soil microorganisms can use soil nitrogen (N) as an energy source, making ammonium as a by-product, viz.,



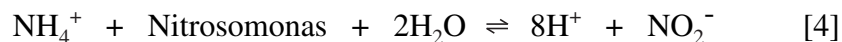
Once produced by “ammonification,” the environmental fate of ammonium can be influenced by electrostatic interactions with the charged surfaces of clays and organic matter (see Nommik and Vahtras, 1982). In fact, ammonium is used as a reagent to displace cations in the determination of the cation exchange capacity of soils. Once sorbed or fixed, it is no longer in solution:



Ammonium may also be converted to either nitrate or nitrite. Nitrobacter bacteria can convert ammonium into nitrate:



Ammonium can be converted to nitrite via Nitrosomonas, although the reaction is slow.



Nitrate can be converted to nitrite without microbes.



Ammonia concentrations greater than about 1.5 to 2.0 mg/L require relatively expensive treatment processes to eliminate the nitrogen species from the water supply. About 5 percent of the public water supply wells in Illinois contain excess ammonium (Larson and Roy, 2000). The possible sources of ammonium in groundwater in east-central Illinois include fertilizers such as anhydrous ammonia and ammonium nitrate, manure, landfill leachate, and wastewater. However, the hypothesis of this study is that subsurface organic zones such as buried soils can contribute ammonium to groundwater. Larson and Roy (2000) proposed that the occurrence of enhanced levels of ammonium in water wells could be linked to buried organic material in Quaternary-age

deposits. They presented evidence of a possible link between relatively large concentrations of ammonium in wells that penetrated topographically low areas of the Robein silt, an interglacial soil that is stratigraphically above the Sangamon soil which is developed in Illinoisan glacial till. Two years later, Schilling (2002) proposed that enhanced levels of ammonium in groundwater in Iowa resulted from the mineralization of *in situ* organic matter. The largest concentrations of ammonium occurred in wells that were open to Quaternary deposits. The objective of this study was to conduct laboratory and field work to gather data to test the proposed link between buried organic material in Quaternary-age deposits and ammonium in groundwater-wells in east-central Illinois. The results of this study may help define the relationship between buried soils/organic zones and enhanced levels of ammonium in groundwater, and suggest what can be done to avoid excessive levels of ammonium in drinking-water supplies.

Methods and Materials

Soil samples were collected from cores in the ISGS Geological Samples Library that contained buried organic material. Emphasis was given to the Robein silt. These soil samples were characterized in terms of (1) organic carbon content; (2) cation exchange capacity using the unbuffered salt extraction method of Sumner and Miller (1996); (3) water-soluble ammonium using an ion selective electrode (APHA, 1992); (4) exchangeable ammonium using a 0.2-M KCl solution; and (5) the alkali-labile nitrogen content (Mulvaney, 1996; Khan et al., 1997) as a measure of the amount of nitrogen available for mineralization. The ability of these soil samples to store ammonium was evaluated by conducting adsorption studies using the methods of Roy et al. (1992).

Groundwater samples were collected from wells that were either associated with the soil samples, or were located in high-ammonium areas. The wells were pumped and sampled using a Hydrolab system. Temperature, pH, and Eh were measured in the field at the time of sampling. A ZoBell solution was used as an Eh reference solution (Wood, 1976). The water samples were characterized in terms of ammonium content using an ion specific electrode. Chloride, nitrate, and sulfate were determined using a Dionex 2110i ion chromatograph, following U.S. EPA Method 300.0 (Pfaff et al., 1991). The concentrations of Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, La, Li, Mg, Mn, Mo, Na, Ni, Pb, S, Sb, Sc, Se, Si, Sr, Ti, Tl, V, and Zn were determined by inductively coupled argon plasma spectroscopy (ICAP).

Ammonium in Groundwater

If enhanced levels of ammonium in groundwater were solely derived from ammonium-containing fertilizers, then well-water data collected prior to the current voluminous use of fertilizers may indicate a scarcity of ammonium in groundwater. However, information published by the Illinois State Water Survey (ISWS) (Hanson, 1950; 1958) indicated that ammonium has been sporadically detected in water wells throughout east-central Illinois before the current heavy use of fertilizers (Table 1). When only those wells that contained ammonium concentrations greater than 2 mg/L were considered, ammonium ranged from 2.8 to 29 mg/L, and averaged 10.7 mg/L. The amount of information given in the well logs was sparse, but the reported presence of buried organic matter in some wells suggested that there could have been a subsurface source of ammonium. The presence of methane suggested reducing conditions that could have promoted the formation of reduced nitrogen such as ammonium.

Table 1. Occurrence of ammonium in water samples collected from water supply wells in east-central Illinois prior to the contemporary use of nitrogen-containing fertilizers (from Hanson, 1950; 1958).

City/village	County	NH ₄ (mg/L)	Depth of well (ft)	Notes	Date
Arcola	Douglas	15.1	128	“Peat” at 90 ft	1948
Arrowsmith	McLean	3.6	228	Methane detected	1957
Buckley	Iroquois	3.3	152		1948
Casey	Clark	6.0	131	“Buried wood” at 114 ft	1948
Chrisman	Egar	15.5	165		1948
Cisco	Piatt	24.2	114		1956
Chenoa	McLean	9.0	194	Methane detected	1948
Clinton	De Witt	4.2	360	Methane detected	1948
Colfax	McLean	13.3	105	Methane detected	1948
Deland	Piatt	19.9	83	Methane detected	1949
Downs	McLean	8.4	95		1956
Farmer City	De Witt	5.7	174	Methane detected	1948
Fisher	Champaign	14.0	240	“Soil at 225”	1948
Flanagan	Livingston	28.8	168		1947
Hammond	Piatt	10.3	152	Hydrogen sulfide detected	1948
Kansas	Edgar	8.1	80	Methane detected	1948
Leroy	McLean	9.5	59		1948
Newman	Douglas	16.6	143	Methane detected	1948
Paxton	Ford	6.0	150		1948
Rantoul	Champaign	2.8	137	Methane detected	1949
Sidney	Champaign	7.2	56		1948
Stanford	McLean	8.7	235		1948
Tolono	Champaign	13.3	157	Methane detected	1948
Weldon	De Witt	3.1	165	Methane detected	1949

Two study areas were chosen for collecting groundwater samples. Groundwater samples were collected from a well field in Champaign County, Illinois, that had been identified as containing anomalously large amounts of ammonium. One sample was collected near Parkland College in Champaign County from a production well owned by the Illinois-American Water Corporation.

The open interval of the well is in the Glasford Formation which is composed of glacial tills and outwash deposits. The Glasford is an Illinoisan-age deposit that is stratigraphically below the Sangamon soil. The second study area was the Allerton Park-Cisco area in Piatt County. This area contains wells that were installed by the ISGS, ISWS, and the City of Decatur. The open interval of the wells is in the Banner Formation which is stratigraphically below the Glasford formation. Previous water quality data from the ISWS and the Illinois Environmental Protection Agency indicated that it would be likely that elevated (greater than 2 mg NH₄⁺/L) levels of ammonium would be present in these areas.

Water-well samples were collected from Champaign and Piatt Counties (Table 2). Ammonium concentrations ranged from 0.47 to 4.67 mg/L. Nitrogen as nitrate was not detected in the samples (<0.1 mg/L). There was no obvious relationship between the amount of ammonium in solution and the concentration of calcium, iron or Eh of the samples. There was no obvious relationship between the amount of ammonium in each well and the depth or thickness of the Robein silt, nor the distance between the Robein in the column and the open interval of the well (Table 3). Schilling (2002) found no correlation between ammonium and well depth in Iowa. Moreover, while well Al-2 contained 2.00 mg/L ammonium, the Robein silt was not detected in well cuttings in the field during the installation of the well. Although there are relatively few observations given in Table 3, these few results did not support the hypothesis that the ammonium in the well was from a buried soil. Additional groundwater sampling is planned at the same wells, and possibly others in the Allerton study area.

Soil Physical Chemical Characterization

Soil samples were collected from cores that penetrated buried organic materials in Champaign, Piatt, and Tazewell Counties. A total of 29 soil samples were collected for this project from six different soil cores. Two of the soil cores were collected from the Parkland College study area in Champaign County, three cores were collected from the Allerton Park study area in Piatt County, (Tables 4 and 5) and one core was collected near Peoria. Each core was examined and described prior to sampling for this project. A total of 23 samples of the Robein Silt were collected. Four samples of the Sangamon soil—which is stratigraphically below the Robein—and two samples of the Morton Loess—which is above the Robein—were also collected.

In general, the Robein silt in the six cores ranged from being a fairly indistinct silty material with weak laminations and sparse root fragments to a distinct, dark peat. It can be described as having a silt, sandy loam or peaty silt loam texture, and a grayish brown, light olive gray, dark grayish brown, or black color. The depths of the samples ranged from about 18.7 to 52.7 meters below land surface. A sample of the Robein silt from the Peoria core dated by the carbon-14 method yielded an age of 27,000 years before present. As shown in Table 4, the reaction pH of the Robein samples ranged from 7.3 to 8.6. The organic carbon content ranged from 0.08 to 17.2%. Based on 19 samples, the mean carbon content was 3.33 ± 4.07 %. Also based on currently available data, the cation exchange capacity of the Robein samples was 3.52 to 7.07 cmol/kg.

Table 2. Chemical composition of six groundwater samples collected in Champaign and Piatt Counties, Illinois. All concentrations in mg/L unless otherwise noted.

Constituent ¹	NIWC-1 ²	Al-1 ³	Al-2 ³	Al-3 ³	D2 ⁴
pH	7.72	7.60	7.68	7.77	7.55
Eh _z (mV)	64	45	102	212	43
Temp (°C)	14.5	12.1	12.2	13.3	12.9
Depth (m)	49.1	80.9	75.4	59.9	98.2
NH ₄ ⁺ (mg/L)	4.67	0.47	2.00	0.54	2.20
Al	<0.01	<0.01	0.09	0.02	<0.01
B	1.75	0.51	0.66	0.10	0.35
Ba (µg/L)	232	39	286	100	185
Ca	56.1	16.2	54.4	88.8	82.8
Cl	<0.02	13.9	6.91	6.63	4.40
Fe	2.43	0.23	2.97	2.42	1.72
K	3.00	3.00	4.00	3.00	3.00
Li	<0.01	0.03	0.04	0.02	<0.01
Mg	31.1	7.78	23.0	40.4	35.4
Mn (µg/L)	16.0	70.0	161	104	29.0
Mo	<0.02	0.03	0.01	<0.01	<0.01
Na	67.0	240	209	42.9	32.2
Ni	<0.01	0.01	<0.01	0.01	<0.01
S	0.10	40.3	31.7	10.5	<0.02
Si	6.58	4.86	6.25	8.23	8.29
SO ₄	<0.02	109	81.4	29.8	<0.02

¹The concentrations of As, Be, Cd, Co, Cu, Cr, La, Pb, Sb, Sc, Se, Ti, Tl, V, and Zn were less than 0.2 mg/ L.

²Northern Illinois Water Corporation (now Illinois-American Water Corporation) well in Champaign County.

³Allerton Park area in Piatt County

⁴Oberservation well for the City of Decatur in Piatt County.

Table 3. Summary of ammonium concentrations, the screened interval in the corresponding well, and the depth of the Robein silt.

Well	NH ₄ ⁺ (mg/L)	Screened interval (m)	Depth of Robein (m)
NICW-1	4.67	84.2 to 90.3	36.9 to 38.3
AI-1	0.47 (0.74) ¹	77.8 to 80.9	About 8
AI-2	2.00	72.4 to 75.4	Absent
AI-3	0.54	55.3 to 59.9	19.8 to 21.0
D2	2.20 (1.95) ¹	91.8 to 98.2	Unknown

¹Data from Walton Kelly, Illinois State Water Survey.

Table 4. Summary of the buried soil samples.

Soil	Core	Depth Interval (m)	Description
Robein silt	NIWC-1	36.9 to 37.0	Silt. Dark grayish brown (10YR4/2). Laminated. 2.23% organic carbon. CEC 7.03 cmol/kg, pH 7.3
Robein silt	NIWC-1	37.5 to 37.7	Silt. Grayish brown (2.5Y5/2). Slightly calcareous. 1.39% organic carbon. CEC 5.10 cmol/kg, pH 7.6
Robein silt	NIWC-1	37.8 to 37.9	Sandy loam. Grayish brown (2.5Y5/2). Laminated. 0.52% organic carbon. CEC 4.93 cmol/kg, pH 7.5
Robein silt	NIWC 2	34.7 to 34.8	Silt. Grayish brown (2.5Y5/2). Weak laminations. 1.92% organic carbon. CEC 3.52 cmol/kg, pH 7.6
Robein silt	NIWC-2	34.9 to 35.1	Silt. Grayish brown (2.5Y5/2). Laminated. Wood/root fragments. 2.12% organic carbon. CEC 3.89 cmol/kg, pH 7.6
Robein silt	NIWC-2	35.1 to 35.2	Silt. Grayish brown (2.5Y5/2). Slightly calcareous. 0.59% organic carbon. CEC 4.93 cmol/kg, pH 7.6
Sangamon profile	NIWC-2	36.4 to 36.5	Silt. Olive (5Y5/3). Slightly sandy. Slightly calcareous. Compact. CEC 5.07 cmol/kg, 0.19% organic carbon.
Robein silt	Allerton 3	19.8 to 19.9	Silt. Grayish brown (10YR5/2) Slightly sticky. Cohesive. Calcareous. Profile is disturbed. 1.99% organic carbon. pH 7.4
Robein silt	Allerton 3	20.7 to 20.9	As above. Mottled. 2.02% organic carbon. pH 7.4
Robein silt	Allerton 3	21.1 to 21.2	As above. 3.01% organic carbon. pH 7.5
Robein silt	Allerton 4	18.7 to 18.8	Silt. Light olive gray (10Y6/2). Moderately clayey. Calcareous. 0.08% organic carbon. pH 8.1
Robein silt	Allerton 4	20.9 to 21.1	As above. Slightly sandy. 0.32% organic carbon. pH 8.6
Unidentified pre-Robein soil/ weathered zone	Allerton 4	29.6 to 28.7	Silt. Grayish brown (10YR5/2). Slightly calcareous. pH 7.3

The water-soluble ammonium content of the samples is being measured by conducting a 48-hour extraction using deionized water at 23° C (Table 6). Based on the results available so far, the amount of soluble ammonium ranges from 2.84 to 82.9 mg NH₄/kg. Water soluble plus exchangeable ammonium is being measured by the application of 0.2 N KCl. The results to date range from 5.75 to 149 mg NH₄/kg with a mean of 79.1 ± 42.6 mg NH₄/kg. Another way of expressing the results is that one ton of Robein silt has the potential to yield 72 grams of ammonium. Hence, the Robein silt can serve as a source of ammonium to groundwater. The amount of alkali labile nitrogen—a measure of the amount of nitrogen in the soil that is susceptible to mineralization—ranged from 99 to 272 mg nitrogen/kg soil. A portion of this nitrogen could occur as ammonium. The analysis of a surface soil sample from core NIWC-1 yielded 126 mg N/kg which was in the range of the buried Robein samples.

The capacity of the Robein samples to store ammonium is being studied by measuring ammonium sorption using a zero-head space, batch technique at 23° C. The results to date conform to a linear isotherm model (Fig. 1), viz.,

$$S = K_d (C)$$

where S = amount sorbed (mg/kg)
 K_d = a sorption constant (L/kg)
 C = equilibrium concentration in solution (mg/L)

The results to date (Table 7) have yielded K_d values of 2.55 to 5.90 L/kg, with a mean of 4.29 L/kg. If the equilibrium concentration of ammonium is 10 mg/L, which is within the range of well samples in Table 1, then 4.29 L/kg x 10 mg/L = 43 mg NH₄/kg of soil. If we let the mean concentration of water-soluble plus exchangeable ammonium equal S, the amount sorbed, then C = S/K_d = (79.07 mg/kg)/(4.29 L/kg) = 18.4 mg/L which is in qualitative agreement with the water-well data given in Table 1.

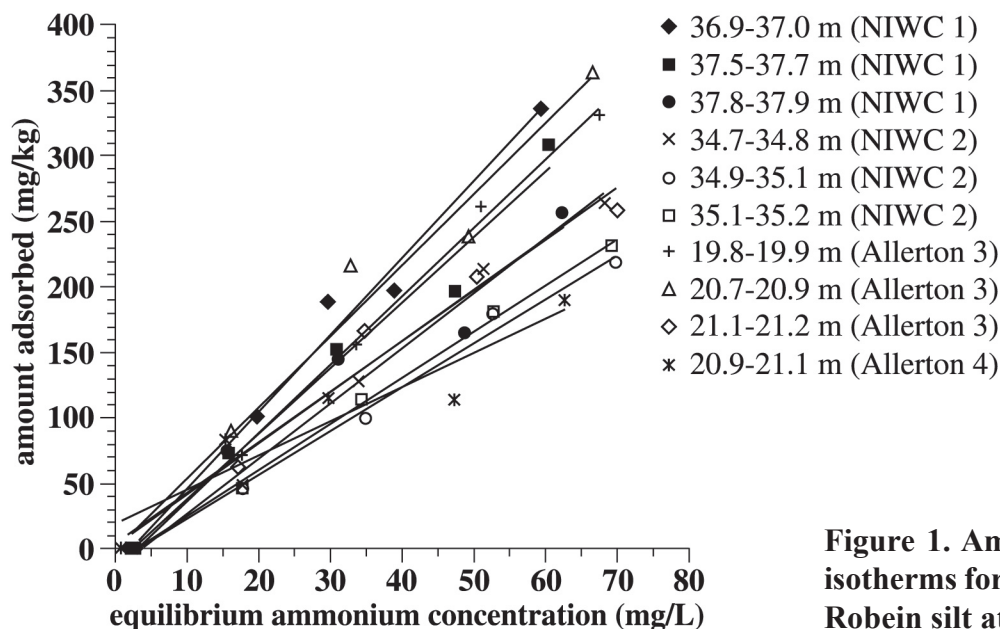


Figure 1. Ammonium sorption isotherms for ten samples of the Robein silt at 23° C.

Table 5. Summary of the buried soil samples from the Peoria Core (SPBY0202).

Soil	Depth Interval (m)	Description
Morton Silt (loess)	54.3 to 54.4	Silt to silt loam. Very dark grayish brown (2.5Y3/2 (wet), 5Y5/3 (dry)). Calcareous. Scattered organic material. 1.19% organic carbon. Fine silt and clay beds.
Morton Silt (mixed with Robein silt)	56.1 to 56.2	Silt to silt loam Very dark grayish brown (10YR3/2 (wet), 10YR5/2 (dry)). Calcareous. 3.37% organic carbon. Scattered wood fragments. Diffuse bedding. Crumbly structure.
Robein silt	56.5 to 56.6	Silt to silt loam Very dark grayish brown (10YR 5/3 (wet), 10YR 5/2 (dry)). 3.32% organic carbon. Scattered wood fragments. Slightly inclined bedding.
Robein silt	57.0 to 57.1	Peat. Woody silt loam. Very dark brown (10Y2/2 (wet), 10YR5/2 (dry)). More than 15% woody fibrous material. Bedding due to parallel-lying material. 4.84.% organic carbon.
Robein silt	57.2 to 57.3	Peat. Peaty silt loam. Black (10Y2/1 (wet), 10YR4/1 (dry)). More than 15% woody fibrous material. Bedding due to parallel-lying material. 8.33% organic carbon
Robein silt	57.4 to 57.5	Peat. Peaty silt loam. Black (10Y2/1 (wet), 10YR4/1 (dry)). More than 15% woody fibrous material. Bedding due to parallel-lying material. 17.2% organic carbon
Robein silt	57.6 to 57.8	Peat. Peaty silt loam. Black (10Y2/1 (wet), 10YR4/1 (dry)). More than 15% woody fibrous material. Bedding due to parallel-lying material. 8.08% organic carbon
Loess below Robein silt	58.4 to 58.5	Silt to silt clay. Very dark brown (10YR2/2 (wet), 10YR5/2(dry)). 1.32% organic carbon.
Sangamon (A horizon)	59.5 to 59.6	Clay loam to silty clay loam. Very dark grayish brown (2.5Y3/2 (wet), 5Y5/1 (dry)). Scattered dolomite gravel and organic matter. Iron staining along fractures. 0.49% organic carbon.
Sangamon (B horizon)	60.3 to 60.4	Sandy loam. Olive (5Y4/3 (wet), 5Y4/2 (dry)). Calcareous. Significant amount of gravel and pebbles. Oxidized. 0.47% organic carbon.
Sangamon (C horizon)	60.8 to 60.9	Sandy loam. Olive gray (5Y4/2 (wet), 5Y6/1 (dry)). Significant amount of gravel with pebbles and cobbles. Pebbles include granites, weathered shale, chert. Thin beds of sandy material. Oxidized. 0.11% organic carbon.
Robein silt	20.9 to 21.1	As above. Slightly sandy. 0.32% organic carbon. pH 8.6
Unidentified pre-Robein soil/ weathered zone	29.6 to 28.7	Silt. Grayish brown (10YR5/2). Slightly calcareous. pH 7.3

Table 6. Summary of the ammonium and alkali-labile nitrogen content of the buried soil samples.

Soil	Core	Depth Interval (m)	Water soluble ammonium (mg NH ₄ ⁺ /kg)	Exchangeable plus water soluble ammonium (mg NH ₄ ⁺ /kg)	Alkali-labile nitrogen (mg N/kg)
Robein silt	NIWC 1	36.9 to 37.0	82.9	149	272
Robein silt	NIWC 1	37.5 to 37.7	72.5	114	208
Robein silt	NIWC 1	37.8 to 37.9	45.6	59.8	99
Robein silt	NIWC 2	34.7 to 34.8	52.3	75.3	176
Robein silt	NIWC 2	34.9 to 35.1	68.7	109	267
Robein silt	NIWC 2	35.1 to 35.2	58.4	87.8	173
Sangamon profile	NIWC 2	36.4 to 36.5	52.4	92.1	NA ¹
Robein silt	Allerton 3	19.8 to 19.9	59.0	87.5	NA
Robein silt	Allerton 3	20.7 to 20.9	63.2	80.4	NA
Robein silt	Allerton 3	21.1 to 21.2	67.5	93.0	NA
Robein silt	Allerton 4	18.7 to 18.8	4.24	8.23	NA
Robein silt	Allerton 4	20.9 to 21.1	2.84	5.75	NA
Unidentified pre-Robein soil/ weathered zone	Allerton 4	29.6 to 28.7	11.0	11.2	NA

¹Not yet available.

Table 7. Summary of the ammonium sorption data for the buried soil samples.

Soil	Core	Depth Interval (m)	Sorption constant, K _d (L/kg)	Correlation coefficient (r ²)
Robein silt	NIWC 2	34.7 to 34.8	3.35	0.991
Robein silt	NIWC 2	34.9 to 35.1	4.20	0.993
Robein silt	NIWC 2	35.1 to 35.2	3.68	0.990
Robein silt	Allerton 3	19.8 to 19.9	5.20	0.997
Robein silt	Allerton 3	20.7 to 20.9	3.90	0.970

Conclusions and Future Research

Enhanced (>2 mg/L) concentrations of ammonium were detected in groundwater samples collected from wells located in two study areas. However, there was insufficient evidence to link the ammonium in the samples with the Robein silt. The open interval of each well was stratigraphically below the buried soil, and the existence of a hydrogeologic connection between the groundwater in the buried soil and the aquifers below remains unknown. Additional water samples will be collected in the two study areas. There may be limitations, however, in interpreting the results because well samples of groundwater in the aquifers do not specifically represent the chemical composition of the *in situ* soil water in the Robein silt. The chemical composition of the soil water could be subject to groundwater dilution, sorption, and a number of biogeochemical reactions that would alter the distribution of ammonium while it is being transported to the open interval of the well.

The Robein silt varied from a gray, silty material with only sparse fragments of organic matter to a peat soil containing 17.2% organic carbon. Based on the results of the first year of this project, the Robein silt *is* a potential source of ammonium in groundwater via dissolution, ion exchange, and mineralization reactions. Moreover, the Robein silt can store ammonium via sorption. Preliminary results suggest that a sorption mechanism (ion exchange) can account for the distribution of ammonium between the solid and liquid phases. On-going research will include the soil physical chemical characterization of recently collected soil samples, and the capacity of the Robein for mineralization (Eq. 1).

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