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**Review paper**

# Mineral carbonation of industrial wastes for application in cement-based materials

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*ABSTRACT*

Mineral carbonation is a way to permanently store carbon dioxide ($CO_2$) in cement-based materials. Demolished concrete waste and other types of alkaline industrial wastes, like iron and steel slags, fly ash, concrete slurry waste and cement kiln dust are prospective candidates for accelerated carbonation application. This paper presents CO2 sequestration potentials, impacts of the accelerated carbonation on the selected properties of wastes and their possible applications in cement-based materials. Based on the analysis of published research in the area it was concluded that porosity, volume stability and heavy metals leaching of different waste types are improved after accelerated carbonation pre-treatment. This increases the effectiveness and broadness their application as substitutes for aggregates and binders in mortar and concrete. The research is however still very limited in the area of the application of the carbonated wastes with highest CO2 sequestration potential, namely recycled concrete powders and iron/steel slags. Besides, for proper conclusions on the environmental benefits, an LCA (Life Cycle Assessment) which includes all the phases of the life cycle must be performed, which is also lacking in the published research.

## 1 Introduction

The carbon dioxide ($CO_2$) emissions have increased drastically above the period 1850-1900 and continue to increase [1], Figure 1. This is mainly a result of the unsustainable energy and land use, lifestyles and patterns of consumption and production of humans all over the planet. Due to high concentrations of $CO_2$ in the atmosphere, global average surface temperature rapidly rises causing climate changes [1], Figure 1. Human-caused climate change has already resulted in rise of the sea level, decrease of Arctic sea ice, ocean acidification, greater frequency and intensity of weather and climate extremes, loss of many spices, etc. [1].

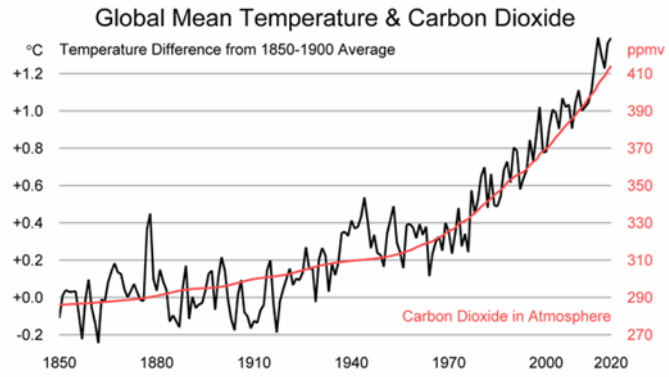


*Figure 1. Rise of $CO_2$ concentration in atmosphere and global mean temperature*

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To prevent irreversible consequences, the Intergovernmental Panel on Climate Change (IPCC) in their 2018 Special Report, titled Global Warming of 1.5°C, recommended that global average temperature increase should be limited to 1.5°C above period 1850-1900, by 2100. For that goal to be reached, it is necessary that global $CO_2$ emissions reach net zero by 2050, while net non-$CO_2$ radiative forcing (other greenhouse gas) is significantly reduced after 2030 [2].

Reaching carbon net zero in such a short time is global task that requires deep and rapid (in some cases immediate) transition in energy, land use, infrastructure (including transport and buildings), and industrial systems. Humankind faces a huge challenge, probably one of the largest ever.

As for cement and concrete industry, there are multiple pathways toward reaching net zero $CO_2$ emissions. They can roughly be classified into: (i) savings in clinker production [3], (ii) savings in cement and binder [4, 5], (iii) efficiency in concrete production [6-9], (iv) efficiency in design and construction [10], (v) re-carbonation ($CO_2$ sink) [11], and (vi) decarbonisation of electricity [3]. However, due to the high degree of process-related $CO_2$ emissions, cement industry is regarded as one of the rare examples where carbon capture and storage or utilisation (CCUS) technologies must be implemented since conventional mitigations levers will not suffice.

## 2. Carbon capture and utilization/storage CCUS

Carbon capture and storage includes the separation of $CO_2$ from industrial and energy-related sources, transformation into a liquid state by compression, transport to a storage location and long-term isolation from the atmosphere [12]. Permanent storage is possible only as geological storage (in geological formations, such as oil and gas fields, coal beds and deep saline formations), ocean storage (direct injection into the ocean water column or onto the deep seafloor) and mineral carbonation [12]. Figure 2 shows the plan of the first industrial-scale carbon capture and storage facility installed at a cement plant in Brewik, Norway. The beginning of the operation is planned by the end of 2024 [13].

Captured $CO_2$ can be utilised in different carbon containing products, in which case it can be permanently (mineral carbonation) or temporarily stored (production of synthetic fuels, urea, methanol, etc.) [14, 15]. Carbon capture and storage is referred to as CCS and carbon capture and utilisation is referred to as CCU. In general, both pathways are together referred to as CCUS.

Mineral carbonation denotes chemical reactions in which $CO_2$ is converted to solid inorganic carbonates. It occurs naturally, where it is known as weathering carbonation of natural silicate rocks, such as serpentine, wollastonite and olivine, rich in magnesium oxide (MgO) and calcium oxide (CaO). The process can be accelerated using alkaline solid waste instead of natural rocks, but in both cases, it permanently stores $CO_2$ in the form of thermodynamically stable carbonate ($CaCO_3$/$MgCO_3$) (Eq. 1):

$$\mathrm{CaO(MgO) + CO_2 \rightarrow CaCO_3(MgCO_3) + heat} \quad (1)$$

Since natural mineral carbonation is a very slow process, current research is oriented towards accelerated carbonation of various industrial waste such as iron/steel slags, coal and fuel combustion products (fly ash), mining/mineral processing wastes, incinerator residues, cement and concrete wastes, pulp and paper mill waste, etc. [16, 17].

### 2.1 $CO_2$ utilization in the cement and concrete industry

There are multiple ways to utilize the captured $CO_2$ from cement plant or any other large point source, and to store it permanently in cement and concrete products. They include $CO_2$ curing of concrete products at early age [18, 19, 20], injection of $CO_2$ in the fresh concrete mix [21, 22, 23] and mineral carbonation of different wastes. The most investigated one however, is mineral carbonation of alkali industrial waste under accelerated conditions including demolished concrete waste, concrete slurry waste, cement kiln dust, fly ash, and steel slags [24, 25, 26].

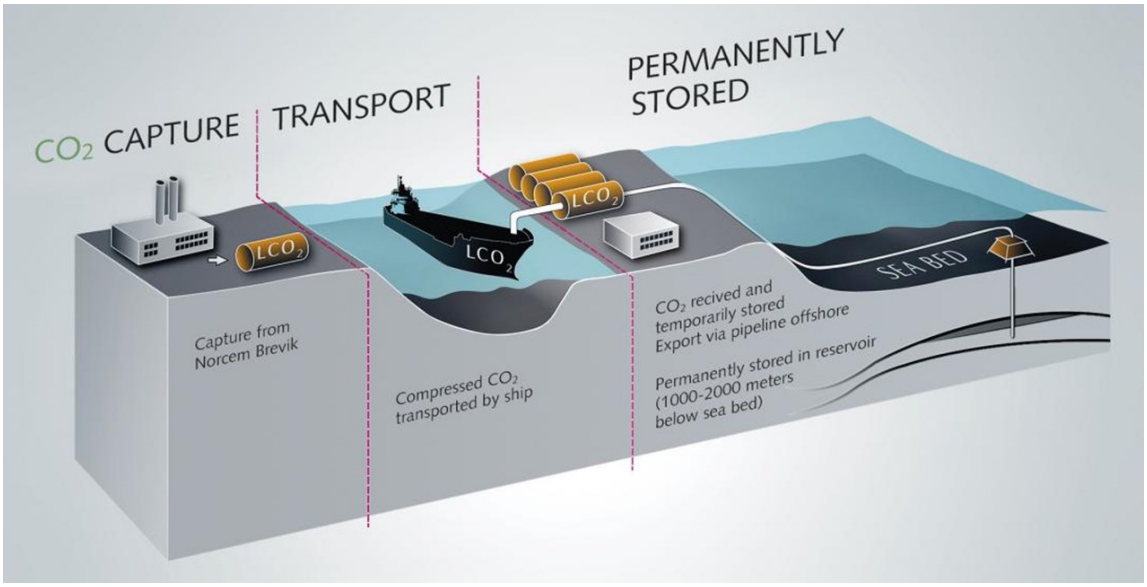


*Figure 2. CCS facility at Norcem Bervik cement plant; capacity 400.000 t per year*

In this work, a state-of-the-knowledge on the mineral carbonation of alkali industrial wastes is presented. Different technologies, $CO_2$ sequestration potentials, and possible applications in mortar and concrete production are discussed.

## 3. Mineral carbonation of demolished concrete waste

On average, industrial recycling of demolished concrete waste results in about 50% coarse recycled concrete aggregates CRCA, up to 40-50% fine recycled concrete aggregates FRCA (particle size <5 mm), and up to 5–10% of recycled concrete powder RCP (particle size <0.15 mm) by weight [27,28,29]. Over the past decade, a lot of research has been performed on the accelerated carbonation of recycled concrete aggregates (RCA). If exposed to accelerated carbonation, these wastes can rapidly absorb $CO_2$ while their properties are improved. The hydrated cement paste in residual mortar reacts with $CO_2$ to form $CaCO_3$ that fills the pores and densifies microstructure of RCA.

Due to the inferior quality of RCA compared with natural aggregate (NA), additional cement is usually required in the recycled concrete (RAC) to obtain performance similar to referent natural aggregate concrete (NAC) [30, 31]. If the properties of RCA were improved, lesser amount of added cement would be needed. Even a small reduction in cement content is important for reduction of $CO_2$ emissions from concrete production. At the same time, a certain amount of $CO_2$ is permanently stored in concrete.

Generally, accelerated carbonation can be performed in two different environments: dry (gas-solid reaction) and wet (liquid-solid reaction) conditions. In the former case, the aggregates are exposed to constant $CO_2$ concentration or flow-through of $CO_2$ under ambient or increased temperature and pressure. In the latter case, aggregates are immersed in aqueous solution with constant flow of $CO_2$ injected into the solution. In dry conditions, the rate of carbonation depends on the amount of pore water in aggregate, while in wet conditions $CO_2$ is dissolved in the solution to form carbonate ions and carbonation reaction is not limited by $CO_2$ diffusion. For that reason, wet carbonation reaction is very fast under ambient pressure and temperature and can be effectively performed with gas that has low $CO_2$ concentration, like for instance flue gas from cement plants [32-35].

In gas-solid conditions, the efficiency of accelerated carbonation depends on the carbonation conditions and RCA properties. Carbonation conditions include $CO_2$ concentration and pressure, relative humidity and temperature of the environment, duration, and gas flow rate. As for properties, it depends on the water content, particle size and calcium content in RCA.

Based on extensive published research [33-43], recommendations regarding the most favorable range of mentioned influential factors are summarized in Table 1. Faster carbonation and higher $CO_2$ sequestration efficiency could be achieved by simultaneous adjustment of several parameters.

### 3.1 Coarse recycled concrete aggregates CRCA

Carbonation of CRCA leads to their properties improvement and therefore to better properties of the concrete made with CRCA. Carbonated CRCA have lower water absorption and higher density relative to non-carbonated. Figure 3 presents the improvement of water absorption due to carbonation of CRCA with particle size 5-20 mm.

*Table 1. Recommended accelerated carbonation conditions and RCA properties*

| Parameter | | Recommendation | Comment |
|---|---|---|---|
| Environmental | Relative humidity (RH) | 50%-70% | Lower or higher RH values both have adverse impacts on carbonation efficiency |
| | $CO_2$ concentration | 20%-50%; | Higher concentration doesn't increase the carbonation degree, but accelerates the process |
| | Gas pressure | Lower than 0.5 MPa | Higher pressure doesn't significantly impact the carbonation efficiency |
| | Gas flow rate | Less than 5 L/min | Can achieve satisfactory carbonation efficiency with low $CO_2$ flue gas (20%) |
| | Temperature | 20-30°C | Positive effect of higher temperature but depends on many parameters; mostly used 20-30°C due to economy and uncertain impact |
| | Duration | Depends on conditions and particle size | Carbonation degree increases with the carbonation duration but the fastest rate is in the first several hours |
| RCA properties | Water content | ~30% of water absorption | Easily affected by RH; water content and RH should be controlled together |
| | Particle size | Smaller particle size exhibits significantly higher carbonation degree | Due to much higher specific surface area and much higher residual mortar (cement paste) content |
| | Calcium content | Adding calcium compounds | Presoaking in $CaOH_2$ solutions, waste water from ready-mixed concrete plant, etc. |

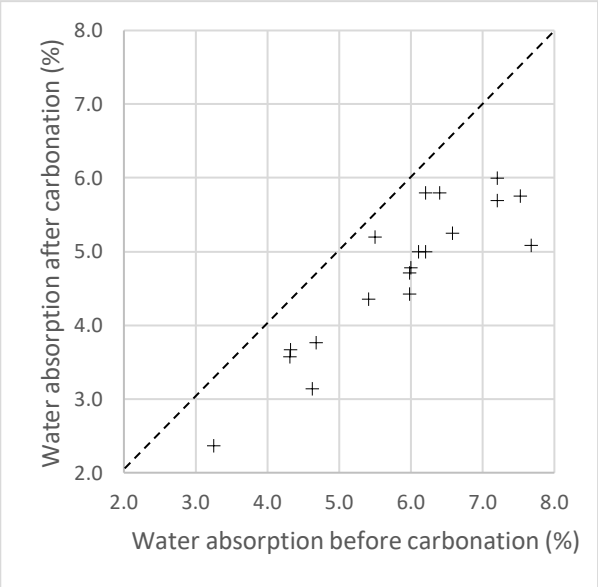


*Figure 3. Water absorption of CRCA (5-20 mm) before and after carbonation [43–54]*

Concrete made with carbonated CRCA shows lower loss of compressive strength compared to concrete made with non-carbonated CRCA. It can be controlled within 10% of the referent NAC compressive strength even with the complete substitution of coarse natural aggregate by carbonated CRCA. Referent NAC is concrete produced with same cement content and effective w/c ratio as RAC but with natural aggregates CNCA. Figure 4 shows the ratio between compressive strength at 28 days of RAC with carbonated CRCA ($f_{cm,RAC}$) and referent NAC ($f_{cm,NAC}$), based on test results from published research. Relatively large range of published results is due to different conditions applied in accelerated carbonation: 20-100% $CO_2$ concentration, pressure 0.1-4 bar and duration 30 min to 72 hours.

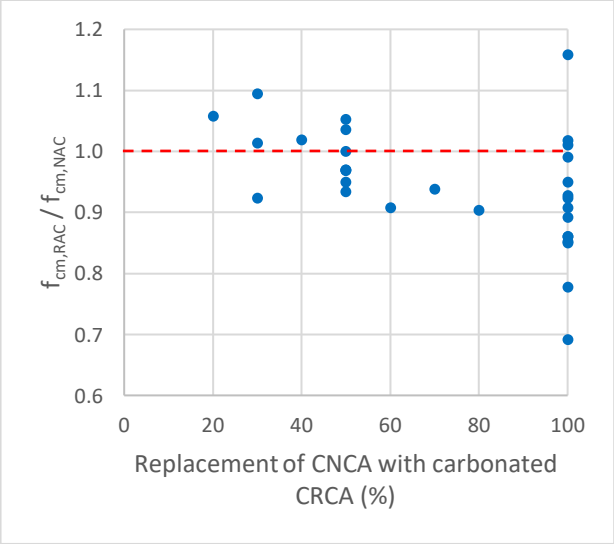


*Figure 4. Ratio between compressive strength at 28 days of RAC with carbonated CRCA ($f_{cm,RAC}$) and referent NAC ($f_{cm,NAC}$) [43, 44, 46, 47, 55-62]*

As for durability related properties, the results reported on the resistance to carbonation of RAC with carbonated CRCA are contradictory. Some researchers obtained lower [56], while some obtained similar or higher carbonation resistance [37, 50] compared to that of RAC with non-carbonated CRCA. The amount of cement paste in RAC, whether with carbonated or non-carbonated CRCA, is higher than in referent NAC concrete. This has two opposite effects: a higher permeability due to higher porosity and higher amount of $CaOH_2$. The former promotes carbonation; the latter hinders it. Finally, the carbonation resistance depends on which factor of these two prevails. Cement paste in RAC with carbonated CRCA has lower porosity but lesser amount of $CaOH_2$ due to carbonation, compared to RAC with non-carbonated CRCA. Therefore, resulting performance depends on many parameters and it cannot be easily predicted. On the other hand, resistance to chloride penetration of RAC with carbonated CRCA is always improved relative to RAC with non-carbonated CRCA, due to refinement of the pore structure and total porosity reduction [34, 44, 47]. Despite the fact that mechanical and durability related properties of RAC with carbonated CRCA are improved compared to RAC with non-carbonated CRCA, they are still on average inferior to referent NAC except for the low replacement ratios, up to 25%.

### 3.2 Fine recycled concrete aggregates FRCA

Since the beginning of the RCA application in concrete, the use of FRCA in structural concrete was not recommended [63]. Today, despite the high availability of experimental data, there is still no consensus on its effect on the concrete properties [64-67]. FRCA contains a large amount of weak and porous mortar, consisting of mostly hydrated cement paste and sand. Reported contents of cement paste in FRCA vary widely, from 18 to 70% [63, 68-70]. High water absorption of FRCA (4-16%) causes problems both with workability and strength of concrete. The FRCA's water uptake in concrete mix is uncertain, with reported values 49-89% of its water absorption [71-74], which makes the effective w/c ratio and therefore concrete performance hardly predictable.

When carbonated, FRCA absorbs much more $CO_2$ than CRCA due to higher specific surface area and higher amount of attached cement paste. The reduction of water absorption of FRCA after carbonation is even more pronounced then in the CRCA case; reported range in published research is 3-60%, Figure 5. Some researchers investigated the possibility of wet instead of dry carbonation [33, 35, 83]. As already mentioned, in wet carbonation FRCA is immersed in water with controlled $CO_2$ concentration or flow rate. Due to the more rapid dissolution of hydrates and the fact that $CO_2$ diffusion is not a limiting factor in this case, the carbonation rate can be significantly increased and carbonation efficiency enhanced. Liu et al. [33] reported similar water absorption reduction after 10 minutes of wet carbonation compared to 24 hours of pressurized dry carbonation. When wet and pressurized dry carbonation of the same duration of 10 minutes were compared, Fang et al. [35] reported a 5 times higher $CO_2$ sequestration in wet carbonation conditions. The reason is that the wet carbonation transformed a gas-solid reaction into a liquid-solid reaction and therefore enhanced the chemical reaction rate [84, 85].

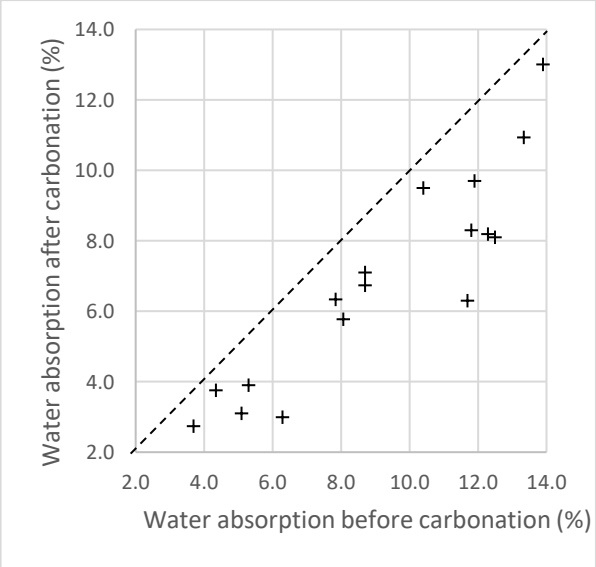


*Figure 5. Water absorption of FRCA (< 5 mm) before and after carbonation [35, 41, 53, 75-83]*

The negative effect of sand replacement with FRCA on concrete properties can be reduced if FRCA is pre-treated with accelerated carbonation. Moreover, recent research is focused on the full replacement of NA (both coarse and fine) with carbonated RCA.

As shown by Xiao et al. [48], with careful choice of water added to concrete mix to compensate for high water absorption values of FRCA, it was possible to produce concrete with full replacement of NA with carbonated CRCA and FRCA with similar compressive strength compared to referent NAC. In the case of RAC, the two-stage mixing method was followed [86] and the amount of additional water was set as 70% of the aggregate water absorption value minus the aggregate moisture content. Both CRCA and FRCA were obtained from real demolished concrete waste having high water absorption values, i.e. 7.67% and 13.34%, respectively. After carbonation, these values were reduced to 5.09% and 10.94%, which is still high compared to water absorption of natural aggregates (around 1%). Recycled aggregates were carbonated in chamber with a 20% $CO_2$ concentration and 75% relative humidity for at least 14 days (until full carbonation, which was proved by phenolphthalein test). Results of compressive strength testing showed that concrete mix with full replacement of NA with carbonated coarse and fine recycled aggregates had slightly higher compressive strength at 28 days, and slightly lower compressive strength at 180 days compared to referent NAC.

Liang et al. [76] also tested concrete mixes with full replacement of NA with carbonated and non-carbonated CRCA and FRCA. The objective was to monitor the improvement of RAC properties due to pre-treatment of recycled aggregates by accelerated carbonation. In this experiment, CRCA and FRCA were obtained by crushing and sieving the laboratory prepared mortar specimens with three different water/cement ratios equal to 0.3, 0.4 and 0.5. Recycled aggregates were carbonated in chamber with a 20% $CO_2$ concentration and 70% relative humidity for 10 days. Upon carbonation, water absorption of both CRCA and FRCA was reduced; for instance, from 12.3% to 8.2% and from 13.9% to 7.6% for FRCA and CRCA, respectively. It can be seen that both FRCA and CRCA had similar water absorption because they were produced from mortar samples and had similar amount of residual mortar. Both non-carbonated and carbonated recycled aggregates were pre-saturated with extra water equal to water absorption value until a saturated surface dry state was obtained and two-stage mixing method was applied. The concrete with carbonated CRCA and FRCA showed the highest compressive strength regardless of the quality of parent mortar. Negative impact of FRCA was therefore annulated by carbonation. Results cannot be compared to referent NAC mix since it was not tested. The resistance of RAC to carbonation was significantly improved by inclusion of the carbonated recycled aggregates. Carbonation depths were measured after accelerated carbonation of concrete samples at 20 ± 3% $CO_2$ concentration, T= 20 ± 2°C, RH=70 ± 5% for 56 days. Based on these results, authors calculated decrease of carbonation coefficient from 1.054 (RAC with non-carbonated RCA) to 0.63 (RAC with carbonated RCA) indicating higher resistance to carbonation. The pore structure refinement and improvement of ITZ properties prevailed over lower alkalinity of old cement paste in this experiment resulting in lower $CO_2$ diffusivity. Again, results cannot be compared to referent NAC mix.

Izoret et al. [57] reported results on the properties of concrete incorporating CRCA and FRCA carbonated in pilot installations implemented on a full scale in two industrial cement plants (rotating drum and fluidized bed). Authors tested several mixes with different replacement ratios of NA with carbonated and non-carbonated CRCA and FRCA with highest replacement ratio of 100% and 40% for coarse and fine aggregates, respectively. All mixes with carbonated aggregates, regardless of the replacement ratio, showed compressive strength at 28 days very similar to that of referent concrete, within 5% [57, 87]. Measurements of chloride diffusion coefficient showed no significant negative or positive impacts associated with the use of carbonated RCA compared to non-carbonated RCA; in both cases, this coefficient is higher than that of the referent concrete by 30-70%.

Chinzorigt et al. [88] tested mixes with 100% replacement of coarse NA with CRCA, and 15, 30 and 50% replacement of fine NA with carbonated and non-carbonated FRCA. Both CRCA and FRCA were obtained from commercial producer, while only FRCA was carbonated at 5% $CO_2$ concentration, RH 60±5%, T=20±2°C and ambient pressure for 72 hours. The loss of compressive strength at 28 days relative to referent concrete was about 10% for all replacement ratios of fine NA with carbonated FRCA. Measured carbonation depths under 56 days accelerated carbonation of all RAC were higher compared to referent concrete. Contrary to [76], in this experiment, concrete mixes with carbonated FRCA showed lower carbonation resistance relative to mixes with non-carbonated FRCA. Carbonation depth of concrete with 50% of carbonated FRCA was about 1.5 times that of referent concrete. The chloride penetration resistance was slightly lower for concrete with carbonated FRCA compared to concrete with non-carbonated FRCA despite lower porosity of the former (and both are lower than that of referent concrete). Possible explanation according to authors lies in the fact that chloride ions, which have been attached to the cement hydrate as Friedel's salt, are released into the pore solution during carbonation causing an increase of chloride ions concentration.

Ha et al. [89] compared mechanical and durability related properties of concrete with 100% replacement of both coarse and fine aggregates with carbonated CRCA and FRCA. Aggregates were carbonated for 4 and 14 days at 20% $CO_2$,

T= 20°C, RH=60% and ambient pressure. Measured compressive strengths at 28 days were practically same for RAC and referent NAC, i.e. full replacement of natural with carbonated recycled aggregates did not decrease the concrete compressive strength. Measured carbonation depths under 28 days accelerated carbonation were only slightly higher in concrete with carbonated RCA (9.2 mm compared to 8.6 mm in referent concrete). The same is valid for chloride diffusion coefficient: $1.47 \times 10^{-11}$ $m^2/s$ in concrete with carbonated RCA and $1.32 \times 10^{-11}$ $m^2/s$ in referent concrete.

Liang et al. [53] reported large improvement of resistance to chloride penetration of RAC with carbonated CRCA and FRCA compared to RAC with non-carbonated aggregates. Recycled aggregates were carbonated in chamber with a 20% $CO_2$ concentration and 70% relative humidity upon full carbonation. Authors tested mixes with full replacement of NA with carbonated and non-carbonated CRCA and FRCA and found decreased chloride permeability and steel corrosion risk in the former case (chloride diffusion coefficient reduced by 68%). Referent NAC was not tested.

*Figure 6. Industrial mineral carbonation plant (two reactor containers, $CO_2$ storage tank and centre for the process control) [90]*

Accelerated carbonation of CRCA and FRCA can be implemented at industrial scale. There are already several concrete plants in Switzerland using this approach and selling concrete containing industrially pre-carbonated recycled concrete aggregates [90], Figure 6.

### 3.3 Recycled concrete powder RCP

The use of recycled concrete powder in its raw form mainly causes the dilution of binder and therefore acts predominantly as filler [91, 92]. Recent research showed however, that recycled concrete powder, if carbonated, might be used as supplementary cementitious material [93, 94, 95]. Its activity as supplementary cementitious material (SCM) depends on the efficiency of separation process during recycling. If it is possible to separate the cement paste from sand and coarse aggregates, clean aggregates are obtained. On the other hand, (mostly) hydrated cement paste readily carbonates and full carbonation can be achieved in few hours under wet carbonation conditions [83-85, 96]. The material obtained in that way has high pozzolanic activity [97] and can be used as clinker replacement by 20% [98, 99] up to 40% [93]. Therefore, there are several benefits from technology – besides producing value-added products such as high quality aggregates and SCM, potentially high $CO_2$ reductions can be obtained through $CO_2$ sequestration as well as clinker amount reduction.

Technology is still at the “laboratory proof of concept” level, and no tests on concrete made with such binders were yet performed. However, some research on the level of paste and mortar properties is available [83, 94, 95]. The impact of partial replacement of ordinary Portland cement (OPC) with carbonated RCP on compressive strength of pastes and mortars, based on the published research, is shown in Figure 7. On average, replacement ratios of up to 20% are beneficial, while higher replacement ratios reduces the compressive strength of the blended paste, with the exception of the results reported in [94]. However, if the part of OPC is replaced with carbonated RCP in mortars, strength is always reduced, reduction being higher with higher replacement ratio.

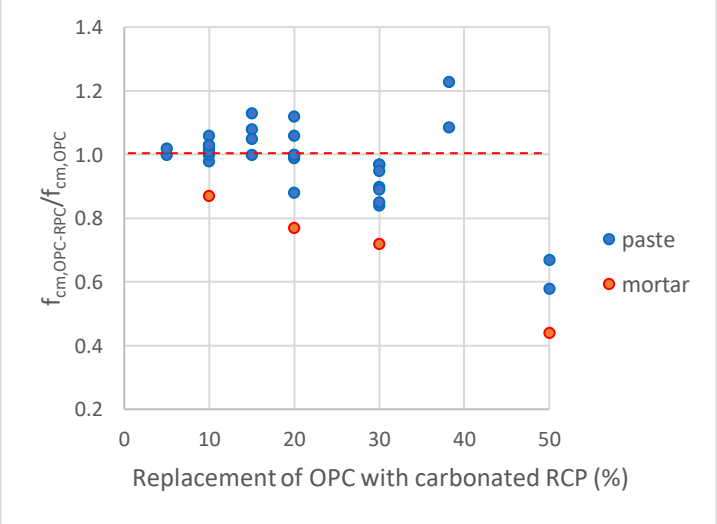


*Figure 7. Ratio between compressive strength at 28 days of paste and mortars with carbonated RCP ($f_{cm,OPC-RPC}$) and OPC paste and mortars ($f_{cm,OPC}$) [83, 94, 95, 98-102]*

As already mentioned, for efficient separation of recycled cement pastes from aggregates, advanced recycling technologies are necessary. Several are already developed. For instance, mechanical comminution with magnetic separation [103], microwave heating [104], advanced dry recovery ADR [105, 106], Heating-Air classification System HAS [107, 108], or pure mechanical comminution by adjusting the crushing force to an intermediate one between the average compressive strengths of the aggregates and the one of the hardened cement paste - Smart Crusher [109]. Both ADR and HAS are already industrially scaled-up mobile technologies and therefore can be used for on-site recycling of demolished concrete waste. ADR can successfully produce coarse (4-16 mm) and fine (0-4 mm) fractions from wet concrete waste by crushing and using air flow for separation, Figure 8. Fine fraction is further processed with HAS in which the input is heated to a temperature of up to 1410 ˚C while simultaneously separating very fine particles (0-0.25 mm) from the rest using air classification, Figure 9. Finely, the obtained 0.25-4 mm fraction is ground in a ball mill with steel balls to separate send from activated cement paste [106, 107].

Some of presented advanced recycling processes are already applied at industrial scale, like the Smart crusher and ADR technology in the Netherlands.

### 3.4 $CO_2$ sequestration potential of demolished concrete waste

The most significant parameter that influences the potential of $CO_2$ sequestration is particle size of the recycled aggregate. Under same carbonation conditions, FRCA absorbs much more $CO_2$ than CRCA, while RCP has the highest potential, Table 2.

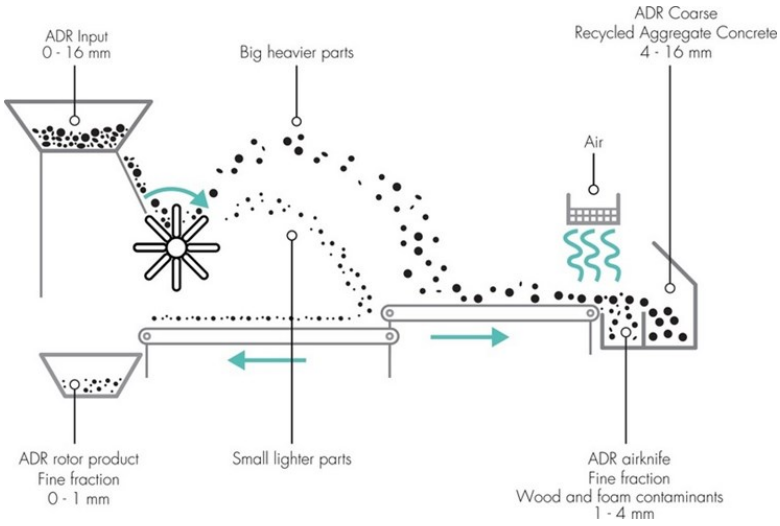


*Figure 8. ADR recycling system [110]*

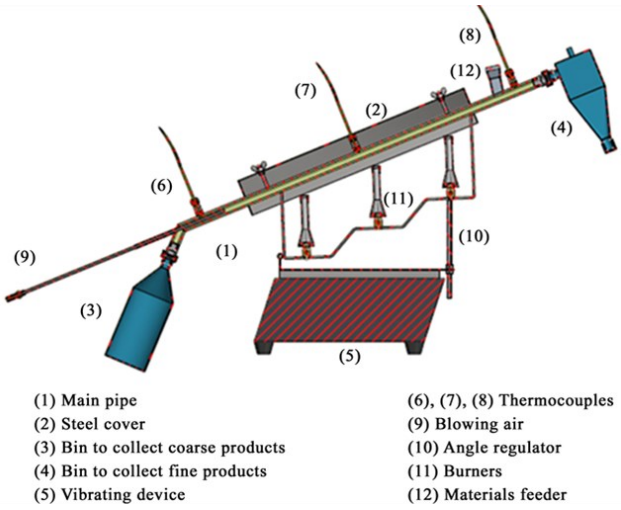


*Figure 9. HAC recycling system [107]*

*Table 2. $CO_2$ sequestration potential of demolished concrete waste of different particle size [42, 57, 61, 80, 111-114]*

| Particle size [mm] | Method | Conditions | Duration h (hour) | CO2 uptake [% of aggregate mass] | Ref |
|---|---|---|---|---|---|
| 5-10 | Gas-solid | $CO_2$ conc.=100%<br>RH=50%<br>T=25°C<br>Pressure =0.1/5 bar | 24 | 2.6/2.8 | [111] |
| 10-20 | Gas-solid | $CO_2$ conc.=15%<br>RH=NA<br>T=20°C<br>Pressure =1 bar | 24 | 0.65-0.96 | [80] |
| 5-10 | Gas-solid | $CO_2$ conc.=100%<br>RH=40-70%<br>T=NA<br>Pressure =0.1 bar/5bar | 24 | 0.74/0.81 | [61] |
| 10-20 | | $CO_2$ conc.=100%<br>RH=40-70%<br>T=NA<br>Pressure =0.1/5bar | 24 | 0.65/0.66 | |
| 4-16 | Gas-solid | $CO_2$ conc.=20%<br>RH=NA<br>T=20-30°C<br>Pressure =1 bar<br>Fluidized bed, industry | 1 | 1.2 | [57] |
| 4-8 | | $CO_2$ conc.=100%<br>RH=NA, 100% WA<br>T=NA<br>Pressure =1 bar | 1.1 | 0.15 | [42] |
| 1-4 | Gas-solid | $CO_2$ conc.=15%<br>RH=NA<br>T=20°C<br>Pressure =1 bar | 24 | 1.4/2.18/5.0 depending on the origin of the aggregate | [80] |
| 0-4 | Gas-solid | $CO_2$ conc.=20%<br>RH=NA<br>T=20-30°C<br>Pressure =1 bar<br>Fluidized bed, industry | 1 | 3.9 | [57] |
| 0-4 | Gas-solid | $CO_2$ conc.=100%<br>RH=NA, 100% WA<br>T=NA<br>Pressure =1 bar | 1.1 | 2.16 | [42] |
| Median diameter 0.0168/0.0962 | Liquid-solid | $CO_2$ conc.=14%<br>Solid/liquid=10g/L<br>T=20°C<br>Pressure =1 bar | 1.5 | 12.0/4.0 | [112] |
| <0.25 | Liquid-solid | $CO_2$ conc.=18.2%<br>Solid/liquid=10g/L<br>T=20°C<br>Pressure =10 bar | 0.17 | 5.0 | [113] |
| <0.5 | Liquid-solid | $CO_2$ conc.=18.2%<br>Solid/liquid=10g/L<br>T=18-25°C<br>Pressure =10 bar | 0.25 | 11 | [114] |

The results in Table 2 show high variability depending on the carbonation conditions and aggregate origin, whether from laboratory concrete and paste or real demolished concrete waste. Generally, aggregates from industry have lower $CO_2$ uptake capacity compared to those produced in laboratory conditions, for several reasons: specific operations at recycling plants, already partially carbonated in natural conditions, various impurities, etc. Under gas-solid conditions, results also significantly depend on the water content of recycled concrete aggregates, which mostly was not reported. The average value of $CO_2$ sequestration, based on this data, is 1.2%, 2.9%, and 8%, for particle size 5-20 mm, 0-4 mm and recycled concrete powder, respectively. If recycled cement paste is separated, much higher $CO_2$

sequestration can be obtained in liquid-solid carbonation, for instance 13.2% [115], 23% [97], 19.7-28.4% [102] (all at ambient temperature and pressure) and 28% (at elevated temperature of 80°C and pressure of 8 bar) [96]. However, these results were obtained on laboratory prepared samples (from pure cement pastes produced in laboratory), not on the hydrated cement pastes recycled in real industrial conditions.

## 4. Mineral carbonation of other industrial waste

Various types of industrial waste are rich in calcium and magnesium and therefore suitable for mineral $CO_2$ sequestration, like fly ash, steel slag, cement kiln dust, concrete slurry waste etc. Their $CO_2$ sequestration capacity, apart from carbonation conditions, depends mostly on the chemical composition, particle size and microstructure. A lot of research has been devoted to this subject. Carbonation under liquid-solid conditions, and at higher temperatures and pressures, was commonly used to accelerate the process [37].

**Fly ash** is product of combustion in coal-fired power plants. Globally, the annual production of fly ash is estimated between 0.75–1 billion tons [116]. Its chemical composition varies a lot, depending on the type of coal from which it was obtained. Fly ash produced by combustion of bituminous coal, sub- bituminous coal, and lignite typically contains 1-12%, 5-30%, and 15-40% of CaO, respectively. It is fine material with particles ranging between < 1 – 500 μm in size and having a tri-modal particle size distribution, the bulk of which is below 75 μm [116].

**Iron and steel slag** is by-product from iron and steel manufacturing. High amounts of this waste is produced each year globally: 330–390 million tons of iron slag and 190–290 million tons of steel slag in 2022 [117]. It is mainly classified into four different types: blast furnace (BF) slag, oxygen furnace (BOF) slag, electric arc furnace (EAF) slag and ladle furnace (LF) slag [118]. Chemical composition of various slags differs a lot depending on the iron/steel manufacturing process [119]. In the case of iron BF slag chemical composition and particle size depend also on whether it is produced with slow cooling (air-cooled slag) or with rapid cooling (granulated slag) [120]. In any case, slag must be further grinded for application in cement or concrete, which helps also in raising the efficiency of accelerated carbonation.

Some of the published results regarding FA and slags $CO_2$ uptake potential are summarized in Table 3.

*Table 3. $CO_2$ sequestration potential of demolished concrete waste of fly ash and different types of iron and steel slag [121-137]*

| Type of waste/ CaO content (%) | Particle size mm | Method | Conditions | CO2 uptake [% of aggregate mass] | Reference |
|---|---|---|---|---|---|
| FA 4.1 | Median diameter d=0.04 | Liquid-solid | $CO_2$ conc.=100% T=30°C Pressure =10 bar Liquid/solid=10 ml/g Duration=NA | 2.6 | [121] |
| FA 7.0 | Specific surface area 4.73$m^2$/g | Liquid-solid | $CO_2$ conc.=100% T=ambient Pressure =ambient Liquid/solid=5 ml/g Duration=24 h | 0.8 | [122] |
| FA 3.4 | NA | Liquid-solid | $CO_2$ conc.=100% T=30°C Pressure = NA Liquid/solid=15 ml/g Duration= 15-55 min | 3.2 | [123] |
| FA 34.1 | NA Petroleum coke ash | Liquid-solid | $CO_2$ conc.=100% T=60°C Pressure = ambient Liquid/solid=80 ml/g Duration=16.7 h | 21.0 | [124] |
| FA 30 | 0.002-0.01 | Liquid-solid | $CO_2$ conc.=100% T=60°C Pressure = 10 bar Liquid/solid=80 ml/g Duration=1 h | 26.4 | [125] |
| FA 31.9 | <0.0212 | Gas-solid | $CO_2$ conc.=100% T=45°C Pressure =18 bar Duration=135 min | 18.2 | [126] |
| FA 10-15 | 0.002-0.006 | Gas-solid | $CO_2$ conc.=100% T=160°C Pressure =8 bar Duration=4 h | 2.3-5.3 | [127] |

| | | | | | |
|---|---|---|---|---|---|
| BF 36.6 | <0.075 | Liquid-solid | $CO_2$ conc.=100% T=150°C Pressure =30 bar Liquid/solid=10 Duration=24h | 28 | [128] |
| BF 22.5 | <0.075 | Indirect | $CO_2$ conc.=98% T=30°C Liquid/solid=10 Duration=60 min ($NH_4NO_3$, $NH_4Cl$, $CH_3COONH_4$) | 12.2 | [129] |
| BF 47.2 | <0.075 | Indirect | T=25°C Pressure =1 bar Liquid/solid=10 Duration=60 min ($CH_3COOH$ + EDTA) | 9.0 | [130] |
| BOF 51.1 | <0.044 | Liquid-solid | $CO_2$ conc.=100% T=60°C Pressure =1 bar Liquid/solid=10 ml/g Duration=1 h | 27.0 | [131] |
| BOF 41.2 | <0.044 | Liquid-solid | $CO_2$ conc.=100% T=25°C Pressure =1 bar Liquid/solid=20 ml/g Duration=2 h | 28.3 | [132] |
| BOF 46.4 | <0.125 | Liquid-solid | $CO_2$ conc.=30% T=30°C Pressure =1 bar Liquid/solid=20 ml/g Duration=20 min | 27.7 | [133] |
| BOF 31.1 | 0.063-0.1 | Liquid-solid | $CO_2$ conc.=40% T=50°C Pressure =5 bar Liquid/solid=5 L/kg Duration=4 h | 46.5 | [134] |
| BOF 29.9 | <0.15 | Liquid-solid | $CO_2$ conc.=100% T=100°C Pressure =10.0 MPa Liquid/solid=5 L/kg Duration=24 h | 32.5 | [135] |
| EAF 33.2 | <0.024 | Liquid-solid | $CO_2$ conc.=18.2% T=25°C Pressure =10.68 bar Liquid/solid=10 ml/g Duration=10 min | 5.2 | [136] |
| EAF 28.3 | <2.0 | Liquid-solid | $CO_2$ conc.=100% T=25°C Pressure =6 bar Liquid/solid=10 ml/g Duration=3 h | 8.2 | [137] |
| EAF 49.3 | <0.15 | Liquid-solid | $CO_2$ conc.=100% T=100°C Pressure =10.0 MPa Liquid/solid=5 L/kg Duration=24 h | 28 | [135] |

Note: BF – blast furnace slag; BOF – basic oxygen furnace slag; EAF – electric arc furnace slag

$CO_2$ sequestration potential of fly ash depends on the CaO content: low calcium FA has much lower $CO_2$ sequestration potential (0.8-3.2%) compared to high-calcium FA (18.2-26.4%). The same is valid for slags, but beside chemical composition, particle size and carbonation conditions, particularly temperature, are important factors. The smaller the particle size, the higher the $CO_2$ sequestration potential due to larger specific surface area. Temperature range between 50°C and 80°C seems to be optimal providing maximal $CO_2$ sequestration capacity [37].

**Concrete slurry waste** (CSW) is result of returned fresh concrete (whether over-ordered or leftovers) which is washed away from truck mixer with water. After washing, aggregates are separated from the slurry waste, which consists of very fine aggregate and cement particles and water. Usually, it is stored in sedimentation tank for a certain period to allow solids to settle and then disposed of in landfills [138]. The waste slurry can be used in wet or hardened state in various applications. If it is in hardened state, it has to be grinded and sieved before any application. The amount of produced slurry wastes is significant, up to 4.0% of freshly mixed concrete annually [25]. It is rich in CaO and can contain between 32% and 53% of CaO by mass [138-140]. Xuan et al. [138] performed a gas-solid carbonation of CSW crushed to a particles <5 mm and pre-treated to reduce the water content from 50% to 10%. Measured $CO_2$ sequestration after 144 hours in chamber with 100% $CO_2$ concentration and 0.1 bar pressure was 11%, or 110 g/kg of dry SCW. Kaliyavaradhan et al. [140] tested several carbonation conditions regarding the water-to-solid ratio (0.1, 0.25, 0.4, 0.55, 0.7) and duration time (1, 24, 72, 120, 168 hours) under same conditions: 20% $CO_2$ concentration, temperature 20°C and 65% RH. Dried CSW was ground and sieved to pass through 0.3 mm. The authors found that maximum $CO_2$ sequestration was obtained at water-to-solid ratio of 0.25 and duration of 72 h, and it was equal to 20.4 % (204.35g/kg CSW).

**Cement kiln dust** (CKD) is generated during the clinker production process in rotary kilns. Being a potentially hazardous waste, it is collected by air-pollution control devices. It is a fine-grained solid material, which consists of uncalcined and partially calcined raw feed, and kiln dust. Cement industry produces 30 million tons of CKD annually [141]. Typical content of CaO is between 38% and 50% [141-143] by mass. In liquid-solid carbonation tests (100% $CO_2$ concentration, 3 bar pressure, T=25°C and liquid-to-solid ratio=5) Medas et al. [141] measured $CO_2$ sequestration of 11.68% (116.8 g/kg of CKD) after 5h. Huntzinger et al. [144] measured $CO_2$ sequestration of 7.7% (77.0 g/kg of CKD) under ambient temperature and pressure accelerated carbonation of CKD from landfill without any pre-treatment.

Figure 10 presents the carbonation efficiency of waste materials obtained from referred published research described in previous section. It can be seen that iron and steel slags, CKD and CSW have comparable potential for mineral $CO_2$ sequestration (10%-30%) and that it depends not only on the CaO content, but also on the accelerated carbonation conditions and particle size. High-calcium FA (over 20% CaO by mass) belongs to that group, while low-calcium FA (below 10% CaO by mass) has small carbonation capacity.

### 4.1 Application

Generally these wastes, similarly to demolished concrete waste, can be applied as aggregate substitutes, Portland cement substitutes (SCM) or as fillers. If pre-treated with accelerated carbonation, their properties are improved and certain amount of $CO_2$ is sequestered, which both can result in the reduction of $CO_2$ emissions in the concrete life cycle.

#### As aggregates

Carbonated iron and steel slag can be used as a substitute for natural fine and coarse aggregates in mortars and concrete. Beside the significant $CO_2$ sequestration potential, accelerated carbonation pre-treatment increases the volume stability, reduces the leaching of heavy metals and improves slag mechanical properties [145-147]. Pang et al. [148, 149] tested the impact of partial replacement of fine and coarse natural aggregate with carbonated BOF slag on concrete properties, keeping same w/c and cement content in the concrete mix. The authors reported similar workability despite higher WA, 20% higher compressive strength at 28 days, improved ITZ zone properties, significantly higher freeze-thaw resistance (2-3 times) compared to reference samples with only natural aggregates. However, production of carbonated fine and coarse slag aggregates included grinding, pelletization, carbonation at 70°C and pressure of 3 bar, and crushing again, which all required a significant energy. Bodor et al. [150] tested the properties of cement mortar with 50% of natural sand aggregate replaced by carbonated BOF slag. Carbonation was conducted in liquid-

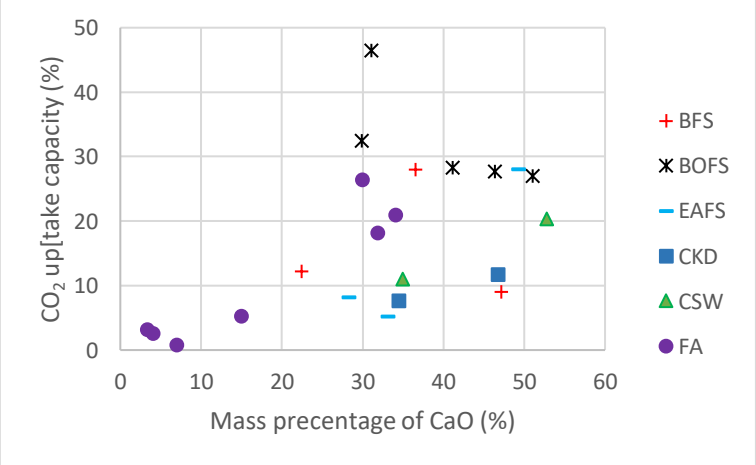


*Figure 10. $CO_2$ sequestration potential of FA, iron and steel slags, CSW and CKD [121-135, 138, 140, 141, 144]*

solid conditions at 90 °C and 20 bar $CO_2$ pressure, for 2 h duration. Mortar mixtures prepared with carbonated BOF slag of <0.5 mm particle size showed similar consistency via flow table test, compressive strength and leaching of toxic elements to referent mortar mixture with only natural sand. In this case, only treatment of slag prior to carbonation was crushing and sieving, while $CO_2$ sequestration equal to 11-19% was obtained.

Several other works showed the applicability of carbonated steel slags as aggregates for concrete: artificial aggregates obtained by wet granulation process of BOF powder [151] or a combination of slag, fly ash and cement [152]. Shen at al. [153] used original slag as coarse aggregate and ball-milled original slag as powder in production of carbonated steel slag pervious concrete. Although commonly tested concrete property – compressive strength - was obtained similar to referent concrete prepared with natural aggregate, it should be kept in mind that significant pre-treatment operations can annul (cancel) the benefits from sequestered $CO_2$ and waste reduction [154].

As SCM

Ground granulated BF slag (GGBS) and FA are already used as clinker replacement in commercial cements. Research on utilization of carbonated slag and fly ash as SCM is limited so far. Ebrahimi et al. [123] and Pei et al. [155] reported that up to 10% replacement of clinker with carbonated FA did not impair the compressive strength of blended cement pastes. Sahoo et al. [156] investigated concrete with 25% cement replacement with carbonated fly ash and reported high resistance against salt, sulfate and acid attack. Regarding carbonated slag as SCM, Chen et al. [157] and Pan et al. [158] reported similar results – up to 10% replacement with carbonated slags provided similar compressive strengths to pure OPC pastes and mortars, while maintaining high volume stability. The volume stability of cementitious materials containing slag is usually a problem, but carbonation consumes the expansion phase in slag (CaO) and improves it effectively. For both fly ash and slag, the main result of carbonation reaction is fine calcite ($CaCO_3$) which fills the pores and reduces pastes and mortars porosity, or becomes nucleus for hydration contributing to strength gain (setting time decrease) of cement pastes and mortars [155, 157, 159].

Utilization of ground but non-carbonated concrete slurry waste and cement kiln dust as SCMs was investigated in the previous research. Replacing clinker with CSW and CKD reduces mechanical and durability related properties of mortars and concretes, and general conclusion is that replacement should be limited to 10% to achieve similar strengths and durability of OPC mortars and concretes [28, 160]. Carbonated CSW and CKD should however improve the mortar and concrete performance due to same reasons as in the case of carbonated FA and slag. Kaliyavaradhan et al. [140] for instance, reported higher strength of mortar prepared with carbonated CSW compared to mortar prepared with fresh CSW, at 20% cement replacement ratio. As concluded by the authors, strength improvement was probably result of the reaction of aluminate C3A and calcite to form calcium carboaluminate hydrate. However, it was still slightly lower compared to pure OPC mortar. Sharma et al. [143] investigated carbonation curing of mortars containing CKD, while Xuan et al. [138] studied the carbonation curing of partition wall blocks made of CSW and fine recycled concrete aggregates. In these cases, accelerated carbonation was applied on the final product resulting in the improvement of their performance.

## 5. Conclusion

Mineral carbonation of alkaline industrial waste is a way to permanently store $CO_2$ in cement-based materials. On the other hand, pre-treatment with accelerated carbonation improves properties of various wastes enabling in that way their application as substitutes for natural aggregates and OPC.

Accelerated carbonation of coarse and fine aggregates obtained by demolished concrete waste leads to decrease of their porosity due to formation of calcite, which fill in the pores and densify the microstructure. Most of the experimental research showed that full replacement of natural aggregates with carbonated ones didn't jeopardize the compressive strength of RAC, meaning that there is no need to increase the cement content to obtain strength similar to that of the referent NAC. As for carbonation and chloride penetration resistance of RAC with carbonated aggregates, different results were reported indicating that those concretes, despite lower permeability, may have lower resistances compared to referent NAC. This may be because of lower alkalinity of their pastes in the carbonation resistance case, or due to increased chloride concentration as a consequence of previous carbonation of aggregates in the chloride penetration resistance case. $CO_2$ sequestration potential depends mostly on the particle size and water content of aggregates under same carbonation conditions: FRCA can absorb up to 5% of their weight depending on the water content, while $CO_2$ uptake potential of CRCA is modest, 1-2% of their weight. Recycled concrete powder has much higher $CO_2$ sequestration potential ranging from 5-11% and in the case of pure recycled cement paste reaches 30% of the weight. Carbonated RCP has good pozzolanic activity and can be used as SCM. In the amounts of up to 20% it even increases the strength of paste, while for higher replacement ratios, the strength is decreased.

Similar to demolished concrete waste, the properties of fly ash, slags, cement kiln dust and concrete slurry waste are improved when pre-treated with accelerated carbonation, including higher volume stability and lower leaching of heavy metals. Except for low-calcium FA, these wastes have high $CO_2$ sequestration potential, between 10% and 30% of their weight, especially iron and steel slags. Carbonated slags can be used as substitute for aggregates, and when ground into powder they can be used as SCM, as well as carbonated FA, CKD and CSW. However, research on the impact of such aggregates and binders on the concrete performance doesn't exist at the moment. The same is valid for the carbonated RCP utilization as partial replacement of OPC in concrete.

Despite the obvious benefits of the mineral carbonation technology, one should kept in mind that these benefits could easily be cancelled by required energy and $CO_2$ emissions during process. Beside the accelerated carbonation itself (energy requirement depends on the applied pressure and temperature), some pre-treatment processes are energy intensive, like drying of recycled aggregates for instance. Wet aggregates are common industrial condition and such high water content practically disables carbonation – so, they must be dried. Preparation of slags for application as aggregates or SCM include various pre-treatments beside accelerated carbonation. Therefore, for proper conclusions on environmental benefits of mineral carbonation application in cement-based materials, an LCA (Life Cycle Assessment) which includes all the phases of the life cycle must be performed.

Regarding $CO_2$ sequestration potential, most promising candidates for mineral carbonation are recycled concrete powders, iron and steel slags, and high-calcium fly ash. The future research should be oriented towards better understanding of the impact of their application as aggregates or SCM in cement based materials, concrete most of all.

**CRediT authorship contribution statement**

Snežana Marinković: Conceptualization; Methodology; Data curation; Formal analysis; Writing.

**Declaration of competing interest**

Author declares no conflict of interest.

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