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Total Recycling of Concrete Waste Using Accelerated Carbonation: A Review

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Abstract

Our world is facing critical environmental challenges due to the overexploitation of natural resources and the extensive release of CO_2. Meanwhile, huge amounts of concrete waste are generated due to accelerated urbanization and redevelopment. Carbonated concrete waste can thus both be used as alternative materials to aggregates and cement, and a carbon sink to reduce CO_2 emission. In this paper, different carbonation approaches and their modified techniques to treat recycled coarse aggregates (RCAs), recycled concrete fines (RCFs), and recycled concrete powders (RCPs) are critically reviewed and discussed. The results suggest that carbonation effectively strengthens RCAs by densifying microstructures and improving physical properties through the precipitation of calcium carbonate (Cc) that fills the pores. Meanwhile, the carbonation reaction also develops surface reactivities on the recycled aggregates due to silica gel and Cc formation, which is particularly beneficial for RCFs because of the high surface area. Re-structuring and re-activation of RCPs through carbonation transforms the waste hydrated cement powders into novel supplementary cementitious materials. In addition to wet carbonation, which has a higher efficiency than dry carbonation, other modified carbonation techniques with the addition of limewater, magnesium ions, ammonium ions, and the use of elevated temperatures are found to produce RCAs/RCFs with better qualities, and facilitate the formation of other high-value products such as nano-silica gel, micro-fiber, and vaterite, etc. Apart from their practical uses in conventional concrete, potential applications of the carbonation products in ultra-high-performance concrete, automated construction, and self-cleaning concrete were also briefly introduced. More importantly, the key issues in upscaling the carbonation technologies and limitations in current research are pointed out. Overall, carbonation could not only facilitate the re-circulating of concrete waste but also help reduce CO_2 emissions and help achieving carbon neutrality by permanently sequestrating CO_2.

Keywords: Recycled concrete aggregates; Recycled concrete fines; Recycled concrete powders; Accelerated carbonation

1 Introduction

Cement and concrete manufacturing relies heavily on natural resources and emits an extensive amount of CO_2 [1]. Re-circulating cement- and concrete-based products back to the construction industry, at the end of their service life, is envisioned as one of the ideal strategies to address the resources and carbon emission crises encountered by the industry. According to official reports from different economic entities [2-6], billions of tons of waste concrete are entering the construction and demolition (C&D) waste stream per year on a global basis, and the amount is still rapidly increasing due to the continuous urbanization in redeveloping activities in many countries. Even though available data featured high geographical variations, it is estimated that 50-90% of the waste concrete is recycled as aggregates [6]. Two size fractions are generally included, namely the coarse fraction above 5mm (recycled coarse aggregate, RCAs) and the fine fraction below 5mm (recycled concrete fines, RCFs). Nevertheless, instead of being reintroduced into concrete production as substitutions of gravels and sand, they have been more practically “down-cycled” as filling materials, e.g., road constructions. The key reason responsible for the situation is widely known as the inferiority of these materials to their natural counterparts due to the higher porosity either inherent in the residual hydrated cement paste or created during crushing [7]. It was also acknowledged that the multiple interfacial transition zones (ITZs) that present among the residual cement paste, virgin aggregate, and new cement paste jointly result in the poor performance of the new concrete. RCFs, owing to the finer particle size and higher amount of residual cement paste, have more defects than RCAs. These defects would particularly worsen the workability, drying shrinkage, and transport properties of concrete. For these reasons, RCAs and RCFs were not widely used in the production of structural concrete, unless they are incorporated at very low dosages, or an extra amount of cement is added for compensation [8-10].

Alongside the interest in recycling RCAs and RCFs, awareness of re-utilizing recycled concrete powders (RCPs) (<0.15mm) has also risen over recent years. The review of the literature has revealed that RCPs contain an even higher amount of residual cement paste than RCFs and are thereby closer to the nature of hydrated cement pastes [11]. Therefore, RCPs, in their raw form, have been utilized in new concrete production as a replacement to cement [12, 13]. However, due to the high porosity of the RCP particles generated during crushing and grinding as well as the extensive amount of gel pores embedded in the hydrated cement paste, the composite binder with RCPs has a high water demand and poor workability at the fresh state [13, 14]. Besides, it also has poor mechanical and durability performance due to the loss of reactivity after hydration. For example, it was reported that a 30% replacement of cement by RCPs could cause a 31% reduction in the 28d-compressive strength [11, 15]. Even though many other applications reported attempts to maximize the recycling value of RCPs (e.g., in foam concrete [16], ultra-high performance concrete [17], and alkali-activated concrete [18]), RCPs are mostly used only as inert fillers.

Intensive research into the carbonation of waste concrete has been carried out over the last decade. As a result, promising results regarding the strengthening of RCAs/RCFs and the re-activation of RCPs were reported [15, 19, 20]. Such a technique is theoretically based on the strong capacity of residual hydrated cement paste to bind CO_2 and form new carbonation products. Moreover, it has been estimated that carbonating RCAs, RCFs, and RCPs exhibit an exceptional capacity for carbon capture that could permanently stabilizes CO_2 by 7.9 kg/tonne, 20 kg/tonne and 190 kg/tonne, respectively [21, 22]. That may be more effective and economical than some of the carbon capture, utilization and storage technologies, such as chemical absorption (15-54 kg/tonne). More importantly, the application of carbonated products back to the concrete industry could reduce CO_2 emissions of concrete manufacturing by over 9% [23].

To provide viable total recycling of waste concrete methods, advances in the carbonation of waste concrete over the last five years are reviewed in this paper. A primary attempt made herein is to highlight the strategic differences of applying the carbonation technique to RCAs, RCFs, and RCPs for their effective uses in the construction industry that can more practically bridge the gap between research and practice.

2 Carbonation on RCAs

2.1 Carbonation strategy and properties of RCAs

2.1.1 Carbonation strategy and improvement

While natural aggregates are almost inert to carbonation, the residual cement paste attached to RCAs is highly CO_2-reactive. Thermodynamic modelling on the phase assemblage and the corresponding solid volume evolution have predicted that carbonating residual cement paste first consumes portlandite and then decalcifies the high-Ca C-S-H. The two processes are characterized by a significant Cc precipitation of over 40 vol% that marks an estimated increase of the overall solid volume by around 3% [24, 25] (see Fig. 1a). Cc of different polymorphs may precipitate upon carbonating various cement hydrates at different carbonation conditions (e.g., relative humidity, carbonation rate, temperature, etc.). In contrast, the effect of Cc polymorphs on the final solid volume change is negligible due to the limited differences in the molar volume of calcite, aragonite, and vaterite [26]. The strengthening of RCAs by carbonation is mainly due to the advantage of this volume increase to fill the pores and microcracks (see Fig. 1b) and thus improve the qualities of RCAs. However, excessive carbonation of the hydration products including partial decalcification of C-S-H and ettringite may result in adverse effects.

It has been observed in some studies that the amount of portlandite in RCAs may be significantly reduced due to natural carbonation during the service life of the parent concrete and after crushing the waste concrete for recycling. For this reason, providing an additional calcium source is a feasible method to enhance Cc precipitation and, thus, the microstructure and properties of RCAs [27]. Specifically, using wastewater from concrete batching plants or prefabrication plants as a calcium source could be an economical and green choice, as previously investigated by some studies [28, 29]. Moreover, as a further response to addressing CO_2 emissions, CO_2-rich flue gas emitted by cement and steel-making plants has also been proposed to be used as a key and green strategy for the carbonation of RCAs.

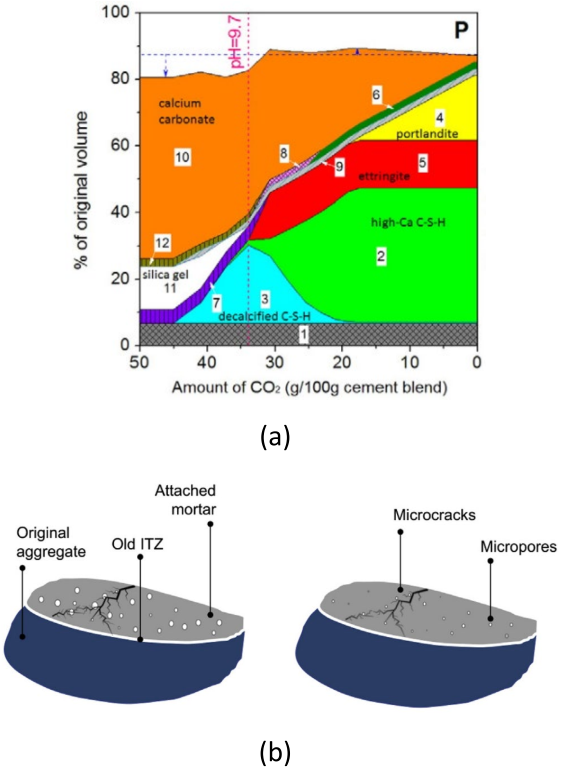


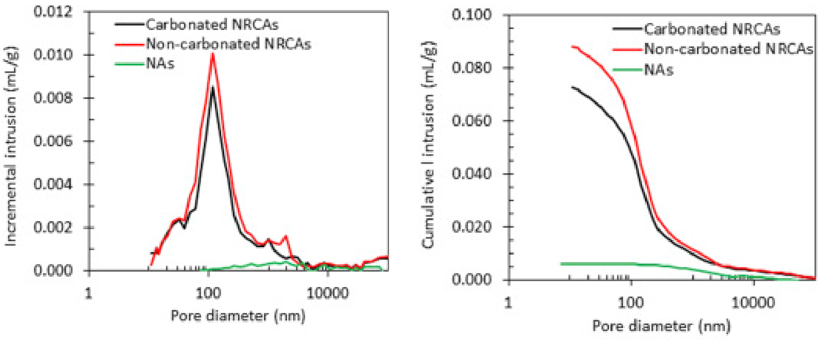
Fig. 1. (a) Changes in phase assemblage and total solid volume with the increasing carbonation (per gram of CO_2 sequestrated) [24] and (b) illustration for the filling of pores and microcracks by carbonation [30].

2.1.2 Properties of carbonated RCAs

Previous experimental studies showed that the microstructure of RCAs could be significantly refined through carbonation; it was reflected by a reduction of the amount of capillary pores as well as the total porosity by up to ~30% [7, 27] (see Fig. 2). The water absorption is the most critical physical property associated with the microstructures. It was found that the water absorption, as summarized from published data (see Fig. 3), decreased markedly by approximately 5-45% after carbonation. Furthermore, evaluation of other transport properties of RCAs demonstrated that the electrical conductivity and the chloride permeability, etc. can also be reduced by up to ~70% after carbonation for 7 days [30].

It should be highlighted that the reduction of porosity and the densification of the microstructure not only occurred in the main old paste matrix, but also in the old ITZs of RCAs [31, 32]. This improvement of old ITZs could homogenise the aggregate and thus mitigate the possibility of internal failure initiated from this region. The massive Cc precipitation could also provide mechanical benefits because Cc had a much higher elastic modulus and hardness than portlandite and C-S-H [33-35]. Accordingly, the carbonated RCAs were also found to perform better in terms of mechanical properties such as having higher microhardness values and lower crushing values, etc. [21, 36, 37].

In addition, it was also noted that pre-soaking the RCAs in waste limewater to provide additional portlandite was more beneficial than carbonating RCAs alone; it was noticed that the porosity and the corresponding water absorption of RCAs could be further decreased by 30% while the crushing value could be reduced by up to 28% [27, 28, 38]. RCAs of different origins, strength grades, and sizes were evaluated [39, 40] and it was found that the improvement by carbonation was independent of these factors, despite that the RCAs sourced from high-performance concrete which naturally had a lower initial porosity might experience less improvement after carbonation [41].



(a) (b)

Fig. 2. (a) Incremental and (b) cumulative pore volume of the RCAs as determined by Mercury Intrusion Porosimetry [42].

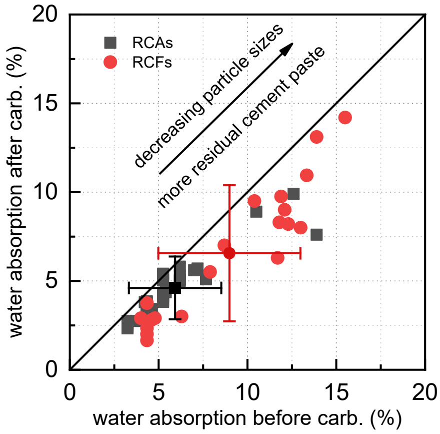


Fig. 3. Water absorption of RCAs and RCFs after carbonation treatment (data for RCAs was extracted from [7, 29, 32, 36, 37, 39, 43-47], while that for RCFs was from [38, 39, 41, 46-53].

2.2 Application in recycled aggregate concrete

2.2.1 Physical, mechanical, and durability properties

Research progress on RCAs over recent years focused predominantly on their use in concrete as coarse aggregates. It was found that the reduction in water absorption of RCAs could increase the flowability of new concrete mixtures and reduce slump loss over time, etc. compared to the use of non-carbonated RCAs [43, 45]. Also, the loss of concrete compressive strength by using carbonated RCAs could be significantly reduced and generally controlled within 10% even with the complete substitution of coarse aggregate by RCAs [44, 54]. The stress-strain curves of the concrete made with carbonated RCAs indicated that the peak stress was increased and the peak strain was decreased, indicating a higher elastic modulus that was closer to the normal concrete [43].

By comparing the results of physical/mechanical properties with durability performance, the carbonation treatment on RCAs has greater beneficial effects on durability. Generally, the permeability to water, gases, and aggressive ions all decreased. As a result, the electrical resistivity could be reduced by 15%, the drying shrinkage could be decreased by up to 25%, and the resistances to chloride and water penetration could be reduced by 36%, etc. [42, 44, 47]. Moreover, due to the high water absorption capacity of carbonated RCAs relative to natural aggregate, they could also serve as moisture reservoirs that facilitate the internal curing of concrete. The pre-saturated RCAs have sufficient water desorption capacity in concrete that gradually release water to favour additional cement hydration and thus counteract the adverse effects of drying shrinkage etc, of the hardened concrete [55, 56]. Furthermore, the use of carbonated RCAs in RAC of different design strengths was also considered. It was found to be compatible even with ultra-high-performance concrete [32, 45], which significantly extended the application scopes.

2.2.2 Mechanisms for the improvements

The improvement of RAC was primarily attributed to the densification of RCAs and the resulting improved physical and mechanical properties. The improvement (lower porosity, stronger old ITZs, and higher strength) of RCAs could provide strong support for the RAC to resist external load and foreign material attacks. Moreover, the carbonated RCAs could not only decrease the total porosity of RAC by 5-16% (see Fig. 4), but also lead to the blocking and tortuosity of pores in RAC, thus contributing to the improvement of the durability performance.

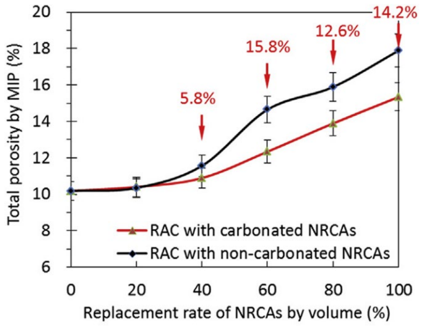
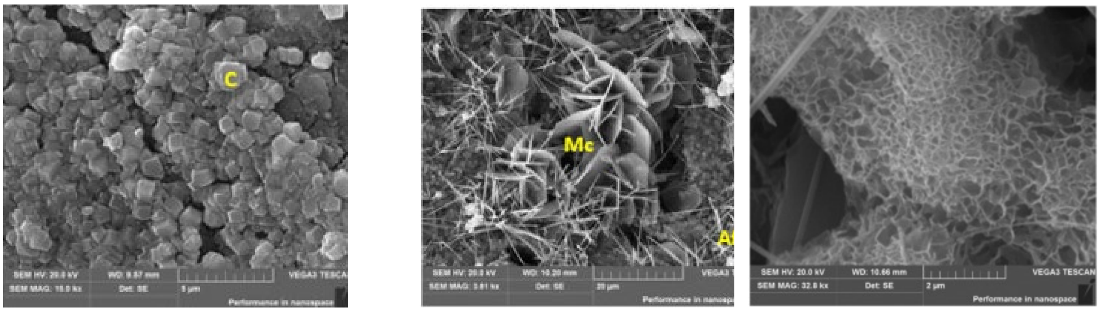


Fig. 4. Total porosity of RAC with non-carbonated and carbonated RCAs [42].

Additionally, it was pointed out by some studies [40, 57] that the Cc precipitated in RCAs not only exhibited physical benefits in terms of filling the pores, but also participated in the cement hydration as per Eq. (1) [15]. The formation of monocarbonate could preserve some sulphate and thus stabilize the ettringite. Besides, the Cc formed on the surfaces of RCAs could act as nucleation sites and thus promote the growth of C-S-H. These two beneficial effects were validated by the SEM images in Fig. 5. It was found that the porosity of the new ITZs (i.e., the region between RCAs and new cement paste) was reduced by ~12% as determined by image analysis. The bonding strength of RCAs with the new cement paste was increased by nearly 90% [40]. Based on micro-mechanical tests, strong linear relationships existed between the modulus and hardness of new ITZs with the mechanical and durability performance of RAC [32]. In summary, the cause of the RAC improvement for RAC was due to the densified RCAs as well as the better ITZs quality.

$$C_3A + Cc + 11\ H \rightarrow C_4AcH_{11} \qquad (1)$$



(a) (b) (c)

Fig. 5. Images of (a) carbonated RCAs surfaces (full of rhombohedral calcite), (b) the formation of monocarbonate (Mc), and (c) the preferential growth of C-S-H on Cc surfaces obtained with Scanning Electron Microscopy.

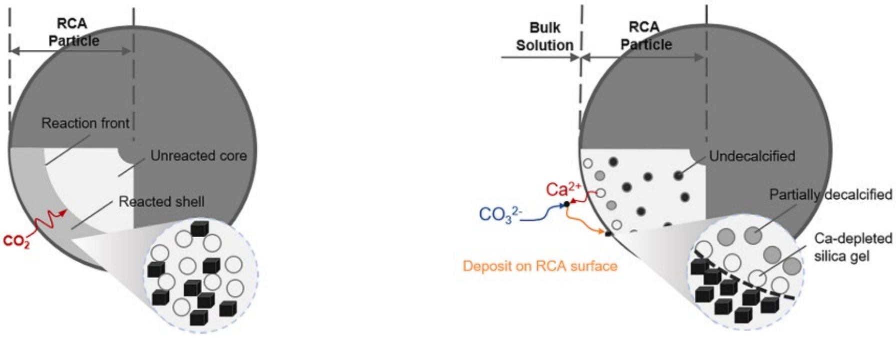
3 Carbonation on RCFs

3.1 Carbonation strategy and properties of carbonated RCFs

3.1.1 Direct carbonation

RCFs, due to the finer particle size and higher amount of residual cement paste, exhibited higher reactivity with CO_2 than RCAs [41, 58]. The carbonation on RCFs thereby could provide more pronounced improvements. The recent experimental results in terms of water absorption for RCFs are also summarized in the same figure with RCAs, as seen in Fig. 3. Raw RCFs were found to have higher water absorption than RCAs. Still, it varied depending on the amount of cement paste present i.e., from as low as 4% for real RCFs derived from a high-performance parent concrete [41] to a max. of 16% for simulated RCFs entirely made of hydrated cement pastes [48]. The reduction of water absorption for the RCFs after carbonation ranged from ~3 to ~60%, which was more significant than RCAs due to the higher amount of Cc formed during carbonation. However, RCFs possessed higher surface areas and thus were more prone to natural carbonation during their exposure to the open air after crushing. To reduce the potential adverse effect of over-carbonation, using limewater to treat the RCFs prior to carbonation was more meaningful than for RCAs [28, 38]. In this manner, the porosity of RCFs after carbonation could be further decreased by ~36% and the corresponding water absorption decreased by ~21% [28]. Additionally, wet carbonation was found to have a higher efficiency than dry carbonation. The RCFs carbonated with the wet carbonation method for 6h could achieve similar water absorption and density values to those carbonated by the dry carbonation method for 24h [59].

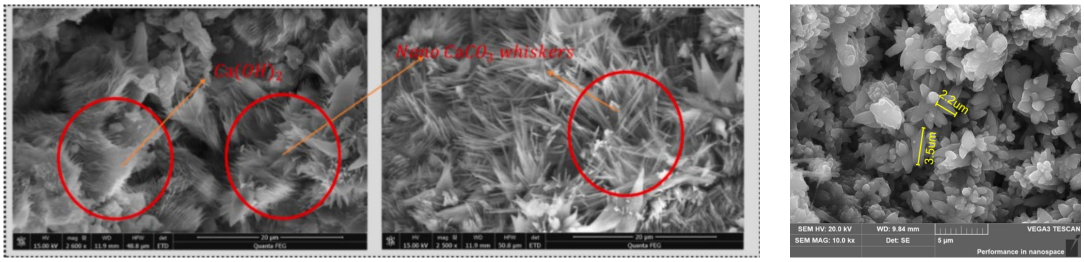
Apart from densifying the microstructure of RCFs by precipitating more Cc, a growing body of literature also demonstrated the significance of silica gel formation with carbonation. The amorphous silica gel generated by carbonation could react with portlandite to form secondary C-S-H (known as pozzolanic reaction). According to previous studies, ~7-17% of the total silicon atoms in the hydration products of the residual cement paste could participate in polymerization and form highly polymerized silica gel mainly consisting of Q^3 and Q^4 silicon coordination [48]. Microstructural analyses via scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy indicated that these gels might be held together by Cc to form inter-mixed agglomerates after dry carbonation. However, the silica gel might precipitate underneath the Cc shell to form a layer-by-layer structure through wet carbonation [59, 60], as illustrated in Fig. 6. In addition, it was noted that the effect of wet carbonation of RCFs was particle size dependent, and the fine fractions (e.g., 0.3mm-0.6mm) contributed more silica gel. In contrast, the relatively coarser fractions possessed a more densified microstructure, thus facilitating the transformation of RCFs into aggregates that have reduced porosity and some reactivity [48].



(a) (b)

Fig. 6. Illustrations for microstructure of RCFs by (a) dry carbonation and (b) wet carbonation [60].

Modifying the surface morphologies of RCFs was another key strategy for carbonation. It was known that rhombohedral calcite (see Fig. 5a) was the primary form of Cc formed upon carbonating RCAs/RCFs; nevertheless, polymorphs and morphologies of the Cc could also be manipulated such as promoting the formation of whisker-like Cc (see Fig. 7a). The Cc whiskers could act as micro-fibers that bridged the aggregate and new cement paste and thus enhance the local bonding strength [45]. Moreover, by introducing an appropriate amount of morphology modifier (e.g., Mg^{2+}), calcite with elongated morphologies [52] could grow on the RCFs surfaces (see Fig. 7b). The robust spear-like Cc on the RCFs surfaces dramatically roughened the surface texture and thus provided interlocking effects when it was used in new mortars or concrete.



(a) (b)

Fig. 7. Formation of (a) nano Cc whiskers and (b) spear-like Cc on the surface of carbonated RCFs [45, 52].

3.1.2 Indirect wet carbonation

Instead of direct carbonation, studies also demonstrated the feasibility of indirect wet carbonation of RCFs [61] (see Fig. 8). It was reported that virgin sand, silica-rich pozzolan, and alkali hydroxides could be extracted from this treatment scenario [61]. The virgin sand produced was equivalent to natural river sand, and the silica gel (in which >50% was amorphous) showed comparable reactivity to coal fly ash. More importantly, additional environmental and commercial values could be acquired by carbonating the residual Ca-rich leachate to sequestrate CO_2 and produce pure Cc in multiple polymorph types [62].

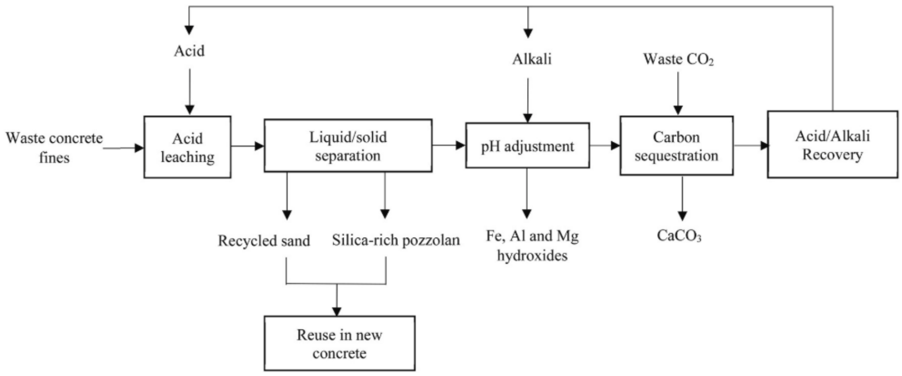


Fig. 8. Flow diagram of the acid treatment process of waste concrete fines to extract clean sand and silica-rich pozzolan and sequester CO_2 [61].

3.2 Applications in concrete

3.2.1 Application in normal concrete

Relative to natural sand that absorbed typically less than 1% water, the water demand of mortars and concretes prepared with carbonated RCFs was still relatively high. This required pre-wetting of the RCAs or RCFs to saturated surface dry conditions [63] or by adding an extra amount of superplasticizer or water to control workability [46]. In practice, considering the water absorption kinetics of RCFs, the additional quantity of water used to compensate for the water demand should be added to RCFs at least 10 min before mixing with cement to mitigate the influence on the effective water-to-cement ratio [64].

Pre-casting concrete containing RCFs in factories would be more efficient for quality control than performing casting on-site. This was not only due to the extra care provided during pre-casting, but also considering the potential influence of the Cc precipitated on RCFs surfaces that could provide extensive new nucleation sites and thus accelerate cement hydration [64]. Extensive studies have indicated that using carbonated RCFs in concrete could significantly reduce the negative impact on strength caused by uncarbonated RCFs and even attain comparable strength to the reference concrete containing entirely natural sand [39]. Nevertheless, recent research progress showed great interest in the joint utilization of carbonated RCAs and carbonated RCFs [39]. A C40 concrete could be directly obtained by fully utilizing carbonated RCAs and RCFs, without adjusting the cement content [46]; the durability properties were also improved based on the decreased chloride permeability and steel corrosion risk, etc.[47]. Moreover, the CO_2 absorbed by these aggregates accounted significantly for reducing the total CO_2 intensity of concrete.

It should be noted that reactive Cc and amorphous gel were introduced into the concrete system via the use of carbonated aggregates that would not cause cement dilution. Therefore, they can be considered as additional binding materials that can refine local pore structures and increase the bonding strength of ITZs [40]. As fine aggregates accounted for a significant amount of the total particle surface area in concrete, using carbonated RCFs was more effective in developing stronger ITZs than RCAs, thus facilitating strength improvement. Furthermore, as compared to other treatment methods aiming at imparting reactivity directly to the weak recycled aggregate such as coating pozzolan slurries on their surfaces, carbonation is more economical and less labour intensive [65].

Moreover, it was suggested that metakaolin (MK) could be used in coordination with carbonated RCAs/RCFs to fully activate the potential of Cc by reacting with the alumina from MK [66]. Similarly, the performance of carbonated RCAs/RCFs in calcium sulfoaluminate cement may also provide enhanced benefits by forming more Mc [67, 68].

3.2.2 Application in automated construction

RCFs have been innovatively selected and utilized for preparing 3D printable mixtures. It was said that the high and continuous water absorption of RCFs could increase the viscosity and static yield stress of the fresh mixture [69]. These changes were found to improve the fresh concrete to resist deformation after printing, thus leading to improved buildability [69]. Besides, the use of carbonated RCFs could also contribute to better strength development including not only the compressive and flexural strength but particularly the bonding strength between printed layers due to the pore refinement both in the RCFs vicinities and the interlayer locations [4].

4 Carbonation on recycled concrete powders

4.1 Novel supplementary cementitious material

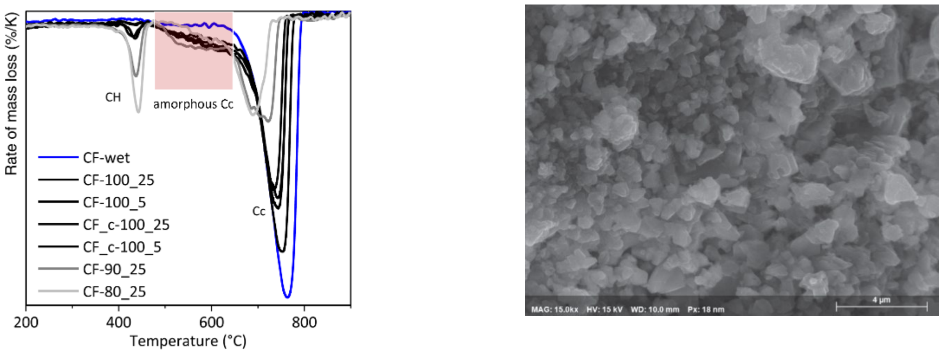
4.1.1 Carbonation strategy

Published results suggested that carbonated RCPs could be used as a novel SCM to replace traditional SCMs such as fly ash and silica fume that are showing decreased availability [25].

Although the use of RCPs in their raw form mainly caused the dilution of binders [70, 71], carbonation could re-generate the reactivity of RCPs by completely changing their phase assemblages. Based on thermodynamic modelling, different mineral phases such as Cc, silica gel, alumina gel, and gypsum, etc. were the end product of carbonation [24, 25]; ideally, they could react with calcium aluminate (present in the clinker) and portlandite (formed from hydration) in the new paste to form monocarbonate, ettringite, and secondary C-S-H, etc. as supplements to cement hydration [72]. Therefore, treating RCPs is to achieve a complete carbonation that transforms all hydration products and re-precipitates the calcium, silicate, aluminate, and sulphate species into un-hydrated and reactive phases.

4.1.2 Dry carbonation

Dry carbonation was one of the primary carbonation methods for carbonating RCPs. The maximum mass yield of Cc using dry carbonation was about 55 wt.% for RCPs made up of pure hydrated cement pastes [14, 15], with the remaining being a carbonation-induced amorphous gel, gypsum as well as uncarbonated clinkers and hydration products. The Cc obtained was mostly calcite, while the formation of vaterite, aragonite, and poorly crystalline Cc (i.e., amorphous Cc, see Fig. 9a) was also highly possible [58, 73]. The difference in moisture conditions during carbonation was critical in affecting the carbonation process, and reducing the moisture content was found to be favourable for attaining less thermodynamically stable Cc polymorphs due to the inhibition effect on their transformation to calcite [73]. It was reported that carbonating RCPs at relative humidity of 80% attained 26% calcite, 45% vaterite, 3% aragonite, and 36% amorphous Cc among all the Cc generated [73]. These Cc had sub-micron grain sizes (Fig. 9b) and higher solubilities [74], thus showing a higher reactivity than ordinary limestone powder [75].



(a) (b)

Fig. 9. (a) DTG curves of RCPs carbonated for 360 min [73], and (b) morphology of carbonated RCPs obtained from SEM [76]

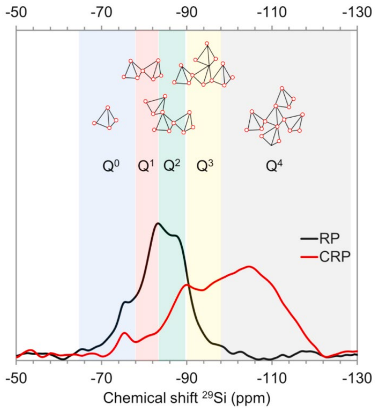


Fig. 10. ^{29}Si NMR spectra for RCPs and carbonated RCPs (as annotated by the RP and CRP respectively in the figure) [77].

The amorphous gel was the other primary carbonation product, and it was mainly silica-based. According to the ^{29}Si nuclear magnetic resonance (NMR) spectra, as shown in Fig. 10, the relative fractions of Q^0, Q^1, and Q^2 coordination of silicon tetrahedrons were reduced while that of the Q^3 and Q^4 significantly increased with carbonation. It indicated the decalcification and polymerization of both anhydrous clinkers (Q^0) and the C-S-H (Q^1 and Q^2) into highly condensed silica gel (Q^3 and Q^4) that was characterized by very low Ca/Si ratios. The silica gel showed comparable reactivity with silica fume and thus contributed to strength improvement by rapidly reacting with portlandite when used as an SCM.

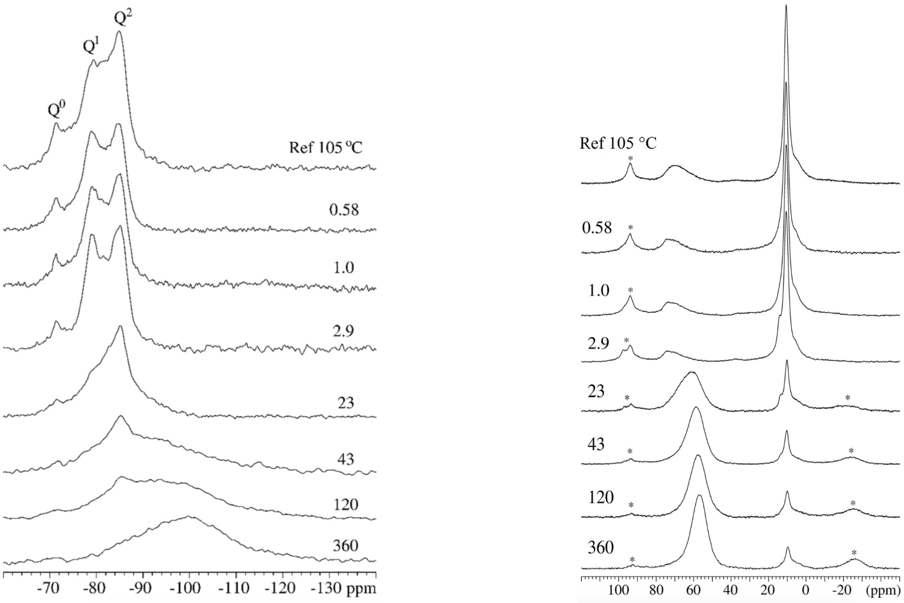
Based on available data, dry carbonation could not attain complete carbonation even with a prolonged carbonation duration of 28 days [15, 77]. Firstly, this was due to the low moisture level of the dry carbonation process. When carbonation proceeded to the internal particles, an insufficient amount of free water made it hard for the calcium-bearing phases and gaseous CO_2 to dissolve and re-precipitate. It might also because carbonation is an exothermic reaction and the heat released evaporates the moisture. Moreover, the internal precipitation of Cc could clog the connected pores, thus gradually impeding CO_2 diffusion and inhibiting complete carbonation. It was supported by the fact that the total pore volume data of RCPs, determined by nitrogen adsorption-desorption analysis, was reduced by ~70% after dry carbonation [78]. The anhydrous cement clinkers located at the interior regions of the hydration cement particles might remain even less carbonated, which was confirmed by backscattered electron microscopy images, X-ray diffraction patterns, and NMR spectra [14, 73].

During dry carbonation, the RCP particles were in close contact with each other due to the lack of good dispersion. Therefore, upon the dissolution of calcium in the surface moisture and the precipitation of Cc, RCPs were very likely to stick together, thus leading to the enlargement of particles [77]. The increase of average particle size by 2-3 times was recorded using laser diffraction as reported in the literature [73, 77].

4.1.3 Wet carbonation

As a comparison, many studies also used a wet carbonation method. It was highlighted by experimental works that the carbonation rate via the wet method could be improved by up to 3 times relative to the dry method [73], and near-complete carbonation could be achieved in several hours [76, 79]. Moreover, the corresponding maximum mass yield of Cc was also improved to over 60%, reflecting a higher degree of carbonation (see the blue line in Fig. 9a) [73, 79]. Cc mainly precipitated as calcite, and the amount of other polymorphs including the amorphous Cc was dramatically reduced compared with the dry carbonation, possibly due to their favourable transformation into calcite in a bulk solution environment [73, 79].

Investigations into the amorphous gel in the carbonated RCPs prepared with the wet carbonation method revealed more details regarding its compositions. The results showed that it mainly consisted of a silica-alumina gel and possibly another alumina gel [25]. The silica-alumina gel was originated from the decalcification and polymerization of C-(A)-S-H. This phase was seen as a broad resonance ranging from ~-80ppm to ~-120ppm with the centre at around ~-100ppm in the ^{29}Si NMR spectra [25, 77, 79] (see Fig. 11), while the presence of alumina in the silicate network as well as the possible existence of alumina gel were supported by the resonance at ~-56ppm in the ^{27}Al NMR spectra which corresponded to the tetrahedral coordination Al(IV) [25].



(a) (b)

Fig. 11. The (a)^{29}Si and (b) ^{27}Al NMR spectra of RCF after subjecting to different carbonation durations.

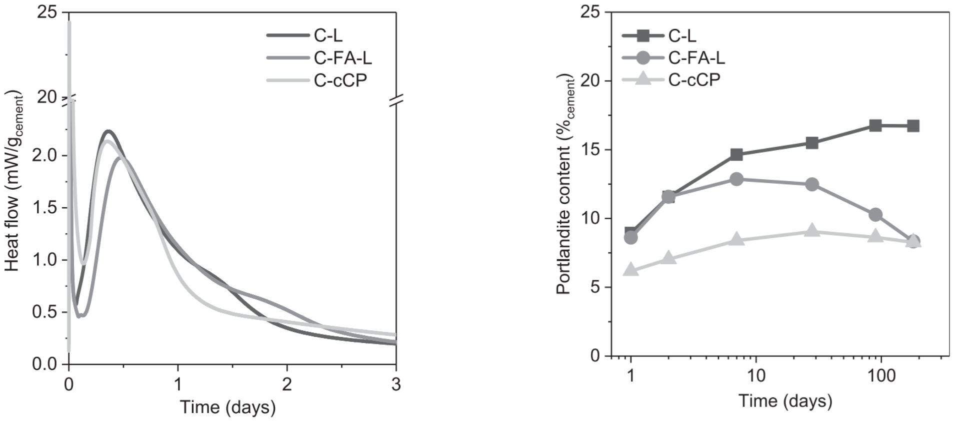
Due to using a high liquid-to-solid ratio, RCP particles were thoroughly dispersed in the wet carbonation system. As such, it enhanced the dissolution by creating a high undersaturation. Significant amounts of ions could leach out from the main particles, followed by the massive re-precipitation in the solution space upon contact with the carbonate species [76]. Generally, it led to the disintegration of the RCPs, which in turn further promoted the dissolution and carbonation of the interior phases, including the anhydrous clinker phases that the dry carbonation could not reach. This phenomenon was particularly obvious when comparing the TGA and NMR spectra of the wet and dry carbonation products, where both the portlandite peaks in TGA and the Q^0 peak in NMR completely disappeared after wet carbonation [73, 77, 79]. In this regard, the changes of the phases in the wet carbonation conformed with the thermodynamic predictions.

Thermodynamic modelling has also pointed out that, due to the loss of almost all chemically bound water, the total solid volume was reduced by ~11% upon complete carbonation [24, 25]. Numerous evaluations on the pore structure indicated that the total pore volume in the carbonated RCPs prepared with wet carbonation could increase by ~65% relative to the raw RCPs [79], and the specific surface area might increase by up to 4 times [73]. This was due to (i) the formation of silica gel with excess gel pores, and (ii) the effect of the decreased total solid volume causing more internal pores inside the particles [79]. Due to the complete decomposition and massive re-precipitation, it was found that RCPs had an increased fineness and decreased average particle sizes after wet carbonation [22], which was contradictory to that of dry carbonation. The significant formation of very fine particles (0-10um) based on laser diffraction analysis well supported the above explanations [22].

4.2 Application of carbonated RCPs in blended cement paste

4.2.1 Effect on cement hydration kinetics

Carbonated RCPs, regardless of the carbonation method, were found to significantly affect the hydration kinetics of blended cements (Fig. 12). It generally intensified the first peak in the calorimetry, which was associated with the clinker dissolution. It also accelerated alite hydration by both advancing the second peak and increasing its intensity [20, 78]. Besides, the third peak linked with the depletion of sulphate also appeared earlier, contributing to the fast precipitation of monocarbonate [20]. In addition to affecting the cement hydration, carbonated RCPs were also found to react rapidly with portlandite to form secondary C-S-H due to the presence of the amorphous gel. This reaction could be much faster than fly ash and proceed significantly at 1 day and almost complete within 28 days [20]. This might be more significant in real applications than fly ash which mainly contributes at later ages (e.g., 90 days).



(a) (b)

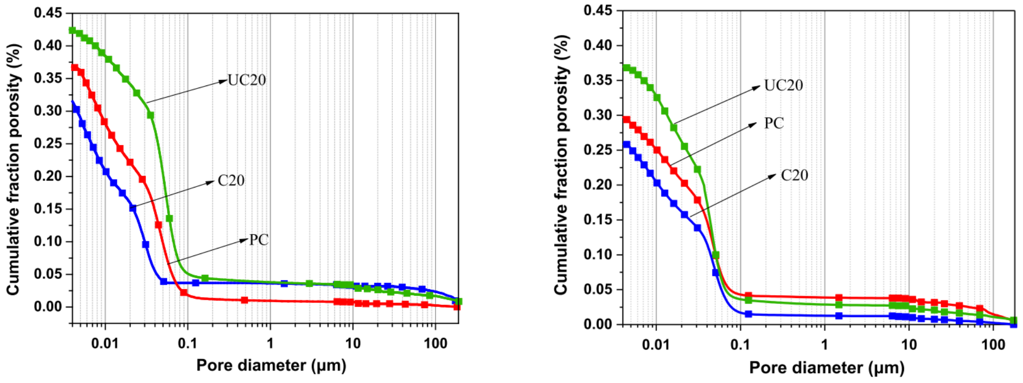
Fig. 12. (a) Heat flow of the composite cement paste with limestone (L), fly ash (FA), and carbonated RCPs (cCP) and (b) the portlandite content in the composite cement paste [20].

4.2.2 Effect on fresh properties

The fresh properties of the blended cement paste were remarkedly affected due to the use of carbonated RCPs. The flowability decreased with the amount of carbonated RCPs addition; a maximum of ~50% decrease in slump flow was recorded at 30% dosage [14]. On testing the rheology, the shear stress increased by up to 180% regardless of the tested shear rate [77]. These results consistently indicated that carbonated RCPs significantly affected the workability, and it was closely linked with the high specific surface area of the carbonated RCPs. The specific surface area for the carbonated RCPs, regardless of the carbonation method, was 20-40m^2/g [20, 58, 78], close to or even higher than the traditional SCMs such as metakaolin and silica fume. Thus more water was required to lubricate the surface of the particles.

4.2.3 Effect on mechanical properties

Based on available studies, the blended cement paste containing carbonated RCPs could attain a compressive strength of ~43MPa, ~50 MPa, and ~61MPa at the water-to-binder ratios of 0.55, 0.40, and 0.3, respectively. This was 10-40% higher than that containing the uncarbonated RCPs [14, 15, 20]. Comparatively, the carbonated RCPs showed equivalent contributions to the strength of the blended cement paste to the cementitious materials that were replaced, and a maximum dosage of carbonated RCPs of 30% was deemed feasible for practical uses. Overall, the higher compressive strength was attributed to the lower porosity of the blended cement paste (see Fig. 13) [15, 80]; and this was associated with the pozzolanic reaction of the amorphous gel as well as the formation and stabilization of other crystalline hydrates such as ettringite, monocarbonate and hydrotalcite [14, 20].



(a) 7d (b) 28d

Fig. 13. Cumulative porosity of blended cement pastes containing 20% raw RCPs (UC20) and carbonated RCPs (C20) and the pure cement paste at different ages (a) 7d and (b) 28d [15].

4.3 Fabrications and applications of value-added products

4.3.1 Calcium carbonate

Targeting at a higher added value, carbonation was also performed by coordinating with other techniques to produce Cc of various polymorphs. For example, aragonite is a metastable polymorph of Cc, which typically grow in a needle-like morphology. To leverage its morphological benefits, Shen et al. [81] proposed to fabricate aragonite-based materials from RCPs by using $MgCl_2$ as the polymorph controller. The results showed, by optimizing the carbonation temperature to >40°C and adjusting the Mg^{2+} concentration to >0.2M, over 50 wt.% of the RCPs were converted into Cc within one hour in which over 95% of the yield was aragonite (see Fig. 14a). This morphology-specialized material could act similarly to micro-fibers in concrete, thereby enhancing the toughness and tensile strength as well as the capacity to resist microcrack propagation [82].

Vaterite is another thermodynamically unstable polymorph of Cc that showed spherical morphology. Mehdizadeh et al. [83] attempted to synthesize vaterite from RCPs using an indirect wet carbonation method. The whole process, as illustrated in Fig. 15, included firstly, the leaching of calcium ions in the RCPs using ammonium chloride (NH_4Cl), and subsequently the carbonation of the ammonium- and calcium-rich leachate. After carbonation, spheroidal vaterite with a purity of ~98% and a mean particle size of ~12µm was obtained (Fig. 14b). The mass yield of vaterite was relatively high that 615g high-purity vaterite could be produced from 1kg RCPs. In terms of its application, it may not only be used as an alternative to limestone powder with a higher solubility, but also act potentially as a lubricant in concrete given its physical appearance as well as for extended applications in many other industrial areas such as drug delivery [84].

(a) (b)

Fig. 14. Calcium carbonate of different polymorphs obtained by carbonating RCPs

(a) need-like aragonite [81] and (b) spherical vaterite [83].

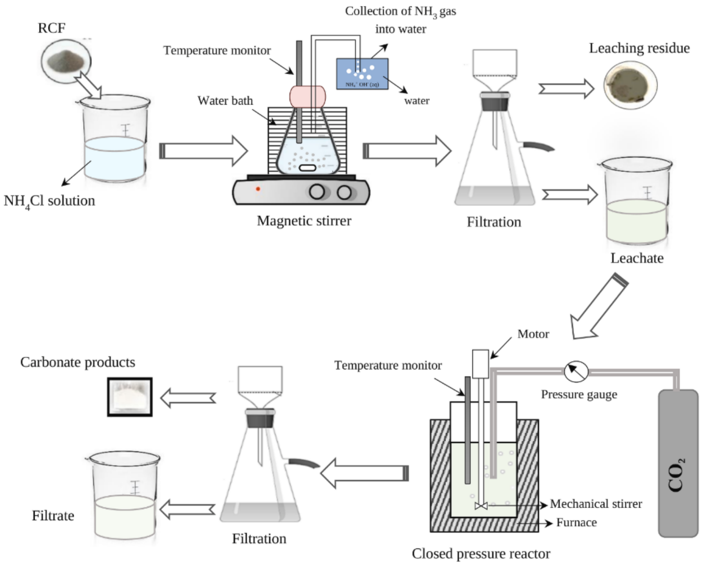


Fig. 15. Illustration of indirect wet carbonation for synthesizing vaterite [83].

4.3.2 Amorphous silica gel

Apart from the Cc, studies [85, 86] also focused on synthesizing silica gel from RCPs using a two-step wet carbonation method. The reaction flow is illustrated in Fig. 16, which includes (i) the first wet carbonation step of RCPs to precipitate the calcium as Cc, (ii) the treatment of the carbonated RCPs using sodium hydroxide to dissolve and purify the silicate phase, and (iii) the second wet carbonation step to precipitate the silica gel. The final silica gel obtained (see Fig. 17) was highly polymerized, nano-sized (surface area=662m^2/g), and thus highly reactive. The theoretical mass yield for this silica gel was 50g/1kg of RCPs.

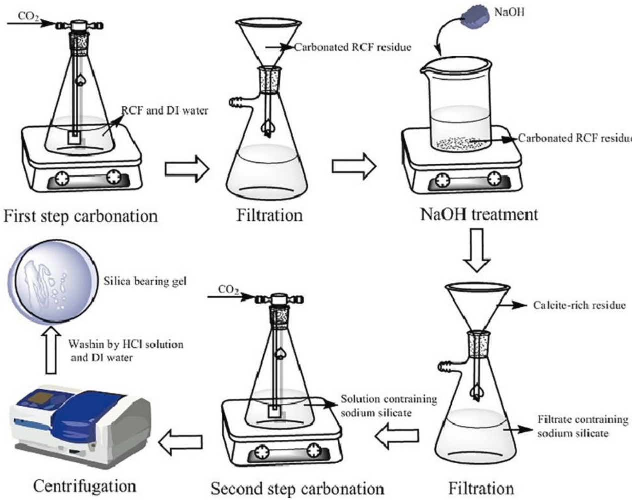
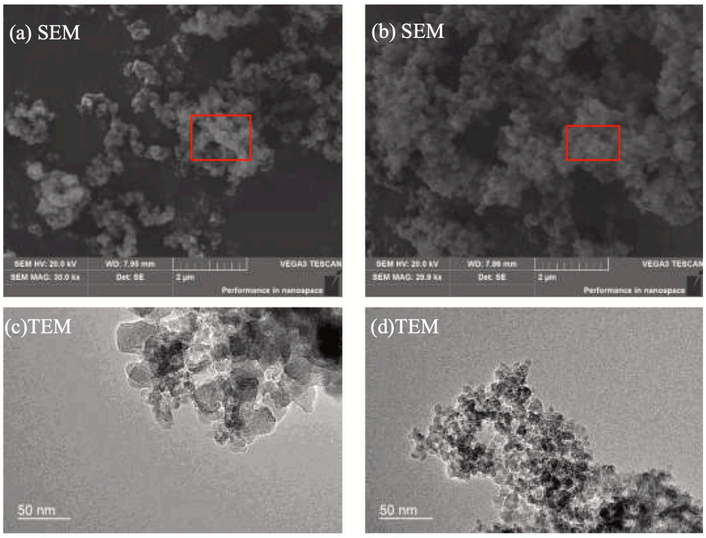


Fig. 16. Illustration of two-step wet carbonation process for synthesizing amorphous gel [85].



S-1M S-washed

Fig. 17. SEM and Transmission electron microscopy (TEM) images of silica-bearing gel extracted from RCPs using two-step carbonation [85].

4.3.3 Superhydrophobic self-cleaning coating

Aiming to improve concrete's ability to retain surface cleanliness and prevent water intrusion, a superhydrophobic self-cleaning coating based on carbonated RCPs was produced (see Fig. 18a) [87]. It was realized by (i) the wet carbonation of RCPs, (ii) the silane modification, and (iii) spraying the product on concrete surfaces. The coating shared similar compositions to the concrete substrate due to the use of RCPs and thus obtained an excellent adhesion and it also had a water contact angle of ~160° and a high surface roughness (see Fig. 18b). Therefore, the coating was considered having effective superhydrophobic and self-cleaning abilities when it was applied to concrete and significantly improved the weathering resistance of concrete.

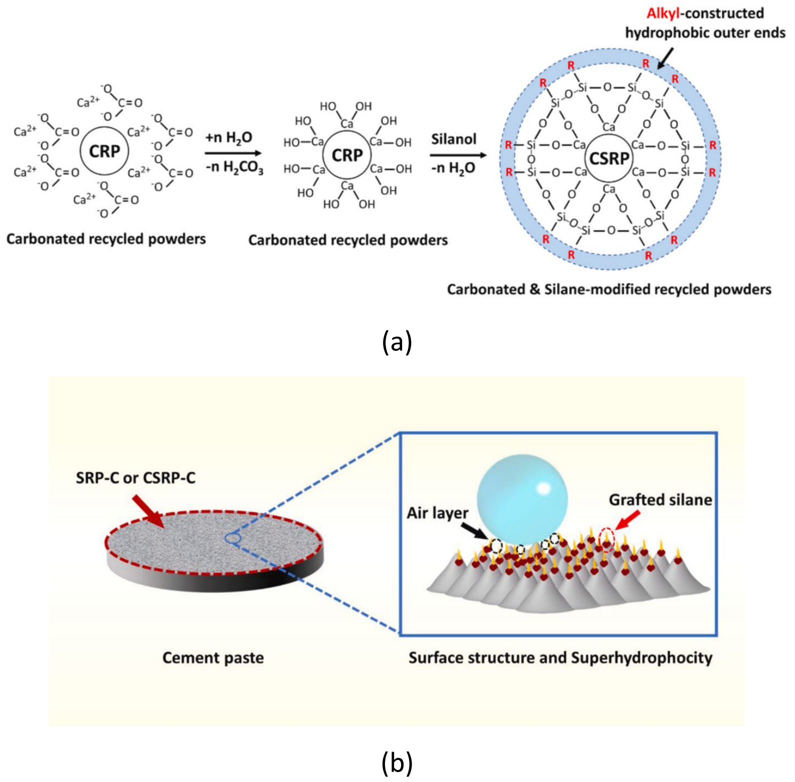


Fig. 18. Illustrations for (a) silane modification on carbonated RCPS and (b) superhydrophobic mechanism [87].

5 Key issues in upscaling

Many studies have focused on improving the carbonation efficiency for RCAs/RCFs/RCPs, as industrial-scale production demands high productivity. Factors controlling the carbonation kinetics were thus investigated and optimized. Generally, for dry carbonation using an autoclave (pressurized vessel), increasing the CO_2 partial pressure was critical. Although the efficiency of carbonation could be increased by increasing the carbonation pressure, a pressure of 1 bar was reported as adequately efficient, and a further increase to 5 bars only led to limited benefits [7, 32]. Moreover, increasing the pressure became even less impactful when a prolonged carbonation duration was applied [7, 21]. However, applying a pressurized environment is extremely difficult for industrialization due to the high operational and safety costs. Compared with using autoclaves, carbonation can also be performed in environmental chambers that regulate the relative humidity and CO_2 concentrations. Optimized conditions by controlling the relative humidity within 50-80% and the CO_2 concentration above 20%

gradually becomes the consensus [88]. Nevertheless, running an environmental carbonation chamber might be a major concern due to its huge energy consumption [89]. Based on the previous literature, pre-conditioning was usually performed prior to dry carbonation due to the interference of excess water on carbonation efficiency (mainly impeding CO_2 diffusion) [90]. However, the moisture contents in RCAs/RCFs/RCPs in a naturally air-dried condition was lower than 1% [7], which was found to have a negligible effect on carbonation. Thus this pre-conditioning procedure may not be considered in industrial production.

Wet carbonation has been increasingly applied in carbonating concrete wastes in recent years; and it was found to have higher carbonation efficiency [59, 76]. This was primarily due to the use of a bulk solution that quickly saturated and homogenized the pore network, eliminating the water-air coexistence that blocked the CO_2 pathway and thus facilitating the diffusion of aqueous carbonate species [48, 76]. Moreover, the full dispersion of RCPs in bulk solution increased the exposed surface area, leading to more complete dissolution, leaching and carbonation. Increasing the concentrations of CO_2 gas [76] or elevating the initial concentration of carbonate species (via the dosage of alkali) [91] could markedly improve the carbonation rate, particularly at an early age. Decreasing the liquid-to-solid ratio was reported to improve the final carbonation degree and the amount of CO_2 sequestrated [22]. Nevertheless, the products freshly obtained from wet carbonation have high moisture contents due to the use of bulk solutions, which may increase the complexity of handling unless other strategies, such as semi-dry carbonation or directly utilizing the wet products, could be implemented. Besides, modifications on wet carbonation and the differences with dry carbonation may result in a different phase assemblage, thus affecting the quality of the final products. Therefore, careful investigation of the quality differences should also be taken into consideration.

Temperature is another main parameter controlling carbonation kinetics. Although contradictory mechanisms had been reported [92], the available studies consistently indicated that increasing temperature up to 100°C was recommended to accelerate carbonation [41, 78]. Instead of using specifically designed heating systems, introducing high-temperature flue gas emitted from industrial processes e.g., flue gas of cement manufacturing, is becoming a popular solution to address the energy issues associated with the use of elevated temperatures.

Besides the efficiency issue, economic and environmental benefits are key determinants of decisions and choices in upscaling. The financial benefit is the difference between total income and cost that consist of the waste concrete removal charge, the trading profit of carbonated products and CO_2 in the carbon emission market, and the cost of operating the recycling platform, including transportation, screening, crushing, and carbonation. Some studies suggested [93] that extensive countries/regions such as China, the U.S., India, the EU-28, etc., could obtain net profit from carbonating waste concrete. But some failed to obtain positive environmental benefits (in terms of CO_2 balance) due to the regional disparity in tax/subsidy policies and carbon emission trading system, etc. By contrast, it was also demonstrated that the cost of producing carbonated RCAs might be several times higher than that for natural aggregates [94]. The primary reason for obtaining these contradictory conclusions lies in the transportation distance as it has been consistently reported to play a major role in influencing economic profitability and carbon emission reduction [46, 93, 94]. Therefore, optimizing the locations of demolition sites, recycling plants, CO_2 sources, and concrete manufacturing sites is a fundamental concern before implementing the carbonation industry. Nevertheless, it was also noted that RCFs/RCPs were more attractive materials than RCAs because they sequestrate significantly higher amounts of CO_2 due to the reduction in particle size and thus indicate higher profitability [94]. However, carbonating and utilizing waste concrete comparatively reduces transportation due to the avoidance of waste concrete to landfills, and the transportation cost of the natural aggregates may further increase upon its exhaustion in the local area. As such, the concrete made with RCAs/RCFs/RCPs may have lower carbon emissions than that made with conventional counterparts [46].

Generally, even though carbonation is technically feasible, it is currently not as low-carbon and sustainable as previous studies claim. Therefore, more studies with pilot-scale and industrial demonstrations that fit local conditions are needed to balance the one-sidedness of laboratory results and promote the establishment of a complete recycling chain.

6 Limitations in current research studies

The use of different carbonation approaches (e.g., dry and wet carbonation) and adjusting carbonation conditions (e.g., relative humidity and temperature) has been observed to result in different rates of carbonation [25, 73, 78]. This may not only alter the polymorphs of Cc, such as favouring the formation of amorphous Cc and vaterite, but also change the carbonation behaviour of other hydrates, such as promoting polymerization etc. that could ultimately cause a different phase assemblage. However, this link between carbonation rates/conditions with phase assemblages has not been adequately investigated and compared.

Besides, the use of SCMs dramatically changed the phase assemblage of concrete which was characterized by a lower amount of portlandite, the increase of Al/Si ratios in C-(A)-S-H, as well as the decrease of Ca/Si ratios. Some studies have reported that carbonation on blended cement paste increased the total porosity [95], adversely affecting the quality of RCAs/RCFs. Nevertheless, minimal experimental studies are available that explicitly investigate the qualities of carbonated RCAs/RCFs sourced from SCM-incorporated parent concrete. Besides, the change of C-(A)-S-H by SCMs may also result in significant differences in the carbonation-induced amorphous gel, and thus its reactivity and modification of hydration products when it is further used for making concrete. It also must point out that although studies on carbonating RCPs have been emerging over recent years, most of the RCPs used were based on hydrated cement paste, and thus the inert phases (e.g., the quartz sand) were not considered. The effect of this inert incorporation on the carbonation kinetics, the compositions, and the influence of carbonated RCPs on workability and strength may affect the conclusions previously drawn. Attempts in this aspect started when Zajac et al. proposed to apply autoclaving to quartz-containing RCPs before its carbonation, aiming at transforming hydraulically unreactive quartz into a reactive gel [80]. In view of the extensive amount of Cc produced, carbonated RCPs may also be suitable for the LC3 system but no literature on this has been published. Moreover, it is also known that flue gas is usually included in the carbonation process design for sustainability considerations; however, it was typically simulated by using CO_2 and N_2. The effect of minor gaseous phases e.g., NO_2 and SO_2 has not been adequately investigated. A pioneering study by Wang et al. has reported possible inhibitory effects of these gaseous phases on the carbonation reaction [96].

Conclusions

The current study critically reviews the recent advances in carbonating concrete waste with the vision of total recycling. Three fractions sourced from waste concrete were highlighted, including the RCAs, RCFs, and RCPs. The strategic differences in carbonating these fractions were highlighted and summarized as follows:

(a) Carbonating RCAs mainly takes advantage of the residual portlandite in the attached cement paste; the precipitation of Cc during carbonation could increase the total solid volume by ~3% and thus decrease the porosity of RCAs by up to ~30%. Furthermore, the precipitation of Cc and the reduction of porosity could enhance the paste matrices attached to RCAs and the old ITZs contained in them, thus leading to a decrease in water absorption and the improvement of macro- and micro-mechanical properties. As a result, the concrete incorporating carbonated RCAs possesses improved mechanical strength and durability such as resistance to water, CO_2, and chloride ion penetrations, etc.

(b) Due to the finer particle size and thus the higher surface area of RCFs, the main purpose of carbonating RCFs is not only for the enhancement of physical properties through the precipitation of Cc, but also the development of surface reactivity of the RCFs through the formation of reactive phases. It was highlighted that ~7-17% of the total silicon might polymerize into silica gel which improved the pozzolanic reactivity of the carbonated RCFs to react with portlandite. Considering the favourable reaction between Cc and aluminate as well as the preferential growth of C-S-H on the carbonated RCFs surface, the overall surface reactivity of the carbonated RCFs could improve the adhesion of RCFs with the new cement paste, and thus also contribute to the improved performance in the new mortars and concretes. Modification of RCFs surfaces such as growing needle-like and spear-like Cc with elongated morphologies, were also reported as beneficial due to the interlocking of ITZs.

(c) Multiple strategies could be applied to the carbonation of RCPs, including transforming RCPs into novel SCMs by complete carbonation, the synthesis of Cc of different polymorphs, and the extraction of pure amorphous gel. As a result, it was feasible to replace 30% of cement or traditional SCMs with carbonated RCPs, and to produce value-added aragonite micro-fibres or spherical vaterite for other productive uses. Most importantly, carbonating RCPs could be an effective carbon sink due to their quick and intensive reaction with CO_2.

(d) Key issues in upscaling such as improving the carbonation efficiency and the use of different carbonation routes were summarized. In addition, the research gaps concerning the influence of carbonation on RCAs/RCFs derived from SCM-containing parent concrete, the influence of quartz-contamination in RCPs, the use of flue gas, and the potential application of carbonated RCPs in LC3 systems were pointed out.

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