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Final
Laboratory Testing of Zeolitic Materials

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LABORATORY TESTING OF ZEOLITIC MATERIALS
Final Report: December 15, 2010

INTRODUCTION

This report summarizes recent results from laboratory experiments performed as part of the study entitled "Laboratory testing of zeolitic materials," under West Valley Environmental Services (WVES) Purchase Order 19-001849-C-JK. The report includes updated results from completed column experiments conducted at the University at Buffalo (UB), as well as cation exchange analysis tests conducted during the construction of the installed PTW (approximately July through November, 2010). This report supplements previous reports (Rabideau et al., 2009a; Rabideau et al., 2009b; Rabideau and Seneca 2010), with a focus on work completed between June and December, 2010.

The UB column experiments and CEC tests were performed by doctoral candidate Ms. Shannon Seneca at the Jarvis Hall Laboratory, under the direction of Dr. Alan J. Rabideau. Additional experiments were conducted at the WVDP site by WVES personnel under the direction of Mr. Michael Pendl and Mr. David Scalise, as reported previously by Rabideau and Seneca (2010). For both studies, outside laboratories were utilized as noted to perform chemical analyses of water samples. The objectives and procedures described in this report are referenced to the approved Quality Assurance Project Plan (QAPP) of February 11, 2009 (Rabideau et al., 2009a). A DOE-WVDP surveillance reviewed adherence to the QAPP requirements and WVES oversight of the testing program. WVES responded to all surveillance findings and comments as documented by Biedermann (2009).

METHODS

Materials

Zeolite test materials were obtained directly from the suppliers: (1) Teague Mineral Products (TMP) of Adrian OR, and (2) Bear River Zeolite (BR) of Preston ID. Because these natural materials are not pure minerals, they are commonly referred to as "zeolite-rich rock." However, for the purpose of this report, the simpler terms "zeolite" or "natural zeolite" are used. For both TMP and BR materials, the requested particle size was 14x40, which corresponds to standard sieve openings of 1.4 and 0.425 mm, respectively. Other properties of the TMP and BR zeolites used in the testing program were reported previously by Rabideau et al. (2009b).

Several of the column experiments were conducted with mixtures of material that included WVDP soil collected from a radiologically non-contaminated area during field characterization performed in fall 2008. Visual inspection indicated that the soil was comprised generally of silty-clay with a soft brown color and silky texture.

The soil was first air-dried overnight, and then sieved to remove particles larger than 1.18 mm. Because the soil was available in limited quantity, further characterization was not performed.

During the period that zeolite was produced for the PTW (approximately May through September, 2010), the UB team performed additional cation exchange testing of PTW construction materials, based on samples provided by Bear River Zeolite, which were designated with Lot numbers that referenced the location and timing of the zeolite mining. The purpose of these tests was to verify that the installed PTW zeolite was consistent with the material used for the batch and column experiments. Typically, CEC tests were performed on replicated Lot samples and a control sample obtained from archived material from the same initial batch that was used throughout the UB experimental program.

CEC testing

The most important zeolite property with respect to removal of Sr^{90} is its cation exchange capacity (CEC), typically expressed in *milliequivalents of exchange capacity per gram of solid* (meq/g). Although standardized washing procedures exist for measuring CEC in soils, for high-capacity materials (such as zeolite) these procedures may underestimate the overall CEC because the washing process is a limited-duration approximation of more sustained exposure to a competitive cation exchange environment. Furthermore, standard CEC procedures quantify the overall capacity but do not indicate the concentrations of individual sorbed cations, which are needed to support solute-specific transport modeling. Thus, for this study, a modified procedure was developed to provide the needed information. The UB method was loosely based on a study by Cerri et al. (2002), which evaluated alternative procedures specifically for application to clinoptilolite-rich rock similar to the materials under investigation; a detailed summary of the washing procedure was given in Rabideau et al. (2009). As discussed below, minor procedural modifications were implemented during the PTW construction phase to reduce the processing time.

Analysis of water samples

The CEC and column test procedures required the analysis of aqueous samples for dissolved cations (Ca^{2+}, Mg^{2+}, Na^+, K^+ and Sr^{2+}). Samples for the UB columns were shipped to Columbia Analytical Services (CAS, Rochester, NY) for analysis by Inductively Coupled Plasma (ICP, EPA Method 200.7). Aqueous sample handling and analytical procedures are summarized in the CAS Quality Assurance Manual, which was included in the QAPP. For the WVDP columns (reported previously), nonradioactive cation analyses were performed by GEL Laboratories LLC (Charleston, SC) using the same method (ICP, EPA Method 200.7), while analyses of aqueous samples for Sr^{90} were performed by WVES personnel as described in Rabideau and Seneca (2010).

Detection limits for the laboratories were summarized previously in Rabideau and Seneca (2010). For the UB column tests, some early-period Sr^{2+} effluent concentrations were observed to fall between the Method Detection Limit (10 ppb) and the Reporting Limit (100 ppb), indicating reduced precision. However, for the purpose of this report, all concentrations are shown without adjustment.

Column Testing

For the UB experiments, ten experimental columns were operated beginning in February 2009, with various durations as summarized in Table 1. Columns denoted UB1, UB2, etc., correspond to the labels C1, C2, etc., that were reported previously by Rabideau et al. (2009b). For the West Valley experiments, six experimental columns were operated beginning in May 2009 (Table 1, denoted WV1, WV2, etc.). Important differences between UB and WV experimental conditions include the use of on-site influent groundwater containing radioactive $Sr^{90}$for the WV column, and variations in column media compositions. Two of the WV columns (WV3, WV4) contained an unrealistically high fraction (50 percent) of WVDP soil; these columns were terminated after approximately 175 days of operation.

Detailed descriptions and photographs of the experimental setup were included in Rabideau et al. (2009b). Both the UB and WVDP columns were constructed of Plexiglas. Each column was approximately 10 cm in length and 3.81 cm in diameter, with column dimensions selected to lessen the potential for substantive wall effects (e.g., Mehta and Hawley, 1969) and structural dispersive mixing (Li et al., 2009). Each column was equipped with a water inlet at the base of the column and a built-in diffuser to evenly distribute the flow of incoming water. Sampling ports located along the column length were not used in this study for aqueous sampling, but facilitated removal of column solids at the end of selected experiments. Further details of the column experimental procedures are provided in the project QAPP. Key experimental design components included:

- At both experimental sites, multi-channel peristaltic pumps were utilized to provide steady flow to the columns, with frequent gravimetric flow measurements used to support minor adjustment (if necessary) to the pump settings. The design flow rate of 0.2 ml/min was selected based on natural groundwater flow conditions observed at the site. The corresponding design Darcy velocity (flow per area) was approximately 0.23 m/day. Based on the results of previous work, this velocity, while near the high end of anticipated field conditions, was considered low enough such that the cation exchange process would achieve local geochemical equilibrium.
- For the UB tests, cation concentrations in the synthetic groundwater (Table 2) were prepared to represent the average concentrations observed in WVDP wells, with the exception of Sr^{2+}, which was increased to the approximate maximum observed value of 1 mg/L.

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After approximately 100 days of column operation, the source concentrations of Ca^{2+} and Na^{+} were increased to enhance the sensitivity of transport model parameters to the effluent data. For the longer running columns, the influent concentrations were subsequently returned to their initial value after approximately 155 days.

- WVDP test procedures were based on and thus similar to the UB experiments, except that the source groundwater was obtained from a well located on the WVDP North Plateau in the vicinity of the proposed PTW. The concentrations of relevant groundwater cations were similar to the UB study (Table 2), but the source water also contained Sr^{90} at an activity of approximately 30,000 to 40,000 pCi/L. The influent groundwater was used as received throughout the experiments.

RESULTS

Column tests: Interpretive Issues

The interpretation presented in this report is primarily graphical and qualitative, as summarized in the plots of column effluent concentrations presented in Figures 1-4. Preliminary modeling of the UB columns was reported previously by Rabideau et al. (2009b); refinement of mathematical models is ongoing and will be documented in a series of journal articles prepared in conjunction with Ms. Shannon Seneca's doctoral dissertation. The primary addition addressed in this report is the presentation of the final sorbed phase data, which were obtained after termination of the UB experiments. For all of the column effluent Figures, concentrations of Sr^{2+} were plotted as μg/L to enhance visual clarity; concentrations of other nonradioactive cations were plotted as mg/L. Data from the WV columns were reported previously in Rabideau and Seneca (2010) and are not repeated here.

An approximate characterization of potential zeolite field behavior can be obtained by converting elapsed time (days) to dimensionless "pore volumes" (PVs) based on the measured or estimated flow rates and the flow-through length (or thickness) of the zeolite zone. However, such comparisons depend on an estimated effective porosity, which varies for different media and column/field emplacement, as well as the estimated groundwater flow conditions in the field, which will vary spatially. For this report, the following assumptions are adopted for illustrative purposes: (1) an effective porosity of 0.5 is assumed for 100% zeolite systems (both TMP and BR, (2) the average column flow per area was quantified at 0.23 m/day as described above, and (3) typical field velocities are assumed to fall within the range of 0.5 to 2 ft/day, (4) the installed PTW is approximately 3 feet thick. These assumptions translate into an estimated normalized groundwater velocity of 4.6 PV/day for laboratory columns and 0.2-0.7 PV/day for field conditions.

Based on these ratios, the results from the longest running UB columns containing 100 percent zeolite (UB3) (606-610 days reported here) represent approximately 13-38 years of field operation, while the WV zeolite columns (320 days reported previously) represent approximately 6-20 years of field operation.

Columns containing soil mixtures are expected to have a much lower effective porosity, on the order of half of the pure zeolite value (0.25) due to the presence of smaller particles; these columns therefore generate a higher number of pore volumes per day of operation. However, because the complete mixing of soil/zeolite is unlikely to occur in the field, extrapolation of the mixture columns is not considered representative. Also, it is likely that the TMP columns could exhibit a slightly smaller effective porosity because of the higher fraction of fine particles; however, such differences are expected to have a minor influence on performance and data were insufficient to support a more refined interpretation.

Although the column zeolite conditions were similar, the UB and WV columns had different influent concentrations, as summarized in Table 2. Of particular importance are differences in Na^+, Ca^{2+}, and Sr^{2+}. Also, UB influent conditions were adjusted during the experiment by increasing and decreasing the Na^+ and Ca^{2+} concentrations. The overall effect of these differences on column behavior is difficult to characterize, but the UB columns probably represent a more conservative representation of field conditions in terms of cation competition for exchange sites. In general, caution should be exercised in extrapolating column conditions to the field.

General Observations

Results from the shorter-duration UB column tests (UB2, UB4, UB6, UB8, UB9, and UB10) were discussed previously in Rabideau et al. (2009b), and results for all WV columns were discussed in Rabideau and Seneca (2010). Results for the >600-day UB columns (UB1, UB3, UB5, and UB8) are summarized in Figures 1-4. It is noted that these reports provide only a few additional effluent data points relative to the previous report (Rabideau and Seneca, 2010). As such, an abbreviated discussion is provided. Data from the analysis of the column solids is presented in Figures 5-8, along with data previous reported for the 142-day columns in Rabideau et al. (2009b).

Throughout the entire duration of the study, both the UB and WV effluent data were consistent with a general conceptual model for PTW behavior that recognizes three distinct “stages” (trends) of effluent cation concentrations: (1) early-time dynamic behavior in which a significant exchange of groundwater and zeolite cations takes place, as expressed by crossing effluent curves influenced by the initial condition of the BR or TMP zeolite, (2) a “quasi-plateau” period in which effluent cation concentrations are similar to the influent concentrations, with the exception of Sr^{2+} and Sr^{90}, which continue to be removed by the zeolite, and

(3) a final equilibration period in which Sr^{2+} and Sr^{90} concentrations increase from the quasi-plateau level to the concentration of the influent groundwater. From a performance standpoint, the functional life of a zeolite PTW will be reached sometime during the third stage of operation, depending on the location of interest, source concentration, and target concentration.

A previous project report (Rabideau et al., 2009b) covered the first six months of the UB column operation, which corresponded to the completion of the first stage of dynamic ion exchange behavior and an extended period of the second stage "quasi-plateau" behavior. During the longer period of operation covered in Rabideau and Seneca (2010), the UB columns and some of the WVDP columns began to exhibit the expected third stage increase in effluent Sr concentrations. The additional data included in this report continue the observed trend in the UB columns. None of the columns, however, have reached the point of "full breakthrough" in which Sr effluent concentrations reach the influent levels. Additional discussion of column behavior was provided previously by Rabideau and Seneca (2010).

Analysis of column solids

After operation of the UB columns was terminated, the zeolite solids were sectioned and analyzed for sorbed cations, as summarized in Figures 5-6. Because of the large number of column samples, only segments from the 100% zeolite columns were analyzed. For both TMP and BRZ materials, comparison of the plots for the 142-day columns with the >600-day columns indicated behavior consistent with the competitive cation exchange conceptual model described above. In general, early time behavior (< 100 days) was dominated by the complex exchange of multiple aqueous bivalent cations for sorbed monovalent cations, while late time behavior was dominated by the more limited exchange of aqueous Sr^{2+} for sorbed K^+, with the other cations at approximate equilibrium. For the 142-day columns, the sorbed potassium concentration at the end of the column were discernibly higher that at the column beginning, indicating that K^+ was still being displaced by Sr^{2+}, which exhibited higher concentrations near the beginning of the column. However, at the end of the >600-day experiments, the spatial distributions for both cations were nearly flat, indicating that the solids were close to equilibrium with the influent water.

Cation exchange testing

During zeolite production for the PTW project (May through September, 2010) approximately 32 samples of the same BRZ materials used for the column experiments (designated UB-BR) were analyzed for cation exchange capacity (CEC), using variations of the procedure summarized in Rabideau et al. (2009b).

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The primary procedural differences for these quality assurance samples were in minor adjustments to the wash durations to reduce the turnaround time. The average measured CEC for the UB-BR samples was 1.54 meq/g, which was consistent with previous work as reported by Rabideau et al. (2009b).

The purpose of the additional CEC testing was to provide side-by-side comparisons with samples of new BRZ material that were being mined for use in the PTW.

These samples represented 45 Lots of material. Additional samples (Lots 45-50) were not analyzed by the UB lab because of the need for fast turnaround, but results provided by BRZ were reviewed by the project team. The comparison of new BR results with side-by-side tests of the archived UB-BR material, combined with other information (visual inspection, moisture content, CEC trends), served as the basis for acceptance or rejection of individual Lots for the purpose of the PTW.

As the production of new BR zeolite advanced, it became clear that the relatively lengthy UB CEC procedure (3-week duration) could impede the process of evaluating newly mined materials in a timely fashion. Consequently, several variations on the established procedure were evaluated, and several additional zeolite samples were analyzed by Hazen Research Inc. (Golden, CO) to aid in the evaluation. Also, some of the Lot samples were also analyzed by BRZ personnel and/or contract laboratories. Key observations from this process included the following:

- Comparisons between UB analysis and Hazen results indicated that the UB procedure yielded similar slightly higher CEC values (average of 1.54 for many samples versus average of 1.46 meq/g for four Hazen samples), which was attributed to the more thorough UB washing procedure.
- Because the air drying of the zeolite material accounted for a considerable portion of the test duration (up to 3 days), it is recommended that future QA/QC work utilize oven-drying (e.g., 1-2 hours at 103 degree Celsius). However, additional preliminary work is needed to characterize the degree of variability introduced by oven-drying, which was observed to produce slightly less consistent results compared with the three-day air drying procedure used for most of the UB tests.
- Although considerable discussion of QA test results focused on the issue of moisture content, repeated tests indicate the initial zeolite moisture has a very minor influence on the final calculated CEC, regardless of the particular drying procedures used at various stages of the test. The lack of sensitivity reflects the fact that the post-drying moisture content (which serves as the basis for CEC calculations) is typically small and less variable than the as-received moisture content, regardless of how drying is conducted.

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In general, future PTW projects would benefit from the development of a modified CEC test procedure designed to facilitate rapid side-by-side comparisons of candidate natural zeolite materials with a reference material. The UB procedure, while rigorous, is probably unsuitable for this purpose because it was designed to quantify the concentrations of individual sorbed cations (in addition to total CEC) and mimic the extended equilibration times that would be experienced by zeolite emplaced in a PTW.

SUMMARY

The column experiments described in this and previous reports represent a substantial expansion of the body of data available to assess the potential of natural zeolites to remove Sr in a PTW system. In particular, the extended duration of the WV and UB columns (142 to >600 days) relative to previously reported results (10 to 180 days in Rabideau et al., 2005) has yielded the first observations of "third stage" behavior in which effluent Sr begins to increase from a low-concentration plateau toward the influent level. Also, the WV columns (reported in Rabideau and Seneca, 2010) provided the first experimental results confirming the removal of Sr^{90} by natural zeolite; all previous work has utilized surrogate Sr compounds. As expected, columns containing zeolite soil mixtures exhibited earlier and more significant increases in effluent Sr concentrations relative to pure zeolite columns, but the column conditions are not considered representative of field PTW conditions.

Of the results discussed in this and previous reports, the most relevant data are for the columns containing 100 percent BRZ material (UB3, UB4, WV5). These results confirm previous observations that the BRZ zeolite will remove both radioactive and nonradioactive Sr by cation exchange over extended periods of operation, following a three stage conceptual model. For the 610-day UB3 (BRZ) column, a gradual increase in effluent Sr^{2+} did not begin until approximately 190 days (approximately 960 pore volumes) of operation, and the effluent concentration did not reach 50 percent of the influent until approximately 450 days (2400 PV). At the end of the 610-day operation (2806 PV), the final Sr^{2+} concentration was approximately 65 percent of the influent value. For the WV column (WV5), significant increases in effluent Sr (radioactive and nonradioactive) were not exhibited during the entire 320 day (1472 PV) experimental period included in this report.

Comparisons between BRZ and TMP zeolites indicated similar conformance with the three-stage conceptual model described above. Differences between the column plots were relatively minor, with the TMP columns exhibiting slightly higher Sr^{2+} concentrations near the end of the 606 days. However, the interpretation of this difference is hindered by the uncertainty regarding the precise value of the effective porosity for each material, which is needed to define the pore volume equivalent of elapsed time; additional testing related to effective porosity is ongoing as part of Ms. Seneca's UB dissertation research.

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As discussed in previous reports, the BRZ material is preferred from a performance perspective due to its slightly higher CEC, the more friable nature of the TMP material, and the presence of a clay mineral in the non-zeolite portion of TMP material.

The large number and diverse nature of the column data have provided a rich data set to develop a more detailed modeling interpretation, which is the focus of Ms. Shannon Seneca's doctoral dissertation (to be completed in late 2011). In conjunction with Ms. Seneca's dissertation, the results of this study will support the development of three-five new journal articles that summarize the entire performance testing program and the development of refined mathematical models (e.g., similar to Rabideau et al., 2005).

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Table 1: Scope of Column Experiments

Column	Description (dup. = duplicate)	Duration (days)	Samples (* = not analyzed)
University at Buffalo columns			
UB1	TMP zeolite only	606	64 effluent, 8 zeolite segments
UB2	TMP zeolite only (dup.UB1)	142	31 effluent, 8 zeolite segments
UB3	BR zeolite only	610	64 effluent, 8 zeolite segments
UB4	BR zeolite only (dup. UB3)	142	31 effluent, 8 zeolite segments
UB5	80% TMP zeolite, 20% WVDP soil	606	64 effluent, 8* zeolite segments
UB6	80% TMP zeolite, 20% WVDP soil (dup. UB5)	142	31 effluent, 8* zeolite segments
UB7	80% BR zeolite, 20% WVDP soil (dup. UB8)	142	31 effluent, 8* zeolite segments
UB8	80% BR zeolite, 20% WVDP soil	610	64 effluent, 8* zeolite segments
UB9	WVDP soil only	131	28 effluent
UB10	70% BR zeolite, 20% WVDP soil, 10% iron	131	28 effluent
West Valley columns			
WV1	80% BR zeolite, 20% WVDP soil	370	39 effluent
WV2	80% BR zeolite, 20% WVDP soil (dup. WV1)	370	39 effluent
WV3	50% BR zeolite, 50% WVDP soil	175	32 effluent
WV4	50% BR zeolite, 50% WVDP soil (dup. WV3)	175	32 effluent
WV5	BR zeolite only	370	39 effluent
WV6	TMP zeolite only	370	39 effluent

Table 2: Influent cation concentrations

Cation	Groundwater conc. from 11 on-site wells		UB synthetic groundwater 2/19 – 5/28/09 (mean)	UB synthetic groundwater 5/29 – 8/4/09 (mean)	UB synthetic groundwater after 8/4/09 (mean)	WVDP 5/13/09 – 5/27/10 (mean)
	Mean	Maximum				
Ca^{2+} (mg/L)	167	230	160.8	205.6	164.8	143.4
Mg^{2+}(mg/L)	26	36	26.5	25.4	26.2	22.1
K^{+}(mg/L)	3	6	5.0	5.0	5.2	3.0
Na^{+}(mg/L)	185	273	178.8	217.8	176.6	234.2
Sr^{2+}(mg/L)	0.3	1	1.0	1.0	1.0	0.32
Sr^{90}(pCi/L)						34,088

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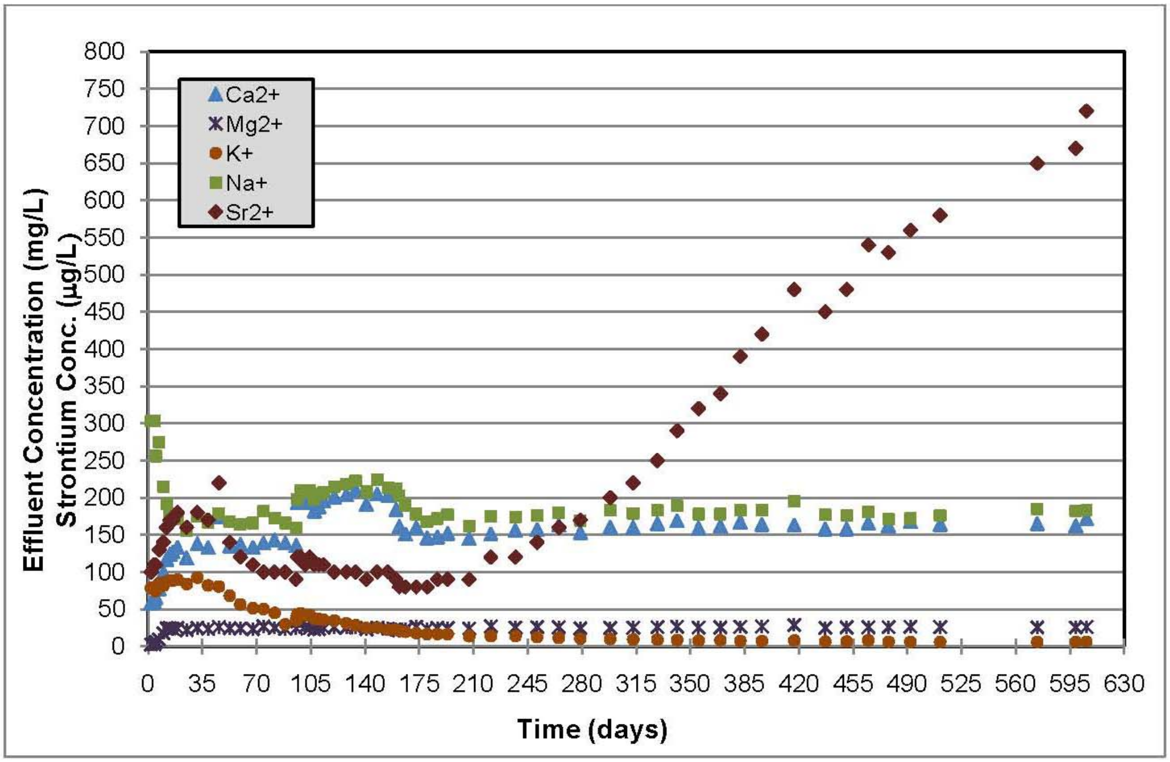
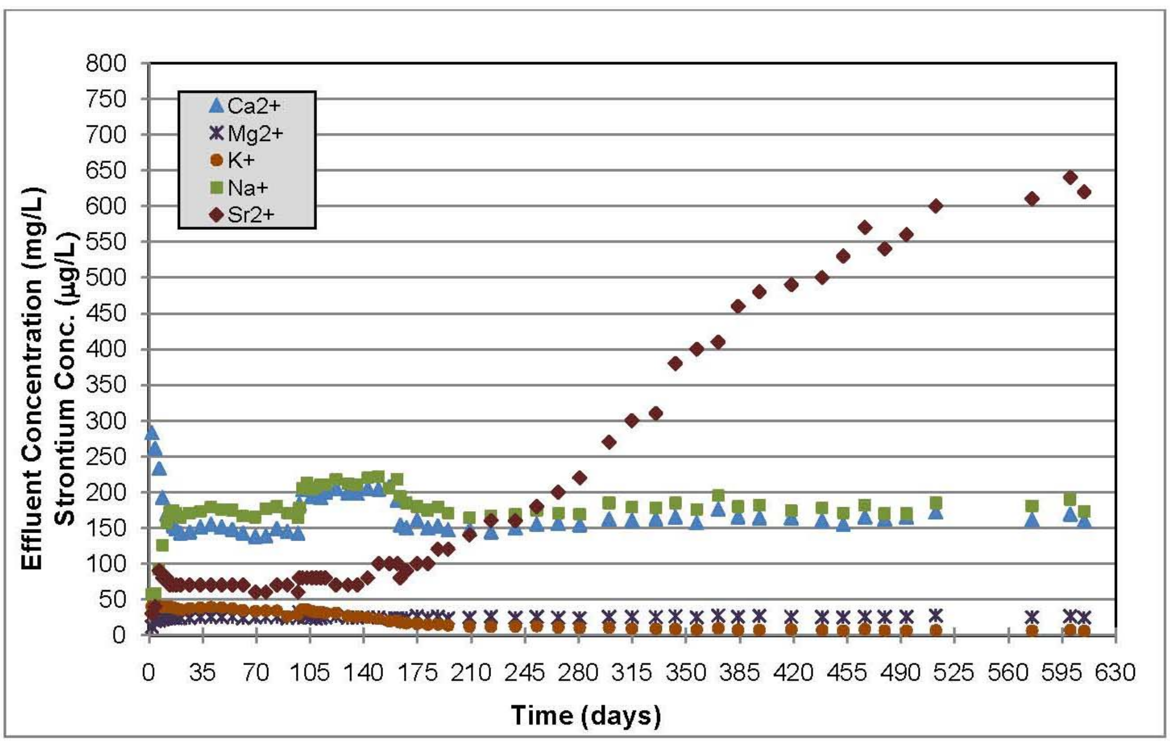


1 Effluent concentrations for Column UB1 (100% TMP zeolite)



2 Effluent concentrations for Column UB1 (100% BR zeolite)

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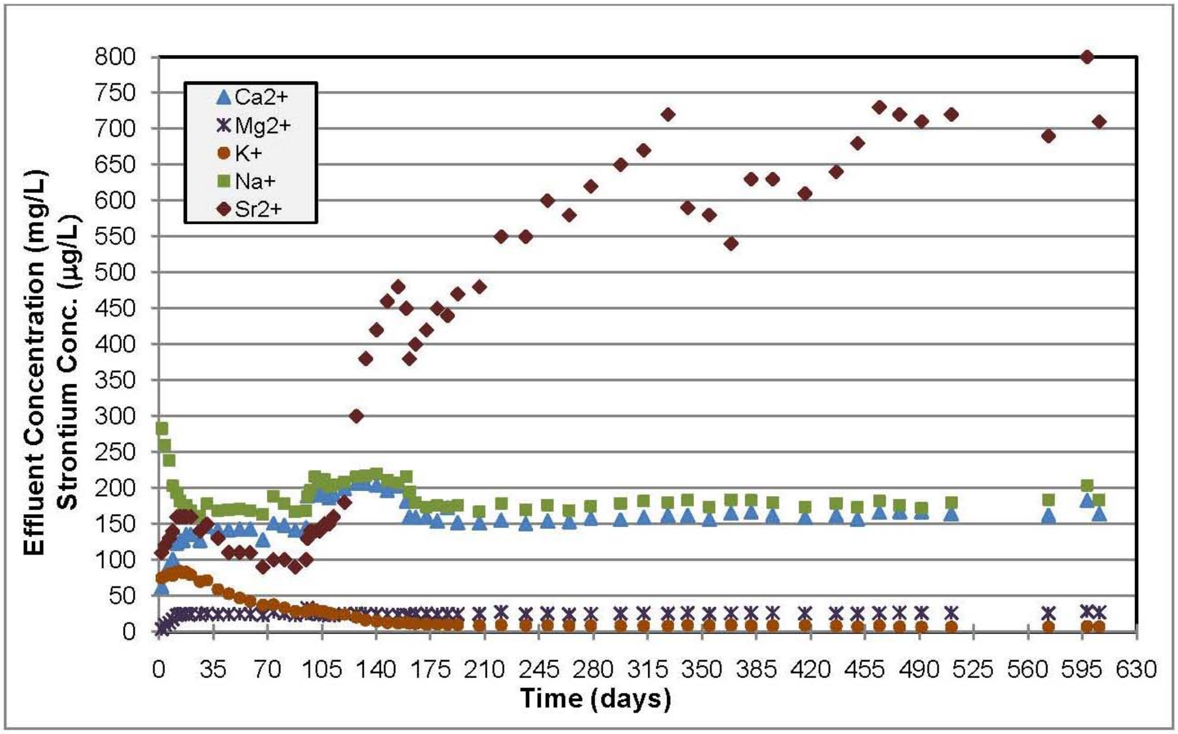
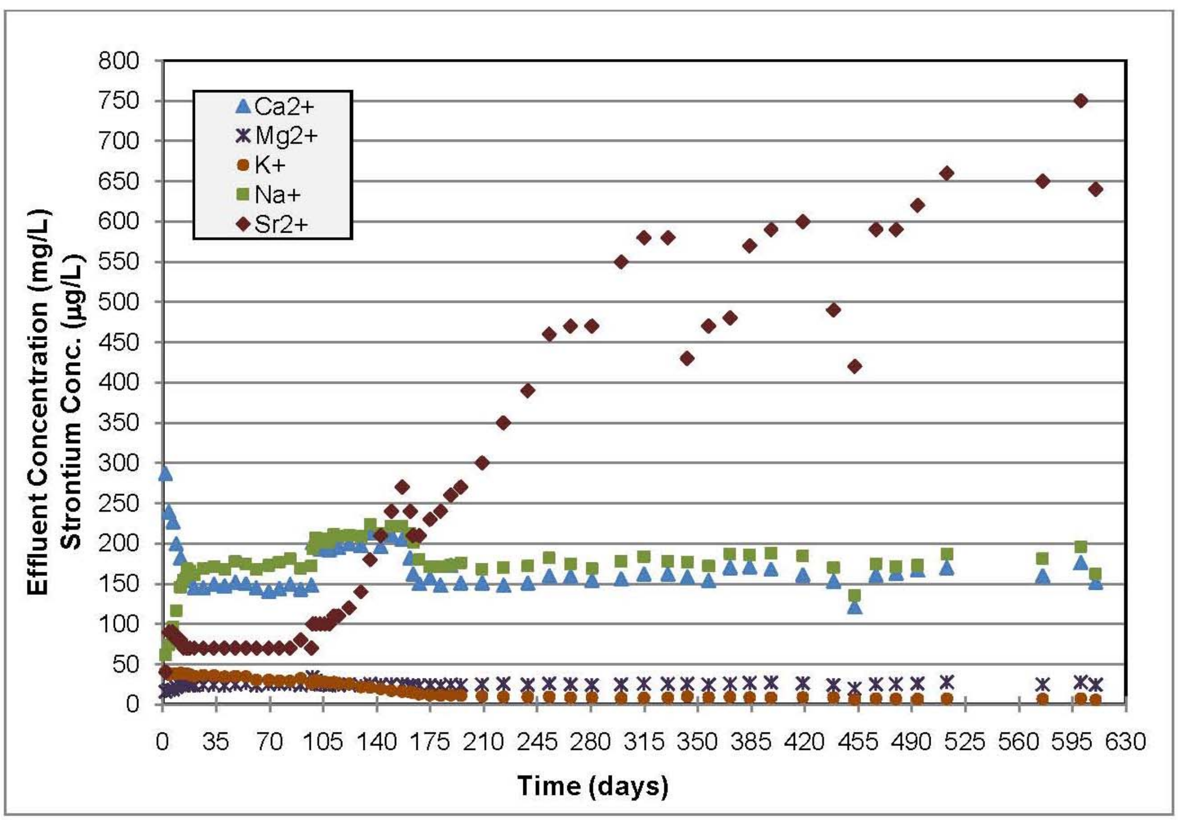


3 Effluent concentrations for Column UB5 (TMP zeolite with 20% WV soil)



4 Effluent concentrations for Column UB5 (BR zeolite with 20% WV soil)

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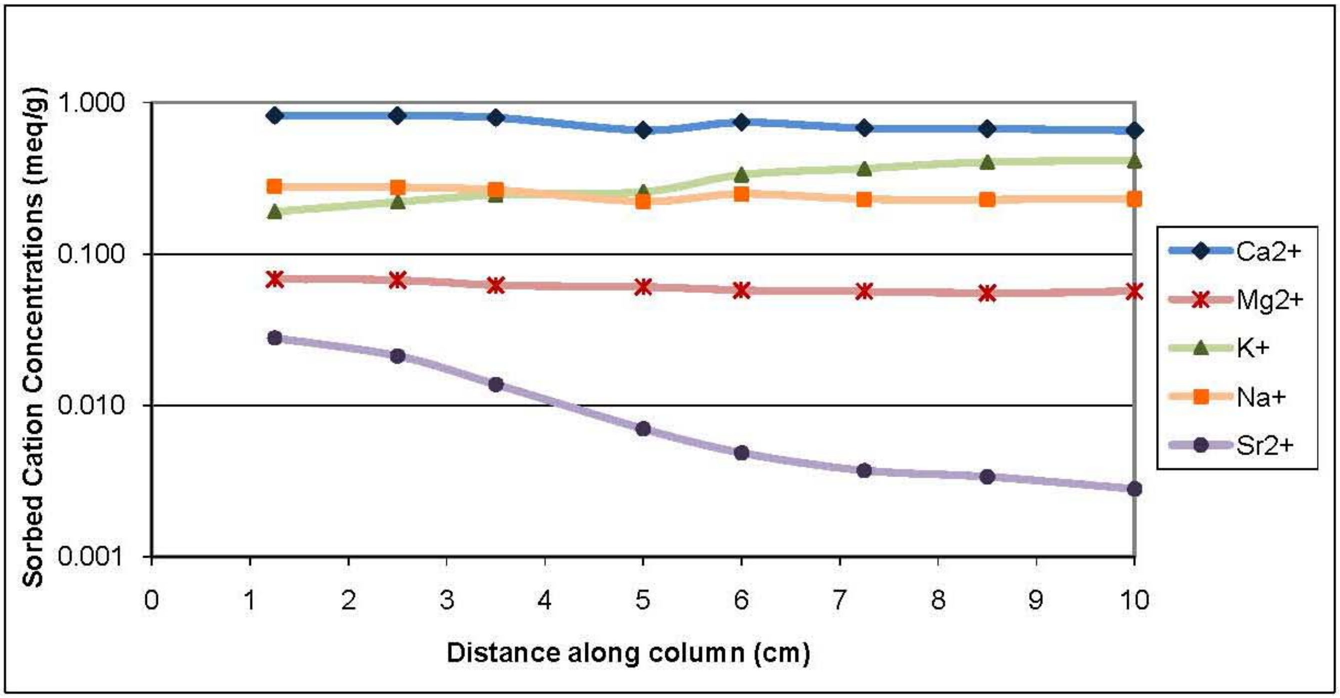
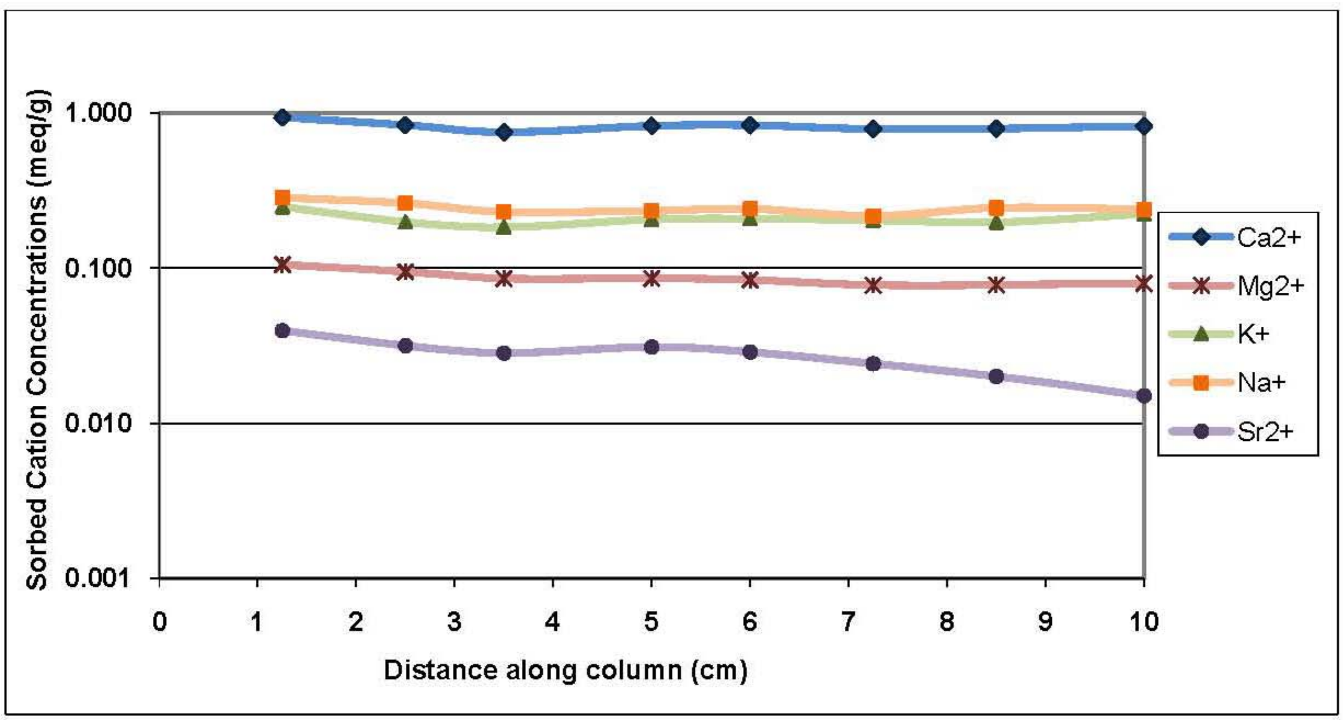


5 Sorbed solute concentrations for Column UB-2 [100% TMP] at 142 days



6 Sorbed solute concentrations for Column UB-1 [100% TMP] at 606 days

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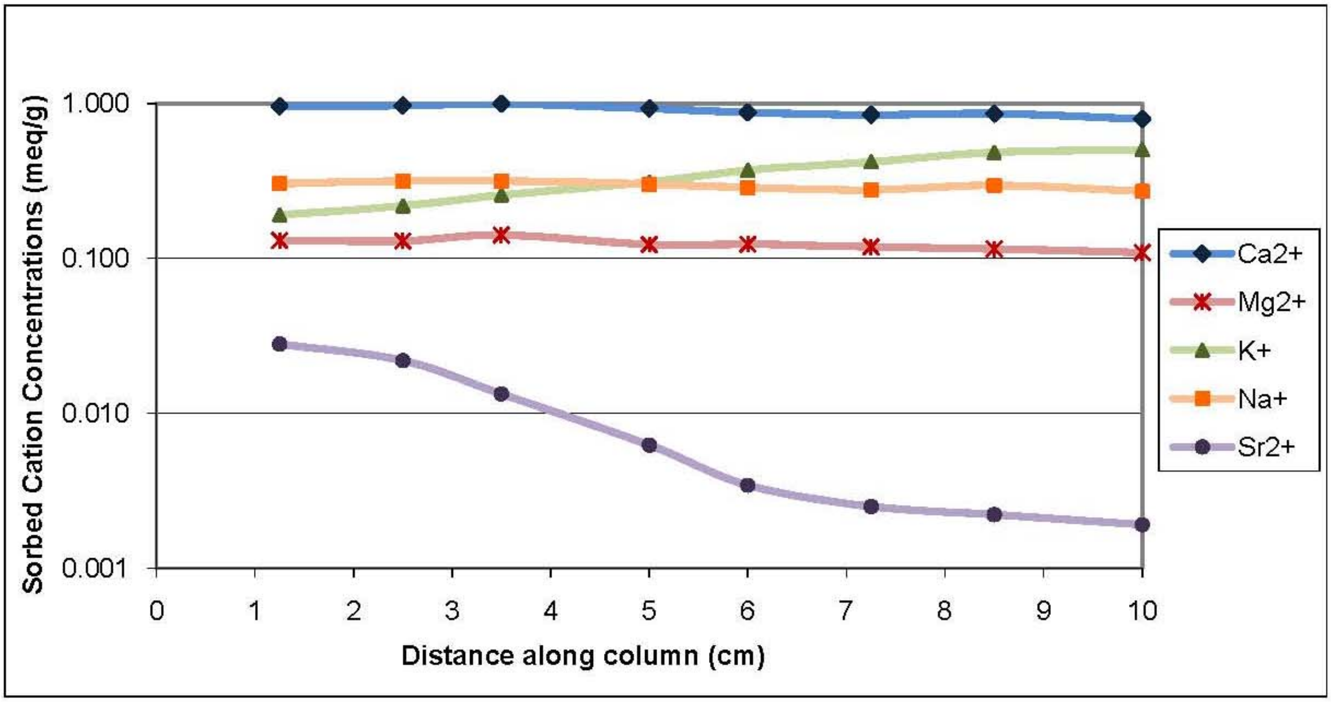
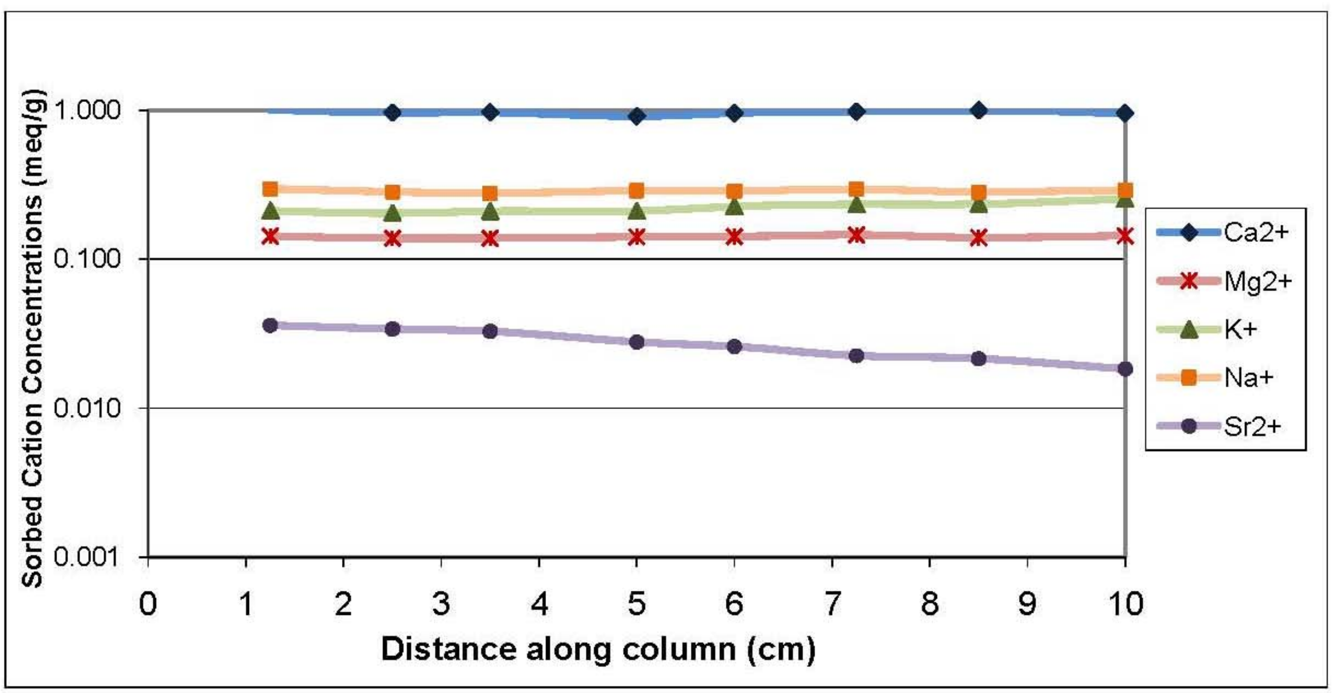


7 Sorbed solute concentrations for Column UB-4 [100% BRZ] at 142 days



8 Sorbed solute concentrations for Column UB-3 [100% BR] at 606 days

WVDP RECORD OF REVISION

Rev. No.	Description of Changes	Revision On Page(s)	Dated
0	Original Issue The State University of New York at Buffalo (UB) Department of Civil, Structural, and Environmental Engineering under contract to WVES conducted the testing of zeolite materials for potential application in mitigation of North Plateau Strontium-90 groundwater contamination.	All	01/27/2011