

The Effects of Liquid Corrosion Inhibitor in Air-Entrained Dry-Mix Shotcrete

By Jean-François Dufour, Simon Reny, Pierre Lacroix, and Richard Morin

In Canada, the main causes of reinforced concrete deterioration are the corrosion of the reinforcing steel and the action of frost, or freezing-and-thawing cycles, in the presence of deicing salts. The City of Montreal's dry-mix shotcrete specification document for concrete repairs requires the use of inorganic corrosion inhibitors, commonly known as calcium nitrites, in air-entrained dry-mix shotcrete. These concrete admixtures are commercially available from most major concrete admixture manufacturers in North America.

A shotcrete rehabilitation condition survey conducted in Montreal in 2004-2005 revealed that most air-entrained dry-mix shotcrete used in the field with the addition of corrosion inhibitors produced significant increases in the compressive strength, but with variations in the air void distribution/spacing factor.¹

As a result of this survey, a test program was initiated to determine the effects of liquid corrosion inhibitors when added to the mixing water of air-entrained dry-mix shotcrete on various plastic and hardened properties. Parameters such as plastic consistency, set time, air void distribution, compressive strength, and freezing-and-thawing cycles in the presence of deicing salts were examined.

The test program was a joint venture between the City of Montreal Materials Laboratory, King Packaged Materials Company (a prepackaged shotcrete materials manufacturer), and Grace Canada Inc. (a manufacturer of corrosion inhibitors).

Test Program

The shotcrete mixture used was a typical air-entrained shotcrete mixture specified by the City of Montreal for vertical and overhead concrete repairs. It was supplied in 2200 lb (1000 kg) bulk bags by the King Packaged Materials Company. It was also the same mixture specified by the Ministry of Transportation of Quebec as referenced in a previous article.² It contained a powder air-entraining admixture preblended by weight of cementitious materials.

The tender specification documents called for a calcium nitrite-based corrosion inhibitor with $30 \pm 2\%$ solids. The current corrosion inhibitor specified dosage rate is 4 gal./yd³ (20 L/m³) of concrete (shotcrete).

Because it was observed on various job sites that the rate of calcium nitrite-based corrosion inhibitors influenced the concrete set time, it was suggested that two series of tests should be performed:

- DCI, regular corrosion inhibitor; and
- DCI-S, introduced in the market as a neutral corrosion inhibitor, with no effect on set times for a normal-density concrete.

Although the current dosage rate of corrosion inhibitor used was 4 gal./yd³ (20 L/m³), a second dosage rate of 2 gal./yd³ (10 L/m³) was also tested. This lower dosage rate is the minimum volume required, according to Grace's technical support, to obtain corrosion protection of the reinforcing steel and avoid the ring corrosion phenomena around the repaired section. The test program is detailed in Table 1.

Béton Projeté MAH Inc., a Montreal-based shotcrete contractor, was selected to shoot the test panels. An ACI certified nozzleman⁴ was used for the shoot. The equipment used was an Aliva 246 with a 3.6 L rotor, with a 1-1/2 in. (38 mm) inside hose diameter and a hydromix nozzle. In a hydromix nozzle,²⁻⁵ the mixing water was introduced through a water ring located approximately 10 ft (3 m) from the end of the nozzle tip. The panels were shot in a vertical position in accordance with the City of Montreal standard specifications³ at King Packaged Materials Company's yard in Blainville, QC, Canada (refer to Fig. 1).

The calcium nitrite content in the corrosion inhibitor solutions was determined by gravimetric analysis (evaporation of the liquid at 220 °F [105 °C] to obtain a constant weight). One liter containers were used for sampling, and a volume of 6.75 oz (200 mL) was used for testing.

Table 1: Test program of air-entrained dry-mix shotcrete with corrosion inhibitor

Mixtures	Control	DCI 2 gal./yd ³ (10 L/m ³)	DCI 4 gal./yd ³ (20 L/m ³)	DCI-S 2 gal./yd ³ (10 L/m ³)	DCI-S 4 gal./yd ³ (20 L/m ³)
Test panels	1	1	2	1	2
Plastic properties					
Air content (CSA A23.2-4C)	1	1	1	1	1
Density (CSA A23.2-6C)	1	1	1	1	1
Water content*	1	1	1	1	1
Plastic consistency [†]	1	1	1	1	1
Corrosion inhibitor—solid contents					
Corrosion inhibitor (CI)	0	0	1	0	1
Solution CI + water	1	1	1	1	1
Hardened properties					
Compressive strength cores (3VM-40)					
3 days	1	1	1	1	1
7 days	2	2	2	2	2
28 days	2	2	2	2	2
Air void distribution (ASTM C457)	1	1	1	1	1
Salt scaling resistance (NQ 2621-900) ³	0	0	2	0	1

* The water content was measured by sampling approximately 1 lb (0.5 kg) of fresh as-shot shotcrete and then evaporating it in an aluminum plate over a burner. The amount of water evaporated was then evaluated by weight.

[†] Portable penetrometer measuring plastic dry-mix shotcrete consistency. Penetration resistance measured using a modified tip of 0.35 in. (9 mm) in diameter.²

Test results

The shotcrete test results are presented in Table 2.

Results Analysis

Corrosion Inhibitor

Solid contents of 33.1 and 34.3% were measured from the original corrosion inhibitor containers of DCI and DCI-S, respectively, before being diluted in water at the specified dosage rates. These values are slightly above the values given on the manufacturer's technical data sheets and the City of Montreal's requirements of $30 \pm 2\%$.

The dosage rates of corrosion inhibitors in the mixing water were obtained based on the use of 39.6 gal. of water per cubic yard (196 L/m³) of shotcrete placed, assuming a water-cementitious



Fig. 1: ACI Certified Shotcrete Nozzleman—test panel shooting

Table 2: Shotcrete test results

Mixtures	Control	DCI 2 gal./yd ³ (10 L/m ³)	DCI 4 gal./yd ³ (20 L/m ³)	DCI-S 2 gal./yd ³ (10 L/m ³)	DCI-S 4 gal./yd ³ (20 L/m ³)
Plastic properties					
Temperature, °F (°C)	74.5 (23.6)	76.8 (24.9)	—	79.2 (26.2)	83.8 (28.8)
Air content, %	4.0	3.2	2.9	3.3	4.0
Density, lb/yd ³ (kg/m ³)	3860 (2294)	3919 (2325)	3897 (2312)	3914 (2322)	3966 (2353)
Water content, %	9.09	8.62	9.10	10.76	8.89
Penetration resistance— 5 minutes, psi (MPa)	299 (2.1)	256 (1.8)	>327 (>2.3)	>327 (>2.3)	>327 (>2.3)
Corrosion inhibitor solid content*					
Corrosion inhibitor, lb/gal. (g/L)	—	—	424 (3.54)	—	443 (3.7)
Corrosion inhibitor, solid contents, %	—	—	33.1	—	34.3
Solution CI + water, lb/gal. (g/L)	Trace (0.1)	0.18 (21.4)	0.32 (38.9)	0.20 (24.4)	0.35 (41.6)
Solution CI + water, solid contents, %	0.01	2.1	3.9	2.4	4.2
Solution CI + water, measured dosage rate, gal./yd ³ (L/m ³)	0 (0)	2.2 (11)	4 (20)	2.6 (13)	4.4 (22)
Hardened properties					
Compressive strength— 3 days, psi (MPa)	4120 (28.4)	5230 (36.1)	5600 (38.6)	5450 (37.6)	7090 (48.9)
7 days, psi (MPa)	5580 (38.5)	6470 (44.6)	7090 (48.9)	6710 (46.3)	8860 (61.1)
28 days, psi (MPa)	6510 (44.9)	8000 (55.2)	9060 (62.5)	8420 (58.1)	10,570 (72.9)
Air content, %	4.9	4.6	3.6, 3.1, 5.1 [†]	3.2	3.2
Air void spacing factor, in. (µm)	0.0098 (250)	0.0102 (260)	0.0094 (240) 0.0106 (270) 0.0126 (320) [†]	0.0087 (220)	0.0094 (240)
Salt scaling resistance— 56 cycles, lb/ft ² (kg/m ²)	—	—	0.43 (2.1)	—	0.39 (1.9)

*Density of corrosion inhibitor for solid contents calculation is 1.27.

[†]Test performed by a third-party laboratory.

Table 3: Percentage of compressive strength versus control mixture at 28 days

Mixtures	Control	DCI 2 gal./yd ³ (10 L/m ³)	DCI 4 gal./yd ³ (20 L/m ³)	DCI-S 2 gal./yd ³ (10 L/m ³)	DCI-S 4 gal./yd ³ (20 L/m ³)
Compressive strength—percent of the control at 28 days					
3 days, psi (MPa) [%]	4120 (28.4) [63]	5230 (36.1) [80]	5600 (38.6) [86]	5450 (37.6) [84]	7090 (48.9) [109]
7 days, psi (MPa)[%]	5580 (38.5) [86]	6470 (44.6) [99]	7090 (48.9) [109]	6710 (46.3) [103]	8860 (61.1) [136]
28 days, psi (MPa) [%]	6510 (44.9) [100]	8000 (55.2) [123]	9060 (62.5) [139]	8420 (58.1) [129]	10,570 (72.9) [162]

material ratio (*w/cm*) of approximately 0.40 and a cementitious content of 760 lb/yd³ (450 kg/m³):

- Dosage: 2 gal./yd³ (10 L/m³) of DCI and DCI-S
 - 13.2 gal. (50 L) of corrosion inhibitor to 264 gal. (1000 L) of water
 - As indicated in Table 2, the solid contents measured equals a field-measured dosage rate of 2.2 and 2.6 gal./yd³ (11 and 13 L/m³).
- Dosage: 4 gal./yd³ (20 L/m³) of DCI and DCI-S
 - 26.4 gal. (100 L) of corrosion inhibitor to 264 gal. (1000 L) of water
 - As indicated in Table 2, the solid contents measured equals a field-measured dosage rate of 4 and 4.4 gal./yd³ (20 and 22 L/m³).

Observations—Plastic Shotcrete

Regardless of the type and dosage of corrosion inhibitors used (DCI or DCI-S versus 2 or 4 gal./yd³ [10 or 20 L/m³]), the addition of calcium nitrite-based corrosion inhibitor significantly accelerated the set time of shotcrete. The penetrometer generally used to measure shotcrete consistency failed to differentiate the effect of dosage from the type of corrosion inhibitor. The maximum capacity of the penetrometer was reached within less than 5 minutes after the placement of shotcrete. As a result, surface finishing must be performed quickly after placement.

Characteristics of Hardened Shotcrete—Compressive Strength

Regardless of the type and dosage of corrosion inhibitors used (DCI or DCI-S versus 2 or 4 gal./yd³ [10 or 20 L/m³]), the addition of corrosion inhibitor increased the compressive strength of the shotcrete (refer to Table 3).

As presented in Table 3, the use of DCI increased the compressive strength at 28 days by 23% and 39% compressive strength at the respective dosage rates of 2 and 4 gal./yd³ (10 and 20 L/m³). The use

of DCI-S had the same effect with increases of 29% and 62% at 28 days at the respective dosage rates 2 and 4 gal./yd³ (10 and 20 L/m³).

Air Content and Spacing Factor of Hardened Concrete

The results shown in Table 2 indicate that as the dosage rate of corrosion inhibitors increased as the air content slightly decreased. For the respective dosages of 2 and 4 gal./yd³ (10 and 20 L/m³) of DCI, 0.3 and 1.3% air was lost. As for the DCI-S, 1.7% air was lost with both dosage rates.

In addition to air content, one of the most important performance criteria for durability remains the air-void distribution as per ASTM C457 for good frost resistance. The in-place, hardened shotcrete requires an average air-void spacing factor of less than 0.0118 in. (300 μm), with no individual results more than 0.0125 in. (320 μm).

The air void distribution analysis revealed that spacing factors ranged from 0.0087 to 0.0106 in. (220 to 270 μm), which met the City of Montreal's specification requirement for air-entrained dry-mix shotcrete. It seems that for the slight loss of air content experienced, the type and dosage of corrosion inhibitor did not impact the spacing factor significantly.

Salt Scaling Resistance (Freezing-and-Thawing Cycles in Presence of Deicing Salts)

The air-entrained control mixture was not tested for salt scaling resistance, as previous data have shown that it constantly meets the City of Montreal's specification requirement of a maximum allowable surface loss of 0.25 lb/ft² (1.2 kg/m²).

Test results of 0.43 and 0.39 lb/ft² (2.1 and 1.9 kg/m²) surface loss were obtained for mixtures containing 4 gal./yd³ (20 L/m³) of DCI and DCI-S,



Fig. 2: City of Montreal—overpass shotcrete repair

respectively. These results, however, mirror the performance of other accelerated, air-entrained, dry-mix shotcrete⁶ with respect to salt scaling resistance. Such accelerated shotcretes, however, are still used for repairs when faster set is required.

Unlike the salt scaling test where horizontal surfaces are critically saturated by the ponding, in the procedure used in this program, most of the shotcrete repairs for the City are generally oriented in either a vertical or overhead position (refer to Fig. 2). These orientations typically do not allow critical water saturation. It should also be noted that the City of Montreal's specifications for the repair of steel-reinforced concrete infrastructure (bridges, overpasses, and tunnels) require application of a pigmented membrane over the entire surface. The membrane acts as a sealant

against water and salt penetration and also provides color uniformity. Thus higher salt scaling surface losses of shotcrete when these types of corrosion inhibitors are used are not critical for such applications.

Conclusions

As stated at the beginning of this article, it was observed during the 2004-2005 field condition survey that most air-entrained dry-mix shotcretes used in the field with the addition of corrosion inhibitors produced significant increases in the compressive strength of the shotcrete but with variations in the air void distribution/spacing factor. Although this test program does not explain such variability, it could be attributed to the different parameters encountered on job sites, such as shotcrete equipment and accessories, volumetric batching systems versus prepackaged shotcrete materials, predampeners versus hydromix nozzles, and dosage rates of corrosion inhibitors.

In the field, however, it was observed that surface crazing was present on some corrosion inhibitor-enhanced shotcrete finished surfaces. Although the microcrack pattern (less than 0.0039 in. [100 μm] wide) pattern was not noticeable on dry surfaces, it could explain the cause of the merely modest performance of salt scaling resistance obtained in the laboratory.

In summary, when calcium nitrite-based corrosion inhibitors (DCI and DCI-S) are used in air-entrained dry-mix shotcrete, the effects on both shotcrete plastic and hardened properties should be considered when designing such mixtures for use in concrete rehabilitation. The following are the effects of the corrosion inhibitors on the properties of air-entrained dry-mix shotcretes:

- Accelerates the set time at the dosage rates tested in this program;
- Significantly increases the compressive strength at various curing times;
- Slightly reduces air content;
- Despite the slight loss of air content (in both plastic and hardened shotcrete), it does not negatively impact the quality of the air void system; that is, it does not increase spacing factors above the maximum allowable limit of 0.0118 in. (300 μm);
- Moderately reduces salt scaling resistance (resistance to freezing-and-thawing cycles in presence of deicing salts). In this respect, it is typical of other accelerated dry-mix shotcrete mixtures;
- The test program demonstrated that there were no notable differences between the corrosion inhibition type (DCI and DCI-S) and dosage rates (2 and 4 gal./yd³ [10 and 20 L/m³]) with respect to shotcrete plastic and hardened properties; and

Based on the results of this study, calcium nitrite-based corrosion inhibitors are specified by the City of Montreal for use in dry-mix shotcrete when corrosion protection is required.

Subsequent to this study, it was observed that when regular predampeners were used to predampen the dry-mix shotcrete materials (4 to 6% by mass moisture content),⁵ the corrosion inhibitor added to the mixing water used in such predampeners seems to increase variability on the shotcrete properties studied in this program. Further data indicated that less variability was obtained when hydromix nozzles were used as a means for dry-mix material predampening.

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Jean-François Dufour, MSc Eng, P Eng, is Technical Director for King Packaged Materials Company, a leading manufacturer of prepackaged shotcrete mixtures in Montreal, QC, Canada.

Most of his experience relates to the field of concrete and shotcrete technology in several disciplines such as new construction, rehabilitation, and mining industries. He received his master's degree in civil engineering from Laval University, Quebec, QC. He is a member of the American Shotcrete Association, ACI Committees 506, Shotcreting; C660, Shotcrete Nozzleman Certification; and the ACI Certification Programs Committee. Dufour is also certified as an ACI Certification Examiner.



Simon Reny, Eng, is a Technical Representative for King Packaged Materials Company. His areas of expertise include applications, mixture designs, rehabilitations and durability, new technologies, and equipment for both dry- and wet-mix methods. He received his degree in civil engineering from Laval University.



Pierre Lacroix, Eng, works for the Division of Expertise and Technical Support of the City of Montreal and specializes in concrete technology and aggregates. He is a graduate of the École Polytechnique de Montréal,

QC, Canada, in geological engineering applied to civil works, and received his master's degree from the same institution.



Richard Morin, Eng, is Head of the Civil Section for the Laboratories of the City of Montreal, QC, Canada, and is in charge of quality control for all civil construction works. He directs all quality control activities for the

construction and rehabilitation of roads, bridges, tunnels, building, and parks in the city of Montreal. He is also involved in the structural evaluation of pavements and expert consultation for civil works and buildings. Before joining the City of Montreal in 1990, he worked for 6 years with a private enterprise involved in consulting and quality control work. He received a degree in engineering in 1979 and his master's degree from the University of Sherbrooke, Sherbrooke, QC, Canada. He is member of ACI as well as a number of CSA and Quebec BNQ committees. As a representative of the City of Montreal, he is actively involved with the Center for Interuniversity Researches on Concrete and devotes time to technological transfer in the field of concrete.