

# OVERVIEW OF LATEST MINERAL CARBONATION TECHNIQUES FOR CARBON DIOXIDE SEQUESTRATION

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**Abstract:** This relatively new domain, of carbon dioxide sequestration for mitigation purposes, is one of the carbon dioxide capture and storage (CCS) alternatives. CO<sub>2</sub> sequestration could be used in the future where geological storage, for example, is not an option due to the lack of suitable locations or could represent a hazard to the surrounding areas. Also could be used in those locations where the needed material (raw minerals or industrial wastes) are abundant.

The interest in this technique of CO<sub>2</sub> storage is growing fast and results of researches on this topic are coming from a larger number of countries every year. What is more interesting is that the research of the industrial wastes carbonation is developing, giving in the future new purposes for these wastes.

**Keywords:** mineral carbonation, carbon dioxide, calcium carbonate, storage

## 1. Introduction

The greenhouse effect is known to be created by different gases in the atmosphere. One of these gases is CO<sub>2</sub> and because its concentration is increasing dangerously for the past one hundred years is now considered to be the main cause of the temperature rise on Earth.

In order to avoid the potentially devastating consequences of global warming and climate change, the CO<sub>2</sub> emissions into the atmosphere caused by human activities should be reduced considerably [1]. For this goal to be reached some carbon dioxide capture and storage (CCS) technologies were proposed but for the moment none of them were efficient enough in reducing the CO<sub>2</sub> emissions. Despite the negative results, researches are still ongoing and in some cases the results are promising as for example the storage in underground cavities. The same is hoped to be happening with the CO<sub>2</sub> storage in minerals and wastes and hopefully all the CCS technologies will contribute together to a more efficient CO<sub>2</sub> reduction.

## 2. Carbon dioxide sequestration techniques

### 2.1. Geological storage

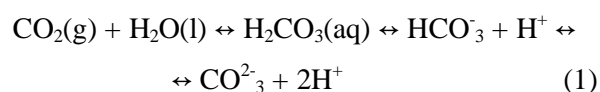
One of the technologies, that have already been employed on a significant scale, but not large enough to have a global CO<sub>2</sub> emissions mitigation

impact, is storage of CO<sub>2</sub> in underground cavities. This includes the so-called Enhanced Oil Recovery (EOR) and also Enhanced Gas Recovery (EGR), which are concepts aimed at improving the oil/gas recovery potential of an oil/gas field by flooding it with CO<sub>2</sub> [2].

Perhaps the greatest problem related to underground storage is the permanency of the solution, as there will always be a risk of leakage. Therefore, this solution would require continuous monitoring of storage sites for thousands of years.

### 2.2. Ocean storage

Another widely studied option for CO<sub>2</sub> sequestration involves injecting CO<sub>2</sub> into the ocean at great depths, where the gaseous CO<sub>2</sub> reacts to form carbonic acid (H<sub>2</sub>CO<sub>3</sub>). The carbonic acid then dissociates into a (bi)carbonate ion and hydrogen ion in accordance with the equation below: [2]



Although ocean storage could provide a fast and relatively easy alternative for CO<sub>2</sub> emissions reduction it has lost its appeal in recent years, largely due to the uncertainty when considering the

environmental consequences (e.g. decreasing pH of ocean water) and the lack of permanency [1].

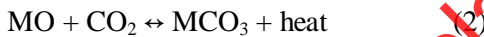
**2.3. Storage below sea bed**

An alternative to both geological storage and ocean storage described above is CO<sub>2</sub> storage below the ocean floor at depths of at least 3000 m of ocean and several hundred meters of marine sediment. In contrast to the previously mentioned options this option does not suffer from lack of permanency (ocean storage) or the demand for monitoring the storage site (geological storage). The idea is based on the fact that CO<sub>2</sub> becomes denser than water at sufficient depths (≈3000 m), but that it still needs to be trapped in order to prevent it from being released by ocean currents or e.g. earthquakes. Therefore, it should be stored below the seabed [3].

This alternative is still new and further research is ongoing in order to verify the theories.

**2.4. Mineral carbonation**

The reaction between a metal oxide bearing material and CO<sub>2</sub> is called carbonation and can be expressed by the following reaction:



where in practice M describes a (metallic) element such as calcium, magnesium or iron. The reaction in Equation (2) is exothermic and the heat released is dependent on the metallic element bearing

mineral at hand (for the magnesium- or calcium-based silicate minerals- olivine: 89 kJ/mol CO<sub>2</sub>, serpentine: 64 kJ/mol CO<sub>2</sub> and wollastonite: 90 kJ/mol CO<sub>2</sub> at 298 K). [2]

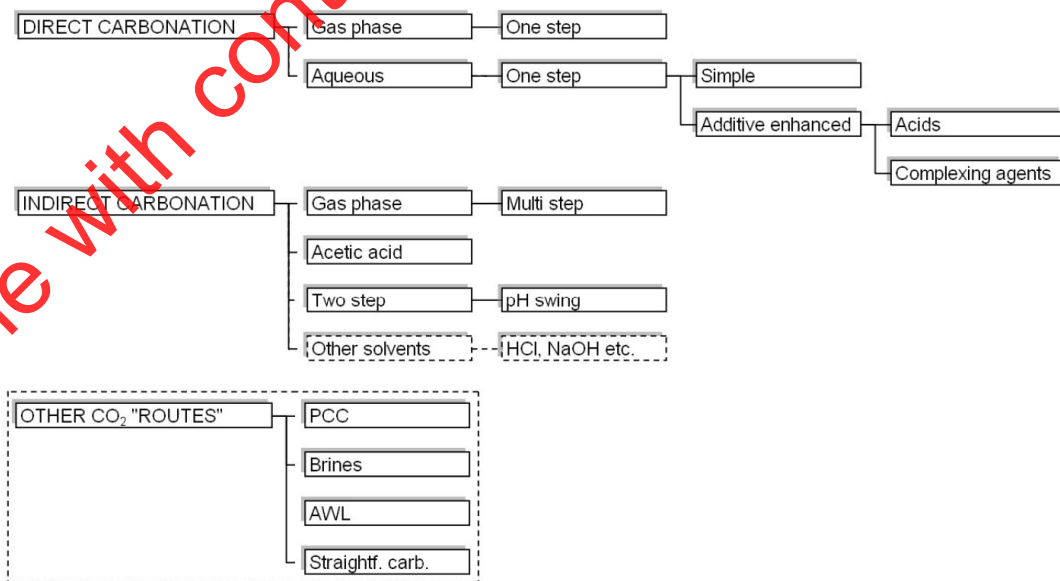
One major benefit of CO<sub>2</sub> sequestration by mineral carbonation consist of the environmentally benign and virtually permanent trapping of CO<sub>2</sub> in the form of carbonated minerals by using abundant mineral resources such as Mg-silicates [2]. Unlike other CO<sub>2</sub> sequestration routes it provides a leakage-free long-term sequestration option, without a need for post-storage surveillance and monitoring once the CO<sub>2</sub> has been fixed.

In addition to the benefits of mineral carbonation, this option is the only CO<sub>2</sub> sequestration option available where large underground reservoirs do not exist and ocean storage of CO<sub>2</sub> is not feasible.

Another benefit of mineral carbonation is that, at least theoretically, the carbonation process could proceed without energy input, but this has not yet been accomplished.

Attempts to speed up the carbonation reaction include using both dry and wet methods, additives, heating and pressurizing the carbonation reactor, dividing the process into multiple steps, pretreatment of the mineral source and more (fig.1).

There are several different elements that can be carbonated, but alkaline earth metals, calcium and magnesium, have proven to be the most suitable due to their abundance and insolubility in nature.



**Figure 1:** Main carbonation processes and variants.

In addition to the abundant magnesium and calcium containing minerals, there are also industrial solid residues that contain large amounts of Mg, Ca and even Fe.

Currently the most investigated mineral resources are olivine, serpentine and wollastonite. From the solid residues, steel slag has recently received a lot of attention but other industrial residues also have been studied: asbestos-mining tailings, electric arc furnace (EAF) dust, cement-kiln dust, waste concrete, coal fly ash etc.

Figure 1 displays the various carbonation routes that are currently being investigated.

### 3. Directions of mineral carbonation

Trapping carbon dioxide in carbonates can be achieved through various process routes as described further, ranging from the most basic accelerated weathering of limestone to advanced multi-step processes.

#### 3.1. Direct carbonation

Direct carbonation is the simplest approach to mineral carbonation and the principal approach is that a suitable feedstock, e.g. serpentine or a Ca/Mg rich solid residue is carbonated in a single process step. For an aqueous process this means that both the extraction of metals from the feedstock and the subsequent reaction with the dissolved carbon dioxide to form carbonates takes place in the same reactor.

##### 3.1.1. Direct gas-solid carbonation

Gas-solid carbonation is a simple approach towards mineral carbonation. Here particulate metal oxides are brought into contact with gaseous CO<sub>2</sub> at a particular temperature and pressure. The dry process has the potential of producing high temperature steam or electricity while converting CO<sub>2</sub> into carbonates.

Unfortunately, the reaction rates of such a process have been too slow and the process suffers from thermodynamic limitations and further studies around this alternative have mostly been abandoned.

One of the important benefits of using industrial solid residues as feedstock for carbonation compared to the carbonation of mineral ores is the possibility of utilizing a waste stream. The

possibility of simultaneously binding CO<sub>2</sub> and lowering the hazardous nature of e.g. municipal solid waste incinerator (MSWI) ash makes this carbonation route interesting [4]. However, the potential CO<sub>2</sub> storage capacity for this option is limited, simply because the amounts of material that may be carbonated are too small [4].

Nevertheless, where these wastes are present the carbonation should be taken into consideration because as shown by R. Santos *et al.* [5] during the carbonation the strength of the stainless steel slag is increased. This will give an added value to the carbonated stainless steel slag wherefore the material can be used in secondary applications. The sale price of this product might increase.

##### 3.1.2. Direct aqueous carbonation

The direct aqueous mineral carbonation-route referring to carbonation performed in a single step in an aqueous solution, appears to be the most promising CO<sub>2</sub> mineralization alternative to date [6]. High carbonation degrees and acceptable rates have been achieved but the process is (still) too expensive to be applied on a larger scale [7]. Ranging from 40–80 €/t CO<sub>2</sub> mineralized (includes energy use) compared to 0.4–6 €/t CO<sub>2</sub> [2] stored for geological storage.

Direct aqueous mineral carbonation can be further divided into two subcategories, depending on the type of solution used. Studies focusing on carbonation in pure aqueous solutions have quickly made way for additive-enhanced carbonation experiments and a common solution type used today, originally presented by O'Connor *et al.* (2000) [8], consists of 0.64 M NaHCO<sub>3</sub> + 1.00 M NaCl. However, it has been reported [9] that there are still improvements to be made regarding the above mentioned solution.

When it is necessary to use additives in carbonation processes it is extremely important to recycle these, due to the large scale of any industrial application [7].

Work on finding optimal aqueous carbonation conditions is ongoing and even though it has been studied extensively, some questions still remain unanswered. For example increasing the liquid/solid ratio (L/S) has been reported to have both a positive and a negative effect on CO<sub>2</sub> conversion.

The advances made to aqueous solution chemistry by McKelvy *et al.* [9] were significant,

but unless the (expensive) additives used cannot be recycled the process route becomes unattractive. Nevertheless, the studies conducted on direct aqueous carbonation have improved the overall knowledge of aqueous carbonation reactions considerably.

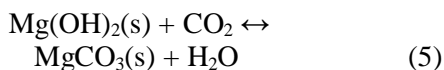
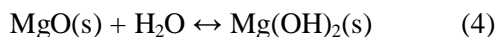
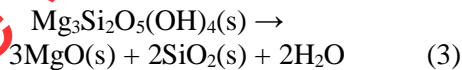
### 3.2. Indirect carbonation

When the mineral carbonation process is divided into several steps it is classified as indirect carbonation. In other words, indirect carbonation means that the reactive component (usually Mg or Ca) is first extracted from the feedstock (as oxide or hydroxide) in one step and then, in another step, it is reacted with CO<sub>2</sub> to form the desired carbonates.

#### 3.2.1. Multistage gas-solid carbonation route

Direct gas-solid carbonation of silicate minerals has been shown to be too slow for any large scale implementations, but a staged gas-solid carbonation process could overcome the slow reaction kinetics. The process involves extraction of magnesium (oxide or hydroxide) in an atmospheric pressure step followed by a carbonation step at elevated temperature (>500 °C) and pressure (>20 bar) [10].

It has been found that the carbonation of MgO is significantly slower than the carbonation of Mg(OH)<sub>2</sub> [11]. Using this observation Zevenhoven *et al.* [10] suggested (noting that Mg(OH)<sub>2</sub> production from serpentine in one step cannot be done because of thermodynamic limitations) that the direct gas-solid carbonation process should be divided into three-steps: 1) MgO production (Equation 3) in an atmospheric reactor followed by 2) MgO hydration (Equation 4) and 3) carbonation (Equation 5) at elevated pressures:



In addition to the faster carbonation kinetics in the three-step gas-solid carbonation route described above, the process is also preferable from an energy efficiency point of view compared

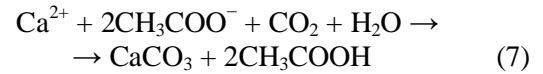
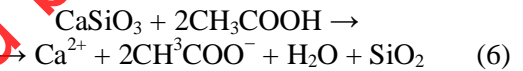
to the two step carbonation of MgO [10]. However, the three-step process is still too slow for large scale implementation, as preliminary tests performed in up to 45 bar pressures have shown.

Progress has been made in enhancing the direct gas-solid reaction rates, primarily by increasing pressure. Experiments showing that all three-steps in Equations 3–5 are fast enough for industrial implementation are still required.

Dividing the gas-solid carbonation route into several steps could be beneficial, but there is not enough evidence yet for industrial viability.

#### 3.2.2. Acetic acid route

In order to speed up the aqueous carbonation process, the use of acetic acid for the extraction of calcium from a calcium-rich feedstock has been suggested by Kalizawa *et al.* [12]. In principal it consists of two-steps as given in Equations 6 and 7:



Equation 6 describes the extraction step and Equation 7 the precipitation step. In principal the acetic acid used in the extraction step could be recovered in the following precipitation step.

Inspired by the concept of binding CO<sub>2</sub> in calcium extracted from a calcium silicate such as wollastonite using acetic acid [12], Teir *et al.* [13] investigated the possibility of producing a high value PCC material from calcium silicates and later from other calcium-containing materials [14].

Steelmaking slags then became the centre of attention as they can contain significant amounts of both CaO and MgO. Eloneva S. [15] reported that 80–90 % pure calcite was produced from blast furnace slag using acetic acid. However, significant amounts of sodium hydroxide were required for promoting the precipitation of carbonates from the acidic solution.

Research in Finland has focused on steel slag carbonation and especially the possibility of producing valuable precipitated calcium carbonate (PCC) [14, 15]. The annual CO<sub>2</sub> binding potential of steelmaking slags is in the order of 70 – 180 Mt CO<sub>2</sub>. Globally the CO<sub>2</sub> sequestration potential for this option is small, but for individual steel plants,

however, the method could provide significant economical benefits.

### 3.2.3. Two step aqueous carbonation

Two-step aqueous carbonation has been investigated because the overall carbonation reaction can easily be divided into two-steps: extraction and precipitation that may be investigated and optimized separately.

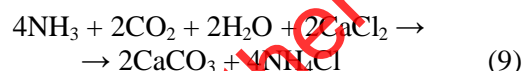
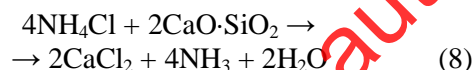
By upgrading a waste product into a product of high commercial value, expensive CO<sub>2</sub> sequestration processes could become economically feasible. One such approach has been investigated by Katsuyama *et al.* [16] who studied the use of waste cement for the development of high-purity CaCO<sub>3</sub> by CO<sub>2</sub> carbonization.

Katsuyama *et al.* [16] studied the feasibility of producing CaCO<sub>3</sub> from waste cement by first extracting calcium from pulverized waste cement in a water slurry at high CO<sub>2</sub> pressure (several MPa), followed by the precipitation of CaCO<sub>3</sub> from the extracted solution at lower CO<sub>2</sub> pressures, producing high purity CaCO<sub>3</sub> (up to 98%) from waste cement at relatively high reaction rates. The estimation of the cost of producing high-purity CaCO<sub>3</sub> could be as low as 105 €/m<sup>3</sup> when compared to the commercial price of 154–263 €/m<sup>3</sup>. In addition, if the produced CaCO<sub>3</sub> could be purified to meet the requirements of ultra-high purity CaCO<sub>3</sub> (>99% CaCO<sub>3</sub>) the potential profits could increase substantially. The current cost of ultrahigh purity CaCO<sub>3</sub> is around 7700 €/m<sup>3</sup>, while Katsuyama *et al.* [16] estimated a production cost of only 250 €/m<sup>3</sup>.

Gorset *et al.* [17], describing a way of producing pure MgCO<sub>3</sub> from olivine, claims that the process consisting of one dissolution step and two precipitation steps is rapid enough for large scale implementation. The process does not require the use of strong mineral or organic acids even though the dissolution step requires an acidic environment. The required acidity (pH 3–5) is to be achieved using pressurized CO<sub>2</sub> (50–150 bar) and a temperature around 100–170 °C, while the following step consisting of MgCO<sub>3</sub> precipitation, takes place in another reactor with preferably a lower CO<sub>2</sub> pressure (50 – 80 bar) and a higher temperature (140 – 250 °C) favoring the precipitation of carbonates. Experimental results showed a high degree of purity, between 99.28 and 99.44% MgCO<sub>3</sub>, of the precipitated carbonate.

The pH-swing process developed in Japan is another two-step aqueous carbonation process where at first the pH of the solution is lowered thereby enhancing the extraction of divalent metal ions. In the second step the pH is raised to enhance the precipitation of carbonates.

The principal reactions taking place inside the extractor (Equation 8) and the precipitator (Equation 9) are:



Equation 9, taking place inside the precipitator, consists of both CO<sub>2</sub> absorption and CaCO<sub>3</sub> precipitation. In their study, Kodama *et al.* [18] investigated a CO<sub>2</sub> sequestration process that utilizes pH swing using NH<sub>4</sub>Cl. The energy input requirement for the investigated process using steel making slag as the mineral source was estimated at around 300 kWh/t CO<sub>2</sub>, but the loss of a chemical additive (NH<sub>3</sub>) was considerable.

Different approaches of a two-step aqueous carbonation process have been presented. All options are good in theory, but it remains uncertain whether or not these processes could lead the way to any significant scale long-term storage of CO<sub>2</sub> in the future. More experiments for large scale viability are required.

### 3.3. Other processes of carbon dioxide sequestration

Beside the process routes mentioned above, there are other processes and applications that resemble mineral carbonation and are described in the following section.

#### 3.3.1. The precipitated calcium carbonate production

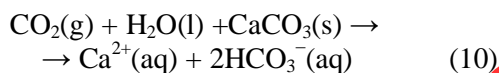
The production of valuable products (*e.g.* PCC) by utilizing CO<sub>2</sub> was studied extensively. An example of this concept is Two-step aqueous carbonation of solid residues. Various methods to obtain a product of desired properties have been used and one of the simplest methods is that of direct aqueous carbonation without the use of additives.

### 3.3.2. Brines used in carbonation

The saline-based solution formed as a waste product during the oil or natural gas extraction is called brine and it is found in large quantities in storage tanks. Due to its large amounts available and a high concentration of elements fit to form carbonates (mostly calcium and magnesium) this brine represents an option for carbon dioxide storage by a carbonation process. Although this brine is capable of forming carbonates, its use in a large scale process is for the moment not appropriate due to the slow kinetics. If the pH of the brine is raised the carbonation process is faster, but unknown factors about the parameters (temperature, pressure, brine composition and pH) need to be further studied.

### 3.3.3. Accelerated weathering of limestone

Another option is carbon dioxide capture and storage by accelerated weathering of limestone (AWL). This option imitates the natural carbonate weathering according to the following reaction:



The product of an AWL plant would be a calcium bicarbonate solution that could readily be released and diluted into the ocean with a minimal environmental impact [19]. However, there are still many issues to deal with, such as the energy demand of transporting large amounts of calcium containing (waste or mineral) material to the AWL plant that preferably should be located near a CO<sub>2</sub> point source as well as a possible disposal site (e.g. the ocean). In an ideal case (with access to free limestone, e.g. waste fines, and a "free" water source, e.g. power plant cooling water) the CO<sub>2</sub> mitigation cost by means of AWL could be as low as 2.3–3.1 €/ton CO<sub>2</sub>. Rau *et al.* [19] suggests that some 10–20% of the United States point-source CO<sub>2</sub> emissions could be mitigated this way.

Despite the potential positive effect of bicarbonate disposal Rau *et al.* [19] concludes that further research is needed to fully understand the impacts of AWL effluent disposal in the ocean.

Some of the most important applications and techniques for mineral storage of CO<sub>2</sub> are described above, yet almost all of these options need further research before starting a

large scale process. Despite the inconvenience given by results lacking, all of them should represent an option when CO<sub>2</sub> emission reduction is taken into account.

## 4. Conclusions

It can be concluded that the carbonation techniques using additives for reactivity rising gave better results than those not using additives and for that, studies were performed with the purpose of a better understanding on the reactions complexity regarding the carbonation. Despite some good results the performed studies have not, for the moment, came with significant discoveries regarding the reactivity in mineral carbonation process.

For the techniques using additives in carbonation the biggest problem represents the recycling of these additives and for the moment no such breakthrough was reported and if this will never be accomplished, maybe method's that don't need them will be implemented.

Besides the cost of the process that is for the moment larger than of those other carbon dioxide capture and storage methods, the direct aqueous carbonation seems to be the most promising option of mineral carbonation. Other studies, however, show that the dissolution and the precipitation steps should be separated, even if the costs will be higher. This separation would take out the need to balance these reactions that are opposite. So, taking that into consideration, the indirect aqueous carbonation is the most attractive option.

Despite the good results on the high purity precipitated calcium carbonate process, for the moment, no large scale production was realized. If this will be accomplished it is expected for the price of carbonation process to be acceptable even if is at a higher rate, due to the great value of the high purity PCC.

Before one or more mineral carbonation techniques could be implemented on an industrial scale, much more research should be done and important improvements of existing options are needed. Another way for the large scale carbonation to become reality would be represented by completely new techniques.

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