

# Ready Mix Technology Trial Results

## TECHNICAL NOTE

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### EXECUTIVE SUMMARY

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The CarbonCure Technologies ready mix concrete system is commercially available as of April 2015. Industrial pilot testing results of the carbonated ready mix concrete are presented in this technical note. The process created nanocrystalline carbonate reaction products that positively effected the early hydration and the compressive strength. The carbonated concrete was as much as 20% stronger than the control concrete without any compromise in pore solution pH or risk for chloride penetrability.

## READY MIX CONCRETE PILOT RESULTS

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CarbonCure Technologies has conducted industrial trials with multiple ready mix producers. The results of a trial conducted in September 2014 are reported herein. CO<sub>2</sub> gas was delivered into the ready mix concrete truck during and after the initial batching and mixing using the CarbonCure control system. The carbon dioxide was bound as stable carbonate reaction products in the concrete products.

### Methods

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The fresh concrete was assessed via on-site measurement of slump and temperature. The concrete was further cast into 4" x 8" cylinders for compressive strength testing. A fresh sample was taken for assessment of the heat evolution via an iCal 8000 isothermal calorimeter set at 22 °C.

Concrete performance was assessed through compressive strength testing at 1, 3, 7, 28, 56 and 91 days. The measurements performed at 1 and 28 days were performed in triplicate, while the measurements at other ages were performed in duplicate. All specimens were subjected to grinding of the bearing faces before testing to ensure planeness. The specimens tested beyond 1 day were subjected to standard moist curing conditions prior to testing. All concrete specimens were prepared and tested in accordance with CSA A23.2. Bulk resistivity was assessed using a Merlin Mark III by Germann Instruments. Testing of the hardened concrete was overseen by Professor Doug Hooton of the University of Toronto Department of Civil Engineering.

### Gas injection

The CO<sub>2</sub> delivery was controlled by a mobile CarbonCure gas injection system. A tank of liquid CO<sub>2</sub> was connected to the control system and manifold. The liquid was metered for injection into the ready mix truck mixing drum whereupon it converted into a mixture of CO<sub>2</sub> gas and solid carbon dioxide snow. The carbon dioxide was delivered, at a specified flow rate over a fixed injection interval, into the truck whereupon it reacted with the hydrating cement before the concrete mix was completed. The concrete was then subjected to assessment and testing.

The injection was completed alongside normal production steps. CO<sub>2</sub> dosages were determined with respect to the cement loading in the mix design. The truck was set to a slow transit mixing speed at any time other than the pauses for sampling and high speed mixing during the CO<sub>2</sub> injections.

### Industrial trial

The trial was conducted in September 2014. The test focused on trucks carrying 4 m<sup>3</sup> of concrete (half full) using the mix design (25 MPa) summarised in Table 1. The cement was understood to be 80% of the total cementitious fraction.

**Table 1. Mix Design**

Component	Type	Quantity	Fraction
Concrete Sand	Fine aggregate	959 kg	42%
20mm Stone	Coarse aggregate	1080 kg	49%
Slag	Binder	53 kg	2%
Cement	Binder	212 kg	9%
Water Reducer	Admixture	190 ml	
Mix Water	Water	155 kg	

The requested load of concrete was first batched into the truck before transport to the wash rack where the batch received final water adjustments by the truck operator. Once the batch adjustments had been completed a sample of uncarbonated (control) concrete was removed, assessed, and cast into specimens for testing.

The truck was then subjected to sequential doses of carbon dioxide with assessment and casting of the carbonated concrete between each round. The time between the start of mixing and the carbon dioxide application was recorded. All of the test samples came from the same truck to maximize the experimental results from a single batch and to minimize any batch-to-batch variation that may have arisen. In practice the dose was intended to represent a truck treated with carbon dioxide at the wash rack. A second truck was processed wherein the gas was added during the initial batching/mixing phase. This represents an alternate CO<sub>2</sub> injection mode wherein the gas is directly integrated into the batching.

## Results

An overview of the conditions produced during the trial is presented in Table 2. The control, CO<sub>2</sub>-1 and CO<sub>2</sub>-2 samples were taken from the same truck. The carbon dioxide was dosed at the wash rack. Batch CO<sub>2</sub>-3 was in a separate truck. The carbon dioxide was dosed during the batching.

**Table 2: Trial production variables, CO<sub>2</sub> settings and fresh results**

Condition	Age at injection (min)	CO <sub>2</sub> Dose (%bwc)	Slump (inches)	Mix Temperature (°C)
Control		-	3.5	22.4
CO <sub>2</sub> - 1	43	0.30	2.5	24.7
CO <sub>2</sub> - 2	52	0.60	1.5	27.3
CO <sub>2</sub> - 3	0	0.30	3.0	23.3

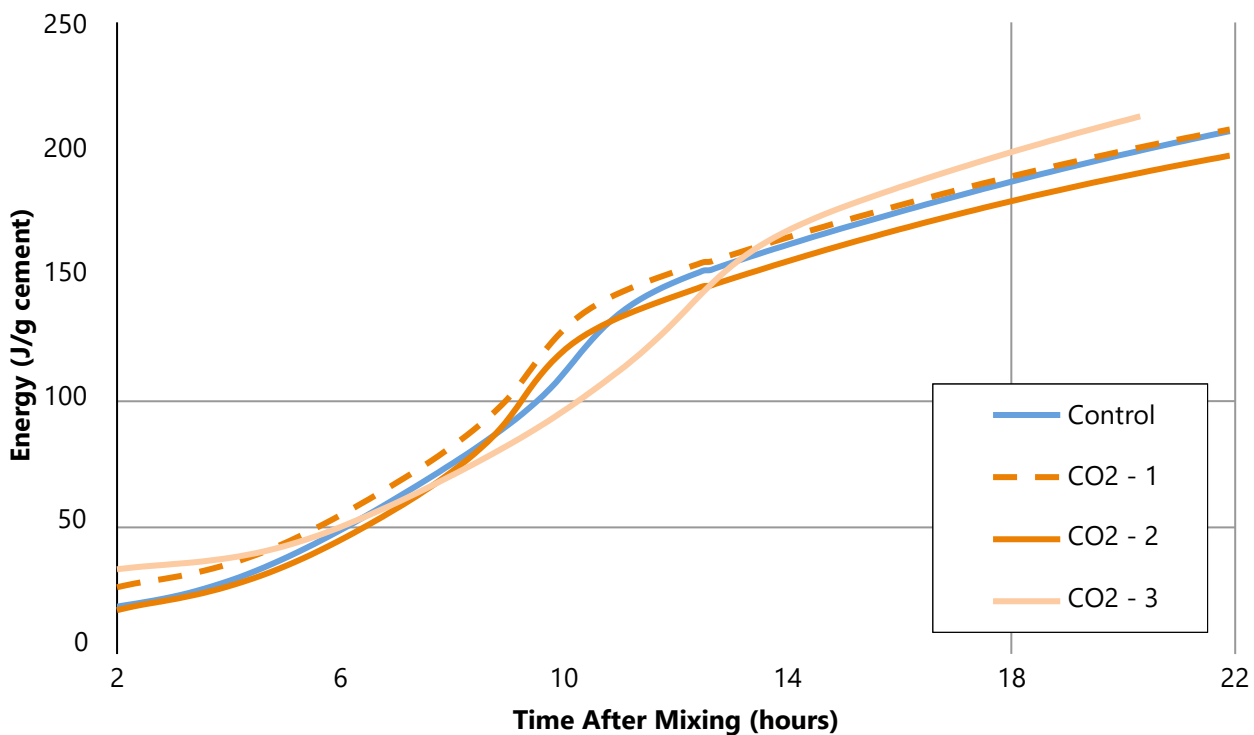
The control concrete was sampled 21 minutes after mixing started. After carbonation it was observed that the temperature of the mix increased and the slump of the mixture decreased. It cannot be concluded whether the entirety of the observed effects were attributable wholly to the carbonation

process, or further represented a natural variation due to the passing of time between measurements, mixing, progress of hydration, increasing ambient temperature, etc.

The second truck showed that a carbonated batch could be created with the target slump. The earlier batches could be produced with the desired slump if the injection was directly integrated with the standard mix finishing.

**Isothermal calorimetry**

The evolution of heat from the fresh concrete was measured from 0 to 20 hours after mixing. The amount of heat released by the concrete in the range of 0 - 24 hours can be used as a proxy for the development of mechanical properties (such as setting or compressive strength) at very early ages. A summary of the energy data (J/g of cement) for the four batches are presented in Figure 1.



**Figure 1: Energy during early hydration via isothermal calorimetry of trial batches**

The carbon dioxide was found to offer an accelerating effect.

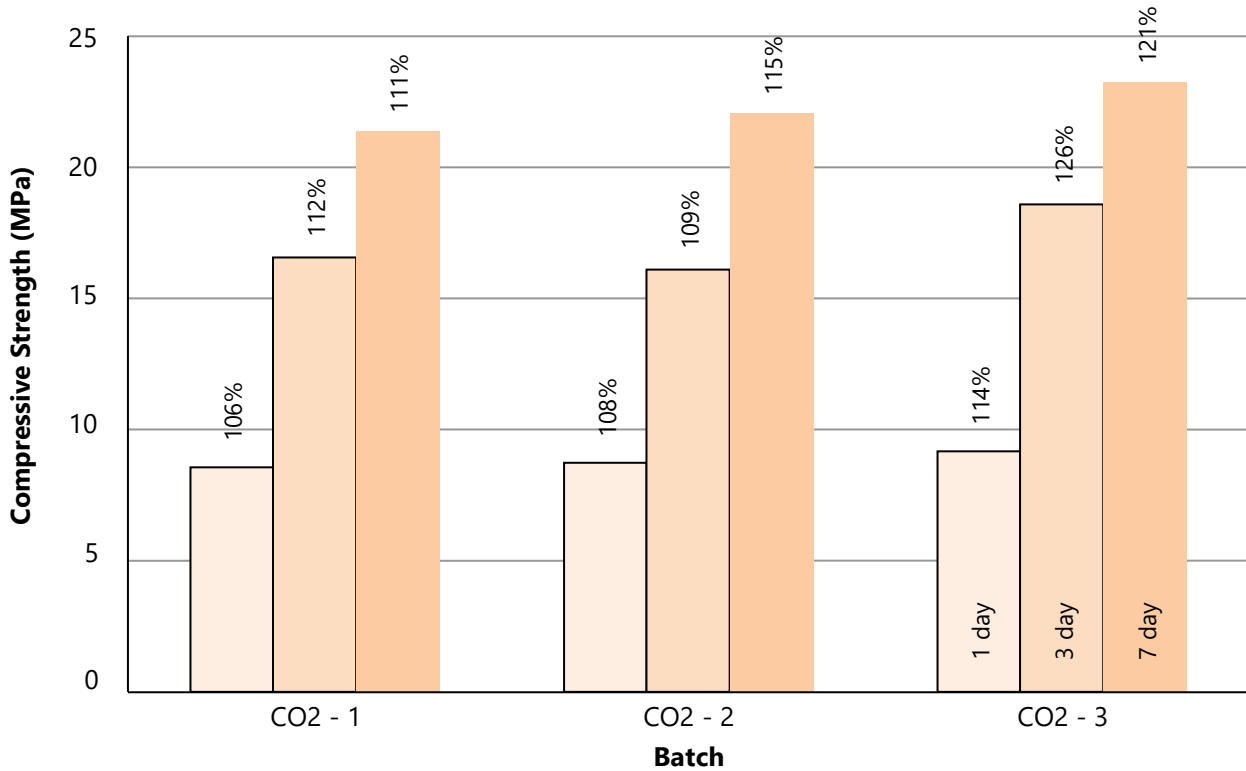
The CO2-2 batch had released 41% more energy than the control at 2 hours. The energy release was 16% ahead of the control at 10 hours before declining to be equivalent to the control. For the highest dose the energy was between 92% and 99% of the control in the first 9 hours before spiking to be 9% ahead and thereafter declining to be equivalent to the control. The implication from the calorimetry is that increased hydration through the first 10 hours can result in short finishing times.

The batch that was dosed with CO<sub>2</sub> during batching (CO2-3) showed a calorimetry response wherein heat evolution was slower than the control across the 7 to 13 hour interval. However, it thereafter accelerated

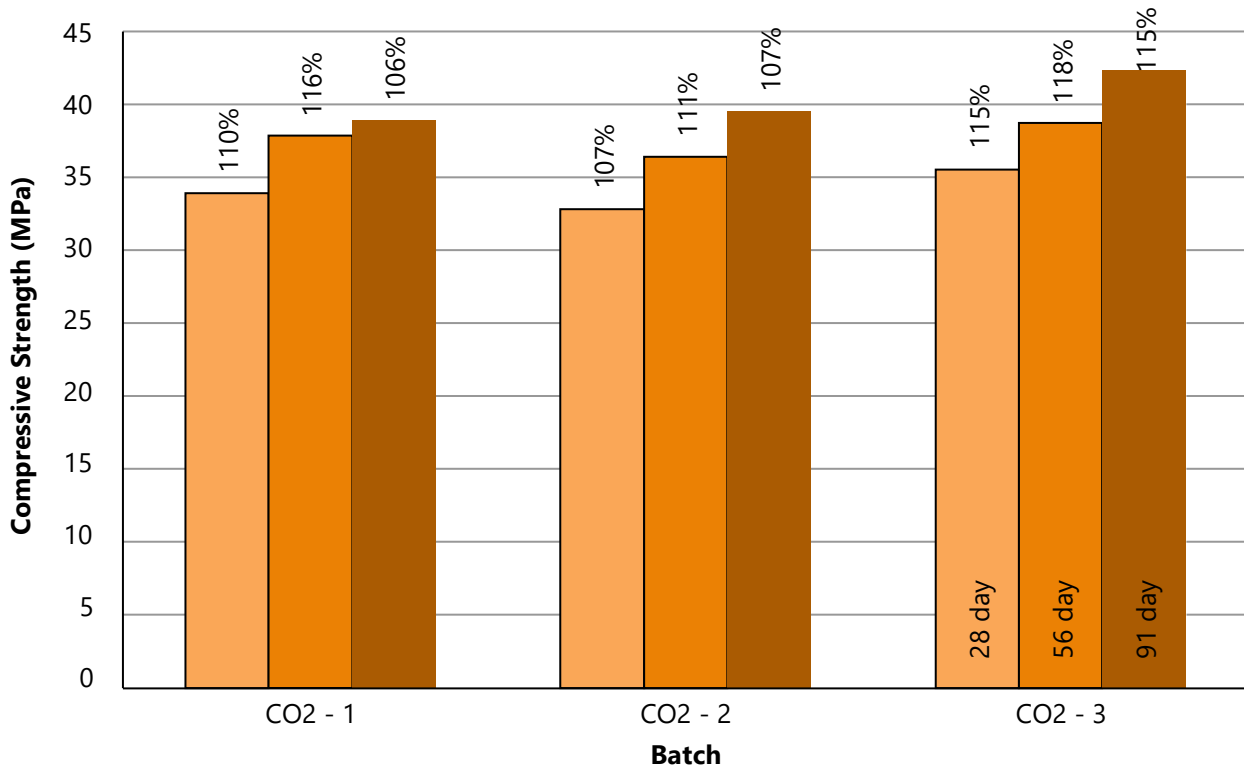
and was 5% ahead at 15 hours and 7% at 20 hours. The higher strength in the 20-24 hour time frame may allow quicker removal of formwork.

**Compressive Strength**

The average compressive strength measured for each carbonated condition across the three early test ages is summarized in Figure 2 while the strengths for the three later test ages is summarized in Figure 3. Each data point represents the average measured compressive strength in MPa with the relative comparison to the corresponding control value displayed as a percentage outside the end of the bar.



**Figure 2: Early compressive strength (1, 3 and 7 days) of carbonated batches**



**Figure 3: Later compressive strength (28, 56, and 91 days) of carbonated batches**

The results confirmed that an increase in the compressive strength could be realized upon carbonation of the concrete mix. The CO2-1 batch was 6% stronger at one day, and 10-12% stronger through 28 days. It finished at 16% stronger at 56 days and 6% stronger at 91 days. The CO2-2 batch was broadly the same with an 8% strength benefit at one day and 7-15% benefits at the later test ages. The final batch (CO2-3) showed the greatest promise given that the strength benefit exceeded 14% at all ages including a 26% benefit at 3 days and 15% at 91 days.

### **Bulk Resistivity**

Bulk resistivity is a measure of the transport properties of concrete. It can be used as an indication of the potential for chloride penetrability in service. The bulk resistivity measurements showed that the carbonated batches were equivalent to the control. The chloride penetrability risk for all samples was assessed to be moderate at 28 days and low at 91 days.

**Table 3: Bulk Resistivity ( $\Omega\cdot\text{m}$ ) and chloride penetrability risk for Trial 1 test specimens at five different ages**

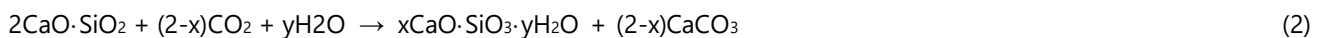
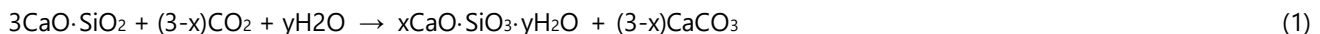
Condition	1 day	3 day	7 day	28 day	91 day
Control	9.6	14.9	21.0	58.3	123.4
CO <sub>2</sub> – 1	9.2	15.9	20.9	59.6	123.7
CO <sub>2</sub> – 2	9.2	16.1	20.7	50.3	112.9
CO <sub>2</sub> - 3	10.1	18.0	23.3	61.8	129.8
Control	High	High	High	Moderate	Low
CO <sub>2</sub> – 1	High	High	High	Moderate	Low
CO <sub>2</sub> – 2	High	High	High	Moderate	Low
CO <sub>2</sub> - 3	High	High	High	Moderate	Low

### Pore solution pH

Lab testing has considered the impact of carbon dioxide on pore solution pH. Cement paste samples were produced to compare a control sample and five samples that were treated with carbon dioxide during mixing. Samples were cured for 28 days. They were crushed and the pore solution was extracted. The pH of the pore solution was measured directly. The control sample had a pH of 13.83. The pH of five carbonated samples ranged from 13.74 to 13.81. The analysis suggests that the carbonation treatment does not increase the risk of ferrous reinforcement depassivation due to reduced pore solution alkalinity.

### Mechanism

The upcycling of carbon dioxide into concrete occurs through a reaction between the hydrating cement and CO<sub>2</sub>. The main cement phases, tricalcium silicate and dicalcium silicate, are known to react with carbon dioxide in the presence of water to form calcium carbonate and calcium silicate hydrate gel (Berger et al. 1972) as shown in equations 1 and 2:



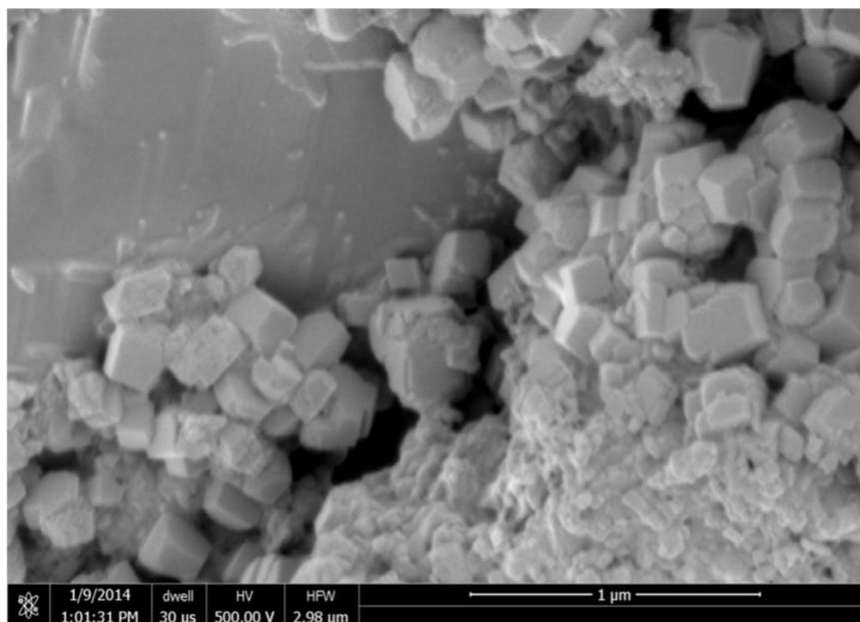
Further any calcium hydroxide present in the cement paste will react with carbon dioxide, as show in equation 3:



The carbonation reactions are exothermic. The reaction proceeds in the aqueous state when Ca<sup>2+</sup> ions from the cementitious phases meet CO<sub>3</sub><sup>2-</sup> ions from the applied gas. The carbonation heats of reaction for the main calcium silicate phases are 347 kJ/mol for C3S and 184 kJ/mol for β-C2S (Goodbrake et al. 1979) and 179 kJ/mol for CaO (Moorehead 1986).

The reaction of carbon dioxide with a mature concrete microstructure is conventionally acknowledged to be a durability issue due to such effects such as shrinkage, reduced pore solution pH, and carbonation induced corrosion. In contrast, a carbonation reaction integrated into concrete production reacts CO<sub>2</sub>

with freshly hydrating cement, rather than the hydration phases present in mature concrete, and does not have the same effects. Rather, by virtue of adding gaseous CO<sub>2</sub> to freshly mixing concrete the carbonate reaction products are formed in situ, at a nano-scale (Figure 4) and homogenously distributed.



**Figure 4. Nano-scale (10-150 nm) rhombohedral calcium carbonate reaction products produced through carbonating freshly hydrating cement**

The performance improvement observed due to the carbonate reaction product formation is analogous to growing an in-situ nanoparticle CaCO<sub>3</sub> addition that impacts later hydration product development. The calorimetric evidence is similar to what can be observed by ex-situ additions of nano-CaCO<sub>3</sub> (10% by mass) to C3S (Sato and Diallo 2010).

One outcome of attaining a consistent strength benefit could be to sell a given concrete mix at a price premium - a 15% strength improvement to a 35 MPa mix is equivalent to producing a 40 MPa mix. Alternatively, the cement content could be reduced and the same 35 MPa performance can be offered. A reduction in cement loading results in an economic saving but also offers a direct and tangible reduction in the carbon footprint of the concrete so produced. Further, a strength boost realized in a concrete containing slag potentially offers a way to increase the use of slag in cold weather.

## Conclusions

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Industrial scale experiments assessed the viability of adding small doses of carbon dioxide to a ready mix concrete production cycle in order develop performance benefits associated with the in-situ development of carbonate reaction products. Isothermal calorimetry indicated that hydration could be accelerated. Compressive strength was increased up to 14% at 1 day, 26% at 3 days and 15% at 28 days. Neither the pore solution pH nor the bulk transport properties were effected by the carbon dioxide treatment. A carbonation-related strength benefit could allow for more economical, lower carbon footprint concrete mixes to be produced.



## References

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