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

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1.0 Introduction

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

A shotcrete rehabilitation condition survey 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 and 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 with the presence of de-icing salts would be examined.

The test program was a join venture between the City of Montreal's Materials Laboratory, King Packaged Materials Company (Pre-packaged shotcrete materials manufacturer) and Grace Canada Inc. (Manufacturer of corrosion inhibitors).

2.0 Test Program

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

The tender specification documents called for a calcium nitrite based corrosion inhibitor with 30 ± 2% solids. The current corrosion inhibitor specified dosage rate is 4 gal/cu.yd. (20 L/m^3) of concrete (shotcrete).

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

- DCI, regular corrosion inhibitor
- 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 is 4 gal/cu.yd. (20 L/m³), a second dosage rate of 2 gal/cu.yd. (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:

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

Mixtures	**Control**	**DCI** 2 gal/cu.yd (10 L/m³)	**DCI** 4 gal/cu.yd (20 L/m³)	**DCI-S** 2 gal/cu.yd (10 L/m³)	**DCI-S** 4 gal/cu.yd (20 L/m³)
- 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

Salts 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 evaporated in 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].

Béton Projeté MAH inc, a Montreal based shotcrete contractor, was selected to shoot the test panels. An ACI certified nozzleman[4] was used for the shoot. The equipment used was an Aliva 246 with a 3.6 L rotor, with a 1½ in. (38 mm) inside hose diameter and a hydro-mix nozzle. In a hydromix nozzle[2-5], the mixing water is introduced through a water ring located at approximately 10 ft (3 m) from end of the nozzle tip. The panels were shot on a vertical position in accordance with the City of Montreal standard specifications[3] at King Packaged Materials Company's yard in Blainville, Quebec, Canada (Fig. 1).

Fig.1: ACI Certified Shotcrete Nozzleman – Test Panel Shooting

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 ounces (200 ml) was used for testing.

3.0 Test results

The shotcrete test results are presented in Table 2.

Table 2: Shotcrete test results

Mixtures	**Control**	**DCI** 2 gal/cu.yd (10 L/m³)	**DCI** 4 gal/cu.yd (20 L/m³)	**DCI-S** 2 gal/cu.yd (10 L/m³)	**DCI-S** 4 gal/cu.yd (20 L/m³)
Plastic Properties					
Temperature °F (°C)	74.5 (23.6)	76.8 (24.9)	-	79.16 (26.2)	83.8 (28.8)
Air Content (%)	4.0	3.2	2.9	3.3	4.0
Density lb/cu.yd (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 min. 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)			3.54 (424)		3.7 (443)
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 Contens (%)	0.01	2.1	3.9	2.4	4.2
Solution CI+Water - Measured Dosage Rate gal/cu.yd (L/m³)	0	2.2 (11)	4 (20)	2.6 (13)	4.4 (22)
Hardened Properties					
Compressive Strength psi (MPa)					
3 days	4120 (28.4)	5230 (36.1)	5600 (38.6)	5450 (37.6)	7090 (48.9)
7 days	5580 (38.5)	6470 (44.6)	7090 (48.9)	6710 (46.3)	8860 (61.1)
28 days	6510 (44.9)	8000 (55.2)	9060 (62.5)	8420 (58.1)	10570 (72.9)
Air Content (%)	4.9	4.6	3.6 3.1 5.1**	3.2	3.2
Air Void Spacing Factor inch (µm)	0.0098 (250)	0.0102 (260)	0.0094 (240) 0.0106 (270) 0.0126** (320**)	0.0087 (220)	0.0094 (240)

Salts Scaling Resistance - 56 cycles lb/sq.ft (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

4.0 Results Analysis

4.1 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 ± 2%.

The dosage rates of corrosion inhibitors in the mixing water were obtained based on the use of 39.6 gallons of water per cubic yard (196 L/m^3) of shotcrete placed, assuming a water/cementitious ratio of approximately 0.40, and a cementitious content of 760 lb/cu.yd (450 kg/m^3):

- Dosage: 2 gal/cu.yd (10 L/m^3) of DCI and DCI-S
  - 13.2 gallons (50 L) of corrosion inhibitor to 264 gallons (1000 L) of water
  - As indicated in Table 2, the solid contents measured equals to a field measured dosage rate of 2.2 and 2.6 gal/cu.yd (11 and 13 L/m^3).

- Dosage: 4 gal/cu.yd (20 L/m^3) of DCI and DCI-S
  - 26.4 gallons (100 L) of corrosion Inhibitor to 264 gallons (1000 L) of water
  - As indicated in Table 2, the solid contents measured equals to a field measured dosage rate of 4 and 4.4 gal/cu.yd (20 and 22 L/m^3).

4.2 Observations - Plastic concrete

Regardless of the type and dosage of corrosion inhibitors used [DCI or DCI-S vs. 2 or 4 gal/cu.yd (10 or 20 L/m^3)], the addition of calcium nitrite based corrosion inhibitor seemed to significantly accelerate 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.

4.3 Characteristics - Hardened concrete

4.3.1 Compressive strength

Regardless of the type and dosage of corrosion inhibitors used [DCI or DCI-S vs. 2 or 4 gal/cu.yd (10 or 20 L/m^3)], the corrosion inhibitor seemed to increase the compressive strength of shotcrete (Table 3).

Table 3: Percentage of Compressive Strength vs. Control Mixture at 28 days

Mixtures	**Control**	**DCI** 2 gal/cu.yd (10 L/m³)	**DCI** 4 gal/cu.yd (20 L/m³)	**DCI-S** 2 gal/cu.yd (10 L/m³)	**DCI-S** 4 gal/cu.yd (20 L/m³)
Compressive Strength - % 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]***	10570 (72.9) ***[162]***

As presented in Table 3, the use of DCI increased the compressive strength at 28 days by 23% and 39% respectively. The use of DCI-S had the same effect with increases of 29% and 62% at the respective dosage rates [2 and 4 gal/cu.yd (10 and 20 L / m³)].

For both DCI's (DCI and DCI-S), the increase in dosage rates resulted also in an increase in compressive strength. From the lower to the higher dosage rates, there was a 13% increase at 28 days for the DCI and a 25% for the DCI-S.

4.3.2 Air content and spacing factor of hardened concrete

The results showed in Table 2 indicate that as the dosage of corrosion inhibitors increased, air content slightly decreased. For the respective dosages of DCI, 0.3 and 1.3% air was lost. As for the DCS-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 C 457. The in-place, hardened shotcrete requires an average air-void spacing factor of under 0.0118 inch (300 µm), with no individual results over 0.0125 inch (320 µm).

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

4.3.3 Salt scaling resistance (freeze-thaw cycles in presence of de-icing salts)

The air-entrained control mixture was not tested for salt scaling resistance, as previous data showed that it constantly meets the City of Montreal's specification requirement. It requires a maximum allowable surface loss of 0.25 lb/sq.ft (1.2 kg/m^2).

The results obtained of 0.43 and 0.39 lb/sq.ft (2.1 and 1.9 kg/m^2) on mixtures containing respectively 4 gal/cu.yd (20 L/m^3) of DCI and DCI-S, are above. These results however, mirror the performance of accelerated, air-entrained, dry-mix shotcrete[6] with respect to salt scaling resistance and are still used for repairs when a faster set is required.

Unlike the salt scaling test procedure used in this program, most of the shotcrete repairs for the City are generally oriented in vertical and overhead positions (Fig. 2). These orientations do not allow a degree of water saturation as important as concrete exposed in a horizontal position such as the test performed. It should also be noted that the City of Montreal's perspective for the repair of steel reinforced concrete infrastructures (bridges, overpasses, tunnels, etc.) is to apply a membrane over the entire surfaces that will act as both sealant and color uniformity. Thus higher salt scaling surface losses of shotcrete when these types of corrosion inhibitors are used are not critical in this case.

Fig. 2: City of Montreal - Overpass Shotcrete Repair

5.0 Conclusion

As stated in the Introduction, it was observed during the 2004-2005 condition survey that most air-entrained dry-mix shotcrete used in the field with the addition of corrosion inhibitors produced significant increases in the compressive strength of the shotcrete and 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 vs. pre-packaged shotcrete materials, pre-dampeners vs. hydro-mix nozzles, dosage rates of corrosion inhibitors, etc.

In the field, it was observed however that surface crazing was present on some corrosion inhibitor enhanced shotcrete finished surfaces. Although the micro-crack [less than 0.0039 inch (100 μm)] pattern was not noticeable on dry surfaces, it could explain the cause of the modest performance of salt scaling resistance obtained in 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 the design of such mixtures are used for concrete rehabilitation. The following are the effects of the corrosion inhibitors on air-entrained dry mix shotcrete mixes:

- Accelerates the set time at the dosage rates tested in the 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 impact the quality of the air void distribution, thus does not increase spacing factors above the maximum allowable limit of 0.0118 inch (300 μm);
- Moderately reduces salt scaling resistance (freeze-thaw cycles in presence of de-icing salts), typical of other accelerated dry-mix shotcrete mixes;
- The test program demonstrated that there was no notable difference between the type (DCI and DCI-S) and dosage rates [2 (10) and 4 gal/cu.yd (20 L/m^3)] with respect to shotcrete plastic and hardened properties.
- Based upon 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 (4-6% 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 mean for dry-mix materials predampening.

6.0 Acknowledgement

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7.0 References

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