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Energy Procedia 1 (2009) 4907–4914

**Energy
Procedia**www.elsevier.com/locate/procedia

GHGT-9

Carbonation of magnesium silicate mineral using a pressurised gas/solid process

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Abstract

Carbon dioxide mineral sequestration is not as widely advocated as CO₂ sequestration by other means such as underground storage alternatives, yet it possesses properties (capacity, permanency, energy economy) that can not be matched by other options. In this paper, our findings and results since GHGT-8 as well as current activities and near-future plans regarding CO₂ mineral carbonation are presented. The focus lies on the use of fluidised bed (FB) reactors for the carbonation of magnesium silicates via magnesium oxide or magnesium hydroxide intermediates, at temperatures and pressures up to 600°C, 100 bar (allowing for both sub- and supercritical conditions for CO₂), supported by earlier experiments using pressurised thermogravimetric analysis (PTGA). In addition, as the production of reactive magnesium from silicate mineral is not straightforward, it receives special attention, and first results of magnesium hydroxide production from serpentine using different methods are presented.

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Keywords: Carbon dioxide storage; mineral carbonation; gas/solid carbonation; Mg(OH)₂; serpentine

1. Introduction

Carbon dioxide capture and storage (CCS) is an important option for mitigating CO₂ emissions from human activities [1]. It comprises separation and compression of CO₂ from industry and power plants and transportation of CO₂ to a storage site followed by long-term storage. The carbonation of natural silicate minerals that contain alkaline-earth oxides like magnesium oxide (MgO) and calcium oxide (CaO) offers a leakage-free alternative to using underground geological formations for storage of CO₂. The fact that the deployment of geological storage of CO₂ is progressing rather slowly (current total capacity 5–10 Mt CO₂/a) and might not offer the necessary capacity soon enough puts pressure on the development of alternatives. As Figure 1 illustrates, (Mg-based) mineral carbonation gives the highest capacity and longest storage time of the currently known CCS options [3], and in

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contrast to carbon dioxide capture and geological storage methods (further referred to as CCGS²) post-storage monitoring of CO₂ won't be needed. Mineralisation of CO₂ has benefits and potential especially at locations where the risk of leakage is considered unacceptable or where underground storage sites are not available while vast resources of suitable magnesium silicates exist, such as in Finland, a country that has emission reduction commitments under the 1997 Kyoto protocol [4].

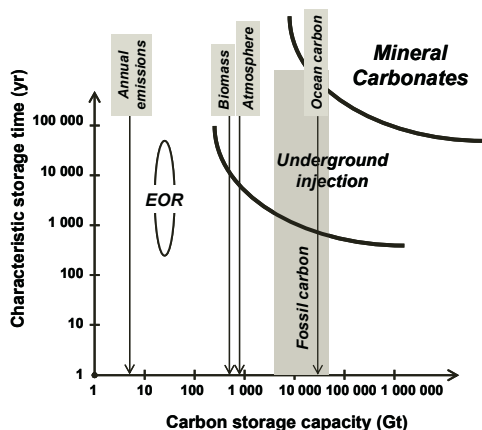
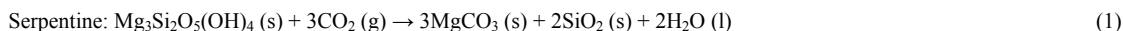


Figure 1. Estimated storage capacities and storage times for various CCS methods. (from [2], after [3])

There is an abundance of suitable minerals that consist of magnesium silicates such as serpentine and olivine containing high concentrations of MgO, while also pyroxenes and amphiboles are a potential source for both CaO and MgO. Also Mg- or Ca- containing mine tailings and by-products or wastes from industry may be used for carbonation purposes – see [2,5,6]. Two silicate minerals that have been extensively investigated in the field of mineral carbonation are serpentine and wollastonite for which the gas/solid carbonation reaction can be described by the following overall chemistry:



Here only the carbonation of Mg-based material is addressed since only this offers the capacity needed for significantly reducing anthropogenic CO₂ emissions. However, no commercial silicate mineral carbonation technology yet exists. Although the overall reactions given by equation (1) and (2) are exothermic (which implies that proper optimization and process integration should allow for operation at zero or negative net energy input) the natural carbonation of silicate minerals is very slow and the reaction must be accelerated considerably to be an economically viable large-scale CO₂ storage technology. In short, besides the implicit requirement of energy efficiency, two problems must be solved to make large-scale mineralisation of CO₂ more attractive [8]:

- extracting or activating the reactive component MgO from silicate mineral, and
- speeding-up the carbonation chemistry kinetics

² Surprisingly, the recent proposal for an EU directive on geological storage of carbon dioxide [7] confuses this CCS method with CCS in general: "...capture CO₂ from industrial installations and store it in geological formations (carbon dioxide capture and storage, or CCS)".

A small yet steadily increasing number of countries are looking into technical solutions to perform the carbonation of magnesium silicate with CO₂ on a large scale; the most important results were reported from the U.S. since the mid-1990s. While most research concentrates increasingly on methods using aqueous solutions, research in Finland still primarily addresses gas/solid (“dry”) methods besides aqueous solution (“wet”) routes. The reason for preferring a gas/solid route is that aqueous processes have temperatures much lower than, say, 500 °C where rate of the gas/solid carbonation of MgO or Mg(OH)₂ appears to become significant. As a result, the energy input requirements of the “wet” processes are high; it should be much lower (or negative) for a gas/solid process with heat recovery. Thus, instead of developing chemical schemes that after some time appear to be unattractive from an energy economy point of view [9,10,11], the Finnish research has always had the energy output from the exothermic carbonation reaction step as the obvious starting point.

While work on gas/solid routes in the U.S. ended already a while ago to make way for the seemingly more promising routes using aqueous solutions, the Finnish work on gas/solid carbonation has continued and produced data on thermodynamic feasibility and chemical kinetics, and identified step-wise processes for serpentine carbonation [12,13,14] which is currently being optimised in an experimental study. Still, the production of reactive magnesium oxide or, preferably, hydroxide for the final carbonation step in a fluidised bed (FB) reactor requires further consideration and results obtained with magnesium hydroxide material produced from serpentine using both a wet extraction process [15] as well as an combined dry-wet extraction process [16] are addressed below as well.

Finally, an issue not to be forgotten when considering CO₂ mineralisation is solids mining and handling and the question of what to do with the carbonate and other product material. It will have impact on the environment, although this won't be very different from a typical ore mining activity, benefitting from the fact that mineral deposit layers to be exploited will be very thick. Although large-scale mineralisation of CO₂ allows for compact storage compared to storage as (super-critical) fluid, the surface disposal of the carbonate material may be problematic and unwanted at certain locations. A metric ton of CO₂ will require 2.5-3 tons of magnesium silicate mineral (for coal combustion-derived CO₂ this implies ~8 tons of mineral per ton coal). Altogether this will result in a mining activity similar to typical commercial mining of coal or metal-containing ore.³ Using MgCO₃-containing material in, for example, building materials or flame retardants may be considered; in paper products however it can't be used [2]. On the other hand, being also a product of natural weathering of silicate mineral, the carbonate is benign and stable when disposed of in nature. Tests showed that MgCO₃ and CaCO₃ are stable in rain water and acid solutions down to pH 1 [17], but a very slow dissolving in water bodies and streams could in fact be beneficial.⁴ It should be noted that other solid products of magnesium silicate carbonation are significant amounts of silicon oxides and iron oxides, the latter offering a serious material resource for iron-and steel production.

2. Gas/solid carbonation of magnesium silicate and magnesium hydroxide

While other routes to mineral carbonation have progressed further, gas/solid carbonation as the final step of a staged process for magnesium silicate carbonation remains the focus of attention at Åbo Akademi University in Finland. This derives from the fact that gas/solid carbonation of magnesium silicates has not been as extensively studied as other options to this date and from the possibility of utilizing the heat of reaction between a Mg containing compound and CO₂. The direct reaction between e.g. serpentine (see equation 1) and CO₂ is too slow for practical applications, but the reaction rates could be enhanced by dividing the process into several steps (referred to as indirect carbonation).

³ A quick calculation based on solid material densities shows that the storage of 1 Mt CO₂ on an area of a 50 m × 100 m football (soccer) field would give a MgCO₃ layer of around 32 m in a 24 m deep hole (from where the mineral was produced); i.e. an 8 m thick layer above the ground.

⁴ Increasing the concentrations of Mg²⁺, Ca²⁺ and HCO₃⁻ ions can provide a buffer to the decomposition solid carbonate material under water, as the equilibrium reactions CaCO₃ (s) + H₂O (l) + CO₂ (aq) = Ca²⁺ (aq) + CO₃²⁻ (aq) + H₂O (l) + CO₂ (aq) = Ca²⁺ (aq) + 2HCO₃⁻ (aq), with HCO₃⁻ being much better soluble than CO₃²⁻, will then be pushed to the left side. Thus, preferable disposal sites for large amounts of MgCO₃ product could be the open sea or ocean.

2.1. Previous work on the gas/solid magnesium silicates carbonation route

In the open literature, almost no data can be found on the kinetics of chemical reactions between magnesium silicates or oxides with CO₂. Langmuir [18] refers to experimental work from the early 20th century that reports very slow chemistry for MgO and CO₂ also the catalytic effect of water on MgO carbonation is mentioned. Since the mid-1990s many results from mineral carbonation work by consortia in the U.S. were published, however for a large part in the form of qualitative work-in-progress reporting. Roughly after year 2000 several other countries followed.

For more information about previous work on mineral carbonation one should refer to literature reviews by Newall *et al.* [9], Huijgen and Comans [19,20], and recently by Sipilä *et al.* [21], dealing with Mg-based as well as Ca-based materials. From there it can be concluded that studies on direct and indirect “dry” (gas/solid) carbonation of Mg (and Ca) containing minerals are unanimous: the reaction rates achieved so far are too slow for large scale applications. However, to completely abandon the “dry” routes might be too early as the use of high temperature supercritical (sc) CO₂ has not been sufficiently tested. The goal our current work is to establish and present the possibilities provided by a high temperature, high pressure CO₂ environment. Although a merely powdered Mg-silicate sample does not react to a sufficient degree even at severe conditions (500°C, 340 bar [22]), the carbonation of Mg(OH)₂ is different. A detailed study on Mg(OH)₂ decomposition and the effect of pressure on Mg(OH)₂ carbonation, studying how dehydroxylation and re-hydroxylation interact with carbonation can be found in [23,24]. Surprisingly, above the minimum CO₂ pressure for stable MgCO₃, the rates of both dehydroxylation of Mg(OH)₂ and of the carbonation of MgO were found to decrease. Elevated pressures apparently slow down dehydroxylation of Mg(OH)₂, generating fewer reactive MgO sites for carbonation.

Concluding that a process that involves carbonation of Mg(OH)₂ is preferable, one challenge is how to extract the magnesium from Mg-silicate mineral without the consumption of chemicals and/or the need for a significant energy input.

2.2. Recent results from pressurised thermogravimetric analysis (PTGA)

Our earlier results from PTGA tests showed carbonation conversion levels of Mg(OH)₂ particles (Dead Sea Periclase, 97% pure, 75-125 µm) increasing from a few % at 370°C, 1 bar to 55% at 510°C, 45 bar, in CO₂/H₂O 99%/1% (v/v) [12,13]. It was shown that after several % carbonation conversion, the diffusion of CO₂ and H₂O through the carbonate product layer becomes rate-determining, motivating our choice for an FB reactor where carbonate material that builds up on the reacting particles (eventually slowing down conversion) will be removed from the particles as fines by attrition and abrasion, which are entrained from the reactor with the exit gas flow. This was confirmed by a few tests in an atmospheric FB reactor, using the same Mg(OH)₂ material.

The latest PTGA test series aimed at carbonation of serpentinite rock from the Hitura nickel mine in Finland, currently owned by Belvedere Resources Ltd. (83 % serpentine, 14 % iron oxides, size fraction 74-125 µm) in three steps, i.e. thermal activation by heat-up to 630 °C in air, followed by hydroxylation of MgO to Mg(OH)₂ at 35 bar, 380°C, followed by carbonation at 35 bar, 495 or 525 °C. A result is shown as a PTGA output graph in Figure 2. It shows that while the release of water from the mineral is fast and straightforward, the subsequent step which could be hydration of MgO to Mg(OH)₂ using pressurised steam is very slow. Apparently the heat-up stage doesn't produce MgO but merely converts serpentinite into olivine, water and quartz. Dehydroxylation/hydroxylation of Mg(OH)₂ (97%, size fraction 75-125 µm) was studied in a separate test, confirming that conversion of MgO (i.e. calcined Mg(OH)₂) to Mg(OH)₂ in pressurised steam is very slow. Hydroxylation of MgO thus presents yet another challenge. Besides faster carbonation kinetics for Mg(OH)₂ compared to MgO also the energy economy of a three-stage process involving Mg(OH)₂ production from MgO and carbonation was shown to be better than that of a two-stage process with MgO carbonation [14]. At the same time it can be concluded that the production of MgO as an intermediate is preferably avoided, since not only the carbonation but also the hydration of MgO is too slow.

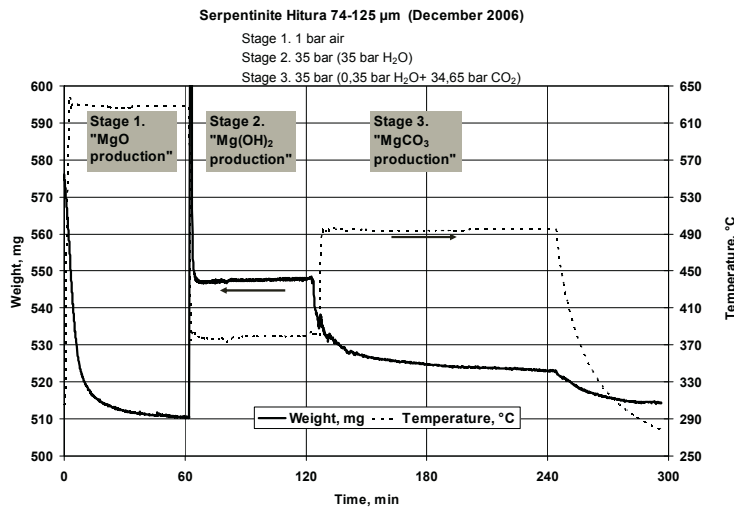


Figure 2. PTGA results of (a – left) a three-step carbonation test with serpentine and (b-right) a calcination – hydration test with Mg(OH)₂

2.3. Current work on the gas/solid carbonation route: carbonation of Mg(OH)₂ in a fluidised bed reactor

Our work at ÅAU focuses on optimising the conditions inside a small pressurised fluidised bed setup as seen in Figure 3. The dimensions of the fluidised bed are: inside diameter = 1.43 cm and height = 40.6 cm, giving a volume of ~65 ml. Besides the electrically heated FB, the setup consists of a preheater which ensures that the right temperature can be maintained inside the FB. A cyclone is placed after the FB to separate most of the solid particles (cut-size: ~10 μm) from the gas, while very small (< 10 μm) particles are collected by the filter following the cyclone.

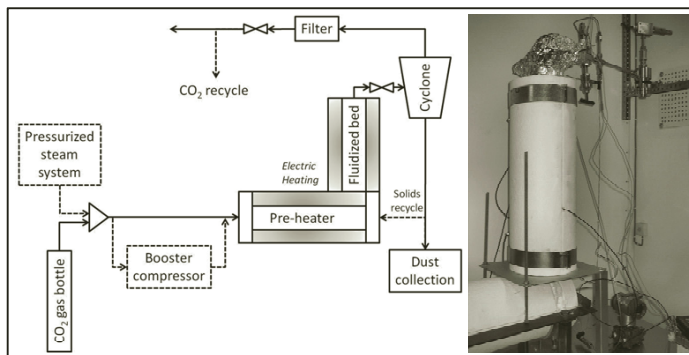


Figure 3. Schematic diagram of the FB-setup together with actual (work-in-progress) setup

As the first start-up and shake-down experiments with the FB setup (as displayed in Figure 3) are currently being conducted, carbonation tests results have not yet been obtained. The purpose is to gradually increase the pressure and temperature of the experiments following the steps taken earlier in a pressurised thermogravimetric analysis (PTGA) reactor system (see [12]), using both pure and wet CO₂. The first experiments will be 1 bar CO₂ pressure and 370 °C, which is just below the equilibrium temperature of the carbonation reaction with Mg(OH)₂. For given

pressure, the temperatures selected are taken equal to $T = 0.95 \cdot T_{eq}$ (K), where T_{eq} is the equilibrium temperature for the $Mg(OH)_2$ carbonation reaction, to allow for some driving force (*i.e.* $-\Delta G/T$) for the chemistry. After for example 12 bar/460°C, 35 bar/495°C, CO_2 pressure will be increased to and above the supercritical pressure of CO_2 , 73.2 bar.

Using a fluidised bed as the carbonation reactor is founded on two important attributes. Firstly, the continuous motion of the bed enhances particle-particle collisions allowing for fresh unreacted core material to be exposed to CO_2 and secondly, the temperature distribution inside a fluidised bed is nearly uniform. Also, the material being chipped off is lighter than the initial particles allowing for it to be carried away by the passing CO_2 . The separation of the particles is carried out in a cyclone from where the particles can be removed for analysis or (in the future) recycled back to the fluidised bed.

Besides the importance of operating with sc CO_2 or not, also the effects of particle size, fluid flow (within the limits provided by minimum fluidizing velocity and terminal velocity) and small amounts of H_2O mixed with the CO_2 will be considered. Most test will be made with Finnish serpentinite from the Hitura site plus tests with for example a serpentinite from Lithuania.

3. Production of $Mg(OH)_2$ from serpentine followed by gas/solid carbonation

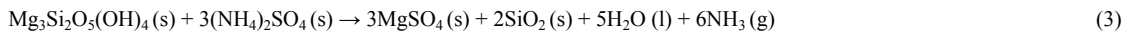
3.1. Magnesium extraction using strong acids

Extensive pre-treatment of feedstock mineral makes the aqueous processes using direct carbonation in fact indirect. As an alternative to energy-intensive pre-treatment research teams worldwide embarked on methods to dissolve or leach Mg or Ca from minerals or industrial by-products and wastes using strong or weak acids, alkali solutions or ligands [19-21]. In Finland, Teir and co-workers [6,15] dissolved the serpentinite from Hitura mentioned above (74-125 μm) in 1 M, 2 M and 4 M aqueous solutions of common acids or bases at room temperature during 1 h. It was found that H_2SO_4 extracted most Mg (24-26%), followed by HCl (17-24%), HNO_3 (12-18%), $HCOOH$ (5-8%) and CH_3COOH (3-4%). None of the acids extracted Mg selectively, extracting also some Fe and Si. Very small amounts of Mg, Fe or Si dissolved in the alkaline solutions. Solutions of 2 M HCl, H_2SO_4 , and HNO_3 were also tested at 30, 50 and 70°C, showing 100% Mg extraction in 1-2 h at 70°C in each acid. Some Fe and Si were also dissolved. Carbonate precipitation experiments were made with two solutions prepared from serpentinite in 4 M HNO_3 and 4 M HCl, respectively, at 70°C for 2 h. After filtration and drying, the remaining salts were dissolved in water and iron oxides were precipitated. The magnesium-rich solutions – mainly dissolved magnesium nitrates or chlorides – were used to precipitate magnesium carbonates by exposing each solution to a CO_2 flow for 30 min, while regulating pH at 7-12 using aqueous NaOH. Regulation of pH was necessary for producing precipitates: for both solutions an optimum of pH 9 was found, precipitating the Mg as 93-99 wt-% pure hydro-magnesite, $Mg_3(CO_3)_4(OH)_2 \cdot 4H_2O$ consuming roughly 2 mol NaOH per mol Mg. The thermal stability of a precipitate was analysed using thermogravimetric analysis (TGA) at 25-900°C in N_2 , showing that hydro-magnesite should be a stable CO_2 storage medium up to 300°C. More detail is given elsewhere [6,15].

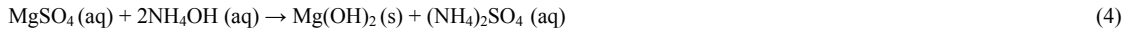
Also, a carbonation test was made in the PTGA device using an $Mg(OH)_2$ produced from the serpentinite as described above, using HCl as the solvent. However, at 495°C/35 bar in in CO_2/H_2O 99%/1% (v/v) gave no measurable conversion after 2 h (the PTGA mass signal suggests that the $Mg(OH)_2$ had calcined to MgO), contrary to the the Dead Sea Periclase $Mg(OH)_2$ sample that gave 50% conversion after 6 h under these conditions.

3.2. Magnesium extraction using salts

In a scheme that is currently being optimised at ÅAU, magnesium hydroxide is produced via a staged process of dry, solid-solid reaction of serpentine and ammonium sulphate at elevated temperatures followed by a precipitation process. Theoretically, serpentine reacts according to:



Thermodynamic equilibrium calculations using the HSC 5.1 software [25] show that the thermodynamics of the above reaction is feasible at temperatures between 200 and 550°C. Magnesium sulphate produced from the reaction is soluble in water and can react with base precipitants, for example, ammonia, to form magnesium hydroxide:



The reaction of the mineral (containing ca. 82% serpentine and 18% impurities, with Fe accounting for the greatest percent of the impurities) with ammonium sulphate together with the precipitation reaction result in the formation of $\text{Mg}(\text{OH})_2$, Fe (II&III) compounds, silica, ammonium sulphate and ammonia gas are other products and by-products of the process. The simplified process scheme that is currently being optimised is shown in Figure 4.

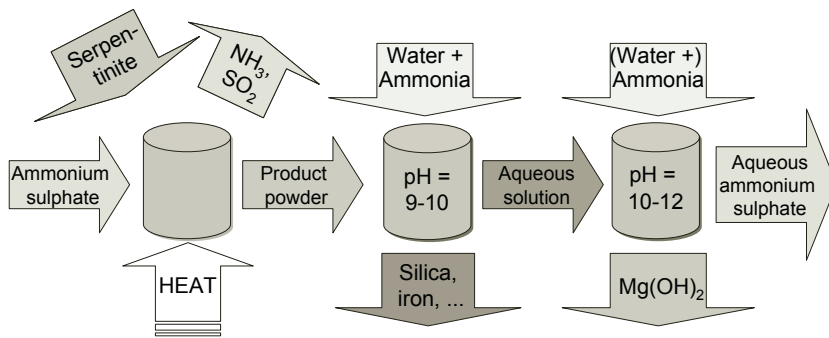


Figure 4. Flow diagram of the $\text{Mg}(\text{OH})_2$ production process currently under investigation

The first result showed that indeed an $\text{Mg}(\text{OH})_2$ product can be obtained by this route, with XRD analysis confirming a high purity. At the same time the significant amount of iron oxide by-product is obtained. Current work on the optimisation of this involves finding the preferable temperature for the first step of the process in Figure 4 and the best levels for pH in the second and third steps, plus optimisation of material amount ratio's while at the same time ensuring (near) 100% recovery of the ammonium sulphate and gaseous products (NH_3 , SO_2).

4. Conclusions

Mineralisation of CO_2 is an interesting (due to its enormous potential at very many locations), urgent (due to the slow deployment of geological storage of CO_2) and leakage-free option for CCS. Motivations for exploring the gas/solid carbonation route and current ongoing efforts towards its utilization have been presented here. Experiments using high pressures and temperatures inside a fluidised bed are being conducted in Finland for carbonation at up to 600 °C, 100 bar (allowing for super-critical conditions for CO_2). In addition, production of $\text{Mg}(\text{OH})_2$ from serpentinite rock is investigated for use in the fluidised bed process, which shows very promising results.

Acknowledgements

This work was funded by Nordic Energy Research (2003-2007), the Academy of Finland Research programme "Sustainable Energy" (2008-2011) and KH Renlund Foundation (2007, 2008).

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