

Carbonate-bonded construction materials from alkaline residues

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Abstract

Accelerated carbonation is a rapidly developing technology that is attracting attention as it uses CO₂ as a binder to make construction materials. Originally stemming from geochemical and environmental research into CO₂ sequestration or waste remediation, accelerated carbonation has been developed into a technology that enables to transform alkaline precursors into products that meet technical requirements for use as aggregates or shaped blocks. Alkaline precursors can be manufactured from primary resources or derived from industrial residues: a.o. metallurgical slags, incineration ashes and concrete recycling residues are prone to carbonate under controlled conditions. Moist carbonation of shaped Ca-silicate rich precursors at elevated curing temperature and CO₂ concentration or pressure has delivered the most promising results so far.

This letter presents an overview of current accelerated carbonation approaches to make carbonate-bonded construction materials from alkaline residues. The general carbonation mechanism is explained and two application routes are exemplified: i.e. production of lightweight aggregates and compact blocks by accelerated moist carbonation.

Keywords: Carbonation; Slags; CO2; Aggregates; Compacts; Curing

1 Introduction

Among low carbon cements, by far the greatest CO_2 reduction potential is offered by cements that actually sequester CO_2 as solid carbonates as part of their solidification process [1,2]. "Carbonate-bonded" construction materials actually mimick their natural limestone counterparts [3,4]. The abundance of limestones in the geological record and their use as traditional building material indicate the potential for long-term durability and preservation of technical quality [5].

In the context of concrete, carbonation is mostly perceived as problematic as it is one of the prime causes of deterioration of reinforced concrete structures by corrosion [6]. In case carbonation is actually desirable then kinetic barriers that slow down the natural carbonation process need to be overcome by applying elevated temperature and CO_2 concentrations in curing chambers or autoclaves. Chemicals such as NaOH can be added to accelerate and enhance the process even further [7]. Most recent research developments in producing CO_2 uptake cements depart from calcium (and magnesium)-rich materials and relate to 1)

accelerated curing of concrete [8-10], 2) carbonation of brines [11-13], 3) carbonation of hydrated lime [14,15] and 4) carbonation of calcium silicates [16-19].

Although in principle similar mechanisms apply for carbonation of different precursors, this review will focus on Ca(Mg) silicate based precursors, mainly because most recent progress was made in this area. The carbonation mechanism, potential precursors and examples of specific product developments are treated in the following sections.

2 Carbonation mechanisms

The basic concept behind carbonation of Ca(Mg)-silicates is the process of rock weathering. In this natural process calcium and magnesium ions are leached from silicate rocks and react with ambient CO_2 to form solid carbonates.

Direct mineral carbonation is the process in which the extraction of metal ions from Ca/Mg-rich rocks or solid residues and the precipitation of carbonate(s) take place in one process step. Indirect carbonation is accomplished through the extraction in a solution of reactive ions (generally Ca and/or Mg) from the minerals in the first step

and carbonation in a subsequent step as a result of the chemical reaction with either gaseous or aqueous CO_2 [20]. The direct carbonation route can yield a variety of valuable construction products while the indirect route, can lead to very pure Precipitated Calcium Carbonate that can find use in the papermaking industry, as a filler in polymer materials or as coating pigment to increase solar reflectance [21].

The overall carbonation chemistry in the direct carbonation route using calcium or magnesium silicates is presented in Equation 1:

$$(Ca,Mg)_2SiO_4 + CO_2 + H_2O \rightarrow 2(Ca,Mg)CO_3 + SiO_2 + H_2O$$
 (1)

The reaction consists of three main subprocesses (Fig. 1): (1) the diffusion of CO_2 through the pore network and dissolution of CO_2 in an aqueous solution to form carbonic acid; (2) the dissolution or leaching of calcium and/or magnesium from the silicates in an aqueous solution, and (3) the precipitation of calcium and/or magnesium carbonates (and silica) [22,23].

In the direct carbonation route steps (1) and (2) occur simultaneously and the rate and extent of carbonation depend on a number of factors such as the precursor composition and moisture saturation degree [24,25], but also on the carbonation conditions such as the CO2 grade and pressure and the relative humidity [26,27]. Saturation degree and relative humidity play a key role in providing CO2 access to the connected pore network. CO₂ diffusion in the aqueous phase is slow and therefore decreases sharply in highly saturated materials, leading to incomplete carbonation. An additional factor to take into account is that the carbonation reaction of Ca-silicates is highly exothermic, e.g. the reaction enthalpy of equation (1) is about -1300 J/g. This heat may lead to internal temperature gradients and pressure build up due to vapour generation. Crucial in product design is therefore to find mix designs and reaction conditions that well balance the ongoing processes to avoid incomplete carbonation of the product interior or failure by fracturing.

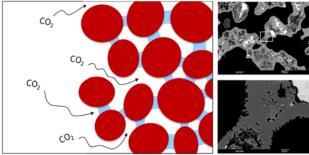


Figure 1. Moist carbonation of unsaturated compacts: schematic view (left), Backscattered electron microscopy images showing precipitation of calcium carbonates at pore necks and particle surfaces.

3 Precursors and CO₂

The precursor Ca(Mg) silicate material can be manufactured in a conventional Portland clinker kiln using the same raw materials as required for cement. Since formation of alite is not required, kiln temperatures can be lowered to about $1150\text{-}1250^{\circ}\text{C}$, saving fuel and lowering CO_2 emissions additional to the CO_2 uptake by subsequent carbonation. The Solidia company is upscaling this approach [28]. However, precursor manufacturing cost and emissions can be avoided if one can use carbonatable mined minerals, [29], or alkaline residues from other industries. The remainder of this letter will treat the latest developments in the latter field

Secondary resources that can be used include in principle all alkaline waste materials rich in non-carbonated CaO and/or MgO. Materials that have been considered for carbonation include ash from coal-fired power plants (CaO content up to 65 wt-%), bottom ash (~10 to 20 wt-% CaO) and fly ash (~35 wt-% CaO) from municipal solid waste incinerators, de-inking ash from paper recycling (~35 wt-% CaO), steelmaking slag (~30-60 wt-% CaO and MgO), and waste cement or concrete [30-34].

Most research on carbonation of alkaline residues has focussed on carbonation as a means for CO_2 sequestration, immobilising toxic elements and heavy metals, as well as improving the structural durability of wastes and byproducts [20]. Far fewer research has been carried out with the purpose of using accelerated carbonation to make construction materials [17,35-37]. Tables 1 and 2 summarize reports in the scientific literature on accelerated carbonation as a means to produce carbonate bonded blocks (Table 1) and aggregates (Table 2) from alkaline residues. Carbonation conditions, efficiency and product performance are compared.

Because alkaline residues and wastes may contain metals and other elements that potentially pose a threat to the environment, the leaching of contaminants from the carbonated products should always be assessed. Carbonation may effectively reduce the leaching of elements such as lead and zinc but may also enhance the leaching of other elements such as chromium, vanadium, copper, sulphate and fluoride in slags or ashes [38-40].

The CO_2 used in the process can be obtained from different sources: e.g. from flue gases from coal-fired power plants or from waste incineration plants, which have CO_2 concentrations of 10 to 20% [41], or from biogas, which typically contains 25-55% CO_2 [42].

4 Case 1: Carbonate bonded aggregates

The idea of combining carbonation with an agglomerating process was first introduced by Gunning et al. [46] to valorize alkaline residues, producing aggregates with suitable properties for applications in the construction sector. The pilot-scale process (see Fig. 2) was made up of a rotary carbonation reactor (pelletizing drum) operated at ambient temperature and pressure fed by the premixed residues with quarry fines and water. The product was discharged into a curing chamber circulated with dehumidified CO_2 for 7 days. Aggregates with properties comparable to natural lightweight aggregates (bulk density below 1000 kg/m³ and

pellet strength above 0.10 MPa) were obtained using at least 40% of alkaline residues such as Cement Kiln Dust (CKD), wood ash or paper ash.

More recently, the process at pilot-scale was tested for MSWI APCr (Air Pollution Control residues) using pure CO_2

[47]. The leaching of the resulting aggregate was reduced with respect to the untreated APCr for most metals (e.g. Pb from 143.9 to 1.8 mg/kg). Based on these results, the process was scaled up and is being commercialized by the Carbon8 Aggregates company [50].

Table 1. Carbonate bonded compacts (blocks): overview of investigated precursors, process conditions, efficiency and product performance.

Material Type	Composition (wt.%)	Precursor particle size	Compact size Compaction pressure	Carbonation conditions				Efficiency			
				Pressure/ CO ₂ conc.	Temp.	Moisture content/ RH	duration	CO ₂ –uptake	Compr. Strength (in MPa)	Referen ce	
Steel slag	CaO: 56.8 % MgO: 3.7 %	Median diameter 610 μm	100 mm diamer x 200 mm heigh 25x25x25 cm 1x1x1 m Bulk density: 2.30 g/cm³	1.005 atm 1 L/min 1.030 atm		L/S= 0.053- 0.063		6±1 weight %	18.3 19	[43,44]	
Stainless steel slag		< 125 μm		0.3 MPa 100% CO₂		L/S = 0.125		18 weight %	9	[17]	
Steel slag	CaO: 41 % MgO: 7.6 %	5 ~ 24 μm	90x40x10 mm 7.75 MPa	0.536 MPa, 100% CO₂		L/S = 0.125 RH: 60~80%	120 min.	108 g CO ₂ /kg slag	45	[36]	
Stainless steel slag (EAF)	CaO: 44 % MgO: 6.8 %	< 500 μm	61x61x40 mm 17.85 MPa	2.0 MPa, 100% CO ₂	140°C	L/S = 0.12	16 hours	177-188 g CO ₂ /kg slag	55 (Tensile splitting strength:2. 7 MPa)	[35]	
Stainless steel slag (EAF)	CaO: 45 % MgO: 9.3 %	5-300 μm D50: ~ 100 μm	300x100x50 mm, 29.42 MPa	2.0 Mpa, 100 % CO ₂	140°C	L/S = 0.10	16 hours	150-200 g CO₂/kg slag	134	[18]	
Stainless steel slag (AOD)	CaO: 55% MgO: 8.0 %	10-200 μm D50: ~ 60 μm	40x40x40 mm Fresh bulk	Atm. pressure 5 vol. % CO ₂	22°C	L/S = 0.15 RH: 80%	3 weeks	4.3 weight %	43	- [37]	
			density: 2.25 g/cm³	0.8 MPa, 100 % CO₂	80°C	L/S = 0.15	15 hours	8.1 weight %	60	[3/]	

Table 2. Carbonate bonded aggregates: overview of investigated precursors, process conditions, efficiency and product performance reported in literature.

Material Type	Composition (wt.%)	Precursor particle size	Carbonation conditions				Efficiency		
			Pressure/ CO ₂ conc.	Temp.	Moisture content/ RH	time	CO ₂ –uptake	Compr. Strength (in N/cm²)	Referenc
Carbide slag	CaO: 69.2% MgO: 1.1 %		0.6 MPa/ 100% CO ₂	55 °C	L/S= 6	125 min	443 g CO₂/kg	224	- - [45] -
Waste lime	CaO: 81.3% MgO: - %	- - 74 - 150 μm					585 g CO₂/kg	277	
Steel slag	CaO: 38.9% MgO: 7.6 %						202 g CO ₂ /kg	198	
Waste concrete	CaO: 23.5% MgO: 1.6 %	_					75 g CO₂/kg	104	
CKD/paper ash/ wood ash/MSWI APCr	CaO: 45-70% MgO: 1-14%	-	1 atm./ 100% CO ₂	22 °C	L/S =	7 days	3.9-8.3 %	120-170	[46,47]
Carbon steel slag (BOF)	CaO: 55% MgO: 14%	0.4 – 0.5 mm	1 atm./ 40% CO ₂	20-50 °C	L/S = 0.2	30 min.	4-10 %	-	[48,49]

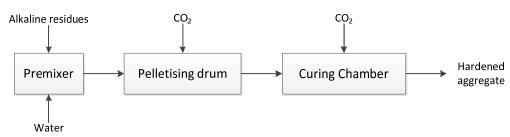


Figure 2. Scheme of a pilot-scale process proposed for aggregates production based on carbonation of industrial residues. Adapted from [46].

The production of carbonate-bonded aggregates from Basic Oxygen Furnace (BOF) steel slags was investigated by Morone et al. [48] in a lab-scale granulation device and more recently by the same group also in a pilot-scale rotating drum [49]. Operating at lab-scale at 40% CO₂ concentration and ambient temperature and pressure, allowed to obtain a CO₂ uptake of 2.5% immediately at the end of the test (30 min) and of 5.5% after 28 days curing in a moist atmosphere. [49]. A remarkably higher CO₂ uptake was achieved for the pilot-scale tests performed in a rotary kiln at 40% CO₂ concentration, atmospheric pressure and temperatures of 30 or 50 °C. Values between 4.4 and 6% CO₂ uptake were measured in samples collected immediately after the treatment, which increased up to 10% in some of the samples after 28 d curing. The increased CO2 uptake in the pilot tests was explained by the higher specific surface area of the granules exposed to the CO₂ flow with respect to labscale tests [49]. Besides the potential increase of the leaching of Cr and other oxyanion-forming elements, the main barrier to produce carbonate-bonded aggregates according to this process was the mechanical properties. Latest developments show that for BOF steel slag this issue can be addressed by using alkaline activators although in this case the leaching of Cr and V did not comply to leaching limits for unbound use, nor did the product pH [51].

5 Case 2: Carbonate bonded compacts

Apart from CO_2 sequestration and mineral stabilization, the formation of carbonates by accelerated carbonation of steel slag can also serve as a consolidation for the slag material. A typical process for direct carbonation of compacts consists of 3 main steps (Fig. 3): (1) pre-treatment of the precursors, including mixing and grinding, if required; (2) shaping of the precursor into blocks, e.g. by (hydraulic) compaction; and (3) CO_2 curing in an autoclave (at elevated CO_2 pressure) or in a climate chamber (at low CO_2 pressure).

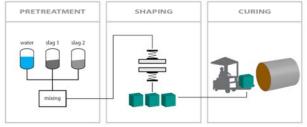


Figure 3. Schematic drawing of the accelerated carbonation process developed for stainless steel slag by [18].

In 1999 large porous blocks (1mx1mx1m) were made using only steelmaking slag and CO_2 [44]. The blocks were produced by moist carbonation in an airtight mold. CO_2 gas saturated in steam was injected through the bottom of the molds at a pressure of 1.03 atmosphere over a period of 12 days. The compressive strength of the resulting blocks reached 18 \pm 3 MPa. The blocks were carbonated homogeneously and the CO_2 uptake was approximately 6 % [44]. The NKK corporation (presently JFE steel) started

marketing these blocks under the registered trade name - Marine Blocks for constructing artificial reefs [52].

Subsequently, Quaghebeur et al. [35] produced stainless steel compacts that reached compressive strengths up to 55 MPa using pure CO_2 at a pressure of 20 bar and 140°C. By optimizing the particle packing and compacting pressure EAF steel slag compacts (300x100x50 mm) were produced with compressive strengths up to 134 MPa after carbonation and that complied to environmental leaching limit regulations [18]. The CO_2 uptake was 10-15 %. Salman et al. [37] made similar compacts of AOD slag that reached compressive strengths of 34 MPa after 3 weeks in a carbonation chamber at atmospheric pressure with 5% CO_2 , and 59.8 MPa after 15 hours in a carbonation reactor at 8 bar CO_2 and 80°C.

Recently, moist carbonation of steel slags was taken to the industrial scale by the DeRuwBouwGroep (DRBG) company for the production of pre-cast building blocks. The products consist of natural sand mixed with fine milled steel slag and are carbonated in a climate chamber at low pressure. The first products were released on the Dutch market early 2017.

6 Conclusions and perspectives

Different alkaline residues or waste materials can be used to make sustainable products for the construction industry by accelerated carbonation. Most studies on accelerated carbonation have focused on the carbonation efficiency and carbonation rate has been studied as a function of L/S ratio, pressure, temperature, particle size, CO_2 concentration and pressure. However, when the main goal is to produce valuable construction products that can compete with traditional construction materials, the technical properties, environmental impact and processing costs are at least as important as the carbonation rate and it has been noted that the highest CO_2 conversion rates do not necessarily result in the highest compressive strengths.

Carbonation coupled with granulation also appears to be a promising treatment for permanently storing CO_2 in solid form, obtaining a product (aggregate) that may be employed for civil engineering applications and also valorize different types of alkaline waste materials that are currently under utilized. Although, there are already full scale examples of application of this granulation process, some issues need to be further elucidated. In particular, the treatment time and the amount and type of waste materials and additives (cements or alkali activators) should be optimized in order to obtain a product presenting suitable mechanical properties and leaching behavior. Future studies will also need to assess the overall environmental, as well as economical, sustainability of these treatment processes as well as the net uptake of CO_2 achieved.

At present few studies have focused on strength development, and factors such as microstructure development that affect strength are not as well investigated as factors affecting carbonation efficiency. Besides carbonation rate the compressive strength that can be gained by compacts through mineral carbonation also

depends on compact size and shape, compaction force, particle packing and carbonate mineralogy.

One of the main challenges for accelerated carbonation is to obtain a CO_2 gas stream at the carbonation site. In most lab studies high purity CO_2 is used, which is costly. New efficient and low cost CO_2 capturing technology may significantly lower these costs. Using flue gases or biogas directly or after a CO_2 pre-concentration treatment can be a viable option, as long as these do not contain any constituents that may affect the CO_2 uptake or mechanical performance of the carbonated compacts.

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