

CO₂ Sequestration by Direct Dry Gas-solid Contact of Serpentinite Mining Residues: A Solution for Industrial CO₂ Emission

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Abstract – Direct dry gas-solid carbonation is a simple approach towards mineral carbon dioxide sequestration. The route theoretically implies the direct reaction of CO₂ with silicates of Calcium and Magnesium in dry condition to form stable, insoluble metal carbonates. The mining regions of southern Québec have a large deposit of serpentinite residues. The current study examines the suitability of serpentinite mining residues to use as feedstock material for mineral carbonation. The focus of the present work is to assess the CO₂ removal efficiency of the residue from a simulated flue gas mixture of a typical cement plant (18 Vol% CO₂). This approach avoids the requirement of separate CO₂ capture and pre-concentration prior to mineral carbonation. The reaction parameters considered are temperature, pressure and time. The optimization of parameters is carried out for the maximum CO₂ removal efficiency (%) from the feed gas. Operating condition for CO₂ removal is optimized at 258 °C, 5.6 barg (pCO₂ ≈ 1) for 310 minutes with a removal efficiency of 37%. Preliminary analysis of reacted solid indicates carbonation is null at optimum condition, possibly a reversible adsorption might be responsible for the depletion of CO₂ from feed gas. The study also checks the importance of pre-treatment options such as grinding, magnetic separation and heat treatment on CO₂ removal. A separate optimization study is carried out for magnetic separation of serpentinite residue and the separation parameters are optimized at an initial pulp density of 40% and magnetic intensity of 7.5*10⁻³ T with about 70% of iron oxide removal from the initial feed.

Keywords: Serpentinite, mining residues, CO₂ sequestration, direct dry gas-solid.

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1. Introduction

The augmentation of greenhouse gases such as carbon dioxide (CO₂) in the atmosphere has led to an increase in global temperature and changes in the climate. In order to mitigate the potentially devastating consequences of that phenomenon, the emissions of anthropogenic greenhouse gases especially CO₂ into the atmosphere should be reduced [1]. Carbon dioxide capture and storage (CCS) is a well-known option for mitigating the unwanted anthropogenic CO₂ emissions. Geological formation and ocean are the widely used sinks for carbon dioxide storage, but they are limited either due to the lack of permanence in storage or due to environmental issues associated with storage [2]. Mineral carbonation is the promising CCS option which guarantees the permanent storage of CO₂ sequestered [3, 4]. This option mimics the process of natural silicate weathering in which CO₂ reacts with the divalent cation (Ca²⁺ or Mg²⁺) of natural minerals to form metal carbonates [5]. The carbonates formed are environmentally benign and geologically stable [2-4, 6]. Mineral carbonation is now in the developing stage, but the cost and kinetics keep it laid-back from other CO₂ storage options [2]. Mafic and ultramafic rocks

containing magnesium (Mg) and calcium (Ca) are generally used for mineral carbonation due to their relative abundance and admissible reactivity with CO₂ [5]. In comparison to other metal silicates, magnesium based minerals such as serpentine and olivine are most abundant in nature [7]. Therefore, these minerals are under research scrutiny to develop as a cheap raw material for mineral carbonation [8, 9]. Besides these natural forms, waste materials contained admissible concentration of Mg and Ca have also been employed as mineral carbonation feedstock. These include mineral tailings of asbestos, industrial waste like stainless steel slag, waste cement, fly and bottom ash from municipal solid waste incinerator etc. [1, 7].

Mineral carbonation could be done either directly or indirectly through aqueous or dry route. Direct gas-solid route is a straight forward approach towards mineral carbonation and exothermic in nature [7, 10, 11]. Since the carbonation rate of pure mineral under direct dry condition was found to be slow, many modifications were recently suggested. Most recent works have been reported using a multistep gas-solid carbonation of magnesium hydroxide [Mg(OH)₂] produced from serpentine [12-16]. Another recent approach has been reported with the addition of a small amount of water or water vapor for carbonation enhancement [17-21]. Most of the previous works on direct gas-solid route was mainly focused on the carbonation of solid material [13, 19, 21, 22] and less work have been reported on the CO₂ depletion potential of minerals [18, 20].

The present is a preliminary laboratory study conducted using a batch wise mode. The objective of the study is to check the feasibility of serpentinite mining residue as a CO₂ removal or carbonation material using direct dry gas-solid route. The mining residue used is from a chrysotile extraction mine in southern Québec. Direct dry gas-solid route was selected because of the simplicity and exclusion of chemical additives. The study adapted a new strategy of directly using a readily available mining residue for the capture and storage of CO₂ from an industrial flue gas. Instead of pure mineral, using a mine residue that contains various mineral phases might provide a better reaction and could avoid the mining and mineral purification requirements. Direct use of a flue gas composition (18% CO₂) could avoid the separate steps of CO₂ capture and pre-concentration.

2. Experimental Section

Serpentinite Mining Residue (SMR) collected from Black Lake mine (Québec) was used for the present study. Collected samples were homogenised and then stored in separate sealed containers. The moisture content of the raw sample was measured by heating the mineral sample at 110 °C for around 24 h. The texture analysis of the sample was carried out by sieving through meshes with different cut sizes between 0.075 mm to 2 mm.

2.1. Material Characterization

Mineral phase of the residue was identified by X-ray diffraction (XRD) analysis using a Siemens D5000 diffractometer with Cu K α radiation. Scans were taken for 2 θ over 2° to 65° at 0.02°/s. Microscopic imaging and a semiquantitative analysis of the sample was done with a scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDS) (Zeiss Evo 50 Smart SEM). Chemical composition of the mineral sample was determined by inductively coupled plasma atomic emission spectroscopy (ICP-AES) (Varian 725-ES, Model Vista-AX CCO, Palto Alto, CA, USA) after an alkaline fusion with lithium metaborate. The specific surface area and average pore diameter of the sample were determined by means of Brunauer–Emmett–Teller (BET) analyzer (BELSORP-max, BEL Japan Inc). For this, the samples were dried and analyzed for N₂ sorption and desorption isotherm at 77 K after degassing at 150 °C overnight and the residual pressure down to 10⁻⁵ Torr.

2.2. Experimental Procedure

2.2.1. Mineral Pre-treatment

The grinding of SMR was done in a shatter-box (BLEULER-NAEF shatter-box, model M04/06) to increase the surface area and thereby enhance the reaction rate. The particle size of the ground SMR was determined by a laser particle analyzer (Horiba laser particle size distribution analyzer LA-950). The others pre-treatments options adapted for the current study were thermal heat treatment and magnetic separation. Temperature for thermal treatment was adapted from the previous studies conducted with similar type materials [23-27]. For this, the ground SMR was heated to 650 °C for 30 min in an air muffle furnace (Thermolyne Furnatrol 133). Magnetic separation was carried out by using a wet high intensity magnetic separator (WHIMS -CARPCO model serial no. 221-02) in order to remove the magnetic impurities; especially

compounds of iron (Fe) from SMR. This was carried out to avoid the formation of a passive layer of iron oxide such as hematite during heat treatment which negatively affects the carbonation process [27, 28]. Conducting magnetic separation prior mineral carbonation can also provide final carbonation products of less magnetic impurities and a separate stream of magnetic by-product with good potential market value. Optimization of parameters for magnetic separation of SMR was separately carried out and the non-magnetic sample at optimum condition was used for direct dry gas-solid experiments. Parameters considered for the magnetic separation study were magnetic intensity (T) and initial pulp density (%).

2.2.2. Direct Dry Gas-solid Reaction

Samples for direct dry mineral reaction were categorized into three types: (1) raw, (2) heat-treated, and (3) magnetically separated samples. All tests were conducted in a batch reactor designed by Parr (Parr 4560 Mini Bench Top Reactor) of capacity 300 mL. The representation of the experimental set-up is given in Figure 1.

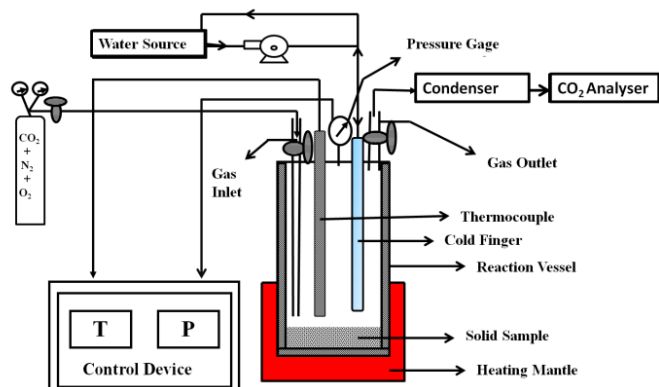


Figure 1. Diagrammatic representation of direct dry gas-solid carbonation.

Direct dry gas-solid reactions were carried out by contacting ground SMR with CO₂ gas mixture (N₂/CO₂/O₂-78/18/4% (v/v)) at varying temperature and pressure. Based on the previous studies on this route, the reaction parameters considered for optimization were temperature, pressure and time [11, 12, 19, 29]. The direct dry gas-solid experiments were conducted by loading a definite mass (g) of ground SMR into the reactor. Then, it was heated to the desired reaction temperature (°C). After reaching the reaction temperature, the air and moisture inside the reaction vessel was purged and filled with the gas mixture to a

desired pressure (bar). After a certain reaction time (min), the non-reacted gas was collected in a *Tedlar Bags* (3.8 L) equipped with an *on/off valve* and subjected to analysis. The concentration of CO₂ (Vol%), before and after reaction, was measured by means of a CO₂ analyzer (Quantec instrument model 906) and the mass of CO₂ in the inlet and outlet gas was calculated using the ideal gas equation (PV=nRT) by knowing the reaction temperature and CO₂ partial pressure.

The reaction deciding factor considered in the parameters optimization was the efficiency of CO₂ removal (%) (Equation 1). Initial experiments were conducted in conventional method (single variable at a time) using raw, heat-treated and nonmagnetic sample. This was carried out to get an approximate idea about the parameter influence on CO₂ removal. Then, the optimization was carried out with a statistical Box-Behnken model (Design Expert with ANOVA: Version 8.0.4, Stat-Ease Inc., Minneapolis, USA). The independent variables considered for the model were temperature, total pressure and time with corresponding ranges of 25 to 280 °C, 5 to 95 bar and 120 to 360 min respectively. The model designed 17 experiments and heat-treated sample of 25 g was used for the each experiment. Based on the experimental results obtained, the model predicted a most desirable interaction of parameters for the maximum response. Experimental validation of the model was carried out on the predicted optimized conditions. Comparative results were also generated with raw and nonmagnetic sample at optimum conditions.

$$\text{CO}_2 \text{ removal efficiency (\%)} = \frac{((\text{CO}_{2\text{in}} - \text{CO}_{2\text{out}}) / \text{CO}_{2\text{out}}) \times 100}{(1)} \quad (1)$$

Where CO_{2in} is the mass of CO₂ in the inlet gas and CO_{2out} is the mass of CO₂ in the outlet gas. The solid samples at optimum result conditions were subjected to both elemental carbon analysis (Leco CHNS-932 auto analyzer) and XRD to assess the formation of carbonates.

3. Results and Discussion

3.1. Characterization of SMR

The average interstitial water content of SMR from Black Lake residue was about 5.3% (±0.5). The texture analysis of SMR shows about 38% of the sample was above 2 mm, 22.9% was between 1 to 2 mm and remaining 39.1% was below 1 mm. Since 60% of the sample was above 1 mm, grinding was adapted to

reduce the size to a micro level. The mean size of the ground SMR given by laser particle analyzer was 75 μm with 90% distribution below 250 μm . The XRD gives major peaks for lizardite associated with minor components such as chrysotile, magnetite, brucite, talc and chlorite. This is in concurrence with mineral compositions reported for similar type materials near the present sampling station [17, 26, 30, 31]. The EDS analysis of the site within the SEM images confirmed the presence of Fe substituted lizardite and magnetite (Fe_3O_4). The chemical composition of SMR in each size fraction is presented in Table 1. The sample is mainly

concentrated with magnesium and silicon with a considerable level of Fe. This confirms the presence of serpentine group mineral (lizardite). The presence of Fe would account for both magnetite and partial substituted Fe within the lizardite structure. The average percentage of MgO ($42.5\pm 0.2\%$) and SiO_2 ($40\pm 0.4\%$) in SMR are admissible range with known average value for lizardite [32]. The BET specific surface area and average pore diameter of the SMR were $11.5 \text{ m}^2/\text{g}$ and 13.7 nm respectively.

Table 1. Particle size distribution and chemical composition of SMR from Black Lake mine.

Mesh size (mm)	Weight (%)	Fe_2O_3 (%)	MgO (%)	SiO_2 (%)	Ni (mg/kg)	Co (mg/kg)	Cr (mg/kg)
≥ 2	38.0	11.1	41.5	37.0	2409	63.9	2493
1-2	22.9	9.0	36.9	37.7	1702	107.6	2010
0.5-1	17.8	10.1	41.3	37.9	2017	74.8	2160
0.3-0.5	9.3	11.9	39.4	36.5	1895	63.3	1991
0.15-0.3	7.5	12.2	34.8	35.9	1696	108.6	2554
0.075-0.15	3.4	18.6	36.9	34.5	1968	73.4	2856
≤ 0.075	1.1	21.9	31.9	32.4	1739	135.3	2545

3.2. Magnetic Separation

The best conditions for magnetic separation of SMR from mine Black Lake was optimized at a magnetic intensity of $7.5 \times 10^{-3} \text{ T}$ and an initial pulp density of 40% with ground SMR of mean size 75 μm . The second pass of magnetic separation products at above mentioned conditions given a maximum non-magnetic mass recovery of 90.0% (± 0.2). The magnetic separation reduces the iron oxide (as Fe_2O_3) concentration of SMR from 10.9% (± 0.4) to 3.4% (± 0.1). From the raw feed about 97% of magnesium has been recovered in non-magnetic fractions. The final recovered magnetic fraction is rich in iron oxide with 79.0% (± 0.3) composition. All the above results are calculated based on the ICP-AES analysis (after alkaline fusion with lithium metaborate). In short, conducting magnetic separation at above conditions resulted in 70% of iron oxide impurities removal from feed SMR.

3.3. Direct Dry Gas-solid Reaction

The maximum CO_2 removal obtained using conventional single variable method was about 36.7% (± 2) with heat treated sample at 200 $^\circ\text{C}$, 5 bar ($p\text{CO}_2 = 0.90 \text{ bar}$) after 360 min (6 h) duration. The response

surface methodology (RSM) graph obtained from Box-Behnken analysis is given in Figure 2. The result shows that the CO_2 removal percentage was increased with increases in temperature and decreases in total pressure. The Model Prob > F value less than 0.05; implies that the model is significant. The value of multiple regression coefficients, $R^2 = 0.98$, shows that the model could explain 98% of the response variability and which was in reasonable agreement with the adjusted R^2 value (0.94). The model predicted an optimum condition at 258 $^\circ\text{C}$, 5.6 bar ($p\text{CO}_2 \approx 1$) for 310 min (5 h) with 40% CO_2 removal and was validated experimentally with 37% (± 0.6) CO_2 removal at these conditions. Direct dry reaction of raw and non-magnetic samples was also conducted at optimum condition (Box-Behnken) and obtained almost equal CO_2 removal. At the maximum removal efficiency (37%), 25 g SMR removed about 0.12 g (± 0.01) of CO_2 from the feed gas with initial CO_2 concentration of 0.32 g CO_2 . The elemental carbon and XRD analysis carried for reacted solid at optimum conditions did not show any variation from the original sample. This indicates CO_2 might be removed from the feed gas due to a reversible adsorption rather than carbonation.

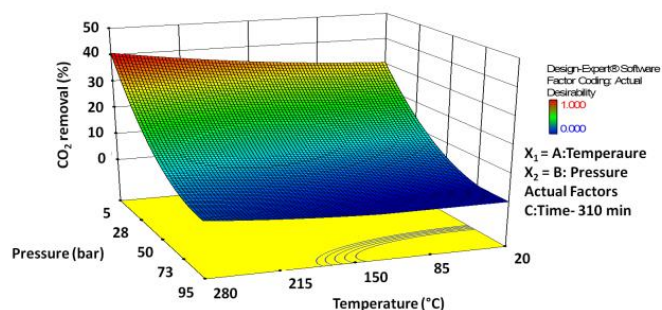


Figure 2. RSM graph showing the interaction of T and P on CO₂ removal efficiency.

The results furnished for CO₂ removal are only based on the CO₂ analyzer measurement. The current removal is too low for practical application. We suggest that a limited CO₂ removal occurred due to the poor gas-solid contact in the batch set-up. Experiments are required to be conducted with the presence of water vapor to enhance carbonation [17-21]. More process improvement is required to enhance the CO₂ removal efficiency of solid such as increasing the gas-solid interaction through the fluidization of the bed and increasing the surface area and pore size of solid through a series of grinding.

4. Conclusion

The present work highlighted a new approach towards direct gas-solid carbonation by incorporating a mine waste for the direct capture or sequestration of CO₂ from an industrial chimney. The usage of a residue rather than pure mineral can help in avoiding the mining and mineral purification cost requirement for mineral CO₂ sequestration. Beside this, the process will revalorize the waste residue into environmentally benign material that can be used for land filling and mine reclamation (only if carbonation occurs). Switching from 100% CO₂ to simulated flue gas composition of 18 Vol% CO₂ can reduce the separate CO₂ capture and pre-concentration cost. The main challenge of the route is achieving an admissible reaction rate with a low cost and energy consuming process.

The preliminary results obtained through the characterization and the parameter optimization for the mineral CO₂ sequestration of SMR shows that SMR can be selected as a feedstock for CO₂ sequestration. The reacted solid analysis indicates that the carbonation

under dry condition is negligible, but that the CO₂ is depleted from the feed gas possibly due to a reversible adsorption. Carbonation possibilities have to be assessed by the addition of water vapor. Studies are further required in a suitable design such as fluidized bed providing better gas-solid interaction to increase the reaction rate and kinetics. More extensive studies are required in this area to reach an admissible reaction rate and kinetics and thereby develop a promising process for an industrial application. The prime goal of the future work is to have an admissible carbonation reaction at low pressure and low temperature within the short reaction period.

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