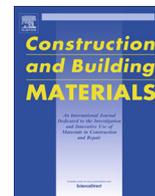




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Carbon dioxide upcycling into industrially produced concrete blocks



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HIGHLIGHTS

- Industrial trial investigated injection of CO₂ in concrete mixer at masonry plant.
- Blocks became less dense and harder to compact; mix water increased in response.
- Concrete block compressive strength and absorption was improved.
- The carbon dioxide was efficiently absorbed into the fresh concrete.
- Approach offers net sequestration of carbon dioxide into useful building products.

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ABSTRACT

A novel carbon utilization trial produced carbon dioxide treated concrete blocks by adding carbon dioxide gas to the concrete during the mixing and forming stages of the block production. The gas was supplied at up to 1.5% by weight of cement. The carbon dioxide was absorbed into the concrete with an average efficiency of about 88% of the gas delivered. The carbon dioxide was shown to reduce the block compaction (and thereby the density) but increasing the mix water was an effective countermeasure. The carbonation process was shown to increase compressive strength (13–33% at ages from 7 to 56 days) and decrease absorption (by 18–36%). The CO₂ upcycling reduced the carbon footprint of the blocks by 1.4% thereby demonstrating a simple approach to utilizing carbon dioxide to make useful construction materials.

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1. Introduction

Carbon dioxide emissions are recognized as a significant issue for the cement and concrete industry. It is estimated that 5% of the world's annual CO₂ emissions are attributable to cement production [1]. Portland cement clinker typically has embodied CO₂ on the order of 866 kg CO_{2e}/t of clinker [2]. Cement production releases CO₂ due to the calcination of limestone that is heated to drive off CO₂ and yield reactive CaO phases. Carbon dioxide emissions are further associated with the energy required to operate the cement kiln. About 40% of the process emissions are associated with the energy consumption and 60% are associated with the calcination.

A number of approaches have been identified to reduce the emissions intensity of the cement produced and used [3]. The thermal and electrical efficiency of cement production can be improved by deploying the best available technology in new cement plants and retrofits. Alternative and less carbon-intensive fuels can be used as the energy source. The rate of substitution in blended

cements can be maximized. Finally, carbon capture and storage (CCS) can capture cement industry CO₂ emissions before they are released and store them permanently.

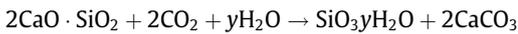
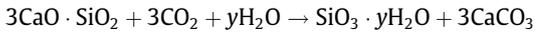
It is clear, however, that practical limits on the impacts of these measures mean that it will be difficult [4] to attain the industry goal to reduce emissions 50% below 2006 levels by 2050 that is outlined in the IEA & WBCSD roadmap [3]. The limitation of conventional approaches is highlighted when Carbon Capture and Storage, a developing but uncertain technology, has been proposed to account for 56% of the sought reductions. Thus, a range of further approaches will also have to be pursued.

One potential method is to upcycle carbon dioxide into concrete products by treating them with CO₂ prior to the end of their processing, such as during the curing stage [5–9]. If an industrial process could successfully use carbon dioxide as a feedstock in the production of concrete blocks there would be widely distributed carbon utilization that would effectively ‘close the loop’ for some of the carbon dioxide emitted during the cement production while simultaneously producing useful building products.

The carbonation of freshly hydrating cement involves the reaction of CO₂ with the main calcium silicate phases to form calcium carbonate and silicate hydrate gel [10]:

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The reaction occurs in the aqueous state when Ca^{2+} ions from the cementitious phases react with CO_3^{2-} ions from the applied gas. The carbonation reaction is exothermic evolving 347 kJ/mol for C_3S and 184 kJ/mol for $\beta\text{-C}_2\text{S}$ [10]. When the calcium silicates carbonate, the formed CaCO_3 is understood to be co-formed with calcium silicate hydrate (C-S-H) gel which itself can lose CaO and water to convert to silica gel [10]. Gel formation has been observed even in the model cases of reacting $\beta\text{-C}_2\text{S}$ and C_3S exposed to a 100% CO_2 . It was found that the amount of calcium silicate that reacted exceeded the amount that would be attributable to the formation of the carbonate products alone [10].

The reaction of carbon dioxide with a mature concrete microstructure is acknowledged as a durability issue given effects such as shrinkage, reduced pore solution pH and carbonation induced corrosion. In contrast, a carbonation reaction integrated into concrete production reacts CO_2 with freshly hydrating cement, rather than the hydration phases present in mature concrete, and does not have the same effects. The potential to integrate beneficial carbon dioxide utilization into industrial block production was investigated.

An industrial scale experiment was performed whereby gaseous carbon dioxide was injected into mixing concrete that was then used to create concrete masonry units. The goal was to assess the potential of using the concrete to sequester carbon dioxide during production albeit via a universally deployable retrofit approach that involves minimal disruption to the conventional production process. Secondly, the experiment determined the attendant material science impacts. Finally, the carbon dioxide uptake was assessed to determine how much gas became locked in the concrete as stable reaction products.

2. Materials and experimental

2.1. Mix design and procedure

A tank of liquid CO_2 was connected to a gas control system and manifold. The liquid was expanded to a gas and metered for injection into the mixer. The gas was delivered, at a specified flow rate over a fixed injection interval, whereupon it reacted with the hydrating cement before the concrete mix was finished.

A medium weight mix design was used to make standard 8" (200 mm) concrete blocks. The mix design contained 8.8% (by mass, excluding mix water) gravel, 55.7% sand, 16.1% silica sand, and 12.9% cement. The mix design also contained an admixture to improve the water repellency of the block. Carbon dioxide was injected at a rate of 0.5 to 1.5% by weight of cement (bwc).

The cement used in the trial was an ordinary Portland cement from the Illinois Cement Company with a Blaine fineness of $501 \text{ m}^2/\text{kg}$. The composition as deduced from analyses done using X-ray Fluorescence (XRF) is shown in Table 1.

2.2. Gas supply

The carbon dioxide used in the experiment was purchased as liquid CO_2 from an industrial supplier. Such carbon dioxide, almost

Table 1
Composition (in wt%) of the ordinary Portland cement found using the Bogue formulas (ASTM C150) and deduced from XRF analysis.

C_3S	C_2S	C_3A	C_4AF	MgO	K_2O	Na_2O
52.7	15.1	10.7	6.8	2.7	1.0	0.3

without exception, is captured as a byproduct from an industrial process (e.g. ammonia, fertilizer or ethanol production). Consequently, carbon dioxide purchased from an industrial supplier is effectively carbon dioxide that has been diverted from an emission stream and captured to meet market demand. Any carbon dioxide captured in the concrete treatment process using industrially supplied carbon dioxide can represent a net sequestration benefit. Any carbon dioxide from the carbonation process that is lost to the atmosphere (as opposed to being absorbed by the concrete) is taken to be returning to the destination it had had prior to the industrial capture.

The use of commercial carbon dioxide does come with an environmental cost given that the gas must be captured, liquefied, and transported. Therefore environmental considerations would have to consider all of the dosed CO_2 (not just the absorbed CO_2) while the net sequestration would have to be assessed on a case-by-case basis according to the specific capture scheme and transportation requirements.

2.3. Experimental approach

The carbon dioxide injection equipment was retrofit onto the conventional block production line (a Besser Ultrapac). The carbon dioxide was injected into the concrete mixer (a Besser Rapid Pan Mixer) through an access hatch in the mixer lid. The mixer was effectively enclosed and ensured that the injected gas was contained.

The mixing scheme, as outlined in Fig. 1, started with delivering the aggregates over 30 s. This was followed by the addition of pre-wet water and cement. The carbon dioxide injection was synchronized with the addition of the cement and lasted 3 min which adhered to the default mixing time given the imposed constraint that the process cycle time remained unchanged. The initial water addition took place between 112 and 145 s after mixing started. The final water addition took place across at 160 s after mixing began and lasted 59 s. If needed, a post- CO_2 water addition was added to the end of the cycle. After the batching was complete the concrete would mix for a few minutes prior to being discharged. In a typical mix, the free water associated with the aggregates accounted for 59% of the total mix water, while the prewet, initial and final water additions were 14%, 5% and 23% respectively.

An example consideration (shown in Fig. 2, as per a control sample) of the change in w/c in the concrete mix over the three distinct water additions shows that at the time of cement addition (60 s) the nominal w/c would be 0.27 before increasing to 0.30 when the cement input is complete. When the carbon dioxide injection is finished the mix has reached a final w/c of 0.43. The motivation of injecting the CO_2 alongside the cement is two fold – to take advantage of the low w/c conditions at that time in the mix cycle (the reaction between hydrating cement and carbon dioxide is known to be accelerated at low w/c [11]), and to maxi-

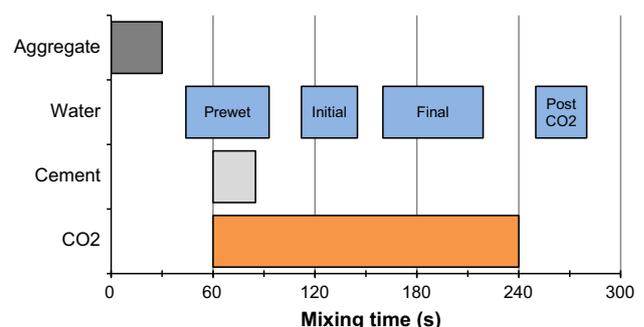


Fig. 1. Schematic representation of batching and CO_2 injection sequence.

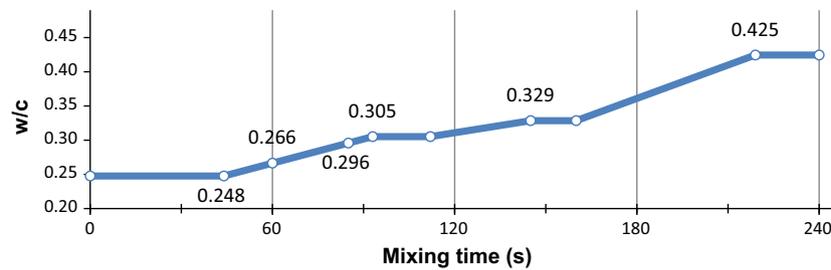


Fig. 2. Evolution of w/c during mixing (CO₂ injection occurred from 60 to 240 s).

mize the cement–CO₂ reaction time without increasing the overall mixing time.

The finished blocks were transported to a curing room heated with forced humid air. The temperature is maintained at 35–38 °C and the relative humidity between 85 and 90%. Blocks were typically held for 3 or 4 days in the kiln. Both the CO₂-treated and the reference blocks received the conventional curing.

2.4. Analytical methods

The fresh concrete was assessed visually and through feedback from the machine (i.e. compaction time) and production personnel. Samples were taken periodically to assess the water content of the concrete and monitor whether the carbon dioxide had any impact on the moisture content (real or apparent) of the fresh product. The w/c is determined by mixer software as a calculation using the free water (the excess water carried in by the aggregates) and added mix water. The “mixer final water” is the proportion recorded from a sensor and reported by the mixer software. The “bakeoff water” is a physical measure expressed as the mass fraction of water in the fresh concrete as determined by evaporation on a hot plate.

The concrete blocks were tested for compressive strength, water absorption and density in accordance with ASTM C140. Performance was assessed through compressive strength testing at 7, 28 and 56 days at the in-house testing lab with five blocks for each carbonated condition at each test age. The control baseline for the production was comprised of two batches contributing eight strength specimens per test age (five from one batch, three from the second). Water absorption and density testing was conducted on three blocks per condition and the reported baseline for the mix is an average of six control specimens sampled across the two batches. The assessment of the amount of mix water found in the control includes an additional two batches that were not subjected to strength or absorption testing.

The samples for analyzing the carbon dioxide content of the concrete were created by taking a fresh sample from the production line, drying the concrete on a hot plate to remove the water, and subsequently sieving the material through a 160 μm sieve. The carbon contents of each sample were then measured and compared to a noncarbonated control sample. All observed increases in carbon content were attributed to the reaction of carbon dioxide

with cement. Consideration of the gradation of the raw materials alongside the concrete mix design allowed for determination of the composition of the mix components that passed a 160 μm sieve and conversion of the measured CO₂ uptake to a percentage of the cement content.

The sieved samples were analyzed via the combustion infrared detection method using an Eltra CS 800 elemental analyzer. The carbon quantification samples were prepared as an A and B sample and each were analysed twice. The CO₂ uptake numbers thus are an average of four measurements.

3. Results and discussion

The batch, water content, temperature, density, absorption and CO₂ uptake data are summarized in Table 2. The water contents of the mixes are presented in three ways.

3.1. Fresh properties

The first trial with the CO₂ injection (batches 102 and 103) did not include any process or mix design alterations to the block making process. A visual assessment of the blocks produced with both doses carbon dioxide (1.0 and 1.5% CO₂) concluded that they appeared dry. The mass of fresh blocks produced in a single four-block cycle indicated that it was likely that the carbonated blocks were also underweight (an operator spot check of one cycle of blocks from batch 103 was found to average 15.8 kg vs 17.3 lbs for a control batch).

The carbonation treatment impacted the apparent water in the block. According to the calculated w/c, a greater amount of water had been added (7% and 8% increases for the two doses respectively), but there was less water detectable by the mixer sensor (reductions of 6% and 10% in the mixer final water). The bakeoff detected 2% more water in the 1.0% dose and 11% less in the 1.5% dose. It can be concluded that the CO₂ caused an apparent drying effect whereby the amount of water that could be detected (i.e. mixer final water) or removed (i.e. bakeoff) from the mix was lower than the input amount (i.e. w/c) would otherwise suggest. Potentially the carbonation reaction had created physically bound water that is neither detected as free water by the sensor nor volatilizable on the hot plate. Such water could be associated with the carbonate reaction products and specifically the accompanying new surface area (i.e. new wetting surfaces). Further, the application of CO₂ has likely created chemically bound water in the form of new hydrates including, according to the earlier presented chemical reactions, a form of C-S-H gel.

Concerns related to the block appearance and the cycle weights are normally addressed through conventional machine adjustments (e.g. increase the mould box feed time) but it was decided to avoid any adjustments that extended the length of a cycle (which would thereby reduce the rate of production) and pursue an increase in the mix water instead. Additional mix water for

Table 2

Test details of the block mixes.

	Control	CO ₂				
Batch ID	Avg	102	103	202	203	204
CO ₂ dose (bwc)	–	1.0%	1.5%	0.5%	1.0%	1.5%
w/c	0.416	0.442	0.444	0.430	0.440	0.455
Water adjustment	–	N	N	Y	Y	Y
Mixer final water	7.58%	7.10%	6.84%	8.25%	8.08%	8.39%
Bakeoff water	6.38%	6.52%	5.67%	6.58%	6.26%	6.56%
Temperature (°C)	26.6	28.8	30.5	28.1	29.6	31.8
Density (kg/m ³)	2093	2042	1987	2143	2098	2088
Absorption (kg/m ³)	136.1	146.0	120.8	86.7	90.5	111.9

subsequent carbonated batches (batches 202 to 204) was added based upon an operator assessment of need. The post-CO₂ increase in water did not slow down production since the mixing cycles were completed before the hopper containing the concrete from the previous mix had been completely emptied.

The manually determined water increases, according to the mixer reports and measured water inputs, for the carbonated mixes were observed to increase along with the increasing CO₂ dose. The total water, according to the w/c calculations, was increased 4% for the lowest CO₂ dose to 7% for the middle dose and 10% for the highest dose. Alternately, the water increases, as calculated based upon the sensor on the batching system, were 9, 7 and 11% higher for the three carbonated batches respectively. In contrast, a bakeoff of the moisture from fresh samples indicates that the observed water was again lower than the other assessment metrics with 3% more, 2% less and 3% more water removable from the fresh samples.

The temperature increase observed in the carbonated batches is indicative of the exothermic nature of the carbonation reaction and can be seen to increase along with the supplied CO₂ dose within each of the two subsets.

3.2. Hardened properties

As shown in Table 2, the carbonated batches without a water adjustment were shown to have a lower density. The density reductions were 2.4% and 5.1% for the 1.0 and 1.5% doses respectively. The average absorption of the blocks was increased 7% at the 1.0% dose and decreased 11% for the 1.5% dose.

The density deficit was satisfactorily addressed through the post-injection water addition. The density of the blocks was 2.4% higher for the 0.5% dose and equivalent to the control for both the 1.0% and 1.5% CO₂ doses. The absorption was improved in all three carbonated batches with reductions of 36%, 34% and 18% over the three increasing dosages. The reduced water absorption suggests that the carbonate reaction products, or their attendant impact on the hydration, have resulted in reduced porosity through either improved pore filling or a denser microstructure.

The strength development of blocks made during the first section of the testing is shown in Fig. 3 and results from the second (water adjusted) test are shown in Fig. 4. The relative strengths of the CO₂-treated blocks compared to the control is noted above the bars.

The strength development of the unadjusted batches, in Fig. 3, shows the impact of producing dry and underweight blocks. The 1.0% dose blocks were 80% of the control strength at 7 days before closing to 91% at 56 days. The higher dose was 62% at 7 days before improving to 76% at 56 days. Clearly, strength was compromised

by not producing blocks with the proper density but it can be noted that the strength development did not run parallel to that of the control baseline but rather reduced the deficit with time. Given that the blocks still met the specifications outlined in ASTM C-90 – a water absorption beneath the upper limit (240 kg/m³) and a strength in excess of the specified lower limit (13.1 MPa) – it is possible that the approach unintentionally offered a means to create blocks with a reduced density and acceptable strength. In practical terms this would increase the batch yield by allowing for a greater number of blocks to be produced per batch of concrete.

The performance of the carbonated mixes was greatly improved through the water adjustment. As shown in Fig. 4, the low dose of CO₂ provided 30% strength increases at the two early ages and 27% at 56 days. The 1.0% dose showed a 33% benefit at 7 days, 29% at 28 days and 13% at 56 days. Finally, the 1.5% dose resulted in a strength increase in the range 18–19% at all three ages. The performance of the two higher doses of CO₂ (7–11% increase in mix water, equivalent density to the control, 18–30% strength increase at all three ages days, 18–34% lower absorption) allows the improvement in properties to be uncoupled from block density and attributed to the carbon dioxide addition.

It is evident that any negative effect on compaction, density and block strength that was associated with the addition of CO₂ can be addressed through a simple mix water increase. If the amount of additional water is known then it could be incorporated into the batch sequencing rather than manually determined.

One potential mechanism for the strength benefit could be that the addition of the CO₂ allowed a higher water content in the block and subsequently increased the overall hydration. Simply increasing the mix water in normal operation would increase the risk of undesirable dimensional variation in the fresh blocks (e.g. edges and corners are not crisp or blocks may slump). Further, operational difficulties may arise related to concrete sticking to the shoe that compresses the blocks from above. If the CO₂-treated blocks contain more water (as per the w/c readings) but do not appear to have more water (either through visual assessment or the water sensor reading) then it is possible that the CO₂ has allowed for a higher true water content (that would factor into hydration) without any change to the effective water content (that would factor into the compaction and appearance of the block). The higher water content may be related to having increased the wetting surfaces through the immediate creation of reaction products.

3.3. CO₂ uptake

The dosed and absorbed CO₂ contents for the carbon dioxide treated batches are presented in Fig. 5. Absorption efficiency is noted above the bars representing the absorbed CO₂ contents.

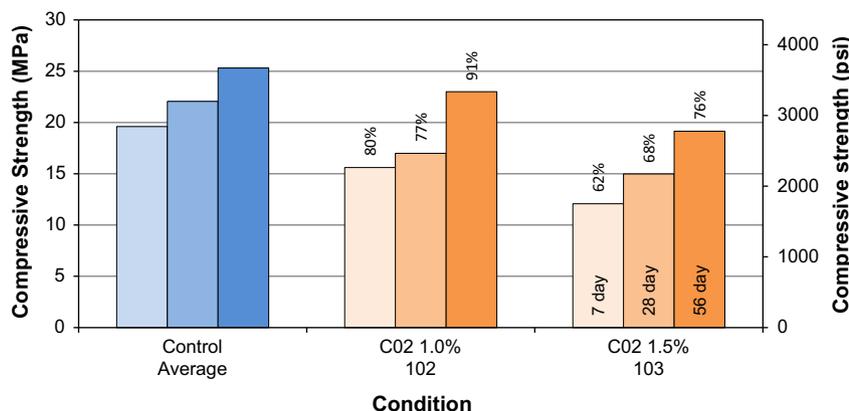


Fig. 3. Compressive strength at 7, 28 and 56 days of the mixes without a water adjustment.

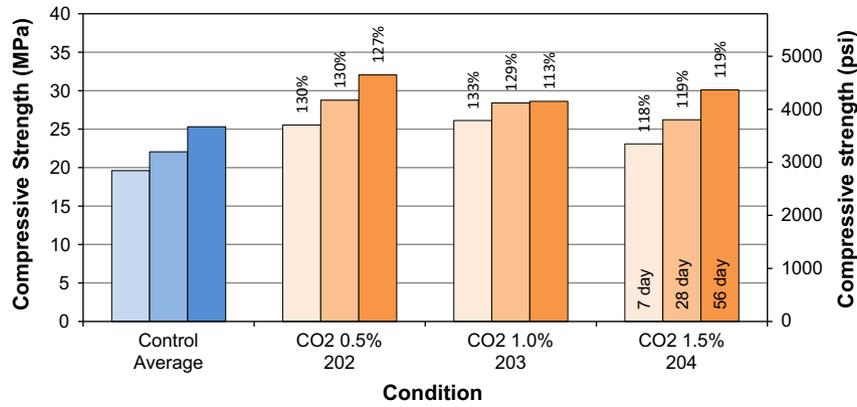


Fig. 4. Compressive strength at 7, 28 and 56 days of the mixes that included a water adjustment.

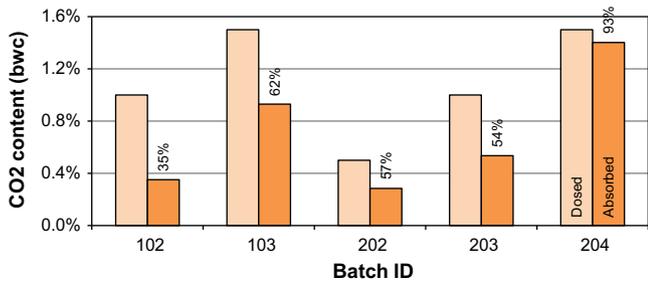


Fig. 5. CO₂ dosed and absorbed for the CO₂ treated batches.

Table 3
Net sequestration details.

Factor	Per 1125 blocks
Cement content (kg)	2569
CO ₂ uptake efficiency	88%
CO ₂ dosed (kg)	38.5
Total CO ₂ absorbed (kg)	33.9
Emissions – gas processing (kg)	4.8
Emissions – gas transport (kg)	0.5
Total CO ₂ produced (kg)	5.3
Net CO ₂ absorbed (kg)	28.6
Net efficacy (Net CO ₂ absorbed/Total CO ₂ absorbed)	84.3%

It was found that both the CO₂ uptake and uptake efficiency increased with CO₂ dose and after employing the water addition. Within the second trial set the carbon dioxide uptake was shown to be most efficient at the highest dose wherein 93% of the supplied CO₂ was found to have been absorbed by the concrete. Conceptually, the gas uptake may have some relation to the empty space (head space) in the mixer above the concrete. After the gas injection is completed but immediately prior to discharging the batch it is likely that some amount of carbon dioxide will remain in the space above the concrete that would not have reacted with the cement. This unreacted head space amount is potentially nominally independent of dose provided that the dose is sufficient to fill the mixer by the time the injection is complete. If there is an efficiency loss attributable to unreacted gas left in the headspace then this fixed amount could, in relative terms, represent a high fraction of a low dose but a low fraction of a high dose. It is noted that, in the set without the water adjustment, the increase from 1.0% to 1.5% CO₂ also resulted in a great increase in the efficiency of the uptake. This preliminary observation would require an extended run of replicates to confirm.

Further tests at the highest dose did explore minor mix and production adjustments but an assessment of the uptake consistency can be made. A dose of 1.50% CO₂ repeated in five other batches

resulted in uptakes of 1.43%, 1.43%, 1.24%, 1.19% and 1.19% bwc. If batch 204 presented herein is included, the average uptake efficiency was observed to be 88%.

3.4. Net environmental benefit

An estimate of the net environmental benefit is summarized in Table 3. The masonry analysis is considered in terms of 1125 conventional 8" blocks, or the nominal number of blocks required to construct a 93 m² (1000 ft²) wall. The analysis considers the amount of carbon dioxide absorbed by the concrete compared to the emissions associated with the processing and transport of the total amount of CO₂ dosed. Further factors would be relevant to an exhaustive carbon footprint calculation (such as emissions associated with manufacture and transport of the gas delivery equipment) but the two most significant factors, by far, are included. No energy usage is associated with the CO₂ liquid-gas phase transition if an ambient gas vaporizer is employed.

The blocks are assumed to be 17.7 kg and contain 12.9% cement by total fresh mass. The total cement usage is 2569 kg. The analysis considers a 1.5% by weight of cement dose of CO₂ that is absorbed at 88% efficiency, or a 1.32% uptake by weight of cement).

The industrial gas processing to create liquid CO₂ from an emissions stream requires on the order of 198 kWh/tonne CO₂ with an electrical emissions rate that is dependent upon the location in which the work is performed. Cargo transport of the total dosed CO₂ in liquid form is estimated according to an emissions rate of 222 g CO₂/ton-mile of cargo [12]. The masonry trial took place 101 km away from the nearest industrial source of CO₂ in Whiting, IN. The 2012 CO₂ emission rate for electrical power produced in that region was 625.7 g CO₂/kWh [13].

It was found that the net CO₂ utilization in a generic wall that would be produced using blocks from the masonry trial would amount to 28.6 kg with a per block amount of 25.4 g. The effectiveness of the approach in creating a net CO₂ sink is calculated as the net efficacy. The proportion of the total absorbed CO₂ that, when accounting for the processing and transport emissions, represents actual CO₂ sequestration is 84.3%.

The CO₂ emissions associated with the production of the cement in the wall (assuming a clinker emissions intensity of 866 kg/t and a clinker content in the cement of 94%) are about 2091 kg. The net upcycled CO₂ (28.6 kg) represents a direct offset of 1.4% of the cement production emissions.

3.5. Industrial suitability

The trial investigated a gas injection system that could be incorporated simply into a conventional block production. The design of

the mixer encountered effectively contained the carbon dioxide and ensured a high conversion. Should the approach be investigated elsewhere the gas containment offered by the mixer would be an important aspect to consider.

In contrast to approaches that upcycle carbon dioxide into concrete by placing pre-conditioned products into enclosed chambers the retrofit mixer injection was investigated for its simplicity and universal applicability (i.e. all block production process would include a concrete mixer but not all would include a suitably enclosable curing chamber). It is acknowledged that carbonation of a pre-conditioned product in a chamber offers, by virtue of extended carbonation times, a strategy to achieve greater carbon uptakes but faces obvious barriers to implementation, mainly due to the attendant process and equipment modifications and gas costs that scale higher as CO₂ dosages are increased.

The marketplace for conventional concrete blocks is commodity based with competition observed mainly on price. The raw material cost of the CO₂, taken generically at 33–44 cents per kilogram (15–20 cents per lb), would mean that the material costs of the block would increase by 0.85 to 1.14 cents. The cost would be appropriately small with respect to a generic price of \$1 to \$1.50 per block. Alternately, the strength benefit could be the basis for a mix design optimization to reduce the cement loading. Such an adjustment, motivated by both material performance and economics, could offset the cost of the carbon dioxide and preserve competitive pricing approaches.

4. Conclusion

It is concluded that a carbon dioxide injection can be simply integrated as a retrofit into a conventional block production line and create concrete blocks with a reduced carbon footprint. The reaction of the carbon dioxide with the concrete during mixing forms carbonate reaction products and unlocks material benefits alongside achieving permanent storage of CO₂ into the blocks.

The addition of the carbon dioxide to the masonry blocks suggested that the creation of nano-scale calcium carbonate reaction products in the fresh concrete made the blocks more difficult to compact. Once a mix water correction was applied the blocks became stronger. When the comparing blocks with equivalent density the CO₂ treated products contained 7–11% more mix water, were 18–30% stronger and displayed 18–34% lower absorption. A 1.50% by weight of cement dose of CO₂ was found to be absorbed at 88% efficiency.

The CO₂ utilization approach can produce material performance benefits but would have an associated emission of carbon dioxide. The effective net absorption was calculated to be 84.3% of the overall CO₂ absorption. A generic wall of 1125 blocks would have a net CO₂ consumption of 28.6 kg which would offer a direct offset of

1.4% of the CO₂ emissions associated with the cement in the blocks. The performance benefit and carbon dioxide consumption is realizable at an economic scale appropriate for the commodity-priced block marketplace.

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