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Review

CO_2 Mineralization Methods in Cement and Concrete Industry

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Abstract: Production of Portland clinker is inherently associated with CO_2 emissions originating from limestone decomposition, the irreplaceable large-scale source of calcium oxide needed. Besides carbon capture and storage, CO_2 mineralization is the only lever left to reduce these process emissions. CO_2 mineralization is a reversal reaction to clinker production—CO_2 is bound into stable carbonates in an exothermic process. It can be applied in several environmentally and economically favorable ways at different stages of clinker, cement and concrete life cycle. These possibilities are assessed and discussed in this contribution. The results demonstrate that when combined with concrete recycling, the complete circularity of all its constituents, including the process CO_2 emissions from the clinker, can be achieved and the overall related CO_2 intensity significantly reduced.

Keywords: recycling; circular economy; carbonation; recycled aggregates; supplementary cementitious material; carbon neutrality

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1. Introduction

In this paper, state of the art of CO_2 mineralization methods applicable in the cement and concrete industry are assessed and discussed with emphasis on the efficiency of their CO_2 saving potential and their synergies at different stages of the value chain.

Portland cement-based concrete is the most used man-made material [1]. The concrete has allowed the construction of buildings, roads, bridges and more for centuries now. This is possible because concrete provides the needed strength and resilience to buildings. Concrete is locally produced and robust as it hardens quickly and can be used by unskilled builders. Concrete is also the main building material because it is relatively cheap, and construction is easy. The application of concrete to a building environment allowed the development of modern developed societies. The ongoing urbanization and social development results in further growing demand for Portland-cement based concrete, even more than that for steel or wood [1]. This is also related to the fact that there is no alternative material that can replace concrete [2–4]. However, this development comes at cost. Concrete production is responsible for 5–8% of anthropogenic CO_2 emissions [3–5].

1.1. Clinker Reduction Measures

To address this challenge, the work of industrial and scientific laboratories focused on the optimization of the production process, cement characteristics and the concrete performance, among the other solutions [4–6]. Concrete gains most of its performance from the reaction of Portland clinker-based cement with water. The Portland clinker is made from readily available raw materials comprising limestone and clay. The production process requires grinding and homogenization of these materials that is followed by calcination, i.e., heating of the raw material blend to high temperature. Here, CO_2 bound in limestone as calcium carbonate is released and the CaO is bound into reactive clinker mineral. This process is called clinkering and results in the formation of Portland clinker. The CO_2

released from calcium carbonate in limestone during clinkering is called process emissions. Portland clinker is ground together with about 5% gypsum to form traditional ordinary Portland cement (OPC). The production of OPC results in emissions of about 850 CO_2/t [7], while 2/3 of these emissions comes from the decomposition of calcium carbonate and the remaining third from fuels needed.

As cement and clinker are also the most expensive constituents, the focus has traditionally been put on the reduction of their content in concrete and cement, respectively, also reducing the overall CO_2 intensity. Yet, these two reductions have several limitations. The improvement of the manufacturing efficiency of Portland clinker is limited at present [5] while new solutions, including the electrification of the kiln, are still under development [8,9]. Modern Portland clinker-based cements are mostly composite cements, i.e., the composite of Portland clinker and supplementary cementitious materials (SCMs) [10]. Though, the clinker-to-cement ratio, which illustrates the use of the supplementary cementitious materials for cement production, has not changed significantly in recent years, and was 0.72 in 2020, as estimated by IEA [11]. The main obstacle to its further reduction is the availability of the suitable SCMs [3]. However, even without availability limitations, these developments, alone or combined, cannot reduce all CO_2 emissions associated with the Portland cement concrete production, as the process emissions cannot be avoided. Limestone is the sole calcium oxide source available at large enough quantities and cannot be substituted [12].

1.2. Carbon Capture and Storage, Carbon Capture and Utilization

Considering these limitations, Cembureau [13] in their carbon neutrality roadmap suggest that the measures related to improvement at the clinker, cement and concrete level will have a limited contribution. Clinker measures include an increased use of biofuels, improvement of production efficiency and others. Cement measures comprise the production of composite cements (clinker factor reduction) and improvement of production and transport efficiency. Concrete level measures include the improvement of concrete mix and transport efficiency. Similar conclusions can be found in the other roadmaps and reports [14–16]. In all these reports, the largest reduction in CO_2 emissions is via carbon capture storage (CCS) or carbon capture and utilization (CCU) (Figure 1).

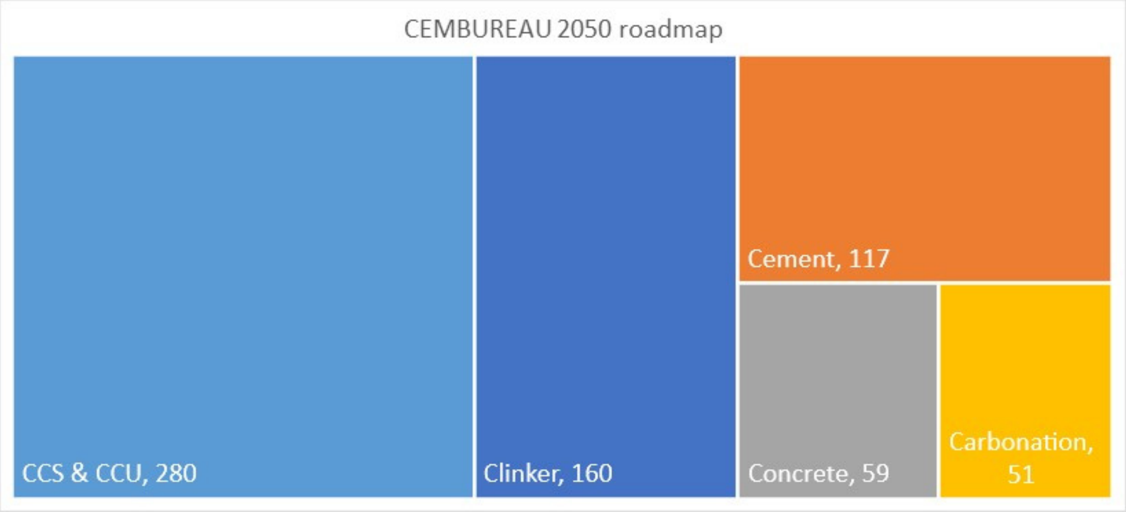

Figure 1. CO_2 reductions measures, area proportional to the potential, along the cement value chain according to CEMBUREAU when comparing to the year 2017, adopted from [13]. The numbers give the reduction in kg CO_2/t of cement. Carbonation is mostly natural weathering of concrete constructions. The biggest potential is in carbon capture and utilization (CCU) and storage (CCS) technologies.

Carbon capture and storage and carbon capture and utilization are an integrated set of technologies that avoid the release of carbon dioxide into the atmosphere. There are three major phases involved in these technologies [17]: (i) capture, i.e., separation of CO_2 gas from other gases produced at industrial process facilities such as cement plants. (ii) Gas treatment and transport including the CO_2 purification, compression, liquefaction and transport. The liquid CO_2 is usually considered to be transported via pipelines, however other modes of transport are also possible, such as in special ships [18]. (iii) Utilization or storage of CO_2. Utilization technologies point toward the use of carbon dioxide to create valuable products, while storage includes the injection of CO_2 into deep underground rock formations, often being depleted gas or oil fields.

The Global CCS Institute listed 65 commercial CCS facilities in the year 2020, with 34 of them either in advanced development reaching front end engineering design or in early development [19]. Only one of them is related to cement production. HeidelbergCement's Norwegian subsidiary, Norcem, with Aker Solutions, develop installations to capture half of the CO_2 from the Brevik cement plant in Norway. However, cement companies announced several new projects in 2021, see for example: [20–24]. In the case of the cement industry, CO_2 capture can be accomplished using post-combustion capture, oxyfuel combustion or direct separation [25,26]. The post-combustion capture method refers to capturing carbon dioxide from a flue gas generated after the production of the cement clinker. The most popular post-combustion technologies include amine scrubbing, membrane separation and calcium looping, while others are possible, such as chilled ammonia processing, and processing using alkali solution instead of amines [26]. One of the examples of the application of such technology is the Northern Lights project [27]. Within this project, the Aker amine technology [28,29] will be applied to one of the cement plants with 400 kt of CO_2 captured annually and transported for permanent storage. The CO_2 storage will be conducted in the depleted oil gas fields under the North Sea [30].

Oxyfuel combustion refers to the combustion of fuel in oxygen instead of air. In such a system, the kiln exhaust gases are mainly composed of CO_2 and steam that can be easily separated out by condensation [31]. However, the gas needs to be further cleaned for transport and storage purposes [26,31]. An example of the direct separation is LEILAC technology (Low Emissions Intensity Lime and Cement) [32]. The gas rich in CO_2 is produced by the separation of the limestone calcination from the main clinker production line. Accordingly, the processing emissions from the limestone calcination are separated from those resulting from fuel combustion for clinkering. This technology was successfully tested in the Lixhe cement plant in Belgium [33] (Figure 2). Currently, it is planned to erect the LEILAC II reactor in the Hanover cement plant, Germany, which will capture 20% of the cement plant's capacity [32]. This short overview indicates that, currently, several options are investigated based on the different physical–chemical processes and engineering solutions. Similarly, the methods of transport and storage of the captured CO_2 are currently being investigated, with the example of the project ACCSESS [34], that aims to develop the connections between the CO_2 producers from mainland Europe to storage fields in the North Sea, or the project Porthos [35]. Porthos is developing a project to transport CO_2 from industry in the Port of Rotterdam and store this in empty gas in fields in the North Sea.

While the project related to the CCS technologies are planned and developed by the cement industry, the CCU projects are less advanced. An example of the ongoing activity is the Hynamics project that targets the production of methanol from captured CO_2 [36]. Additionally, there is no consensus about the environmental impact of these type of projects. Hepburn et al. [37] listed several CO_2 utilization pathways and conduct analysis of them. Technologies that involve the production of chemicals, fuels and microalgae might reduce emissions of carbon dioxide but have limited potential for its removal. This is because CO_2 is emitted to the atmosphere during the usage, or at the end of the service of, these materials. The technologies that involve the carbonation of the construction materials or, more generally, the CO_2 mineralization of the alkaline feedstocks [38] can remove carbon dioxide, and the products of the carbonation can be used as the added value products for the construction. Various CCU technologies require purification of the CO_2 gas and high pressures or liquefication before use [39]. For example, the captured CO_2 is planned to be transported either in the supercritical or the liquid state at high pressure, approximatively 10 MPa [40]. That is why the separation, cleaning, compression, and liquification are essential methods within CCU technologies. This means that the costs of the CCU technology are important [18].

Figure 2. Picture of the LEILAC I reactor in Lixhe cement plant in Belgium Picture credits: HeidelbergCement/Paul Poels fotografien, Meerlo, NL.

1.3. Role of CO_2 Mineralization in Decarbonization of Cement Production

CO_2 mineralization, also called mineral carbonation, is a process of CO_2 storage in the form of calcium and magnesium carbonates [38,41–43]. Calcium and magnesium carbonates are poorly soluble in water, thermodynamically stable at atmospheric conditions [44] and, thus, they provide a permanent CO_2 storage solution. CO_2 mineralization is also a form of CCU and is together with the CCS the only potential way to remove process emissions associated with cement production.

The advantage of the mineralization technologies is that the flue gases from the plant can be directly used for CO_2 sequestration [45,46]. This indicates that the gas with the CO_2 concertation in the range 15–30%, typical for the exhaust gases from power plants and cement kilns [46–48], can be used for these technologies.

The second important advantage of the CO_2 sequestration by mineralization is that this method mimics naturally occurring weathering processes [39,49]. As shown in Figure 3 when CO_2 reacts with alkaline rock or alkaline wastes, it will yield carbonates such as $CaCO_3$ and $MgCO_3$, and the change in the standard molar free energy of formation is strongly negative. This is a spontaneous reaction that needs to be accelerated for industrial purposes, which can make the reaction proceed with a relatively small amount of energy. This is contrary to the production of the fuels or chemicals which also require, beyond the pure CO_2 feedstock, an energy source such as hydrogen or syngas for the chemical conversion of CO_2. This makes CCU products with CO_2 conversion more expensive than conventional products based on fossil feedstock, although the situation is improving due to technology maturing, as well as customer driven demand for such CCU products.

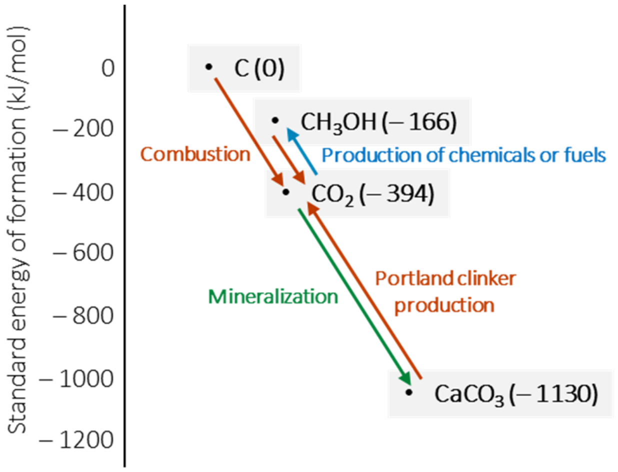

Figure 3. Standard molar free energy of formation for several carbon-containing substances at 298 K. The changes corresponding to the combustion, mineralization, utilization technology with a focus of chemicals or fuel production (example of methanol) and clinker production are highlighted (note that energy change corresponds only to the carbon-related substance). Based on data published in [39,49].

In mineral carbonation, CO_2 reacts with calcium or magnesium originating from different natural or industrial origin materials. When applied to natural materials, rocks rich in alkaline earth silicates such as olivine [$(Mg,Fe)_2SiO_4$], wollastonite ($CaSiO_3$), and serpentine [$Mg_3Si_2O_5(OH)_4$] [41,42] are considered. A disadvantage of this process, when applied industrially, is that the CO_2 mineralization reaction requires high temperatures and pressures to be accomplished, particularly in the case of the olivine and serpentinite carbonation [41,50–52]. Contrary to the carbonation of the natural rocks, the carbonation of the alkaline waste materials and construction materials can be conducted at mild conditions [38,41,49,53,54] as these materials are calcium rich. Examples of these materials include coal fly ash, steel and stainless steel slags, cement and lime kiln dusts, and red mud [38,41,55]. The availability of industrial alkaline construction and waste materials is significantly lower when compared to the calcium- and magnesium-bearing silicate minerals. Sanna et al. [41] evaluated that the global CO_2 sequestration potential of waste materials and by-products of industrial origin is 200–300 Mt CO_2 per year, while calcium- and magnesium-bearing silicates can store CO_2 volumes in the order of thousands Gt, depending on the accessibility of these materials (Figure 4).

CO_2 mineralization technologies can be divided into the two groups, i.e., in situ and ex situ methods [41,42]. In situ mineralization uses ultramafic and mafic geological formations for permanent, solid storage. In these technologies, CO_2 is injected into these reactive geological formations. In ex situ mineral carbonation, CO_2 is converted to carbonates in an engineered process. A number of processes have been developed to achieve ex situ mineral carbonation with acceptable kinetics. In general, ex-situ mineral carbonation can be divided into two categories (Figure 5):

- direct carbonation,
- indirect carbonation.

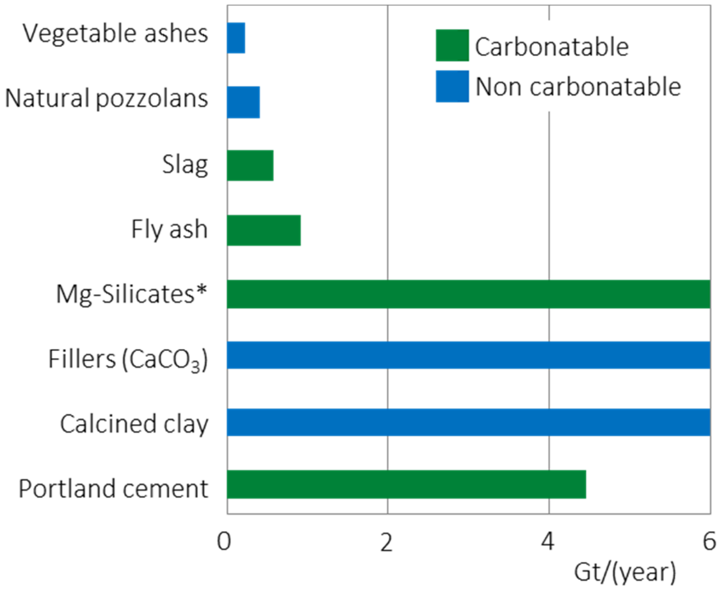

Figure 4. Use and estimated availability of possible cementitious materials, supplementary cementitious materials and fillers used in the cement industry and Mg-silicate minerals. Carbonatable material contains CaO that can react with CO_2 while non carbonatable material does not. Note, however, that $CaCO_3$ contains a significant amount of CaO and is not carbonatable. The availability of Mg-silicate minerals, fillers and (calcined) clays is believed to be significantly greater than 6 Gt/year. Data plotted based on [4,41,56]. *Mg-Silicates are non-cementitious materials.

Direct carbonation can be accomplished via direct gas–solid reactions or mineralization in aqueous solutions, while the aqueous route is the focus of several investigations, being more efficient than dry processing [57]. Indirect carbonation concerns mostly the aqueous processes. The carbonation processes take place in more than one stage. The process can be accomplished through different technologies and process routes, such as indirect multistage gas–solid mineral carbonation, the pH swing process, HCl extraction, the molten salt process, other acid extractions, bioleaching, ammonia extraction and caustic extraction [38].

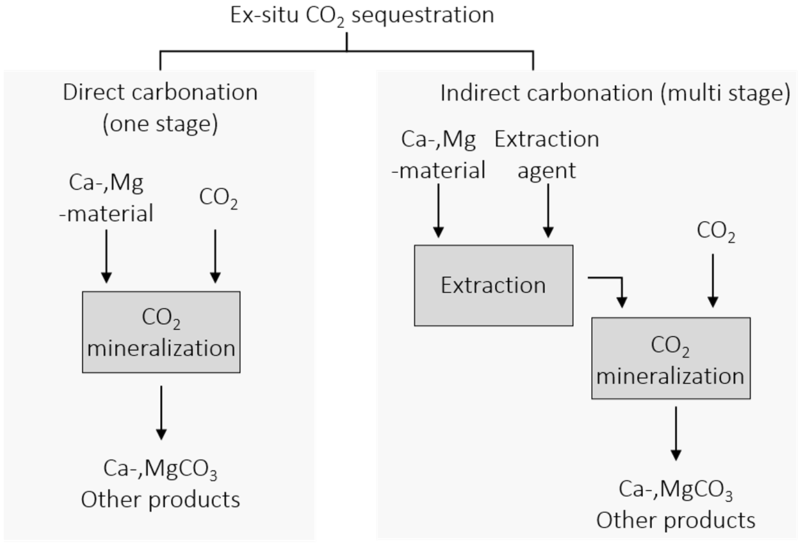

Figure 5. Mineral carbonation technologies, adopted from [38].

2. CO_2 Mineralization within Cement and Concrete Production

As discussed above, all alkaline by-products offer a yearly CO_2 mineralization potential of 200–300 Mt CO_2 [41]. Global yearly process emissions from cement production have exceeded 1500 Mt CO_2 [58]. These process emissions correspond to the CO_2 mineralization potential of cement and concrete, respectively, making it ideal feedstock for CO_2 mineralization. Below, various mineralization approaches at different stages of concrete service life are reviewed and assessed.

2.1. Carbonation Concrete during Mixing

Recently, a CO_2 utilization approach for ready mixed concrete production has been developed [59,60]. The addition of carbon dioxide to concrete during the mixing of dry components with water results in a reaction with the main cement phases, tricalcium silicate and dicalcium silicate, to form calcium carbonate and calcium silicate hydrate gel. The formed calcium carbonate is characterized by nanometre size and acts as a fine filler formed in situ [60]. This results in the acceleration of the Portland cement hydration and increased concrete compressive strength [61]. However, in this approach, the CO_2 dosage is in the range 0.1% to 1% by weight of cement [60], and so has limited sequestration potential.

Overall, the carbonation of fresh concrete during mixing is limited to <1% of its carbonation potential as higher carbonation rates would impair the hydraulic reactivity and resulting performance of hardened concrete.

2.2. Carbonation of Fresh Waste Concrete

The direct carbonation methods were suggested to beneficiate the concrete slurry waste (CSW) [62,63]. CSW is generated from ready-mixed concrete plants during concrete production and from the cleaning of the concrete trucks. It is a mixture of fine aggregates, cement hydration products and residual cement particles [64]. This material is classified as corrosive hazardous due to its high pH value, as well as heavy metal contamination and accumulation. The carbonation was proposed to neutralize the high pH of the slurry, while the carbonated solid material may be used as supplementary cementitious material [62].

This technology being important from the perspective of the waste material valorization can only bring a limited saving of the overall CO_2 emissions of the construction industry. This is because the availability of the fresh concrete waste material is limited to 1–3% of the ready mix concrete production [65,66].

2.3. Carbonation Hardening

Early CO_2 binding by fresh concrete combines the benefit of CO_2 storage via mineralization with the utilization of the reaction products including calcium carbonate, silica gel or decalcified C-S-H phase to improve the early strength of concrete products. Carbonation hardening technology can be applied in the concrete pre-cast plants since it requires installation of the carbonation chamber that can replace the curing chambers in the precast production location [67–70].

Carbonation hardening was already proposed in the 1970s by Berger et al. [71–73] as the alternative method to the steam curing. However, this technology is only presently gaining more interest, which is associated with the effort on reduction of building industry CO_2 emissions. Existing experience with carbonation hardening is documented in the latest reviews [74–77].

Carbonation of the fresh concrete relies on the fact that the calcium silicate phases react easily with the dissolved CO_2, similarly to other CO_2 sequestration techniques applied in the cement and concrete technology. Since CO_2 curing also activates calcium silicates that are normally not hydraulically active (γ-C_2S, C_3S_2, CS: γ-belite, rankinite and wollastonite respectively) [73,78,79], it opens the way for the application of the new materials for the production of concrete in precast plants. These can be classified according to the Ca/Si ratio in the three basic groups [78–80]:

- Wollastonite and Rankinite type systems with Ca/Si of approximatively 1.5 and lower. These phases are not hydraulic.

- Belite type systems with Ca/Si of approximatively 2, with significant difference between β-C_2S and γ-C_2S [81,82]. γ-C_2S is considered as not a hydraulic phase, while β-C_2S reacts slowly with water.
- Portland clinker-based systems that are dominated by alite (C_3S) with Ca/Si of approximatively 3. Alite is rapidly reacting with water.

These materials can be either tailor made clinkers, or waste stream materials can be applied, while research has focused mainly on the different type of the steel slags. Similar to Portland clinker, most steel slags contain tricalcium silicate (C_3S) and dicalcium silicate (C_2S) among the other phases [83,84]. Since CaO content is significantly lower when compared to Portland clinker [83,84], these systems contain more belite than alite and thus can be classified as belite type systems with Ca/Si of approximately 2 [85]. The dried concrete slurry waste [86,87] was proposed to be used as carbonatable binder. Additionally, naturally occurring wollastonite can be excavated and used to produce the building materials [88]. Laboratory studies have enabled the investigation of the characteristics of different calcium silicates [73,78–80], while in the semi-industrial and industrial clinkers and waste stream materials, these phases are co-existing [83,84,89–91]. The reactivity with CO_2 and the resulting performance is strongly affected by Ca/Si of the material used. C_2S is the most reactive, followed by C_3S, reaching a degree of reaction between 50% to 90% in 24 h depending on the carbonation conditions, while C_3S_2 and CS are characterized by the lower carbonation kinetics [79,80]. However, the compressive strength is not directly related to the carbonation degree [79]. Carbonated C_2S compact attains the highest compressive strength, CS is characterized by the lowest compressive strength [80].

The carbonation curing of the concrete element in the precast plant consists of several technological steps [74,76]:

- Concrete mixing and moulding

The concrete block forming is important, since needs to allow the penetration of CO_2 into the concrete element. This is a vital factor since CO_2 needs to enter the pores of cement-based materials from the external environment and reach the reaction zone in the middle of the concrete body. This can be achieved in two ways. The porosity can be increased by the high starting water to cement ratio, however, the samples need to be dried before the carbonation curing [92,93]. Alternatively, the modification of the water to cement ratio and/or the aggregates grading enables the proper pore systems, similar to the case of pervious concrete [94,95].

- Pre-curing

Pre curing is, in general, used to ensure a proper moisture content in the specimens, which, in turn, allows CO_2 penetration in the pores filled by gas [96]. Different curing methods are applied to remove water from the samples: vacuum curing [97–100], drying at low relative humidity (40–60%) [70,92,100,101] or oven drying [93]. In addition to the water removal from the samples, extended preconditioning has an impact on the hydration state of the hydraulic binders [96,98,101,102]. The prolonged pre-hydration limits the carbonation extend.

- Carbonation

During this period, CO_2 penetrates into the concrete body and the main carbonation reaction occurs. Similar to in the case of the RCA carbonation, in the case of the carbonation hardening there are two types of reactor systems: enclosed and flow-able chambers. For the carbonation procedure, CO_2 gas is typically used at high concentrations of CO_2 (10–99%) [70,78–81,93,96,100,101], while the enclosed reactors enable the application of the high pressures, generally 1–5 bars, however, higher pressures are also possible [70,80,81,93,100,103,104]. The experiments conducted with the carbonation of the industrial flue gas prove that it can be directly applied for carbonation curing [105,106].

- Post curing applies only to the hydraulic binders

Further hydration reactions are possible from the cement clinker and other cement components that were not carbonated. The post curing is general conducted in water saturated conditions at a normal temperature and pressure. The formation of the final mechanical performance and durability takes place [70,99–101], along with a pH increase to passivate steel reinforcement, if present.

The reactions involved in the carbonation hardening are more complex when compared to the hydration process. Competitive reactions between the hydration and carbonation reactions take place depending on the specific reactivity of the phases and CO_2 availability [107]. This phenomenon is pronounced when Portland cement is applied for carbonation hardening, while the system is simplified to the carbonation reactions only in the case of a non-hydraulic system, i.e., rankinite and wollastonite. The reactions involved are summarized in Figure 6. The hydration reactions are initiated directly after the contact of cement water, and result in the formation of a C-S-H phase, ettringite and portlandite [108]. The carbonation process is initiated by the diffusion of CO_2, which is usually provided as a gas, into the body of the cementitious material. Consequently, CO_2 dissolves in the pore solution and then reacts with the calcium from the anhydrous cement components or the early hydrates. In the case of Portland cement, the calcium silicates are the main source of calcium. Alite and belite phases react with CO_2 in the presence of water, while C_3A and C_4AF phases do not react considerably [54,73,74,76,101]. In the case of small and non-reactive clinkers, belite, rankinite and wollastonite dissolve and provide calcium for the carbonation reactions [73,78,79,81,82,103,104,109]. Carbonation accelerates the reaction of the calcium silicates when compared to the hydration. This phenomenon is explained by the filler effect of calcite precipitation during carbonation curing [99]. However, another plausible explanation was suggested, whereby CO_2 dissolved in the pore solution causes a strong undersaturation of the anhydrous calcium silicates [54,81]. The strong undersaturation leads to a fast dissolution rate [110,111] and so accelerates the reactions. The carbonation and hydration results in the formation of calcium carbonate and a silica-rich phase. Calcium carbonate may precipitate as different polymorphs depending on the reaction conditions. The available literature reports that the primary polymorph of $CaCO_3$ is calcite [54,99,109,112–114]. The formation of significant amounts of amorphous calcium carbonate is also reported [100,101]. The silica-rich phase is considered to form as either a C-S-H phase, decalcified C-S-H phase or silica gel [54,72,96,99,101,112,115,116]. The available data suggest that the composition of the phase is related to the balance between the hydration and carbonation reactions. In the carbonated cements based on the wollastonite and rankinite, mostly calcium modified silica gel is observed by NMR [103,104,112,117]. The calcium content of the silica gel depends on the initial calcium silicate phase which undergoes carbonation hardening [112]. In the carbonated cements based on the belite, the effect associated with the C-S-H phase (peaks of Q^1, Q^2 silica sites) and silica gel (Q^3 and Q^4 effects, respectively) are observed [81,82,109,112] by NMR. In the case of the carbonated Portland cements, the silica-rich phase is dominated by the decalcified C-S-H phase [101,116]. Furthermore, in the case of the carbonated Portland cement, several different C-S-H characterized by different Ca/Si ratio gels can co-exist [101]. This phase assemblage can be further modified by the post carbonation hydration reactions, thought only in the case of hydraulic systems. The progress of these hydration reactions depends on the extent of the carbonation reaction [70,99–101]. The post carbonation hydration reactions have a pronounced impact on the phase assemblage. The calcium aluminate phases, delayed by the carbonation reaction, hydrate which results in the precipitation of ettringite and monophases. Furthermore, the carbonated systems, rich in the C-S-H phase with low Ca/Si ratio and silica gel, transform into systems dominated by the typical C-S-H phase formed during a normal cement hydration [70,99–101].

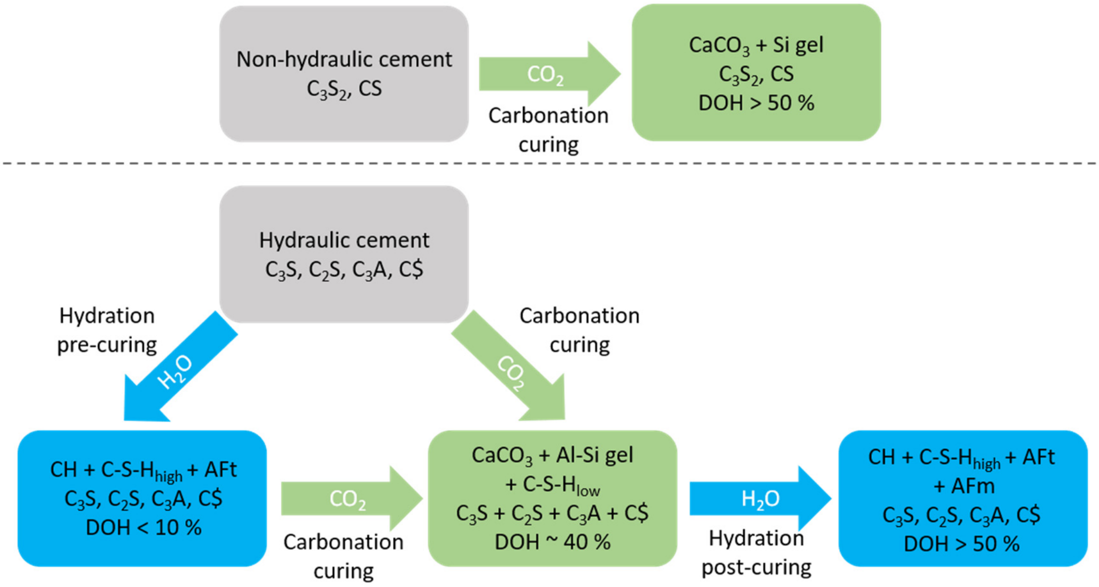

Figure 6. Reactions and their products involved in the carbonation hardening technology of a hydraulic cement and non-hydraulic cement, DOH degree of hydration of cement/clinker, C_3S, C_2S, C_3S_2, CS, C_3A—phases building cement clinker, C$—calcium sulfate, CH—portlandite, Al-Si gel and Si-gel, silica rich gels, C-S-H_{low}—C-S-H phase characterized by low Ca/Si C-S-H_{high}—C-S-H phase characterized by Ca/Si typical for hydrated Portland cement, AFt—ettringite, AFm—mono- and hemicarbonate.

The carbonation curing results in a limited reaction degree of the cement clinkers used. This is in general lower than 50% [80,99,101,109]. This phenomenon is related to the formation of the dense microstructure that limits the reaction progress [118].

The carbonation curing of the fresh cement paste results in the formation of a microstructure that is different when compared to the hydrated samples, however, depending on the starting material. In the case of wollastonite, rankinite and belite systems, calcium carbonate acts as the skeleton of the carbonated matrix with the encapsulation of unreacted grains peripherally bordered by a thin rim of calcium-modified silica gel [80,81,103,117]. In the carbonated Portland cements, less separation is observed and the fine composite is formed, made by co-precipitating decalcified C-S-H, silica gel and calcium carbonate [101]. In both cases, the carbonation hardening results in the formation of a dense microstructure that is characterized by appreciable mechanical performance [80,93,99–101,109,119].

Carbonation curing has a positive impact on the compressive strength evolution. Carbonation curing can improve the compressive strength by 20–100% at an early age and by 5–20% after 28-d subsequent water curing, when compared to the hydrated analogues [74,76]. Furthermore, it was demonstrated that carbonation curing can improve several durability properties of concrete [76]. Still, practically achievable carbonation degrees are typically below 50% as the dense microstructure required to satisfy durability prevents achieving higher carbonation degrees under economical conditions and production rates.

2.4. Carbonation of Recycled Concrete Aggregates

Even after decades of service life, concrete remains largely non-carbonated. Metanalysis [120] shows that about 23% of the carbonation potential is used during the service life and demolition of concrete structures. Old concrete hence presents a large potential CO_2 sink. After the demolition, concrete is typically crushed to recycled concrete aggregates (RCA) and used as road base, for soil stabilization or as aggregate in concrete.

Despite the successful application examples of RCA for concrete production [121,122], RCA is also known to have inferior properties compared to the virgin aggregates [123–125].

The application of RCA may result in the worsening of the concrete characteristics. Recycled aggregates differ from the natural aggregates by presence of the adherent cement paste and mortar. Since the cement paste is more porous than the aggregate itself, and often has inferior mechanical performance, RCA is characterized by higher porosity, higher surface area, higher water adsorption and lower strength. Several methods were proposed to enhance the properties of the RCA [126–128]. Among them, the carbonation was evaluated as an efficient and feasible method [126,128]. Indeed, the carbonation treatment of recycled concrete aggregates has recently gained much attention due to its RCA performance enhancement and environmental benefits [129–133].

Carbonation curing of RCA uses the fact that CO_2 reacts easily with the hydrated cement paste, in a similar way as the carbonation of recycled concrete paste. The improvement of RCA characteristics and the performance of the resulting concrete is ascribed to the densification of the adherent cement paste to the surface of the original aggregate [119,134–136]. It is important to notice that the enforced carbonation of the recycled concrete aggregates has a different purpose when compared to the enforced carbonation of the recycled cement paste. The treatment of the aggregates focusses on the increase in the product volume in the adherent cement paste and not on the optimization of the carbonation degree. The excessive carbonation may result in the pore volume increasing and cracking [137–139], which is contra productive. This is in contrast to the carbonation of the recycled concrete paste that maximizes the carbonation degree and so targets complete decomposition of the hydrated phases, as described in the next section.

Independently of the carbonation conditions, the reaction occurs over the solution, i.e., this is the dissolution precipitation reaction [55] which was verified in [140,141]. It happens in the suspension in the case of aqueous carbonation or in the absorbed water in the particles of aggregates at semi-dry conditions. For the samples carbonated under dry conditions (relative humidity lower than 95%), the following mechanism of the carbonation was proposed [119,142]: the carbonation is initiated with the reaction of CO_2 with the portlandite and C-S-H phase on the surface of the particle. This results in the liberation of the water from the hydrates and enhanced carbonation. Ca ions migrate from the inner part of the paste to outer regions. The consequent carbonation of calcium results in the deposition of calcium carbonate at the surface, intermixed with the decalcified C-S-H phase. This results in the formation of the peripheral zone with improved performance and reduced porosity. The importance of the Ca ions' diffusion is strengthen by experiments where the RCA were impregnated with the Ca rich solution before carbonation [143–145]. Such conditioning improved the results of RCA's carbonation curing. The prolonged carbonation in aqueous conditions or prolonged dry carbonation may result in the leaching of calcium, and decomposition of the hydrates that finally result in an increase in the porosity, similarly to that observed for the aqueous carbonation of concrete paste [146]. However, the performance of recycled aggregates can be improved by a short (i.e., 10 min) liquid–solid carbonation due to the formation of calcium carbonate and silica gel on the surface of the samples [147]. This short curing reduces total porosity and densifies the microstructure. Furthermore, it improves the performance of the surface of the treated recycled aggregates by increasing the bonding of these materials in concrete and finally improving concrete properties [147].

The carbonation treatment effect is strongly influenced by the carbonation conditions and the characteristics of the recycled aggregates. The CO_2 pressure, ambient temperature and relative humidity, carbonation duration, and aggregate moisture condition appear to be the governing carbonation factors [130–132]. However, there are no established general rules and relationships. The majority of the RCA carbonation curing studies were conducted at semi-dry conditions. The pressure of CO_2 containing gas depends primarily on the experimental setup used. The increased curing pressure is used in a pressurized chamber while the experiments in flow-through reactors are conducted at normal pressure. The increased pressure is believed to accelerate the carbonation curing [148]. The gas pressure generally used is in the range 1 to 5 bars [134,135,142,144,145,149–151]. The appreciable reaction degrees in the pressurized chamber are achieved after several minutes or hours [142,147,150–153] while the treatment in flow-through reactors may take several days [134,154–156]. Since the dry carbonation is strongly influenced by the diffusion of the CO_2 into the paste adherent to aggregates, the moisture of the aggregates and RH have an important effect [157]. The water saturation of the paste needs to limited to opening the porosity for the gaseous diffusion since CO_2 diffusion through liquid-water is orders of magnitude slower than in the gaseous phase [158]. The application of the sealed carbonation chambers enables the application of the vacuum treatment that is believed to accelerate the carbonation [134,135,144,149,151]. Another pre-treatment method includes the drying of the samples [159–161]. The optimal relative humidity is 40–70% when the fast carbonation rates are obtained [152,162]. It is reported that increased gas concentration accelerates the carbonation rate extension, however, to about 20–40% which makes the direct application of the cement plant exhaust gases possible [155,163]. However, several carbonation tests were conducted when using 100% purity CO_2, particularly in a pressurized chamber [134,145,149,151]. Overall, the optimal carbonation conditions and treatment performance depend on the moisture content of the recycled aggregate and other aggregate properties [148]. Lately, a modification of the thorough flow reactor was proposed, which combines carbonation and grinding [164]. The higher the content of adherent cement paste is, the bigger the positive impact of the carbonation curing is [134,136,153,165]. The higher content of the paste is generally associated with either a smaller size of the material used, or a greater strength of the concrete or higher strength class of used cement. On one hand, the high variability of the optimal conditions can be related to the different starting materials or experimental setup used. On the other hand, they could be related to the nature of the carbonation treatment that balances between the filling of the porous matrix by the carbonation products and the decomposition of this matrix by carbonation.

A proper carbonation curing improves the properties of the recycled concrete aggregates and consequently the characteristics of the concrete with the aggregates. The concrete workability is improved when compared to the concrete with the uncarbonated recycled material [150,153,166,167]. The evolution of the compressive strength of the concrete with carbonated aggregates is higher when compared to the uncarbonated ones, though it is still lower than the concrete based on virgin aggregates [136]. Carbonation curing improves the compressive strength of the concrete by 5% to 50%, when compared to the concretes with the uncarbonated recycled aggregates [136,143,150,153,154,160,165–168]. Furthermore, the durability of the concrete is improved by the carbonation treatment of the recycled aggregates [149,166,169].

2.5. Carbonation of Recycled Concrete Paste

According to the available literature data, the treatment of the recycled old concrete by means of carbonation can be conducted by direct and non-direct methods. These are described in this section separately.

2.5.1. Direct Carbonation Methods of Recycled Concrete Paste

The same carbonation potential as in RCA can be exploited in the so-called enforced carbonation [170] of recycled concrete paste (RCP) also called recycled concrete fines. Contrary to RCA produced by a simple size reduction, advanced recycling enabling separation of the old concrete into aggregates, sand and RCP rich in cement hydrates is needed. The advantage is that the RCP removal not only improves the quality of the aggregates and sand, but also significantly simplifies and accelerates the rate of the CO_2 mineralization as RCP is a powder with fineness comparable to the original cement used.

The ex situ carbonation of RCP can be conducted with the dry or wet methods (Figure 5). While the aqueous carbonation technique allows close to full carbonation degree [54,146,171], the dry methods enable significantly lower carbonation levels. The direct gas solid carbonation experiments [86,140,172,173] demonstrated that about 1/3 of the total carbonation potential can be achieved, whereas the carbonation temperature and water content has an important effect. The lower efficiency is attributed to the dense carbonation layer on the surface of particles, which prevents the further diffusion of CO_2 and thus reaction [173,174]. Still, the origin of the difference among the dry and aqueous carbonation is not fully understood.

The direct aqueous carbonation methods applied to valorize the recycled concrete paste was extensively investigated in Japan from the beginning of the 21st century [175–177]. In these methods, the high CO_2 pressure was used to extract calcium and accelerate the carbonation process. These pioneering works were later on followed by the carbonation experiments at normal conditions and other research groups [54,146,171,172,178–184]. At ambient conditions, the carbonation reaction proceeds via a gas—liquid—solid pathway [55,185]. The carbonation reaction is a complex multistage process including the dissolution of CO_2 into the liquid phase, formation of carbonic acid, dissociation of the acid that in turn influences the dissolution of the Ca and Mg rich minerals, and finally, nucleation and growth of carbonate precipitates. During the enforced carbonation of the hydrated cement paste that constitutes the RCP, cement hydrates convert into calcium carbonate and an amorphous alumina–silica gel, as schematically represented below [54,146]:

$$Ca(OH)_2 + CaO - SiO_2(Al_2O_3) - H_2O \rightarrow CaCO_3 + SiO_2 - Al_2O_3\text{-gel} + H_2O \quad (1)$$

The pH evolution during the carbonation experiments appears as a useful tool to investigate the mechanisms of the reaction [146,171,178,179,183,186]. Initially, the pH is high due to the dissolution of CaO from RCP and the consumption of CO_2 from the solution and from the gas. After passing the maximum, the pH decreases to reach a steady state evolution at longer carbonation times where the pH is dominated by the CO_2 dissolved in the solution. Analysis of the evolution of the carbonation solution concentrations and the CO_2 concentration in the gas revealed that the carbonation reaction of the RCP can be divided into two stages [171,178]:

(i) An initial stage, which is limited by the rate of the dissolution of CO_2 into the solution and precipitation of carbonation products. This stage takes place until the main peak in the evolution of pH is reached. Furthermore, the kinetics of the calcium carbonate precipitation plays an important role in the first kinetics stage [171].
(ii) The second stage concerns the time after the main pH peak where the rate is limited by the dissolution kinetics of the hydrates.

One notes that the mechanism of the aqueous reaction is the same for the Portland cement clinker and the hydrated cement paste [54,171]. The carbonation reaction is very rapid. The carbonation degrees close to the full carbonation potential are reached only in a few hours at normal temperature and pressure [146,178]. However, the other studies report significantly lower carbonation degrees in the range of 40 to 50% [178,179,182,183]. The possible high reaction degree is associated with the fast dissolution rate of the cement clinker and hydrates under the action of CO_2 [54,171]. This is the main difference to the carbonation of Mg-silicate natural minerals that are characterized by the low dissolution kinetics [187] that limits the leaching of magnesium from the minerals [188]. The kinetics of the overall process depends on numerous factors and parameters, such as temperature, pressure, pH of the solution, solid solution ratio, CO_2 concentration in the gas, phase composition and particle size distribution of the reactive solid material [185]. The partial pressure of CO_2 and alkali concentration accelerate the initial stages of the carbonation reaction considerably. Increased concentration of the CO_2 in the gas and alkali concentration in the carbonation solution enables a higher dissolution of CO_2 into the carbonation solution and a higher undersaturation with respect to the hydrates and clinker phases. Both phenomenon result in a faster reaction [171,180]. However, the effects depend on the solid solution ratio [178,179].

In general, the direct aqueous carbonation of the cement paste is conducted at normal pressure and temperature. The CO_2 gas concentration used varies between 10 and

100%, while the solid–liquid ratio varies between 10 and 100 [54,146,171,178–180,182,183]. Additionally, the literature reports experiments conducted at higher pressures of 10 to 20 bars [189,190].

As mentioned above, the main product of the carbonation reactions for recycled concrete paste is calcite [146,178]. Furthermore, the content of the amorphous calcium carbonate is limited. At normal temperature, calcite is the dominating polymorph, independently of the carbonation solution concentrations [171,181]. These findings are in contrast to the natural carbonation process where several of the calcium carbonate polymorphs are formed [191,192]. The second product of wet carbonation is an amorphous alumina–silica gel as revealed by NMR and XRD techniques [146,171,181,193]. This gel is formed from the gradually decalcified C-S-H phase and calcium alumina hydrates [146]. The structure of the gel is similar to the gel formed during the natural and accelerated carbonation of the cement pastes [191,194]. These carbonation products form a specific microstructure in the aqueous carbonation [146,171,181]. Calcite mainly precipitates in the space occupied by the solution reflecting that calcium is leached from the cement paste grains and combines in the solution with the dissolved CO_2. Only at the later stages of the carbonation process, does a co-precipitation of calcium carbonate with the alumina–silica gel take place in the space initially occupied by the cement paste grains. Alumina silica gel is formed in the space occupied by the initial cement paste grains. Because of calcium leaching from the cement paste grains, an open microstructure is formed.

The direct carbonation methods have several advantages, including the simple process that can be easily upscaled industrially, as demonstrated during an industrial trial [45]. Nevertheless, currently there is no full scale application of this process while the technology is being developed [195].

The carbonated cement paste can be used as an SCM to produce a composite cement [196–199]. The pozzolanic activity of the alumina-silica gel results in the formation of a phase assemblage which is similar to those found in composite cements containing siliceous fly ash and limestone.

2.5.2. Indirect Carbonation Methods of Recycled Concrete Paste

Indirect or multi-stage carbonation is usually an aqueous process involving using acidic reagents to dissolve cementitious material or waste to extract calcium, and then exposing the Ca rich solution to a gas or solution that contains CO_2 to precipitate $CaCO_3$ [200]. As extracting agents, it was proposed to use either pure water [201–203] or organic and inorganic acids like HCl, HNO_3, CH_3COOH [204–209], alternatively NH_4Cl and NH_4OH can be used [206,207]. Using the strong acids allows for the high rate of the calcium extraction from RCP, including the extraction of calcium from limestone aggregates as presented in the RCP [207]. However, the acids application requires the addition of alkalis before the calcium carbonate precipitation step, this is the so-called pH swing method [206–208]. After the extraction, the suspension is frequently filtered to separate the calcium rich solution from a residue. The produced residue is an XRD amorphous material rich in silicon and calcium [203,207]. It was proposed to use this residue as the phosphorus purification agent [210]. The calcium carbonate precipitation step is realized either by reaction of the calcium rich solution with the gaseous CO_2 [201–203,206], or with the alkali carbonate solutions [204,205,207,208]. It is possible to directly use the flue gas from a cement plant for these carbonation technologies [202,203]. The main product of the indirect carbonation is calcium carbonate separated from the other process products [204,206,208] with a purity that can be higher than 95% [202,203]. Complex processes are frequently involved targeting at material treatment or recalculation of the solutions or treatment of the waste solutions [204,205,207]. These processes enable the separation of the enforced carbonation products and so the production of the pure calcium carbonate and more concentrated alumina–silica gel, respectively. However, when compared to the direct carbonation, indirect carbonation is characterized by much higher complexity and costs, especially resulting from the regeneration of spent extraction agents. These carbonation treatments are conducted at ambient pressure and temperature while the duration of the extraction step is between 1 and 10 h.

Recently, a new process was proposed to convert recycled concrete paste to a Ca-rich residue and a Si-rich gel by using a two-step carbonation process [211,212]. This two-step technology includes reacting RCP with a Na_2CO_3 solution to precipitate a calcium-rich residue consisting of calcite. Consequently, after filtration, the solution containing Na_2SiO_3 and NaOH is exposed to CO_2 gas to obtain the suspension with a silica-rich gel and the Na_2CO_3 solution rich in silica and alumina. The XRD and NMR investigations confirmed that the silica gel is an amorphous phase similar to that formed during the direct carbonation experiments [146]. The authors proposed that these materials can be used as filler to replace the limestone powder used in cement production and as an additive with pozzolanic properties, respectively [211].

In general, the multi-step carbonation experiments of recycled concrete paste are conducted at ambient pressure and temperature. This is in contrast to the carbonation of the natural minerals containing Ca and Mg like olivine or serpentinites [41]. Still, the research is ongoing to apply indirect carbonation methods to produce the supplementary cementitious from the natural rocks. It was proposed to use lizardite for the synthesis of reactive silica residues that can then be used as supplementary cementitious material [213]. In this process, the lizardite needs to be activated at 630 °C for 4 h [213,214]. Within this process [213], the mineral dissolution stage is repeated three times. Each stage comprises the treatment of heat-activated lizardite in a batch reactor with CO_2 at 6.5 bar and 45 °C for 2 h to extract Mg and to produce a silica-enriched residue. The solid residue is separated and wet milled, and then the process is repeated. These additional milling and dissolution steps are performed to maximize Mg extraction from heat activated lizardite. Finally, an amorphous silica which is characterized by 88% purity and may be used as pozzolanic cement replacement is produced [213]. A similar material can be obtained by one step carbonation experiment of thermally activated serpentinite at hydrothermal conditions (150 bar and 150 °C) and the acid treatment of the obtained residue [215].

Overall, the indirect carbonation enables achieving higher grade products at the expense of a two-step process involving extraction agents resulting in higher costs and complexity. From the perspective of the cement industry, such technology has only limited applicability, as the higher-grade products are not necessarily better performing in applications within the industry and the volumes needed in other industries are limited. Additionally, the complexity makes the technology more sensitive to impurities in feedstock material as well as gas, making it economically as well as technically challenging. The main part of the paper will therefore focus on direct carbonation as the most suitable CO_2 mineralization method.

3. Discussion

3.1. Circular Economy and CO_2 Mineralization

Rapid urbanization processes generate significant quantities of construction and demolition (C&D) waste. European countries and the United States contribute over 800 and 550 million tons every year, respectively [216–218]. In China, over 2.3 billion tons of C&D waste is produced every year [219,220]. Most of this waste is dumped or downcycled [221]. In addition, the demand of natural aggregate resources for concrete is still rising, which generates negative impacts on the surrounding environment [123]. As discussed above, CO_2 mineralization has the potential to be integrated with concrete recycling and to make concrete fully circular.

A circular economy consists of prolonging the life cycle of materials, and results in saving non-renewable natural resources through the reduction, reuse, recycling, and recovery of already used materials. This principle can be effectively introduced to the cement and concrete industry as schematically shown in Figure 7, utilizing the fact that concrete is 100% recyclable. At the end-of-life phase, the concrete constructions are demolished.

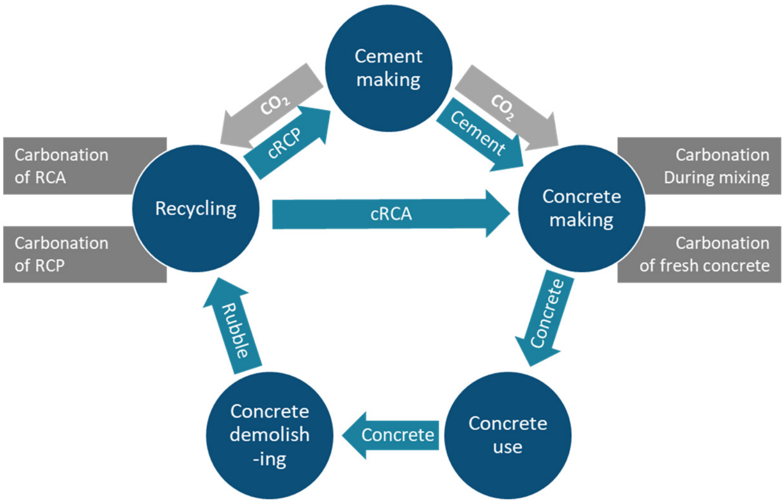

Figure 7. Circular economy concept applied to the cement and concrete production. Application of the carbonation technology is highlighted. cRCP—carbonated recycled concrete paste, cRCA—carbonated recycled concrete aggregates.

Today, old concrete is mainly landfilled or used for ground stabilization, i.e., downcycled [221]. Even in countries with the best recycling practices, only a fraction of the claimed "recycled" volumes are actually used back in concrete production. Proper recycling technology enables, however, the use of recycled materials back in concrete associated with several environmental benefits [222,223], while contributing to the circular economy. Proper recycling of the concrete enables the reuse of recycled concrete paste and aggregates, preserving virgin resources [170,221]. The recycled aggregate's properties can be improved by accelerated carbonation enabling the application of this material for new concrete production [129–133]. The recycled concrete paste can be valorized by means of carbonation and used for new cement production [196,197,200,211]. Another important advantage of this process is that the carbonation of the recycled concrete paste enables nearly full sequestration of the of CO_2 released from the decomposition of the limestone during the manufacture of the Portland cement clinkers used for the production of the original concrete [146], and enables recycling of the original cement back to fresh cement. As indicated for similar materials and applications, the carbonation of demolished concrete can significantly reduce the climate impacts of the industry [224].

3.2. Deployment of CO_2 Mineralization and Related Challenges

Most of the CO_2 mineralization approaches discussed are not yet broadly industrially applied. In the following, the technology readiness levels and challenges for further deployment of the technologies are discussed, based on the core elements of the technologies listed in Table 1.

Direct carbonation of the recycled concrete paste to create the SCM has the potential to offer a cost competitive CCU for the cement industry, as the raw gas can be used and SCM is a valuable and locally applicable product. The biggest obstacles are the missing technology for continuous RCP carbonation at a scale integrated into the plant. Furthermore, cement plants are typically located far away from urban centres where the demolished concrete is concentrated, and RCP needs to be transported to the CO_2 source, presenting a logistical challenge.

Carbonation of recycled aggregates can be done close to the demolished concrete source, hence reducing the logistical challenge of the CO_2 transport. However, for that, a CO_2 source or CO_2 infrastructure are needed at the location where the carbonation takes place. An inherent challenge of the technology is low efficiency, as recycled aggregates only contain a small fraction of carbonatable material and hence the specific CO_2 uptake is low, making all specific energy demands and costs high. As the product, i.e., the carbonated recycled aggregate, replaces common aggregates of relatively low value, it is challenging to cover the high specific costs of the treatment.

Table 1. The carbonation technologies discussed within this work.

Carbonation Method		**Direct**				**In Direct**
Starting material	Ca source	Recycled Concrete paste	Recycled aggregates	Fresh concrete products	Concrete during mixing	Recycled Concrete paste
	CO_2 concentration	5–100%	5–100%	5–100%	~100%	5–100%
Process		Aqueous or dry	Aqueous or dry	Dry	Aqueous	Aqueous
Conditions		Possible at normal temperature and pressure				
Sequestration potential (% process emissions)		30–95%	~30%	30–90%	Few %	40–100%
Products		SCM	Improved aggregates	Hardened concrete	Fresh concrete	Added value products
TRL		4	8	8	9	4–9
Challenge		Technology not yet available Logistics	Low efficiency CO_2 infrastructure Low value of products	Reinforcement Corrosion Missing experience	Limited sequestration CO_2 infrastructure	Cost

Carbonation of fresh concrete products faces two related limitations: missing long-term experience with the final product, especially regarding durability, and missing protection of conventional reinforcement from corrosion. While the first challenge can be overcome in future, the second limitation is inherent to the technology, preventing its use for reinforced elements. Alternative reinforcement materials could be used; however, their cost is generally much higher than the mild steel normally used.

Fresh concrete carbonation is straightforward and mature. The largest inherent limitation is its limited extent, as an excessive carbonation would impair the hydraulic reactivity of fresh concrete and hence also its final properties and value.

The indirect carbonation is inherently associated with the highest costs as it requires several steps, complex plants and chemicals to moderate and catalyse the reaction. It might be profitable provided a high value of the products made. However, such high quality and value is typically not needed in the building materials industry and hence application within the cement and concrete industry is expected to remain limited.

4. Conclusions

Carbon capture, utilization and sequestration solutions will play an increasingly important role in addressing climate change by the cement and concrete industry. While there are several projects developing CCS technologies, the application of CO_2 mineralization in the cement industry is less advanced, perhaps with the exception of the first applications of the carbonation hardening technologies at industrial scale, see for example [225]. However, the synergies between the direct CO_2 mineralization methods discussed in Section 2, with complete recyclability of concrete illustrated in Figure 7 have the potential to significantly reduce the CO_2 emissions associated with concrete production and, together with CCS, ultimately lead to carbon neutral concrete production. The analysis of the reactions involved in CO_2 mineralization applied to cementitious materials revealed that the processes can be conducted at normal temperatures and pressures, achieving high CO_2 sequestration potential. Consequently, the development of the industrial solution is technologically straight forward, which could facilitate a fast deployment of full-scale projects in the near future, provided there are favorable market and legislation conditions. The CO_2 mineralization methods provide several solutions that can be combined in different ways and applied depending on different environments and local circumstances. Finally, one notes that the application of the CCS and CCU will changed the whole value chain of the cement and concrete industry. It requires significant reconfiguration of the existing sites to accommodate the infrastructure for the CO_2-containing gas treatment, as well as the development and deployment of new business models.

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