



Research article

Effect of $p\text{CO}_2$ on direct flue gas mineral carbonation at pilot scale

Ikbel Mouedhen, Nassima Kemache, Louis-César Pasquier*, Emmanuelle Cecchi, Jean-François Blais, Guy Mercier

Institut National de la Recherche Scientifique, Centre Eau Terre et Environnement, 490 rue de la Couronne, Québec, QC, G1K 9A9, Canada

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ABSTRACT

Concerns about global warming phenomena induced the development of research about the control of anthropogenic greenhouse gases emissions. The current work studies on the scaling up of aqueous mineral carbonation route to reduce the CO_2 emissions at the chimney of industrial emitters. The reactivity of serpentinite in a stirred tank reactor was studied for several partial pressures of CO_2 ($p\text{CO}_2$) (0.4, 0.7, 1.3 and 1.6 bar). Prior to carbonation, the feedstock was finely grinded and dehydroxylated at 650 °C by a thermal treatment. The major content of magnetite was removed (7.5 wt% · total weight⁻¹). Experiments were carried out under batch mode at room temperature using real cement plant flue gas (14–18 vol% CO_2) and open pit drainage water. The effect of the raw water and the $p\text{CO}_2$ on the carbonation efficiency was measured. First, the main results showed a positive effect of the quarry water as a slight enhancement of the Mg leaching in comparison with distilled water. Secondly, a $p\text{CO}_2$ of 1.3 bar was the optimal working pressure which provided the highest efficiency of the carbonation reaction (0.8 g CO_2 · g residue⁻¹). Precipitation rates of dissolved CO_2 ranged from 7% to 33%. Pure precipitate was obtained and essentially composed of Nesquehonite. At a $p\text{CO}_2$ of 1.3 bar, additional physical retreatment of the solid material after being contacted with 6 batches of gas enhanced considerably mineral carbonation efficiency (0.17 g CO_2 · g residue⁻¹).

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1. Introduction

The emissions of anthropogenic greenhouse gases have continuously increased during the industrial period and largely contributed to the global warming phenomena (IPCC, 2014). The concentration of CO_2 registered in the atmosphere is gradually rising from 280 ppm in the 19th century to reach 400 ppm nowadays. This is especially due to the intensive use of fossil fuels as a primary energy sources (MacKenzie et al., 2004). Among the different solutions proposed, geological storage and mineral carbonation (MC) processes represent interesting solutions for long term CO_2 disposal. While to date, large-scale capture and storage projects using geological storage are underway, large-scale applications of MC are still limited. In fact, even if MC offers the safest way to store CO_2 , its large energy requirement still stand as a major barrier.

The application of MC for the reduction of anthropogenic CO_2 was first proposed by Seifritz (1990). It is based on the magnesium/

calcium (Mg/Ca) silicate weathering (Lasaga and Berner, 1998). The reaction is exothermic with a slow kinetic, taking place on a geological time scale (Pan et al., 2012). However, the obtained products are known to be stable for a large range of temperature and pressure (Sipilä et al., 2008). Several feed stocks are known to be efficient for MC, such as natural minerals like wollastonite ($\approx 48.3\%$ /wt CaO), olivine ($\approx 49.5\%$ /wt MgO) and serpentine ($\approx 40\%$ /wt MgO) or industrial wastes or mine tailings (Sanna et al., 2014). The aqueous route is the most effective and can be described by three mechanisms. First, the CO_2 dissolution into the liquid phase, then the Mg/Ca leaching and finally the carbonate precipitation (Huijgen et al., 2006). All steps are critical and contribute to the overall reaction efficiency. Nevertheless, the solid dissolution remains the limiting step which control the cation availability for the carbonate precipitation. For this reason, the particle size is a key point. A fine grinding of the feedstock will increase the specific surface area of solid particles and thus promotes the cation dissolution (Alexander et al., 2007).

Among the different feedstocks studied, serpentinite minerals were intensively studied. As MC was first viewed as an alternative to geological storage, pure CO_2 stream treatment was investigated. The processing of supercritical CO_2 in high temperature and

* Corresponding author.

E-mail address: louis-cesar.pasquier@ete.inrs.ca (L.-C. Pasquier).

pressure conditions (155 °C, 115 bars) with a carrier solution (0.64 M NaHCO₃ and 1 M NaCl) and a reaction time of 60 min led to a satisfying conversion rate of 73.5% (Gerdemann et al., 2007). Nevertheless, these hydrated minerals required a heat activation at 650 °C for 30 min to remove hydroxyl groups and thus to increase the material reactivity (McKelvy et al., 2004). In addition, the use of high temperature and pressure conditions globally limits the potential industrial application due to global high energy demands. To counteract the heat activation step, different indirect methodologies were proposed in order to extract Mg prior to carbonation, such as “pH swing” (Nduagu et al., 2012; Park and Fan, 2004). Nevertheless, feasibility of such processes depends on the ability to recover chemicals/acids used and still require high temperatures and/or pressure operating conditions. Another alternative is to implement MC as an integrated capture and storage process. This route has the advantage to avoid a capture step which clearly simplifies the process scheme. In this way, different studies focused on using a diluted CO₂ stream, similar at some industrial flue gas (Pasquier et al., 2014a; Werner et al., 2014). Results showed that reaction mechanism, kinetics were similar as observed for pure CO₂ treatments and led to promising results (Hariharan et al., 2014; Pasquier et al., 2014b). In addition, dissociation of gas treatment steps and carbonates precipitation leads to the production of high purity products giving significant incomes.

The use of alkaline residues as MC feedstock present several advantages. Mostly, the use of an already ground material allows the decrease energy requirements for the particle size reduction (Bobicki et al., 2012). In addition, some of them are available on CO₂ emitting industrial sites such as steel slags or cement kiln dust. This approach allows to answer GHG mitigation jointly with waste valorization and reuse. In the case of serpentine, about 2Gt of chrysotile mining residues (mostly composed of lizardite or antigorite) were generated in Quebec's southern province. Those are known to react spontaneously with CO₂ contained in the air but with geologic scale kinetics (weathering process) (Huot et al., 2003; Wilson et al., 2009). Their reactivity in stirred reactor was also demonstrated using diluted gas (Pasquier et al., 2016). Recently, experiments were performed using such material for the treatment of a real cement plant flue gas at a pilot scale (Kemache et al., 2016). Results showed that the scaling effect was neglectable and that water recirculation enhances carbonates precipitation. The present work is in the continuity of the latter work. The impact of *p*CO₂ variations and the use of raw industrial water on the reaction efficiency are discussed.

2. Materials and methods

2.1. Analytical methods

For each sample, chemical composition was characterised, before and after each process steps/reaction in order to follow the material and liquid and gaseous stream variations. Solid samples were melted using alkaline fusion with lithium metaborate (Claisse method) (Tertian and Claisse, 1982). The resulting liquids were analyzed by inductively coupled plasma spectrometry of emission (ICP-OES, Varian). Liquid samples were also analyzed using ICP-OES. The inorganic carbon analysis in the solid samples was done with the carbon hydrogen nitrogen sulfur analyser (Leco CHNS). Inorganic carbon concentration was measured in liquid samples using a Total Organic Carbon analyzer (Shimadzu TOC-VCSH). Furthermore, the loss on ignition was performed at a temperature of 1020 °C for 390 min. The sample crystallography was determined using X-RD analysis (Siemens diffractometer D5 000). A copper radiation of ($\lambda = 1.54 \text{ \AA}$) was used with a scan range from 1 to 65°, a step of 0.02 mm and a count time of 1.2 s.

2.2. Methods

The global methodology is illustrated by Fig. 1. Both pre-treatments steps and carbonation procedure are presented and a complete description is provided below.

2.2.1. Material pre-treatment

MC experiences required a mass of 170 kg of solid tailings sampled from the chromite mine of Montreal Coleraine situated in Saint Joseph (Coleraine). The initial granulometric size was around 20–100 mm. Prior to physical separation, the raw material was crushed with a jaw crusher (C-RC-335) and grinded with an oscillating mill discs (Retsch-RS200). The grinding conditions were set at 700 tours.min⁻¹ during 75 s. Consequently, the particle size of the solid was determined by a laser particle sizer (Horiba LA-950V2, Laser Scattering Particle Size Distribution Analyzer). The particle sample granulometry was around 142 ± 34 μm. The particles were mixed with water in order to create a pulp. A first separation using a spiral (5LL400 model type) with a water flow of 150 L min⁻¹ was made. The light fraction containing various silicates material was not used. The dense fraction obtained was a mixture of magnesium silicates and magnetite and undergone a second separation step by the Wilfley table (Outokumpu Technology, model SA-13A) using a longitudinal inclination of 14°, a speed of strokes of 500 strokes.min⁻¹, a stroke length of 14 mm, a water flow of 0.12 m³ h⁻¹ and a throughput of solid of 5 kg h⁻¹. Both magnesium silicate and magnetic fractions densities were determined by a pycnometer (AccuPyc 1330, Micromeritics).

The resulting silicate fraction obtained after physical separation was finely ground through a series of three grinding steps. An average granulometry of 57 μm with a median of 25 μm were measured. The material was then heat treated at 650 °C using a modified electric rotary kiln (Pyromaître Pyro 106-HE). The thermal treatment was carried out in continuous mode with a feeding rate of 200 g min⁻¹ of dry solid. The tube was inclined to an angle of 9.45° to create combined tumbling and rolling down effect to respect a residence time of 30 min. The treated solid was characterized by XRD to monitor the change in its mineral structure with the following scan parameters.

2.2.2. Mineral carbonation procedure

The experimental unit was installed at a cement plant site. The gas was directly pumped from one of the plant chimneys. It was at first cooled and condensed in a plate heat exchanger. The cooling fluid used was tap water. At this point, most of the NO_x and SO_x gasses were condensed. Then, once cooled, the gas was compressed using a double stage compressor to 12 barg and stored in the compressor tank (model 2340L5-V from Ingersoll Rand the company). Prior to experiments, the gas composition was measured using an infra-red analyzer from MRU Instrument (NOVA plus IV) calibrated to measure the amounts of CO₂, NO_x, SO_x, and oxygen O₂. The experiences were carried out at ambient temperature in a stirred reactor type “Floor stand 4555 reactor” (Parr Instrument Company) with a total volume of 18.7 L. A mass of 705 g of solid was mixed with 4.68 L of raw water coming from the quarry of the cement plant. The resulting pulp has a 15 wt% of solid. Before each experiment, the pH of the pulp was measured using a pH meter (Orion star A111). Once filled with the pulp, the gas was introduced into the vessel until reaching the desired working pressure. As the experiments were realized following a batch mode, all the valves were closed during the tests. The stirring speed was set to 600 rpm, and the reaction started. After 15 min of the reaction, the gas was removed, and its composition measured. Usually, the same pulp was contacted with a second batch of gas for another 15 min as previously described in the previous laboratory conducted work

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