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CARBON DIOXIDE SEQUESTRATION BY DIRECT MINERAL CARBONATION: RESULTS FROM RECENT STUDIES AND CURRENT STATUS

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Carbon Dioxide Sequestration by Direct Mineral Carbonation: Results from Recent Studies and Current Status

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Abstract

Direct mineral carbonation has been investigated as a process to convert gaseous CO_2 into a geologically stable, solid final form. The process utilizes a solution of sodium bicarbonate ($NaHCO_3$), sodium chloride (NaCl), and water, mixed with a mineral reactant, such as olivine (Mg_2SiO_4) or serpentine [$Mg_3Si_2O_5(OH)_4$]. Carbon dioxide is dissolved into this slurry, by diffusion through the surface and gas dispersion within the aqueous phase. The process includes dissolution of the mineral and precipitation of magnesium carbonate ($MgCO_3$) in a single unit operation. Optimum results have been achieved using heat pretreated serpentine feed material, with a surface area of roughly 19 m^2 per gram, and high partial pressure of CO_2 (P_{CO2}). Specific conditions include: 155°C; P_{CO2}=185 atm; 15% solids. Under these conditions, 78% stoichiometric conversion of the silicate to the carbonate was achieved in 30 minutes. Studies suggest that the mineral dissolution rate is primarily surface controlled, while the carbonate precipitation rate is primarily dependent on the bicarbonate concentration of the slurry. Current studies include further examination of the reaction pathways, and an evaluation of the resource potential for the magnesium silicate reactant, particularly olivine. Additional studies include the examination of various pretreatment options, the development of a continuous flow reactor, and an evaluation of the economic feasibility of the process.

Introduction

Carbon dioxide gas generated by the combustion of fossil fuels is considered the most significant of the greenhouse gases due to the quantity of its emissions. Carbon dioxide emissions are estimated at 6 GtC/year from fossil fuels alone (Freund and Ormerod, 1997). Means to reduce the carbon dioxide (CO_2) emissions from the energy and/or process industries has become increasingly emphasized as a primary environmental concern. This can be achieved by efficiency improvements in fossil-fuel-fired power plants, use of alternative energy technologies, sequestration of CO_2, or some combination of these methods. Due to the availability of fossil fuel resources, it is anticipated that they will continue to play a significant role in the world's energy economy well into the next century. Thus, emphasis must be placed on improving the combustion efficiency of the fossil-fuel-fired power plant, as well as on methods to sequester the CO_2 emitted from these plants. Sequestration can take many forms, such as:

1) forestation, where CO_2 is removed from the atmosphere by biological activity; 2) aquifer storage, in which the CO_2 is injected into terrestrial aquifers where it is trapped hydrodynamically; 3) deep sea storage, where the CO_2 is injected into the ocean at approximately 3,000 meter depths, where it is believed to remain stable; and 4) mineral carbonation, in which the CO_2 is reacted with minerals to form solid carbonates. This final form of sequestration is truly a permanent method, because the mineral carbonates are stable over geologic time periods (millions of years), rather than the hundreds to thousands of years of stability expected for the first three forms of sequestration.
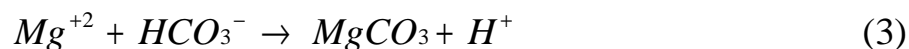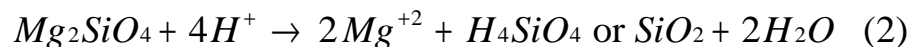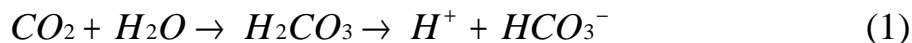
Objective

The objective for the subject research is to develop a technically feasible process for mineral carbonation, one suitable for retrofit on existing fossil-fuel-fired power plants, or included in the design for future plants. The process must be scalable to meet the requirements of significant reduction in CO_2 emissions. Determination of the economic feasibility of the mineral carbonation process requires the development of engineering parameters, such as process temperature and pressure, solids concentration, ore preparation (size reduction and pretreatment), solution chemistry, and post-treatment separation and recycle. The subject process is essentially at this stage, with process development and feasibility studies progressing co-currently.

Project Description and Approach

The Albany Research Center (ARC) of the U.S. Dept. of Energy (DOE) has been conducting a series of mineral carbonation tests at its Albany, Oregon facility over the past 2 years as part of a Mineral Carbonation Study Program within the DOE. Other participants in this Program include the Los Alamos National Laboratory (LANL), Arizona State University (ASU), Science Applications International Corporation (SAIC), and the DOE National Energy Technology Laboratory (NETL).

Lackner, et al. (1996, 1997) discussed CO_2 storage as mineral carbonates in prior literature, although these publications describe a process that differs dramatically from the subject process. The direct mineral carbonation process utilizes a slurry of fine particle sized mineral in water, at solids concentrations from 15-30%. Dissolution of the mineral and subsequent carbonation occur in a single unit operation. The theorized reaction equations follow.

$$CO_2 + H_2O \rightarrow H_2CO_3 \rightarrow H^+ + HCO_3^- \qquad (1)$$

$$Mg_2SiO_4 + 4H^+ \rightarrow 2Mg^{+2} + H_4SiO_4 \text{ or } SiO_2 + 2H_2O \quad (2)$$

$$Mg^{+2} + HCO_3^- \rightarrow MgCO_3 + H^+ \qquad (3)$$

The CO_2 is dissolved in water to form carbonic acid (H_2CO_3), which dissociates to H^+ and HCO_3^- (Eq. 1). The H^+ ion hydrolyzes the mineral, liberating Mg^{2+} cations and forming silicic acid or free silica and water (Eq. 2). The free Mg^{2+} cations react with the bicarbonate ions to form the solid carbonate (Eq. 3). An example process flow diagram is shown in Figure 1. The process has been described in greater detail by Dahlin, et al. (2000) and O'Connor, et al. (2000a, b). The direct mineral carbonation tests have focused thus far on ultramafic minerals as the primary reactants. Geologic studies by Hunter

Figure 1: Process flow diagram for the direct carbonation process

(1941), the
IGCP (1977) and Goff, et al. (1997, 2000) indicate that reserves of these ultramafic minerals are sufficient to provide raw materials for the mineral carbonation of all annual CO_2 emissions for many years. Additional studies conducted at ARC indicate that the mining and milling costs would be similar to those in the porphyry copper mining industry, at approximately 4-5 U.S. dollars per ton.

Experimental Results

Feed Material Analyses

Analyses were acquired for the feed materials used for the direct carbonation tests, with the results reported in table 1. These materials included: naturally occurring olivine (forsterite end member); and two varieties of naturally occurring serpentine, antigorite and lizardite. The product analyses from a magnetic separation step on the antigorite serpentine are also included in Table 1, along with analyses for the heat treat product from the non-magnetic antigorite serpentine fraction.

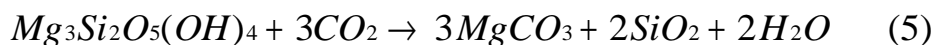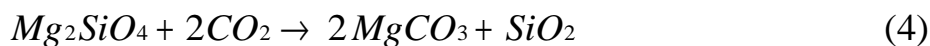*Table 1: Chemical composition of the feed materials (wt pct)*

Oxide	Olivine (forsterite)	Serpentine (antigorite)	Non-mag lizardite	Non-mag antigorite	Magnetic antigorite	HT, non-mag antigorite
Al_2O_3	0.208	0.167	1.540	0.164	0.189	0.255
CaO	0.070	0.077	0.326	0.044	0.074	0.152
Cr_2O_3	0.044	NA	0.171	0.013	0.399	NA
FeO	5.966	3.499	2.730	1.880	17.000	2.211
Fe_2O_3	2.558	3.274	3.440	2.560	27.900	1.914
MgO	49.677	40.623	39.500	38.500	25.700	49.677
K_2O	0.007	0.003	0.017	0.002	<0.002	<0.002
SiO_2	41.357	36.155	38.800	37.200	19.900	42.646
Na_2O	0.099	0.010	0.033	0.007	0.006	<0.002
Volatiles						
C, CO_2	<0.300	0.460	0.200	0.810	0.470	0.190
C, fixed	0.021	0.020	0.055	0.039	0.042	0.015
C, total	0.021	0.480	0.255	0.849	0.512	0.205
H_2O^1	0.380	0.310	0.850	0.360	0.150	0.890
H_2O^2	0.000	13.61	13.100	13.400	7.680	2.215
Total	100.387	98.208	100.762	94.979	99.510	100.165

[1] Water of dehydration (free moisture).
[2] Chemically-bonded (interstitial) water.
NA: Not analyzed

Magnesium oxide content in the olivine, antigorite, and lizardite were measured at 49.7 weight percent (wt pct), 40.6 wt pct, and 40.6 wt pct, respectively. The difference is largely attributable to the high concentration of water (13.6 wt pct) in the serpentines. Adding the magnetic separation step prior to heat treatment proved most effective. The magnesium oxide content in the heat treat product from the non-magnetic antigorite fraction, at 49.7 wt pct, duplicated that of the naturally occurring olivine. Emphasis is placed on the magnesium oxide concentration of the feed material, because it is this oxide that reacts with the carbon dioxide to form the carbonate. Thus, feed materials with higher magnesium oxide concentration are preferred. Calculation of the extent of reaction for the direct carbonation tests was based on the concentration of magnesium oxide in the appropriate feed material, and the stoichiometry of the following theoretical equations.

$$Mg_2SiO_4 + 2CO_2 \rightarrow 2MgCO_3 + SiO_2 \tag{4}$$

$$Mg_3Si_2O_5(OH)_4 + 3CO_2 \rightarrow 3MgCO_3 + 2SiO_2 + 2H_2O \tag{5}$$

Solution Modification

Modifications to the carbonation solution chemistry were also investigated. This included the use of a bicarbonate/salt mixture, rather than distilled water, which improved the reaction rate dramatically. It is theorized that the bicarbonate ion hydrolyzes the silicate, forming the carbonate, hydroxide (OH^-) ions, and free silica (SiO_2), by the following reaction (Eq. 6).

$$Mg_2SiO_4 + 2HCO_3^- \rightarrow 2MgCO_3 + SiO_2 + 2OH^- \qquad (6)$$

$$OH^- + CO_2 \rightarrow HCO_3^- \qquad (7)$$

The OH^- ion is believed to react immediately with the additional CO_2 being injected into the solution to reform the bicarbonate (Eq. 7), maintaining a relatively constant solution chemistry. This may be described as a buffered solution. The in-situ pH of the solution can not be measured, but pre- and post-test pH measurements remain relatively constant, in the range of 7.7-8.0. This supports the assertion that the solution chemistry remains relatively constant, and verifies that the bicarbonate addition is not consumed in the reaction, but acts as a catalyst.

The salt (NaCl) addition is believed to provide a complexing ion (Cl^-), which combines with Mg^{2+} cations to form several intermediate magnesium chloride compounds. This complexing ion effectively reduces the activity of the Mg^{2+} cation in solution, increasing the solubility of the magnesium silicate mineral.

The use of the sodium bicarbonate and sodium chloride solution, in place of distilled water, resulted in carbonation of naturally occurring olivine to approximately 84% of the stoichiometric maximum in 6 hours, at 185˚C and P_{CO2} of 115 atm (Table 2, test SC-40).

Table 2: Test summaries for the direct mineral carbonation tests

Test	Test time, h	T, ˚C	P_{CO_2}, atm	Feed material	Particle size, μm	Solution chemistry	Pct stoich. conv.
SC-25	24	185	115	olivine	-37	distilled water	91
SC-33	24	185	115	antigorite	-37	distilled water	34
SC-39	6	185	115	HT antigorite[1]	-37	0.5 M NaHCO$_3$, 1 M NaCl	83
SC-40	6	185	115	olivine	-37	0.5 M NaHCO$_3$, 1 M NaCl	84
SC-73	3	185	115	HT antigorite[1]	-37	0.5 M NaHCO$_3$, 1 M NaCl	79
SC-83	1	155	150	HT antigorite[1]	-37	0.64 M NaHCO$_3$, 1 M NaCl	82
SC-84	0.5	155	185	HT antigorite[1]	-37	0.64 M NaHCO$_3$, 1 M NaCl	78
SC-100	1	155	150	HT antigorite[2]	-37	0.64 M NaHCO$_3$, 1 M NaCl	79
SC-163	1	155	150	HT lizardite[3]	-75	0.64 M NaHCO$_3$, 1 M NaCl	49
SC-166	1	155	150	HT antigorite[2]	-75	0.64 M NaHCO$_3$, 1 M NaCl	59
SC-168	---	155	10	HT lizardite[3]	-75	0.64 M NaHCO$_3$, 1 M NaCl	49

[1] Antigorite serpentine heat treated in CO_2.
[2] Non-magnetic antigorite serpentine fraction, heat treated in air.
[3] Non-magnetic lizardite serpentine fraction, heat treated in air.

Olivine Carbonation

The initial series of olivine carbonation tests included no pretreatment options, other than size reduction to the desired particle size. These tests, exploratory in nature, were intended to produce a baseline set of test parameters for the carbonic acid process, utilizing distilled water as the liquid medium and continuous agitation of the slurry. The addition of gas dispersion in the water column resulted in significant improvement in the extent of reaction, culminating in 91 pct stoichiometric conversion of the silicate to the carbonate in 24 hours, at a temperature of 185°C and P_{CO2} of 115 atm (Table 2, test SC-25). Formation of the carbonate was confirmed by the identification of magnesite ($MgCO_3$) in the reaction products by X-ray diffraction (XRD) analysis.

Particle size was recognized as a major factor determining reaction rate and extent of reaction, because most mineral dissolution reactions are surface controlled. Although coarser sizes can be used effectively, optimal reaction conditions include a particle size of minus 37 microns.

Serpentine Pretreatment and Carbonation

During investigations conducted on naturally occurring serpentine, it was demonstrated that heat treating the serpentine, to remove chemically-bonded water and activate the mineral, improved the carbonation results (Table 2, tests SC-33 and 39). Differential thermal and gravimetric analysis, coupled with experimental results from a series of heat treatment and carbonation tests, identified the optimum temperature range for heat treatment to be 600-650°C. The relationship between heat treatment temperature, weight loss from the serpentine, and extent of the carbonation reaction are depicted graphically in Figure 2. These tests were conducted on the antigorite variety of serpentine.
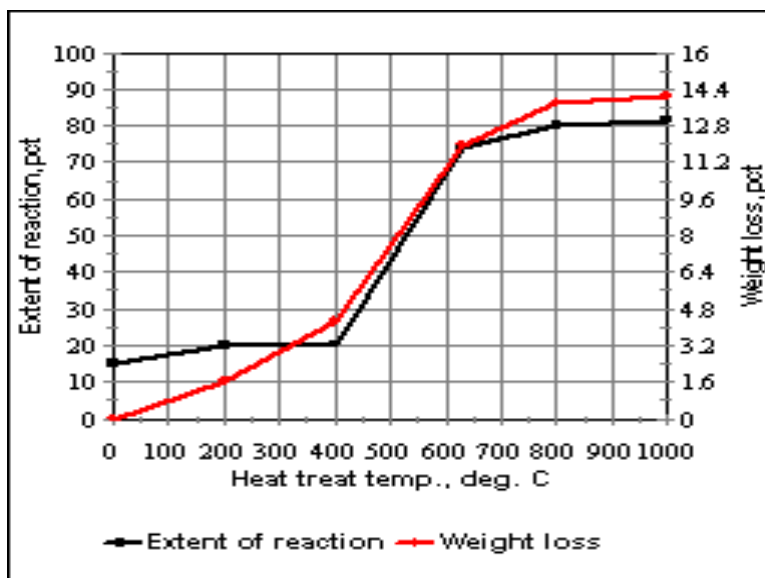

Figure 2.- Relationship between heat treatment temperature, weight loss, and extent of carbonation.

Weight loss (or water removal) increases significantly above 600°C, with heat treatments conducted below this temperature proving ineffective. Heat treatment temperatures above 650°C can remove up to 1-2 wt pct additional water, but are not considered cost effective. Energy requirements for heat treatment at 600-650°C are estimated at roughly 200 kW•h/ton of serpentine. Steam generation from the heat treatment process represents potential recoverable energy, although current studies are insufficient to determine specific values.

Magnetic separation was conducted on the serpentine to remove the magnetite (Fe_3O_4) that occurs naturally with the serpentine. This was done for two reasons: 1) the magnetite concentrate represents a marketable product; and 2) heat treatment of the serpentine can be conducted in an oxidizing atmosphere after the magnetite is removed. Oxidation of the magnetite during heat treatment has been shown to inhibit the carbonation reaction. The magnetic separation step proved effective. The

concentration of iron oxides in the magnetic concentrate (~45 wt pct total iron oxides) compares favorably with typical iron ore. Additional processing could produce a high grade concentrate. Heat treatment tests on the non-magnetic serpentine fraction were conducted under non-oxidizing and oxidizing atmospheres. Results from the subsequent carbonation tests (Table 2, tests SC-83 and 100) demonstrate that the heat treatment atmosphere is no longer critical, once the majority of the magnetite is removed. This simplifies the heat treatment process, and should reduce costs. However, recent tests have indicated that removal of the iron oxide has reduced the reactivity of the heat treated serpentine, suggesting that the iron catalyzes the dissolution/carbonation reactions. Additional study is required to determine the effect of iron oxide, which perhaps acts as a control on redox conditions.

The serpentine feed material is comprised of three minerals in the serpentine group, antigorite, lizardite, and chrysotile. In the ore samples collected, chrysotile occurs as a secondary mineral, with either the antigorite or lizardite the more abundant of the two. In general, antigorite and lizardite do not occur together. Chrysotile is the asbestos variety of serpentine, and is the primary industrial mineral used for the manufacture of asbestos products. Chrysotile was not identified by XRD in any of the carbonation reaction products from tests using the heat treated serpentine as feed material. This suggests that the favored process utilized for serpentine, heat treatment followed by direct carbonation, destroys the chrysotile, making it an effective method for the remediation of asbestos wastes.

Variations in the LOI for the heat treatment products appear to play the predominate role in determining the extent of carbonation, when all other parameters are held constant (Figure 3). Significant variations in LOI for the various heat treatment products, from ~3 to ~6 wt pct, were identified as the primary reason for the reduced extent of carbonation. These irregularities in LOI occurred even though all heat treatment tests were conducted at "identical" conditions. At the bench-scale, minor variations in operating conditions from heat to heat are unavoidable, and they appear to be significant enough to effect the final LOI. Because the serpentine head material contains ~13.5 wt pct chemically bonded water, every 1% LOI in the heat treatment product represents about 7% of

Figure 3.- Relationship between residual LOI and extent of carbonation.

mineral that is still hydrated. The ARC studies strongly indicate that the serpentine containing chemically-bonded water will not carbonate, at least not at the more rapid rate. Thus, every 1% LOI in the heat treatment product (carbonation feed) reduces the effective carbonation potential by about 7%. The relationship between the residual LOI and extent of carbonation appears to be linear, so it may be possible to compare all prior and subsequent tests at a normalized LOI, perhaps 3.5 wt pct. This would eliminate the apparent variations in extent of reaction due to imperfections in heat treatment.
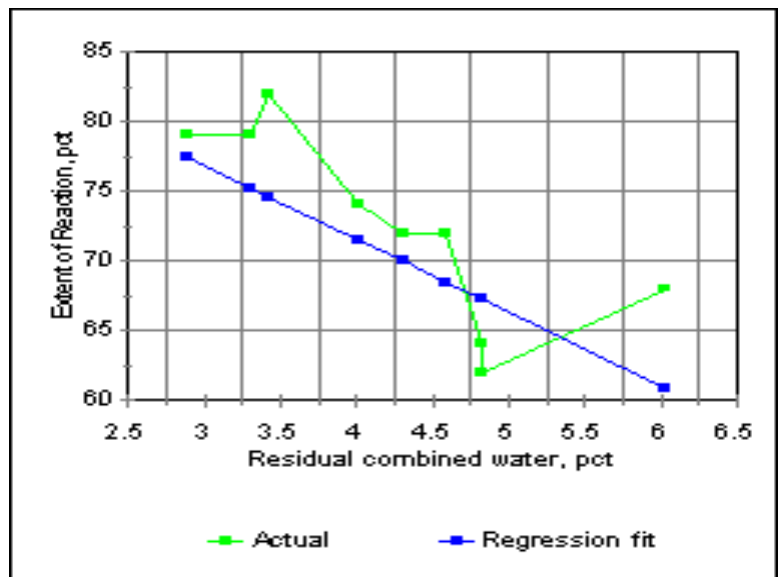
Reaction Kinetics

The reaction rate for serpentine carbonation has been continuously improved by slight reduction of the reaction temperature, increases in the P_{CO2}, and additional modifications of the solution chemistry (Table 2, tests SC-73, 83, and 84). The optimum reaction conditions identified to date for heat treated serpentine include a sodium bicarbonate and sodium chloride solution chemistry, and reaction temperature of 155˚C at a P_{CO2} of 185 atm. These conditions resulted in serpentine carbonation to approximately 78% of the stoichiometric maximum in 0.5 hours (Table 2, test SC-84). Magnesite was again identified by XRD as the primary phase in the reaction products.

Three series of carbonation tests were conducted to determine rate curves for the direct mineral carbonation reaction. The time series included tests conducted for 0, 0.5, 1, 3, 6, and 12 hours, on a heat pretreated, nonmagnetic fraction of antigorite serpentine, a heat pretreated, nonmagnetic fraction of lizardite serpentine, and foundry grade olivine. Test conditions were kept constant: particle size of 90% minus 200 mesh (75 microns); 155˚C; P_{CO2} =150 atm; stirring speed 1,000 rpm; and 15% solids, in a solution of 0.64 M $NaHCO_3$, 1 M NaCl. The series are included in Figure 4.
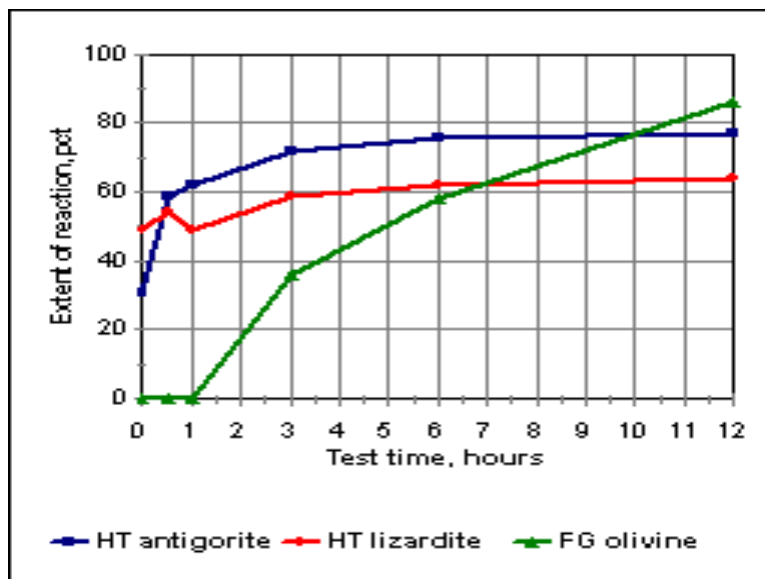

Figure 4.- Reaction rate curves for the direct mineral carbonation reaction.

The much higher reactivity of the heat pretreated serpentines is apparent from the curves. The "zero" hour tests were conducted to determine the extent of reaction that occurs while heating the system to operating temperature. After purging the vessel, a residual P_{CO2} of ~10 atm remains. This, along with the bicarbonate solution, provide sufficient reactant to carbonate a significant portion (~31%) of the heat pretreated antigorite serpentine, and ~49% of the heat pretreated lizardite serpentine, before reaching final conditions. In fact, the lizardite curve shows little improvement in the extent of carbonation, even after 12 hours. This suggests that some portion of the heat pretreated serpentines are highly reactive. These results are of great interest, because they indicate the potential for significant mineral carbonation at much reduced P_{CO2}. Characterization of the meta-stable serpentine compounds produced by heat pretreatment is a high priority for future studies.

This is not the case for the olivine, however. No reaction was apparent after 1 hour on the olivine, at a stirring speed of 1,000 rpm. This can be overcome to some extent by increasing the stiring speed to 2,000 rpm. But in any case, it is obvious from Figure 4 that either some pretreatment of the olivine, or a more aggressive leach solution, seems necessary to shift its rate curve to the left.

Applications

The primary application for the direct mineral carbonation process is the reduction of CO_2 emissions from fossil-fuel-fired power plants. Integration of mineral carbonation into other advanced processing techniques, such as cryogenic oxygen injection and flue gas recycle, could make complete flue gas cleaning, including CO_2, a possibility. Other secondary applications have been alluded to previously, such as magnetic separation to produce an iron ore by-product, and asbestos waste remediation by combined heat treatment and carbonation. Potential product uses include mine reclamation and agricultural amendments. The magnesite product, which comprises ~65% of the total product solids, could potentially be used as a substitute for conventional soil liming agents. The free silica, which comprises ~25% of the carbonation product, is extremely hydrophyllic, which could improve soil water retention in arid environments. Beneficial product uses are to be investigated as part of future project studies.

Future Studies

Future studies fall into three main categories: (1) technical process development; (2) reactor design and scale-up, and (3) in-situ applications. Process development studies are to continue the investigation of reaction kinetics. Mineral pretreatment options, such as ultrasonic and/or microwave treatment, will be investigated as means to produce a more readily soluble mineral reactant. Especially important is the characterization of the meta-stable serpentine compounds produced by heat treatment. Because these compounds appear to be readily carbonated at reduced P_{CO2} (~10 atm), they represent a tremendous potential for improved process economics. The identification of means to produce these compounds at a lower energy penalty than the current process is critical.

Reactor design studies will focus on the development of a flow-type, or pipeline reactor. The non-conventional, 3-phase nature of this system requires unique studies into the processing of gas/liquid slurries with gas dispersion. However, the continuous flow reactor is expected to prove far more economical than batch-type systems. Development, construction, and demonstration of a bench-scale flow reactor (~5 lb/h) is scheduled over the next 18 months.

Finally, application of these ex-situ studies to in-situ mineral sequestration could lead to promising results. Targeting the large ultramafic sequences for CO_2 injection studies is intriguing. Current geologic sequestration studies have focused on sedimentary formations with little potential for mineral carbonation. However, the ultramafic rocks contain abundant reactant, in the form of magnesium silicates, which the subject studies have shown to be amenable to carbonation in bicarbonate solutions. The next logical step is to attempt these reactions in-situ, where formation depth (~ 1 km) could provide sufficient heat and pressure to drive the reaction, with reaction time no longer such a critical factor.

Conclusions

An aqueous process for the direct carbonation of silicate minerals has been developed as a method for CO_2 storage in solid form. Reaction rates have been improved by utilization of supercritical CO_2, at P_{CO2} of 115-190 atm, temperatures of 155-185°C, and a solution chemistry including sodium bicarbonate and sodium chloride. Up to 78% stoichiometric conversion of the silicate mineral to the carbonate has been achieved in 30 minutes under these conditions. The carbonation reaction product consists essentially of magnesite (~60 wt pct), free silica (~25 wt pct), and residual silicate (~15 wt pct). Potential uses for the magnesite/silica product include soil amendments, replacing materials such as lime (CaO), limestone ($CaCO_3$), and/or dolomite [$(Ca,Mg)CO_3$]. The hydrophillic nature of the free silica may also improve soil water retention where necessary. A potential by-product from the process is the production of an iron ore concentrate of comparable grade to typical iron ore concentrates. In addition, the results from these studies suggest that the process may be an effective method for the remediation of asbestos wastes, due to the apparent destruction of chrystotile by combined heat treatment and direct carbonation. Future studies are intended to investigate various mineral pretreatment options, alternative reactants, scale-up to a continuous process, and process economics.

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