

The Effect of SO₂ on Mineral Carbonation in Batch Tests

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ABSTRACT

CO₂ sequestration is a key element of future emission-free fossil-fueled power plants. Other constituents of flue gas must also be captured and rendered innocuous. Contemporary power plants remove SO_x from exit gases, but next-generation plants may simultaneously treat CO₂, SO_x, and other pollutants. Pioneering tests at the U.S. Department of Energy's Albany Research Center investigated the combined treatment of CO₂ and SO₂ in a mineral-carbonation process. SO₂ was removed from the gas stream, and as a small fraction of the total volume of mineralizing gas, it did not inhibit the carbonation reaction. The results indicate that this approach to CO₂ sequestration could be used to treat multiple pollutants.

INTRODUCTION

Fossil-fuel-fired power plants produce flue gas that is a mixture of CO₂, NO_x, SO_x, particulates, and other components. Emissions-free plants have been proposed that would capture all emissions or render them environmentally neutral (Ochs et al., 2003; Ziock et al., 2001). CO₂ is an unavoidable product of coal and other hydrocarbon combustion, and it is under scrutiny as a potential contributor to global warming. A broad range of research within the U. S. Department of Energy is addressing capture of CO₂ and its storage in various sequestration schemes (DOE Office of Fossil Energy, 2003).

SO₂ is another natural gaseous product of coal combustion. Desulfurization of flue gas is commonly done in the U.S. and other countries to reduce acid rain and its environmental damage. Current techniques for desulfurization leave traces of SO₂ in the treated gas. New technologies under study may eliminate the need for a separate desulfurization step, instead capturing SO₂ with CO₂ simultaneously for use as feed to co-sequestration processes (Abdelmalek, 1992; Kreutz et al., 2003; Oryshchyn et al., 2004).

Albany Research Center (ARC) has conducted considerable research on the concept of mineral carbonation as a means of CO₂ sequestration (O'Connor et al., 2001, 2002, 2003, 2004). As envisioned by ARC, mineral carbonation is the reaction of suitable minerals (primarily magnesium silicates such as forsterite olivine and serpentine, but also calcium silicates and ferrous-iron silicates) with aqueous CO₂ to form geologically stable mineral carbonates. The reaction product is a stable, natural mineral, but the process entails significant disadvantages, such as large-scale mining and unacceptably high energy and economic costs in its current stage of development (O'Connor et al., 2004).

Some of the disadvantages associated with the *ex-situ* process may be mitigated by *in-situ* mineral carbonation, in which CO₂ is injected into underground aquifers within suitable host geological formations. One such candidate type of formation is a massive flood basalt province, an example of which is the Columbia River Basalt Group in the Pacific Northwest of the United States (Reidel et al., 1989). This huge potential resource consists of an extensive aquifer system within multi-layered basalt flows. Basalt has much lower carbonation potential and exhibits limited reactivity compared to olivine, serpentine, and other silicate minerals investigated for the *ex-situ* process, but the greater reaction time required for carbonation to occur in basalt is of less consequence if the reaction is carried out in the aquifer rather than in a reaction vessel. Basalt-hosted aquifers could be favored over typical sandstone-hosted aquifers because of the potential for both hydrodynamic trapping and mineral trapping of injected CO₂ (O'Connor et al., 2003).

Researchers at ARC have conducted more than 600 batch tests to evaluate the technical and economic feasibility of mineral carbonation as a method of sequestering CO₂ from power-plant emissions. Most of the tests used essentially pure (99.99%) cylinder CO₂. The carbonation reaction is dependent on the partial pressure of CO₂. The current studies looked

Table 1. Composition of TSO feed and solid products of carbonation tests. Analyses in weight percent.

Oxide Species	Average TSO feed	Test products		
		SC-352	SC-359	SC-361
Al ₂ O ₃	0.15	0.13	0.08	0.10
CaO	0.11	0.04	0.10	0.11
Cr ₂ O ₃	0.28	0.15	0.24	0.16
FeO	7.89	5.34	5.22	5.27
Fe ₂ O ₃	<0.01	0.37	0.04	<0.01
MgO	49.80	34.49	32.33	32.33
NiO	0.28	0.29	0.22	0.20
K ₂ O	<0.01	<0.01	0.01	0.01
SiO ₂	40.29	27.17	26.96	26.96
Na ₂ O	0.05	0.60	0.54	0.51
SO ₄	0.30	N/A	0.11	0.15
S (excess) ¹	0.00	N/A	-0.01	0.01
CO ₂	0.29	30.10	32.10	31.60
C (excess) ¹	0.03	0.29	0.32	0.23
H ₂ O, dehyd. ²	0.24	0.23	0.33	0.32
H ₂ O, chem. ³	0.79	1.18	0.75	1.30
Total	100.49	100.38	99.25	99.01
LOI ⁴	1.55	31.80	33.60	33.60

N/A = Not analyzed.

¹ Excess S and C are calculated by subtraction of S in SO₄ and C in CO₂ from total S and total C, respectively.

² Water of dehydration (free moisture), measured as weight loss after heating for 1 hour at 105° C in air.

³ Chemically bonded (interstitial) water, calculated as the difference between LOI and all other volatiles.

⁴ Loss on Ignition, measured as weight loss after heating for 1 hour at 1,000° C in argon.

at the effect of a small percentage of SO₂ in the CO₂ stream on the mineral-carbonation process. If contaminants can be tolerated, then the potential exists to simultaneously remove multiple contaminants from the flue gas within the mineral-carbonation process.

In the course of the studies at ARC, sample preparation and test methods were developed to maximize the reactivity of the cations available in the mineral feeds for carbonation. Similar grinding and carbonation conditions were used in this study, but 1.5% SO₂ by volume was added to the CO₂ gas reactant to determine its effect on carbonation.

Samples and Methods

Tests were conducted on olivine from the Twin Sisters deposit in northwestern Washington State (TSO) and on basalt from an outcrop of the Columbia River Basalt Group (CRB) in north-central Oregon. TSO is forsterite, the magnesium-rich member

Table 2. Composition of CRB feed and solid products of carbonation tests. Analyses in weight percent.

Oxide Species	CRB Feed	Test Products	
		SC-516	SC-517
Al ₂ O ₃	13.28	11.58	12.60
CaO	8.17	8.03	8.56
Cr ₂ O ₃	0.08	0.07	0.08
FeO	10.37	9.74	9.92
Fe ₂ O ₃	1.69	2.80	2.47
MgO	4.94	4.86	4.71
NiO	0.01	0.01	0.01
K ₂ O	0.90	0.83	0.84
SiO ₂	51.56	50.27	48.99
Na ₂ O	2.36	3.41	4.10
SO ₄	0.03	0.02	0.28
S (excess) ¹	0.00	0.00	-0.01
CO ₂	<0.1	1.83	2.39
C (excess) ¹	0.01	0.03	0.05
H ₂ O, dehyd. ²	0.65	0.76	0.49
H ₂ O, chem. ³	0.95	1.36	1.80
Total	95.09	95.61	97.29
LOI ⁴	1.74	4.00	5.00

¹ Excess S and C are calculated by subtraction of S in SO₄ and C in CO₂ from total S and total C, respectively.

² Water of dehydration (free moisture), measured as weight loss after heating for 1 hour at 105° C in air.

³ Chemically bonded (interstitial) water, calculated as the difference between the LOI and all other volatiles.

⁴ Loss on Ignition, measured as weight loss after heating for 1 hour at 1,000° C in argon.

of the olivine family. Magnesium (Mg), calcium (Ca), and ferrous iron (Fe²⁺) in the samples are considered potentially available for carbonation. Magnesium is the most abundant cation available to carbonate in forsterite, although ferrous iron is also present. Basalt contains lesser but significant amounts of all three cations, and the source minerals include oxides as well as silicates. The average of three analyses of feeds for tests on TSO is shown in table 1; analysis of the feed to the CRB tests is shown in table 2.

The TSO and CRB feed materials were initially ground to a nominal size of minus 75 microns in laboratory-scale rod and/or ball mills. Each was then ground in a high-intensity attrition mill for one hour; the TSO was attrited dry, and the CRB was attrited in a 50%-solids slurry with tap water. These materials were used as feed in carbonation experiments using test procedures and conditions standardized at ARC.

In each test, 167 grams of mineral were combined with a 0.64M NaHCO₃ and 1M NaCl carrier solution in a 15%-solids slurry and fed to a 2-liter autoclave. The system was then purged with CO₂, pressurized to 150 psi (10 atm) gas (CO₂ or CO₂+SO₂), stirred at 1000 rpm, heated to the test temperature of 185° C (the warm-up period was 1 hr), and held at temperature and pressure for 1hr (TSO) or 6 hr (CRB). After the test, the slurry was cooled to 85° C, depressurized to atmospheric pressure, and then drained from the autoclave. Solids and liquid were separated in a pressure filter, the solids were dried at 105° C and weighed, and samples of the dried product solids and product liquid were submitted for chemical analysis. The combined Mg, Ca, and Fe²⁺ analyses of the feed were used to calculate maximum CO₂ capture potential; CO₂ content of the solid carbonation product was compared to this number to give a percent stoichiometric conversion to carbonate (O'Connor et al., 2002).

In order to determine which constituents of the basalt were affected by reactions with CO₂ and/or SO₂, solid products from the tests on CRB material were examined with a scanning electron microscope (SEM). Spot analyses were performed on some of the mineral grains using energy-dispersive X-ray analysis (EDX).

RESULTS

Extent of Carbonation

The extent of carbonation for all the tests is summarized in table 3. Based on the data in this table, the mixed CO₂-SO₂ gas was slightly more effective than CO₂ alone in the carbonation reaction. Tests with the TSO feed showed extents of carbonation improved from 72% to 79% with SO₂ in the reaction gas; with CRB feed, the extent of carbonation was 10% without SO₂ and 14% with SO₂.

Table 3. Extent of carbonation.

TSO 1 hr tests ¹	SO ₂ in reaction gas? (+/-)	Percent Stoichiometric Carbonation
SC-352	-	71.7
SC-359	+	78.8
SC-361	+	77.0
CRB 6 hr tests ²	SO ₂ in reaction gas? (+/-)	Percent Stoichiometric Carbonation
SC-516	-	10.3
SC-517	+	13.6

¹ Tests on olivine used material attrited dry for one hour after various preliminary grinding treatments.

² Tests on basalt used the same batch of ground material attrited wet for 1 hour.

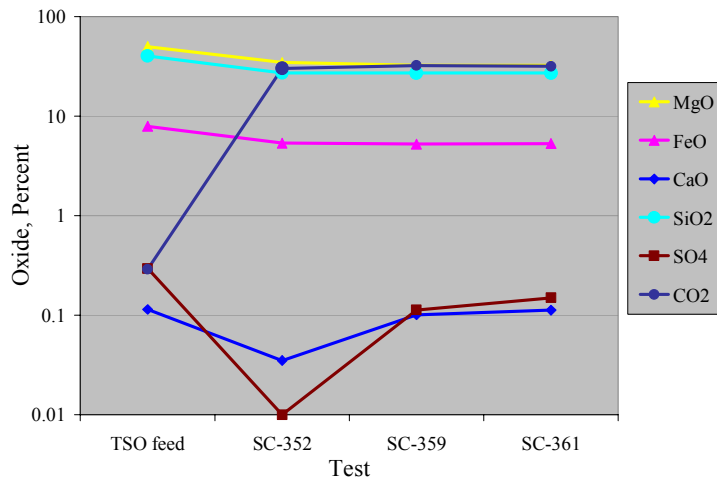


Figure 1. Analysis of oxide constituents in TSO feed and carbonation test products. SO₄ was not analyzed for SC-352.

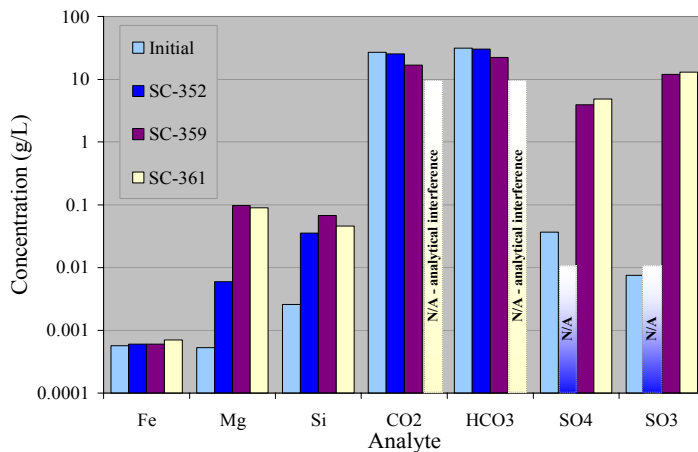


Figure 2. Chemistry of the solutions from TSO carbonation tests. The initial solution was a NaHCO₃/NaCl solution. N/A = Not analyzed.

Chemistry of products

Olivine tests

Changes in chemistry of the solid products of olivine carbonation tests are primarily the result of CO₂ added in the mineral-carbonation reaction. After carbonation, the solid products contained more than 30% CO₂. Figure 1 shows percents of oxides of Mg, Fe²⁺, Ca, and Si, as well as CO₂ and SO₄ in the feed and the solid products. The dilution effect is most clearly seen in the analyses of the most abundant oxides (FeO, MgO, and SiO₂). Table 1 shows more complete analyses for the feed and solid products.

The SO₂ component of the gas improved the overall extent of carbonation, perhaps by improving solubility of the targeted cations. The CO₂ contents of products of the two tests using spiked gas (SC-359 and SC-361) are slightly higher than that of the test using pure

CO₂ as the reaction gas. The SO₄ content of the solids was used as a measure of the uptake by the solids of SO₂ from the gas. The SO₄ content of the feed material is an average from two olivine feed samples (table 1); the feed and product of test SC-352 were not analyzed for SO₄, because SO₂ was not used in that test. The SO₄ contents of the two solid products of the SO₂ gas tests fall very near (within one standard deviation) of the average SO₄ content of the feed. Some SO₄ is expected in the solid products in the form of evaporated salt residue from incompletely washed liquors, and the amounts determined by analysis are probably due to this and not to sequestration.

Chemistry of the post-carbonation liquid products is interesting. Figure 2 and table 4 show the initial and final concentrations of elemental and ionic constituents in the liquids. The initial solution contains significant CO₂ and HCO₃, as expected; analytical interference with SO₃ prevented determination of the levels of these analytes in two of the SO₂ product samples. The SO₂ may have improved Mg solubility; Mg was present in somewhat greater amounts in the final solutions from SO₂-spiked tests (SC-359 and SC-361). Fe was present in both the feed and test liquids in similar but very small amounts; Si increased in all product liquids but was still a minor constituent. SO₂ dissolved into the carrier solution during the carbonation tests. Post-carbonation analyses of SO₃ and SO₄ in the liquids from the tests using SO₂ showed about 12 g/L SO₃ and 4 g/L SO₄.

Table 4. Carrier solution compositions before and after carbonation tests, g/L.

Analyte	TSO solutions				CRB solutions		
	Average Initial Composition	SC-352	SC-359	SC-361	Average Initial Composition	SC-516	SC-517
Cl	36.7	36.2	35.7	36.4	35.7	36.2	35.5
Fe	0.0006	0.0006	0.0006	0.0007	0.0002	0.0004	0.0004
Mg	0.0005	0.0596	0.0970	0.0892	0.0003	0.0060	0.0035
Si	0.0026	0.0350	0.0675	0.0458	0.0023	0.0438	0.0378
CO ₂	26.9	25.4	16.7	AI	27.1	26.4	AI
HCO ₃	31.2	30.3	22.3	AI	32.6	33.5	AI
SO ₄	0.0367	N/A	3.9	4.83	0.0015	0.0030	3.64
SO ₃	0.0075	N/A	11.9	13.0	<0.0001	<0.0001	1.98
K	0.0022	0.0065	0.0060	0.0054	0.003	0.380	0.386
Na	32.7	31.0	31.6	30.8	32.7	32.4	30.8
pH	7.8	7.7	7.5	7.4	7.8	7.8	7.6

N/A = Not analyzed.

AI = Analytical interference.

Basalt tests

Figure 3 and table 2 show concentrations of oxides in feed and products of carbonation tests using CRB as feed material. Test SC-516 was a baseline test using best conditions and CO₂ only in the reaction gas; SC-517 used all of the same conditions but included SO₂ in the gas. Because the extent of carbonation was much lower in these tests compared to those using TSO feed, the effect of dilution by CO₂ is not as evident. Concentrations of MgO, FeO, CaO, and SiO₂ are similar in the feed and products. CO₂ content in the solids was similar for both tests, although the level increased slightly when SO₂ was used (SC-517). SO₄ increased in the

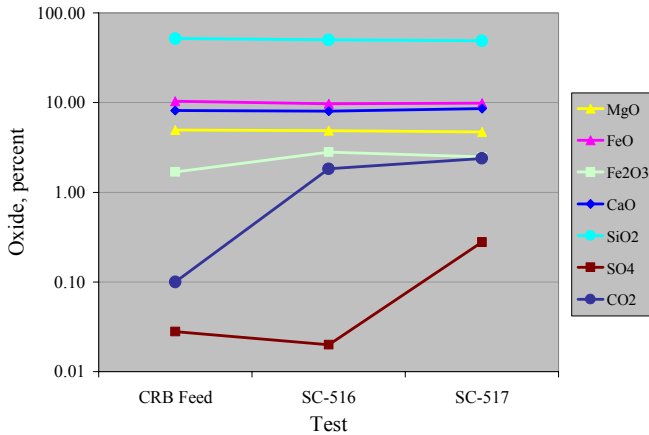


Figure 3. Analysis of oxide constituents in CRB feed and test products.

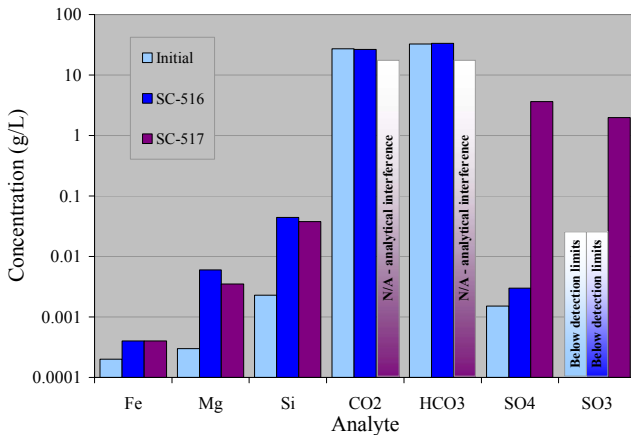


Figure 4. Chemistry of the solutions from CRB carbonation tests. The initial solution was a NaHCO₃/NaCl solution. N/A = No analysis.

ensures an excess of CO₂ compared to the concentration of cations available for carbonation. The experimental system is designed to replenish CO₂ and maintain constant pressure as the carbonation reaction consumes CO₂ and/or gas is lost through leaks in the system. The small percentage of SO₂ does not significantly affect the P_{CO2}.

In contrast to CO₂, which is sequestered as a solid mineral-carbonate product, SO₂ is removed from the gas stream primarily into the product solution. The SO₃ and SO₄ contents of the product solutions increased dramatically from their levels in the initial solution. Although the SO₄ concentrations were similar in the olivine and basalt tests, the SO₃ concentration in the solutions from the olivine tests was significantly higher than in the solution from the basalt test.

solid product of the test for which SO₂ was used, again perhaps as a residue from evaporated solution in the filtered solids.

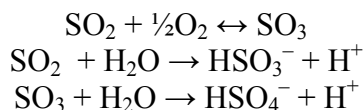
Figure 4 and table 4 indicate levels of constituents in the product liquids for the two CRB tests. Cl, HCO₃, CO₂, and Na analyses reflect the use of the NaHCO₃/NaCl carrier solution. SO₂ again dissolved into the carrier solution, although to a lesser extent than in the olivine tests. Mg content was not increased in the basalt test solution when SO₂ was used in the gas.

Fate of SO₂ and CO₂

The focus of this investigation was the effect of SO₂ on the carbonation reaction and the fate of the SO₂ in the CO₂ reaction gas. The results in table 3 suggest that the presence of SO₂ at a level of 1.5% in a gas stream of CO₂ does not hinder the mineral-carbonation reaction and may actually enhance it slightly.

The carbonation reaction is dependent on the partial pressure of CO₂ (P_{CO2}), and the high P_{CO2}

Were all of the conditions and reactants in these tests identical, the same ratios of SO₃ to SO₄ would be expected in the analyses. Since they are not, we need to examine the behavior of SO₂ in the tests. In the presence of oxygen, SO₂ oxidizes to SO₃ to a limited degree, and both species may dissolve, hydrolyze, and dissociate, producing a solution that contains HSO₃⁻ and HSO₄⁻ ions. The summary reactions are:



SO₃ is much more soluble than SO₂, so that production of HSO₄⁻ is limited by SO₃ availability. The similar amounts of SO₄ in the solution analyses imply that the first and third of these reactions are occurring at the same rates in all tests. Thus, dissolution of SO₂ must be inhibited in tests using basalt as the solid reactant.

Table 5 shows the weights of CO₂ and S removed from the gas phase into the solid and liquid phases during the CO₂+SO₂ carbonation tests based on analyses of the product solids and solutions; the total amount of S in the products (solids plus solution) was calculated based on analyses of all sulfur species (S, SO₄, and SO₃). The weights of CO₂ and S in the products are compared to the weights available in the gas, which are calculated from molar volumes of CO₂ and SO₂ by using the following assumptions:

- (1) Autoclave head volume of 1 liter;
- (2) Ideal gas behavior at test temperature and pressure;
- (3) A make-up volume of gas sufficient to replace the moles of CO₂ converted to solid; and
- (4) No gas leakage during the test.

Because the gas supply was constantly replenished during the test, it would not have been meaningful to analyze a gas sample after the test to determine residual CO₂ and SO₂ contents and conversion efficiencies.

Table 5. Removal of CO₂ and SO₂ from gas.

	CO ₂ in product solids, g	CO ₂ available in gas ¹ , g	Pct CO ₂ removed from gas	S in product solids and solution, g	S available in gas ¹ , g	Percent S removed from gas
TSO - calculations based on CO₂ sequestered						
SC-359	77.9	250.9	31.0	3.23	2.78	116.2
SC-361	76.1	249.2	30.5	4.23	2.75	153.8
TSO - calculations based on SO₂ sequestered, assuming 100% capture						
SC-359	77.9	291.7	26.7	3.23	3.23	100.0
SC-361	76.1	381.4	20.0	4.23	4.23	100.0
CRB						
SC-517	4.1	176.8	2.3	1.57	1.95	80.5

¹Assumes initial weight based on the moles of gas at test temperature and pressure in 1-liter head space of autoclave, replacement volume of gas necessary to replace CO₂ converted to solid, and no gas leakage during test.

With the above assumptions, about 30% of the available CO₂ was sequestered in the olivine tests and only 2% in the basalt test. Most of the available SO₂ was removed to the liquid phase; indeed, the percentage of S removed exceeds 100% for the olivine tests. If recalculated to assume that the S found by chemical analyses in the products of the olivine tests was 100% of that available in the gas, then the volume of gas (and thus CO₂) available for sequestration must increase. Because the weight of CO₂ converted to solid is known (calculated from the chemical analysis) and remains the same, a recalculation on this basis shows that the percentage of CO₂ sequestered drops to 20-27% of the available CO₂.

When calculated based on the initial assumptions, about 80% of the S was removed from the gas in the basalt tests. When the gas volume was recalculated based on 100% SO₂ removal, it was less than that assumed in the one-liter head space available in the autoclave at the known pressure and temperature conditions; these results are not reported in table 5. The lower S removal (80%) is consistent with the hypothesized poorer SO₂ solubility in the basalt solution as described above.

Scanning Electron Microscopy

SEM examinations using backscattered electron imaging were done on the solid products of the carbonation reactions with CRB feed. They showed that the dominant mineralogy of the basalt is plagioclase feldspar [(Ca,Na)Al(Al,Si)Si₂O₈], pyroxene (a Mg-Fe-Ca silicate), and ilmenite (FeTiO₃). Each of these minerals contains one or more cations (Fe²⁺, Mg²⁺, and Ca²⁺) that is appropriate for carbonation.

Visible grain characteristics were similar in the CRB feed and carbonation test product samples. All were angular, and there was no visible evidence of reaction, such as dissolution textures or reaction rims, in the test products. Ilmenite and feldspar grains were typically unfractured and smooth-surfaced, whereas many of the pyroxene grains were fractured and contained a darker gray material in the fractures. When EDX analyses were performed on the material in the fractures of the CRB feed pyroxene grains, no carbon was detected. However, analyses performed on the fracture-filling material in carbonation test product samples (including those from tests using SO₂ in the reaction gas) showed significant carbon content. Sulfur was not detected by the EDX system in the fracture-filling material. From this limited investigation, it was concluded that pyroxene and/or alteration products of pyroxene are carbonated in the basalt, and carbonation occurs in the presence of SO₂ gas.

CONCLUSIONS

In carbonation tests performed with pure CO₂ and mixed CO₂-SO₂ reactant gases, SO₂ in the mixed gas did not inhibit the carbonation reaction and may have enhanced it slightly, perhaps by increasing the solubility of Mg. SO₂ had little effect on the chemistry of the solid products of the carbonation reactions; solid sulfates did not form to any significant extent.

SO₂ was readily scrubbed from the gas into the liquid phase, and the amount of SO₂ taken into solution is determined by the volume and solubility limits of the solution. In contrast, the

quantity of CO₂ sequestered is dictated by the quantity of cations available for carbonation and the kinetics of the carbonation reaction.

The poorer carbonation of basalt indicates that its Mg²⁺, Fe²⁺, and Ca²⁺-containing minerals are less reactive than olivine for mineral carbonation. Pyroxene shows evidence of carbonation in basalt tests, but the reaction is localized to fractures in the mineral grains. The lower brightness of the fracture filling in SEM images indicates a very different molecular weight for this material, so that what is being carbonated may be a secondary silicate mineral formed by alteration of pyroxene rather than the pyroxene itself.

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