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COUPLING BETWEEN FLOW AND PRECIPITATION IN HETEROGENEOUS SUBSURFACE

ENVIRONMENTS AND EFFECTS ON CONTAMINANT FATE AND TRANSPORT

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EXECUTIVE SUMMARY

The Environmental Management Science Program (EMSP) project (number 1020913) entitled "Coupling Between Flow and Precipitation In Heterogeneous Subsurface Environments and Effects on Contaminant Fate And Transport," George Redden (Idaho National Laboratory) Principal Investigator, was a multi-institutional collaborative effort among Idaho National Laboratory (INL), Pacific Northwest National Laboratory (PNNL) and University of Idaho (UI). During the performance period of the UI grant, the INL program was reorganized as the "Subsurface Immobilization of Metal Contaminants by Amendment-Driven Mineral Precipitation" Scientific Focus Area (SFA). The ultimate goals of the EMSP project and the continuing INL SFA are to provide a scientific basis for the development of methods of amendment distribution in porous media that leads to sequestration of metal contaminants (e.g. strontium-90), and to determine how macroscopic field-scale modeling can be applied to predict the outcome of remediation activities. Using calcium carbonate as a model system, physical experiments, in addition to modeling at the pore-scale and continuum-scale, were and continue to be used to improve the conceptual approach to predicting the impact of flow-precipitation coupling on solute migration. The University of Idaho has responsibility for physical experiments aimed at characterizing calcium carbonate precipitation kinetics, polymorphs formed, and metal contaminant (i.e., Sr) co-precipitation over a wide range of saturation indices

and ion activity ratios. Results from these experiments support intermediate-scale reactive-transport experiments being conducted at the Idaho National Laboratory.

University of Idaho conducted calcium carbonate and strontium co-precipitation experiments that attempted to maintain conditions of constant solution composition and levels of supersaturation that are likely to be relevant in the implementation of subsurface remediation strategies. These experiments stand in contrast to our previous investigations using microbially drive precipitation in batch experiments in which individual experiments exhibit wide ranges in composition, saturation state, and precipitation (and co-precipitation) rates. The results from this study provide the basis for developing generalized precipitation and co-precipitation kinetic models that explicitly account for the extent of supersaturation and variations in solution compositions. The application of the kinetic models will enable the more effective deployment of DOE relevant subsurface remediation strategies.

COUPLING BETWEEN FLOW AND PRECIPITATION IN HETEROGENEOUS SUBSURFACE ENVIRONMENTS AND EFFECTS ON CONTAMINANT FATE AND TRANSPORT

INTRODUCTION

The U.S. Department of Energy (DOE) is committed to aggressively cleaning up the environmental legacy of nuclear weapon and civilian nuclear research and development programs at numerous sites throughout the United States. In total, these sites represent millions of cubic meters of contaminated media with recurring contaminants. To meet this challenge, better remediation technologies, long-term stewardship options, and alternate cleanup approaches are required. One engineering approach growing in acceptance by DOE and regulators is containment and stabilization of contaminants by precipitation of co-precipitation in the subsurface.

Engineered remediation strategies for inducing mineral precipitation in the subsurface typically involve the introduction of at least one reactant either by direct injection or by *in situ* generation[1]. The localization of reactant sources means a wide range of saturation states and ion ratios will be created as reactants are mixed: These conditions together can result in a wide range of precipitation rates, as well as impact which mineral phase precipitates. This is potentially important for the capacity of the precipitates to take up of trace metal contaminants, for their long term stability. Aragonite, for example, is able to sequester a larger amount of Sr than calcite. However, aragonite is less stable under typical groundwater conditions, and so may release sequestered Sr over time as the aragonite transforms to a more stable phase. In addition, previous experimental studies (Lebron and Suarez, 1996, 1998) have indicated that other system constituents may influence calcium carbonate precipitation and consequently the Sr uptake potential of a system. For example, dissolved organic carbon (at levels typical of groundwaters) can suppress crystal growth. As a result, the continuous nucleation of small crystals, rather than growth of existing crystals, may be the dominant mode of precipitation. This has the potential for greater uptake of Sr because the smaller crystal sizes associated with nucleated calcite may more readily accommodate the distortion resulting from substitution of the larger Sr ion for Ca

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[1] An example of *in situ* generation that we continue to study is the microbial hydrolysis of urea and the associated precipitation of calcite and co-precipitation of Sr (Fujita et al., 2000; Colwell et al., 2005).

ions than can larger crystals (Fujita et al., 2004). However, these smaller crystals may also be less stable and over the long term release Sr as a result of Ostwald ripening. To better understand the formation and composition of relevant calcium carbonate mineral phases two related series of mineral precipitation experiments were conducted. The first series of experiments, conducted at steady state rates of precipitation was focused on understanding the influence of pH and $(NH_4)_2CO_3$ (the hydrolysis product of urea: ureolytically driven calcium carbonate precipitation has been demonstrated to be a promising method of inducing mineral precipitation in the field) on calcium carbonate polymorph and Sr co-precipitation. The second series of experiments, conducted at constant pH and saturation state, was focused on understanding the influence of aqueous $[CO_3^{2-}]/[Ca^{2+}]$ ratios on the precipitation rate of calcite.

METHODS

Continuously Stirred Tank Reactor Experiments

A Braun Biostat MD bench scale fermentor with a DCU3 control system (Sartorius Group, Allentown, PA) shown in Figure 1a was used as a continuously-stirred, flow-through tank reactor (CSTR). The reaction vessel has an operating volume of 5 liters. A constant volume of fluid was maintained within the reactor using a high level sensor that triggered the outflow or "harvest" by a peristaltic pump to remove fluid. Three peristaltic pumps delivered solutions into the reactor. All pumps were calibrated prior to the start of each experiment per the manufacturer's instructions. The total volume delivered by each of the three inflow pumps during the course of an experimental run was counted by the DCU3 control system. The flow rate of the input pumps were verified daily and adjustments made as necessary to maintain the flow rate within 5% of the target rate. Reactor pH was monitored and maintained
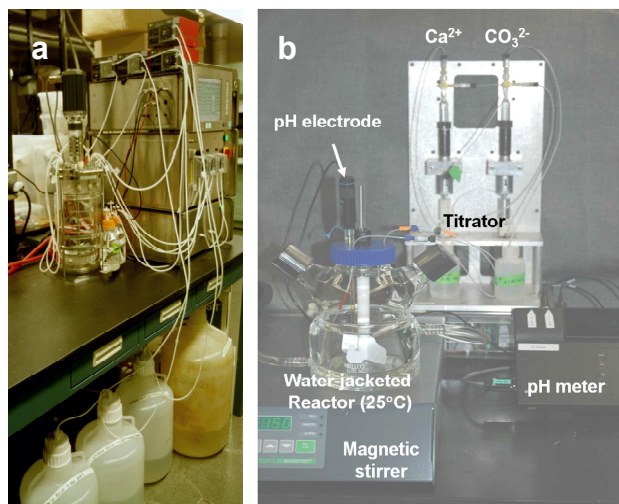
Figure 1. a) Braun Biostat MD fermentor with DCU3 control system configured as a Continuously Stirred Tank Reactor used to conduct steady state calcium carbonate experiments. b) Water jacketed reactor and automated titration system used to conduct ion ratio calcite precipitation experiments.
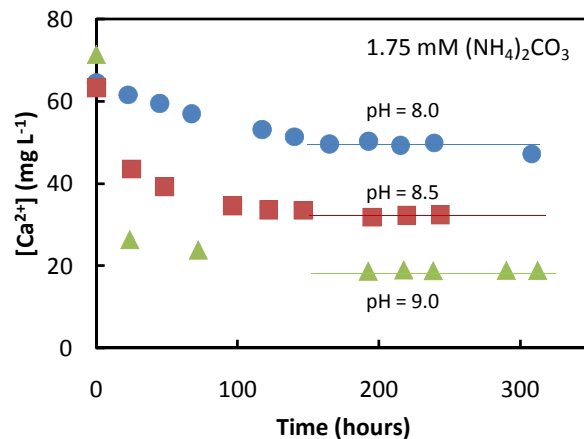
constant at the target level by an automated titration system. Fluid temperature within the reactor was monitored with a thermocouple connected to the DCU3 controller and was 22.5 ± 0.9 °C.

Artificial Ground Water (AGW, Ferris et al., 2004; prepared at 4 times the target reactor concentration), $(NH_4)_2CO_3$, and nanopure water were delivered at a rates of 0.915 ± 0.041 ml min^{-1}, 0.907 ± 0.050 ml min^{-1}, and 1.908 ± 0.121 ml min^{-1}, respectively. The total flow into (and out of) the reactor was 1:1:2, AGW:$(NH_4)_2CO_3$:nanopure water resulting in an overall flow rate of 3.73 ± 0.14 ml min^{-1} with a residence time of 22.34 ± 0.87 hours. Individual experiments lasted approximately 300 hours and were conducted at 3 pH levels (8.0, 8.5, 9.5), two concentrations of $(NH_4)_2CO_3$ (1.75 mM, 8.75mM), and with (0.1 mM) or without added strontium. Several glass microscope slides were placed on the bottom of the reactor vessel before filling to collect settling precipitates for later analysis. Precipitation of calcium carbonate was self-nucleated; i.e., no calcium carbonate seed material was added.

Individual experiments reached steady state (as assessed from the solution saturation indexes) within 72 hours; at steady state the rate of calcium carbonate precipitation can be determined from

$$\frac{dc}{dt} = 0 = Q \times \left(c_0 - c_{steady\ state}\right) - R_{precipitation}$$

Where Q is the fluid flow rate into and out of the CSTR, c_0 and $c_{steady\ state}$ are the inflow and the steady state outflow concentrations of Ca^{2+}, respectively, and $R_{precipitation}$ is the calcium carbonate precipitation rate. Figure 2 shows an example of the change in Ca^{2+} concentration as steady state is achieved in the CSTR.

Ion Ratio Precipitation Experiments

The second series of experiments employed a constant composition technique (Tomson

Figure 2. Change with time of the Continuously Stirred Tank Reactor outlet Ca concentration for three individual experiments showing the approach and achievement of steady state.

and Nancollas, 1978; Hoch et al., 2000; Lin and Singer 2005) to precipitate calcium carbonate. Each experiment is conducted at 25ºC, pH 8.5, and Q/K = 12.8. Although the saturation state was held constant, the carbonate to calcium ion ratio, $[CO_3^{2-}]/[Ca^{2+}]$, ranged from 0.004 to 4. Some of the experiments include low levels Sr^{2+} (0.1mM), allowing the evaluation of the effect of the ion ratio on Sr partitioning and of the effect of the Sr on the overall precipitation rate as well as on the products.

The experiments were conducted in a temperature controlled (water jacketed) stirred reactor, into which a metastable calcium and carbonate containing solution was introduced; the reactor is shown in Figure 1b. Once metastability had been verified for at least 30 minutes (by monitoring pH) reagent-grade calcite seed powder was introduced to the reactor to initiate precipitation. A constant composition was maintained in the reactor with the use of an automated titration system; the decrease in solution pH due to calcium carbonate precipitation triggered the automatic titration system to add solutions containing the cation and anion in equal amounts until the pH returned to the target pH.

The precipitation rate was calculated by normalizing the moles of calcite precipitated over time (based upon the volume of titrant added) by the surface area of the seed crystal, according to the equation:

$$R_{precipitation} = \frac{dv}{dt}\frac{c}{A}$$

where dv/dt is the slope of titrant volume versus time curve, c is the concentration of the titrant, and A is the surface area. The rates reported below were normalized by the initial surface area. Previous workers have assumed that if a titrant volume versus time plot is linear, then the surface area has not significantly changed. However, this is not the case in our experiments as significant amounts of calcite were precipitated (up to 30% of the initial amount) and our titrant volume versus time plot departed from linearity. As a result, we evaluated the tangent of the titrant volume versus time plot to estimate the slope for early times. An example of a titrant volume versus time plot is shown in Figure 3.
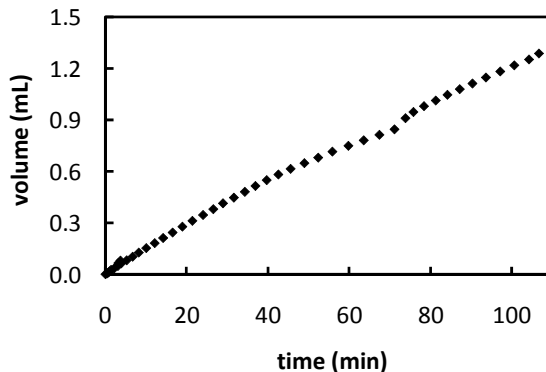
Solution and Solid Characterization

Total alkalinity was determined by titration. Major cations (including NH_4^+) were determined by ion chromatography (Dionex IC25). Additionally, major, minor and trace metals were determined by inductively coupled plasma mass spectrometry (Agilent 7500). Scanning electron microscopy was conducted using a Phillips XL3 ESEM

Figure 3. Example of a titrant volume verses time curve for an ion ratio calcite precipitation experiment.

equipped with an EDAX Genesis energy dispersive spectrometry system. Mineral identification by X-ray diffractometry was conducted using a Bruker D8 system. Quantitative elemental analysis of precipitated solids were conducted using a New Wave UP 213 laser ablation system coupled to an Agilent 7500 inductively coupled plasma mass spectrometer. Geochemical modeling was conducted using The Geochemist's Workbench® Professional (GWB; RockWare, Inc.) and the default thermodynamic data base.

RESULTS AND DISCUSSIONS

Continuously Stirred Tank Reactor Experiments

Results of the 12 CSTR experiments (three pH levels, two $(NH_4)_2CO_3$ levels, and two levels of strontium [with and without]) are summarized in Figure 4. We found that lower pH values and $(NH_4)_2CO_3$ concentration promoted the precipitation of calcite and the higher pH values and $(NH_4)_2CO_3$ concentration promoted the precipitation of aragonite (as determined by X-ray diffraction). Overall, the rate of calcium carbonate precipitation increased with
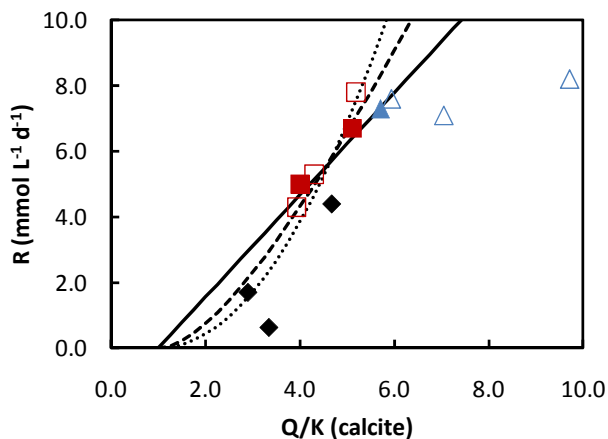
Figure 4. Calcium carbonate precipitation rates as a function of the steady state value of Q/K (for calcite) as determined from the Continuously Stirred Tank Reactor experiments. Open symbols represent experiments using 8.75 mM $(NH_4)_2CO_3$, Closed symbols represent experiments using 1.75 mM $(NH_4)_2CO_3$. Diamond represent experiment in which only calcite was observed, square represent experiments in which both calcite and aragonite was observed, and triangles represent experiments in which only aragonite was observed. The lines represent 3 affinity based rate expressions as described in the text.

increasing pH and $(NH_4)_2CO_3$ concentration, consistent with increasing values of Q/K. Intermediate conditions resulted in the precipitation of a mixture of calcite and aragonite. There was no discernible effect of strontium on the rate of precipitation or the phase precipitated.

Figure 4 shows the steady state rate of calcium carbonate as a function of Q/K (calcite). For experiments in which calcite (diamonds) or both calcite and aragonite (squares) precipitated the precipitation exhibited a positive correlation with Q/K. In experiments that only produced aragonite (triangles) the precipitation rate was essential independent of Q/K. Affinity based (i.e, dependence on Q/K) rate expressions are commonly used to represent calcite precipitation rates (Shiraki and Brantley 1995). These rate expressions can be generalized to

$$ R_{precipitation} = k\left(\left(\frac{Q}{K}\right)^n - 1\right)^m $$

Where k is an operational rate constant and n and m are user defined exponents. Shown on Figure 4 are three commonly used implementations of this model with exponent values of {n=1; m=1} (solid lines), {n=0.5; m=2} (dashed line), and {n=1; m=2}(dotted line) with the values of k estimated from a least squares fit of precipitation rates for experiments in which at least some calcite was formed (experiments that produced only aragonite shown by triangles in figure 4 are not considered in this analysis). Shiraki and Brantley (1995) suggest that inferences on precipitation mechanism can be made based on values of m and n. For {n=1; m=1} they suggest adsorption controlled growth, for {n=1; m=2} they suggest that precipitation is by spiral growth, and for systems with differing aqueous concentration of precipitating cation and anion (expected for Ca^{2+} and CO_3^{2-}) they suggest {n=0.5; m=2} (based on their analysis of Davies and Jones 1955). As may be seen from the figure (as well as the correlation coefficient, r^2, not shown) both models with m=2 better represent the precipitation rates measured in the first series of experiments than does the model with m=1. Both models with m=2 appear to represent the precipitation rates equally well. In our previous work (Fujita et al. 2004; Ferris et al. 2004) with ureolytically driven calcite precipitation we found that the results are well represented by a model in which {n=1, m=2}; our results here are consistent with that finding.

In our experiments we precipitated rhombohedral calcite, lath-shaped aragonite and inter-grown calcite-aragonite mixtures (Figure 5). Using LA-ICP-MS we characterized the composition of
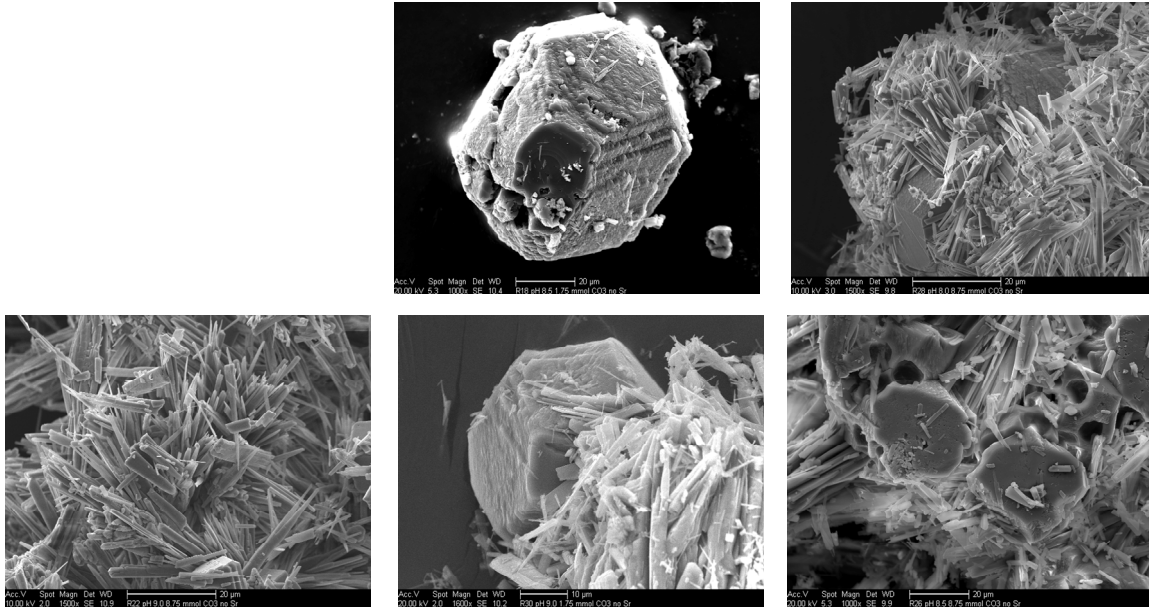
Figure 5. Scanning electron microscopy photomicrographs of precipitates from the Continuously Stirred Tank Reactor experiments (without strontium) showing in order of increasing abundance of aragonite (clockwise from top left to bottom far left) only calcite through inter-grown calcite aragonite mixtures to

solids from an experiment in which both calcite and aragonite precipitates were identified by X-ray diffraction (pH=8.5, 1.75 mM $(NH_4)_2CO_3$). The results are shown in Figure 6. Because the calcite and aragonite are inter-grown, obtaining an analysis of a single phase by LA-ICP-MS (25 μM spot size) was difficult and the compositional range seen in Figure 6 reflects individual analyses that are not single phases but captured varying amounts of calcite and aragonite. From the figure we see a range in composition from a high Sr and low Mg phase (inferred to be aragonite) to a coexisting lower Sr and higher but variable Mg phase (inferred to be calcite). The intermediate compositions shown in the figure are postulated to be mechanical mixtures of calcite and aragonite. A key empirical measure of the distribution of trace metals between aqueous and solid phases is the distribution coefficient ($D_{ex,Sr}$), which for strontium partitioning into calcium carbonate can be represented by

$$D_{ex,Sr} = \frac{\chi_{SrCO_3} \cdot [Ca^{2+}]}{\chi_{CaCO_3} \cdot [Sr^{2+}]}$$

where χ is the mole fraction of the solid phase. By assuming that the highest measured Sr/Ca values are associated with aragonite and that the lowest values are associated with calcite, values of $D_{ex,Sr}$ of 1.1 and 0.2 for aragonite and calcite, respectively can be estimated from the data.
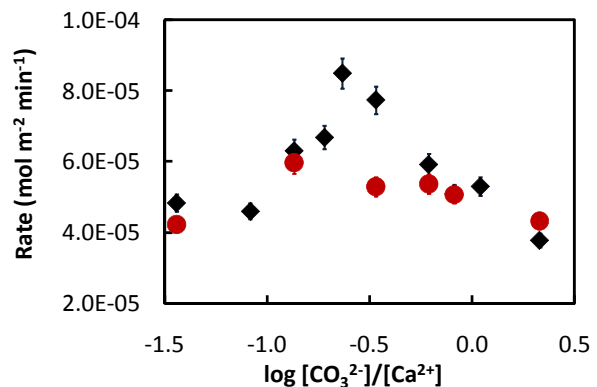
These values compare to $D_{ex,Sr}$ values of 1.1 and 0.1 for aragonite and calcite, respectively, determined from bulk analysis of precipitates from experiments in which only calcite or only aragonite were detected. These results for $D_{ex,Sr}$ in calcite are lower than reported by Fujita et al (2004) and Mitchell and Ferris (2005) both of which relied upon microbial hydrolysis of urea to generate $(NH_4)_2CO_3$ in batch experiments with significant variation in the aqueous Sr/Ca ratio and much higher values of Q/K than the current study.
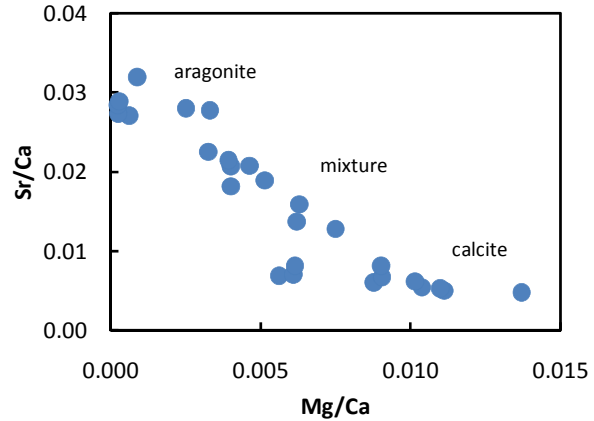
Figure 6. Solid phase ion ratios (wt.) as determined by LA-ICP-MS of calcium carbonate precipitates collected from a CSTR experiment in which both calcite and aragonite was detected (ph=8.5, $(NH_4)_2CO_3$=8.75 mM).

Ion Ratio Precipitation Experiments

Figure 7 shows the influence of solution composition on precipitation rate (at a constant value of Q/K). As may be seen in the figure, the precipitation rate varied by approximately a factor of 2 over the range of conditions considered, with a maximum rate observed at a $[CO_3^{2-}]/[Ca^{2+}]$ molar ratio of approximately 0.2. Precipitation kinetics at the "extremes" tested in this study exhibited interesting behavior. At the lowest ion ratio ($[CO_3^{2-}]/[Ca^{2+}] = 0.004$), a metastable solution was not achievable. At the highest ion ratio ($[CO_3^{2-}]/[Ca^{2+}] = 4$), the solution was indefinitely metastable: no amount of seed material added initiated a drop in pH that would indicate the onset of precipitation. Under conditions considered to date, we cannot definitively quantify the influence (if any) of Sr at 0.1 mM on the measured precipitation rates (work continues in this area through the INL SFA).

Our observed calcite precipitation rate maximum occurs at $[CO_3^{2-}]/[Ca^{2+}] < 1$. Kowacz et al. (2007) report similar results for

Figure 7. Calcite precipitation rates as a function of $[CO_3^{2-}]/[Ca^{2+}]$ molar ratio for experiments with (circles) and without (diamonds) strontium.

the barite precipitation rate maximum under similar supersaturation conditions. However, our result stands in apparent contrast to Nehrke et al. (2007) who reported a maximum calcite precipitation rate at an ion ratio of approximately 1. The primary differences between Nehrke et al. (2007) and the current study are the experimental pH (10.2 vs. 8.5) and the use of single crystals versus fine-grained calcite seed material as the precipitation substrate. Nehrke et al. (2007) selected a pH of 10.2 because CO_3^{2-} would be the dominant dissolved carbon species, which "facilitated the preparation of stoichiometric $[Ca^{2+}]$ to $[CO_3^{2-}]$ ratios ranging from less than one to greater than one, while maintaining constant ionic strength." They concluded from their experimental results that precipitation rates exhibit a maximum at $[CO_3^{2-}]/[Ca^{2+}] = 1$. Examination of their plot of rates vs. solution composition, however, do not conclusively prove or disprove this statement, as none of their experiments were conducted at a ratio of exactly 1 (the nearest was 0.6). Discussions with the authors of Nehrke et al. (2007) subsequent to their publication indicate that their conclusions may in fact be similar to ours.

Previous investigations have recognized an increase in $D_{ex,Sr}$ with increasing calcite precipitations rate in both abiotic (Lorens 1981; Tesoriero et al., 1996) and microbially mediated (Fujita et al., 2004, Mitchell and Ferris 2005) systems. However, in contrast we did not find a definite relationship between calcite precipitation rate and $D_{ex,Sr}$ in either of the two sets of experiments that we conducted. This is likely a consequence of the narrow range of precipitation rates that we investigated (approximately a factor of 2) in comparison to other investigators (cited above) who have considered precipitation rates that varied factors of 10s to 100s.

CONCLUSIONS

We conducted calcium carbonate precipitation and Sr co-precipitation experiments that attempted to maintain conditions of constant solution composition and levels of supersaturation that are likely to be relevant in the implementation of subsurface remediation strategies. In these experiments we found (as have others) that calcium carbonate precipitation rates and contaminant co-precipitation rates (e.g., Sr) are a complex function of level of the supersaturation, solution chemistry (e.g., ion ratios), and the primary carbonate polymorph formed. The results from this study provide the basis for developing generalized precipitation and co-precipitation kinetic models that explicitly account for the extent of supersaturation and

variations in solution compositions. The application of the kinetic models will enable the more effective deployment of DOE relevant subsurface remediation strategies.

ACKNOWLEDGEMENT

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