## Aggregate Freezing-Thawing Performance Using the Iowa Pore Index

Final Report October 2016



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<ul> <li>The following results were observed:</li> <li>The aggregates with a non-carbonate origin outperformed the carbonate aggregates in all three tests.</li> <li>The Iowa pore index was found to correlate fairly well to the aggregate performance measured by the unconfined freezing-thawing test using ASTM C666 conditioning and the CSA A23.2-24A test. The correlation of the Iowa pore index to the ASTM C88 test was poor.</li> <li>The aggregates with high volumes of micropores performed poorly compared to the aggregates with low volumes of micropores. The correlation between the volume of micropores in an aggregate and the freezing-thawing performance was fairly strong.</li> </ul>					
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### AGGREGATE FREEZING-THAWING PERFORMANCE USING THE IOWA PORE INDEX

#### Final Report October 2016

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#### **EXECUTIVE SUMMARY**

In cold climate regions, the use of non-durable aggregate leads to premature pavement deterioration due to damage caused by freezing-thawing cycles. Repair of such distress is expensive, and agencies may sometimes end up replacing the damaged pavements. Differentiating durable and non-durable aggregates is a crucial yet challenging task. The frost durability of coarse aggregate has been reported to be related to its pore structure; however, existing test methods to identify pore structure are often not cost-effective. There is a need for a quick, reliable, and cost-effective aggregate test whose results correlate well with aggregate freezing-thawing performance.

The Iowa pore index test has been used by the Iowa Department of Transportation (DOT) for four decades as a supplemental decision-making tool. This study investigated the relationship between the Iowa pore index and the freezing-thawing performance of aggregates as measured by various methods. These methods included Canadian Standards Association (CSA) A23.2-24A, Test Method for the Resistance of Unconfined Coarse Aggregate to Freezing and Thawing; ASTM C88, Standard Test Method for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate; and an unconfined freezing-thawing test using conditioning according to ASTM C666, Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing.

In the experimental program, 15 carbonate and 12 non-carbonate aggregate sources from Minnesota were tested. The CSA A23.2-24A test included 16 hours of freezing at 0°F and 8 hours of thawing at room temperature as a full cycle. In the ASTM C88 test, one cycle involved immersion in a saturated sodium solution for 16 hours and oven drying until the sample achieved a constant mass. In the third method, which is based on ASTM C666 conditioning, unconfined aggregate samples were subjected to cycles of freezing from 40°F to 0°F and thawing from 0°F to 40°F in 4 hours.

The following observations were made:

- The aggregates with a non-carbonate origin outperformed the carbonate aggregates in all three tests.
- The Iowa pore index was found to correlate fairly well to aggregate performance as measured both by the unconfined freezing-thawing test using ASTM C666 conditioning and the CSA A23.2-24A test. The correlation of the Iowa pore index to the ASTM C88 test was found to be poor.
- The aggregates with high volumes of micropores performed poorly compared to the aggregates with low volumes of micropores. The correlation between the volume of micropores in aggregate and the freezing-thawing performance was found to be fairly strong.

#### PROBLEM STATEMENT AND SCOPE

Freezing-thawing or frost resistance of coarse aggregate significantly affects the durability of concrete pavement in cold climate regions, where high numbers of freezing-thawing cycles occur yearly. The use of non-durable aggregate leads to premature pavement deterioration, often referred to as D-cracking, which manifests itself as pop-outs, cracking, and spalling, particularly at the joints. Repair of such distress may be costly, and agencies sometimes end up replacing the damaged pavements early. For this reason, highway agencies usually specify strict limitations for aggregate (e.g., low absorption and limits on aggregates with questionable carbonate origins), though these restrictions also eliminate potentially well performing aggregate.

Differentiating durable and non-durable aggregate is a crucial yet challenging task. The frost durability of coarse aggregate has been reported to be related to its pore structure; however, existing test methods to identify pore structure are often not cost-effective. The Iowa pore index test has been used by the Iowa Department of Transportation (DOT) for four decades as a supplemental decision-making tool. While researchers have investigated this method, nonetheless a firm correlation between the parameters of the Iowa pore index test and aggregate performance has never been established.

There is a need for a quick, reliable, and cost-effective aggregate test whose results correlate well with aggregate freezing-thawing performance. The study described in this report was designed to analyze the relationship between the Iowa pore index test results and aggregate freezing-thawing performance as measured by three methods:

- Canadian Standards Association (CSA) A23.2-24A, Test Method for the Resistance of Unconfined Coarse Aggregate to Freezing and Thawing
- ASTM C88, Standard Test Method for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate
- An unconfined freezing-thawing test using conditioning according to ASTM C666, Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing

#### BACKGROUND

D-cracking was first discussed in the 1930s; the mechanisms of deterioration have been studied since then (Verbeck and Landgren 1960). D-cracking occurs when water in susceptible aggregate freezes and the resulting hydraulic pressure fractures the aggregate particle. It is noted that this process includes two aspects: first, the stress created by the freezing water inside the aggregate particle is large enough to disrupt the aggregate, and, second, the water expelled from the aggregate particle during freezing exerts a pressure in the surrounding cement paste at a rate that may cause cracking.

Aggregate-related freezing-thawing damage in concrete requires three conditions, as follows:

- Existence of non-durable aggregate
- Critical degree of saturation
- Freezing-thawing cycles

Naturally, aggregate characteristics control these conditions. Particle size, pore structure, absorption, mineralogy, and impurities directly or indirectly control the freezing-thawing performance of an aggregate.

Reducing the maximum aggregate size is known to limit or eliminate frost damage (Janssen and Snyder 1994). This is because when concrete is under a freezing condition, the unfrozen water in smaller aggregate particles is expelled quickly without developing damaging pressure.

Pore structure (i.e., pore size, pore shape, and pore distribution) has been identified as the most influential property that affects the durability of aggregates used as construction material. Pore structure not only affects strength but also determines absorption and permeability (Rhoades and Mielenz 1946). Pore structure also dictates whether an aggregate can become critically saturated in drained and undrained conditions and thus controls D-cracking susceptibility. Verbeck and Landgren (1960) classified aggregates based on pore structure in relation to freezing-thawing performance as follows:

- Low-permeability aggregates—these have a low porosity ( $\leq 0.3$  percent) and are strong enough to absorb the stress resulting from freezing water within their elastic limit.
- Intermediate-permeability aggregates—these contain a significant portion of small pores (i.e., ≤ 500 nanometers). The capillary forces in such small pores can cause the aggregates to become saturated easily. At a certain rate of freezing, water in the pores cannot move out and thus develops internal pressure high enough to fracture the aggregate particle.
- High-permeability aggregates—these mostly contain large pores, which permit easy water movement. During freezing water is expelled from aggregate without generating stress.

Aggregate absorption provides insight to permeability and pore structure to some degree. Low absorption is a sign of low permeability, and aggregates with such characteristics generally perform well. High absorption may or may not indicate that an aggregate is freezing-thawing

resistant. If the pore structure consists mostly of large pores, water can move in and out easily, and the aggregate is potentially sound under freezing-thawing conditions. If the pore structure consists mostly of fine pores, which absorb water quickly but dry out slowly, then the aggregate is likely to have durability problems (Verbeck and Landgren 1960).

While igneous (e.g., basalt or granite) and metamorphic (e.g., gneiss or quartzite) rocks perform well in terms of freezing-thawing durability, many sedimentary rocks are problematic. Most aggregates susceptible to D-cracking are composed of limestone, dolomite, or chert (Stark 1976). The presence of deicing salts exacerbates the potential for D-cracking for certain carbonate aggregates (Dubberke and Marks 1985).

#### TEST METHODS FOR FREEZING-THAWING SUSCEPTIBILTY/PERFORMANCE

As in most durability testing, replicating the field conditions for freezing-thawing is challenging. Numerous methods have been proposed, and a significant number of these have been standardized. Some of these test methods directly measure aggregate performance in a simulated freezing-thawing environment, whereas some evaluate the aggregate indirectly by relating pore structure to performance. The most commonly used test methods are summarized in this chapter.

### Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing (ASTM C666)

In ASTM C666, the aggregate in question is tested in a concrete mix. Concrete beams (e.g.,  $4 \times 3 \times 16$  in.) are subjected to freezing-thawing cycles between 40°F and 0°F. Two different protocols can be used: Procedure A, where specimens are kept in water during both freezing and thawing, and Procedure B, where specimens are frozen in air and thawed in water. Material loss and durability factor are used as measures of performance. Length change may also be used. This method is probably the most well-known and widely used test. It measures coarse aggregate performance in concrete under freezing-thawing conditions. The procedure may take several months and is often criticized as subjecting aggregates to conditions harsher than real field conditions.

### Standard Test Method for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (ASTM C88)

In ASTM C88, aggregate is immersed in a prepared solution of sodium sulfate  $(Na_2SO_4)$  or magnesium sulfate  $(MgSO_4)$  for 16 to 18 hours at room temperature, then oven dried to constant mass, and finally cooled to room temperature before the next immersion. When the cycles are completed (often five cycles), the aggregate is washed by circulating water at 110°F. After drying, the percentage mass loss is calculated as a measure of soundness. In this test method, the expansion of crystallizing salt is used to simulate water freezing. This procedure is easy to run, directly tests aggregates, and requires little sample preparation and test equipment.

#### Washington Hydraulic Fracture

The Washington Hydraulic Fracture test was developed by Janssen and Snyder (1994). It simulates the effects of freezing-thawing cycles on saturated aggregate particles by forcing water into and out of the pore structure of dried aggregate particles in a water-filled pressure vessel. The water is forced into the aggregate pores using a pressurized nitrogen source, and rapid release of the pressure allows compressed air trapped within the aggregate pores to expand, expelling water from the aggregate and creating internal stresses similar to those produced during freezing and thawing. Aggregate fracture occurs when the aggregate pore structure does not allow rapid dissipation of the pore pressures and the aggregate particles are relatively weak. The amount of fracturing that results from this test has been shown to be an indicator of the freezing-thawing susceptibility of an aggregate (Embacher and Snyder 2003). The procedure

takes eight days and is inexpensive to run compared to ASTM C666. However, the test is highly sensitive to pressure release (Issa et al. 1999).

### Standard Method of Test for Soundness of Aggregates by Freezing and Thawing (AASHTO T 103-08)

ASTM T 103 is a combination of the methods described above in this chapter. Clean aggregate samples are soaked with various fluids, depending on the procedure, and subjected to freezing-thawing cycles. There are three different freezing-thawing procedures: Procedures A, B, and C. In Procedure A, aggregate is soaked with water for 24 hours, and 50 cycles of freezing at -9°F and thawing at 70°F are run. In Procedure B, aggregate is soaked with an alcohol-water solution under pressure, and 16 cycles of freezing-thawing are run. Procedure C involves vacuuming the aggregate with water and running 25 freezing-thawing cycles.

### Test Method for the Resistance of Unconfined Coarse Aggregate to Freezing and Thawing (CSA A23.2-24A)

In CSA A23.2-24A, aggregate samples are placed in separate plastic containers filled with 3 percent by mass of sodium chloride (NaCl) solution. After soaking 24 hours at room temperature, the aggregate samples are transferred to a freezer at -0.4°F for 16 hours, then thawed for 8 hours at room temperature. After five cycles of freezing and thawing, the aggregate samples are washed with tap water and oven dried to a constant mass. The percentage mass loss for each aggregate sample due to freezing-thawing cycles is used as a measure of freezing-thawing performance. The test is reported to have better correlation with field performance than other tests (Mummaneni and Riding 2012).

### Test for Thermal and Weathering Properties of Aggregates: Determination of Resistance to Freezing and Thawing with/without Salt (NT BUILD 485 - Edition 2)

NT BUILD 485 - Edition 2 (Nordic Innovation Center 2004) is a variant of CSA A23.2-24A. Aggregate is soaked in either pure water or 1 percent NaCl solution for 24 hours prior to testing. The aggregate then is cooled from 68°F to 32°F over a period of 150 minutes. The aggregate is maintained at 32°F for 210 minutes, and the temperature is further reduced to 0°F over a period of 180 minutes. After the freezing regime, the samples are thawed at 68°F for 10 hours. The freezing-thawing cycle is repeated 10 times. The aggregate is washed and oven dried, and the percentage mass loss is calculated as a measure of freezing-thawing performance.

#### **Iowa Pore Index**

Iowa DOT developed a simple aggregate test in the 1970s in order to identify the D-cracking potential of coarse aggregates, particularly limestone aggregates. For the Iowa pore index test, water is pushed into 1/2 to 3/4 in. aggregates in a sealed system (Figure 1) under a pressure of 35 psi for 15 minutes.



Figure 1. Iowa pore index apparatus

The amount of water that enters the aggregate in the first minute is called the primary load, and the amount of water that enters the aggregate in the following 14 minutes is called the secondary load, or pore index. The primary load reflects the quantity of large voids (or macropores), while the secondary load reflects the quantity of small voids (or micropores) in the tested aggregate. It is believed that the micropores are closely associated with the aggregate's freezing-thawing durability. Aggregate having a secondary load greater than 27 is believed to have poor freezing-thawing durability. The test is quick and simple.

After testing aggregates with 10 or more years of service life, Myers and Dubberke (1980) concluded that the pore index test is sufficiently reliable for determining the D-cracking potential of limestone aggregates in all but a few cases in which marginal results are obtained. Several other states have evaluated the test and found a strong correlation between Iowa pore index and the field performance of aggregates (Thompson et al. 1980, Shakoor and Scholer 1985, Koubaa and Snyder 1996).

#### MATERIALS AND METHODS

The experimental program was designed as the continuation of a previous project in which concrete aggregates from Minnesota were tested (Bektas et al. 2015). The same aggregate batches were used for this project. Samples included 12 crushed gravels and 3 manufactured limestone gravels. The Iowa pore index test utilizes 1/2 to 3/4 in. particles; these same aggregate sizes were used in this experimental program. The aggregates were washed and dried. The gravel aggregates were then separated into carbonate and non-carbonate groups. The sorting process included the following three steps:

- Whitish/light-colored particles, possible carbonates, were separated visually (Figure 2, left and middle).
- Whitish/light-colored particles were first subjected to a hardness test using a steel knife; carbonate is a soft mineral and a steel blade can easily scratch the rock.
- Particles that could not be sorted by the scratch test were subjected to further testing, i.e., the fizz test. A weak acidic solution makes carbonates bubble and fizz because of the release of carbon dioxide as the carbonate dissolves. A 10 percent hydrochloric acid solution was used for the fizz test (Figure 2, right).



Bektas et al. 2015

# Figure 2. Aggregates separated into carbonate and non-carbonate groups: bulk aggregate sample (left), light-colored particles of suspected carbonate origin (middle), and fizz test (right)

Three tests were performed to evaluate the freezing-thawing performance of the aggregates:

- CSA A23.2-24A
- ASTM C88
- Unconfined freezing-thawing test using ASTM C666 conditioning

For CSA A23.2-24A, the aggregates were first washed and oven dried. Then, 2,500 g samples of aggregate were immersed in 3 percent NaCl solution in individual containers (Figure 3).



Figure 3. Aggregates stored in individual containers during testing

The aggregates were stored with the lid on the container at room temperature for  $24\pm 2$  hours. After the soaking period, the solution was drained from each container by rapidly inverting the container over a #4 sieve. The containers were then transferred to the freezer and conditioned for  $16\pm 2$  hours at 0°F. The samples were thawed at room temperature for  $8\pm 1$  hours. After five cycles, the aggregate was thoroughly washed with fresh water (Figure 4) and oven dried to constant mass.



Figure 4. Aggregates thoroughly washed over sieve after conditioning

The aggregate was sieved over a 1/2-inch sieve for three minutes and weighed to determine mass loss. The procedure was repeated for another five cycles, and the mass loss after the 10th cycle was determined.

For ASTM C88, 1,000 g of washed and oven dried aggregate was immersed in a sodium sulfate  $(Na_2SO_4)$  solution prepared using anhydrous sodium sulfate. The aggregates were completely covered by the solution to a depth of at least  $\frac{1}{2}$  in. The container was covered to prevent evaporation and stored at  $70\pm2^{\circ}F$  for 16 to 18 hours. After the immersion period, the aggregate sample was removed from the solution, permitted to drain for about 15 minutes, and placed in the oven. The aggregate then was dried to constant weight. The immersion-drying process was repeated for five cycles. After the completion of the fifth cycle, the aggregate was cooled and washed until it was free from the sodium sulfate, as determined by the reaction of the wash water with barium chloride (BaCl<sub>2</sub>). The samples were then dried and sieved over a 3/8-inch sieve by hand. The mass loss was calculated as the performance measure.

For the third performance test, approximately 2,000 g of aggregate from each source was tested. The aggregate samples were placed in containers in which they were completely covered with water. The aggregate containers were then placed in a chamber specified by ASTM C666. The nominal freezing-thawing cycle of this test method consisted of alternately lowering the temperature from 40°F to 0°F and raising it from 0°F to 40°F over 4 hours. After 50 freezing-thawing cycles, the aggregate was dried and sieved and mass loss was calculated. The samples were subjected to another 50 freezing-thawing cycles, and the mass loss was calculated after the 100th cycle.

#### RESULTS

Tables 1 through 3 give the results obtained from the unconfined freezing-thawing test using ASTM C666 conditioning, the ASTM C88 test, and the CSA A23.2-24A test, respectively. In the Aggregate ID column, a C or N following the dash indicates carbonate or non-carbonate origin, respectively. Additionally, Table 4 summarizes some aggregate characteristics (i.e., Iowa pore index, level of moisture absorption, and the amount of 0.1 to 1  $\mu$ m pores according to mercury intrusion porosimetry) that were obtained in a previous project (Bektas et al. 2015).

Aggregate		Mass, g			Mass loss, %		
ĪD	Initial	50th cycle	100th cycle	50th cycle	100th cycle		
A-C	2033	1866	1790	8.2	12.0		
B-C	2043	1835	1676	10.2	18.0		
C-C	2109	1922	1841	8.9	12.7		
D-C	1938	1706	1617	12.0	16.5		
E-C	2159	1878	1836	13.0	15.0		
F-C	1947	1752	1619	10.0	16.9		
G-C	2080	1968	1840	5.4	11.5		
H-C	2109	1888	1856	10.5	12.0		
I-C	2135	1963	1874	8.0	12.2		
J-C	2028	1675	1663	17.4	18.0		
K-C	2041	1856	1766	9.1	13.5		
L-C	1911	1860	1814	2.7	5.1		
M-C	1998	1786	1726	10.6	13.6		
N-C	1971	1777	1731	9.8	12.2		
O-C	2158	1898	1778	12.1	17.6		
A-N	2104	2077	2069	1.3	1.7		
B-N	2123	2077	2065	2.2	2.8		
C-N	2203	2075	2070	5.8	6.0		
D-N	2103	2002	1975	4.8	6.1		
E-N	2103	2051	2023	2.5	3.8		
F-N	2052	1789	1753	12.8	14.6		
G-N	1978	1815	1800	8.3	9.0		
H-N	2105	2061	2058	2.1	2.2		
I-N	2087	2022	1965	3.1	5.8		
K-N	2058	1981	1967	3.8	4.4		
M-N	2159	2074	2048	3.9	5.2		
O-N	2067	1995	1966	3.5	4.9		

Table 1. Results of the unconfined freezing-thawing test using ASTM C666 conditioning

Aggregate	Mass, g		Mass loss, %	
ID	Initial	5th cycle	5th cycle	
A-C	1001	985	1.6	
B-C	1002	994	0.8	
C-C	1000	995	0.6	
D-C	702	684	2.6	
E-C	n/a	n/a	-	
F-C	991	975	1.6	
G-C	1002	995	0.6	
H-C	1001	992	1.0	
I-C	1000	946	5.5	
J-C	999	980	1.9	
K-C	522	510	2.2	
L-C	1001	996	0.6	
M-C	1002	997	0.5	
N-C	1000	993	0.7	
O-C	1002	984	1.8	
A-N	1002	1001	0.0	
B-N	1003	999	0.4	
C-N	1000	999	0.1	
D-N	1000	997	0.3	
E-N	1000	1000	0.0	
F-N	1001	995	0.6	
G-N	1003	985	1.7	
H-N	1000	999	0.1	
I-N	1001	1000	0.1	
K-N	1000	998	0.2	
M-N	1000	998	0.1	
O-N	1001	995	0.6	

 Table 2. Results of ASTM C88

Aggregate	Mass, g			Mass loss, %		
ID	Initial	5th cycle	10th cycle	5th cycle	10th cycle	
A-C	2498	2331	2292	6.7	8.3	
B-C	2499	2271	2241	9.1	10.3	
C-C	2502	2275	2220	9.1	11.3	
D-C	1250	1190	1123	4.8	10.2	
E-C	1251	1193	1114	4.7	10.9	
F-C	2251	2028	1884	9.9	16.3	
G-C	1250	1145	1102	8.3	11.8	
H-C	1002	952	900	5.0	10.2	
I-C	2500	2437	2399	2.5	4.0	
J-C	2500	2342	2191	6.3	12.4	
K-C	1251	1197	1147	4.3	8.3	
L-C	2502	2296	2238	8.2	10.5	
M-C	2501	2422	2360	3.2	5.6	
N-C	2499	2361	2314	5.5	7.4	
O-C	2499	2326	2298	6.9	8.0	
A-N	2503	2459	2418	1.7	3.4	
B-N	2501	2475	2420	1.0	3.2	
C-N	2502	2473	2408	1.2	3.8	
D-N	2500	2442	2354	2.3	5.8	
E-N	2501	2407	2273	3.8	9.1	
F-N	2498	2432	2306	2.7	7.7	
G-N	2502	2433	2360	2.8	5.7	
H-N	2502	2482	2350	0.8	6.1	
I-N	2502	2450	2439	2.1	2.5	
K-N	2502	2419	2345	3.3	6.3	
M-N	2501	2461	2405	1.6	3.8	
O-N	2501	2382	2321	4.8	7.2	

Table 3. Results of CSA A23.2-24A

Aggregate	Absorption,	Iowa	0.1–1 µm pores,
ID	%	<b>Pore Index</b>	% volume
A-C	1.87	35	0.0119
B-C	1.89	31	0.0125
C-C	1.84	31	0.0125
D-C	2.69	21	0.0141
E-C	2.66	33	0.0136
F-C	2.35	32	0.0134
G-C	1.85	29	0.0129
H-C	1.91	31	0.0132
I-C	2.54	23	0.0153
J-C	3.20	53	0.0166
K-C	2.07	32	0.0141
L-C	1.02	18	0.0068
M-C	2.28	27	0.0143
N-C	1.47	22	0.0060
O-C	2.49	29	0.0161
A-N	0.65	7	0.0009
B-N	0.52	7	0.0016
C-N	0.54	7	0.0031
D-N	1.01	17	0.0022
E-N	1.04	18	0.0030
F-N	0.74	9	0.0016
G-N	0.72	11	0.0022
H-N	0.51	5	0.0012
I-N	1.38	17	0.0081
K-N	0.87	14	0.0030
M-N	0.89	15	0.0032
O-N	1.00	15	0.0017

**Table 4. Aggregate characteristics** 

Source: Bektas et al. 2015

All three performance test results show a clear distinction between the carbonate and noncarbonate aggregates, with few exceptions: in general, non-carbonate aggregates performed better than carbonate aggregates. Examples of aggregate deterioration can be seen in Figure 5.



Figure 5. Fraying after freezing-thawing cycles in the CSA A23.2-24A test (1/2 inch aggregate particles)

The average mass loss values for the carbonate and non-carbonate aggregates in the unconfined freezing-thawing test using ASTM C666 conditioning were 9.9 percent and 4.5 percent after 50 cycles, respectively. The t-test shows that this difference is extremely statistically significant. Aggregate L-C, which experienced a comparably low mass loss (i.e., 2.7 percent), and aggregate F-N, which experienced a comparably high mass loss (12.8 percent), were the outliers. The performance of aggregate L-C can be attributed to its good pore characteristics, namely its low water absorption and low Iowa pore index value. On the other hand, the poor performance of F-N cannot be explained other than as a testing anomaly.

The results of the ASTM C88 test also differentiate the carbonate and non-carbonate aggregates. The average mass loss values were 1.6 percent and 0.4 percent for the carbonate and non-carbonate aggregates, respectively. The t-test shows that this difference is very statistically different. The values seem unconventionally low, particularly for the carbonate aggregate; there might have been a procedural error during testing. Nonetheless, the values can be used for comparison purposes.

As in the other two performance tests, the carbonate aggregates performed poorly compared to the non-carbonate aggregates in the CSA A23.2-24A test. The average mass loss values after five cycles were 6.3 percent and 2.3 percent for the carbonate and non-carbonate aggregates, respectively. Based on the t-test, this difference is extremely statistically different.

In the following sections, aggregate freezing-thawing performance is based on the mass loss after 50, 5, and 5 freezing-thawing cycles in the unconfined freezing-thawing test using ASTM C666 conditioning, the ASTM C88 test, and the CSA A23.2-24A test, respectively. The main objective of this part of the study was to investigate the relationship between aggregate freezing-thawing performance and Iowa pore index. Figures 6 through 8 show the correlations between the Iowa pore index and the different performance tests used in this study.



Figure 6. Unconfined freezing-thawing test using ASTM C666 conditioning versus Iowa pore index



Figure 7. ASTM C88 versus Iowa pore index



Figure 8. CSA A23.2-24A versus Iowa pore index

There is a fairly good correlation between Iowa pore index and the test using ASTM C666 conditioning, and a similar finding is true for CSA A23.2-24A. Although the linear regression is poor between the Iowa pore index and ASTM C88, the general trend is clear: as the Iowa pore index increases, mass loss increases. If an Iowa pore index of 27 is considered to indicate that an aggregate will not experience D-cracking, a mass loss of 8 percent (Figure 6) and 5 percent (Figure 8) can be recommended as limits for the unconfined freezing-thawing test using ASTM C666 conditioning and the CSA A23.2-24A test, respectively.

Pore size distribution has been reported to relate to aggregate freezing-thawing performance. Larger pore volumes and smaller pore sizes lead to poor freezing-thawing durability (Rhoades and Mielenz 1946, Verbeck and Landgren 1960). Marks and Dubberke (1982) found that aggregates associated with D-cracking exhibit a predominance of pore sizes that range from 0.04 to 0.20  $\mu$ m in diameter, and the Iowa pore index test was very effective in identifying those problematic aggregates. The relationship between the Iowa pore index and the quantity of micropores is plotted in Figure 9.



Figure 9. Relationship between micropores and Iowa pore index

A strong correlation is observed: non-carbonate aggregates having low volumes of micropores also have low Iowa pore index numbers. The relationship between the freezing-thawing performance test results and micropore volume is given in Figures 10 through 12. It can be seen that there is a fairly good correlation. This finding confirms the results of previous research.



Figure 10. Results of unconfined freezing-thawing test using ASTM C666 conditioning versus the amount of micropores



Figure 11. Results of ASTM C88 versus the amount of micropores



Figure 12. Results of CSA A23.2-24A versus the amount of micropores

#### CONCLUSION

In this experimental program, three aggregate freezing-thawing performance tests were performed on 15 carbonate and 12 non-carbonate aggregates. The main objective was to investigate the relationship between these freezing-thawing performance tests and Iowa pore index. The following observations can be made as a result of this research:

- There was a clear difference in performance between the carbonate and non-carbonate aggregates: the non-carbonate aggregates outperformed the carbonate aggregates in all three tests.
- The Iowa pore index was found to correlate fairly well to aggregate performance as measured by the unconfined freezing-thawing test using ASTM C666 conditioning and by the CSA A23.2-24A test. The correlation between Iowa pore index and the ASTM C88 test was poor.
- There was a fairly strong correlation between the volume of micropores in an aggregate and the aggregate's freezing-thawing performance. The aggregates with a high volume of micropores performed poorly compared to the aggregates with a low volume of micropores.

#### REFERENCES

- Bektas, F., K. Wang, and J. Ren. 2015. *Carbonate Aggregate in Concrete*. Report MN/RC 2015-14. Minnesota: Minnesota Department of Transportation, St. Paul, MN.
- Dubberke, W. and V. J. Marks. 1985. The Effect of Salt on Aggregate Durability. *Transportation Research Record: Journal of the Transportation Research Board*, No. 1031, pp. 27–34.
- Embacher, R. A. and M. B. Snyder. 2003. *Refinement and Validation of The Hydraulic Fracture Test.* Report MN-RC 2003-28. Minnesota Department of Transportation, St. Paul, MN.
- Janssen, D. J. and M. B. Snyder. 1994. *Resistance of Concrete to Freezing and Thawing*. Report SHRP-C-391. Strategic Highway Research Program, Washington, DC.
- Issa, M. A., M. A. Issa, and M. Bendok. 1999. Evaluation of Washington Hydraulic Fracture Test (SHRP) for D-Cracking Aggregate. Report ITRC FR 95/96-5. Illinois Transportation Research Center, Edwardsville, IL.
- Kaneuji, M. 1978. Correlation between Pore Size Distribution and Freeze-Thaw Durability of Coarse Aggregate in Concrete. Report JHRP-78-15. Joint Highway Research Project, Indiana Department of Transportation and Purdue University, West Lafayette, IN.
- Koubaa, A., and M. B. Snyder. 1996. Evaluation of Frost Resistance Tests for Carbonate Aggregates. *Transportation Research Record: Journal of the Transportation Research Board*, No. 1547, pp. 35–45.
- Marks, V. J., and W. Dubberke. 1982. Durability of Concrete and the Iowa Pore Index. *Transportation Research Record: Journal of the Transportation Research Board*, No. 853, pp. 25–30.
- Mummaneni, S. K. and K. A. Riding. 2012. *Evaluation of Canadian Unconfined Aggregate Freeze-Thaw Tests for Identifying Nondurable Aggregates*. Report K-TRAN: KSU-10-9. Kansas State University Transportation Center and Kansas Department of Transportation, Topeka, KS.
- Myers, J. D. and W. Dubberke. 1980. *Iowa Pore Index Test*. Interim Report. Iowa Department of Transportation, Highway Division, Office of Materials, Ames, IA.
- Nordic Innovation Center. 2004. Test Method for Thermal and Weathering Properties of Aggregates: Determination of Resistance to Freezing and Thawing with/without Salt. NT BUILD 485 - Edition 2. Nordic Innovation Center, Oslo, Norway.
- Rhoades, R. and R. C. Mielenz. 1946. Petrography of Concrete Aggregate. *Proceedings of American Concrete Institute*, Vol. 42, No. 6, pp. 581–600.
- Shakoor, A., and C. F. Scholer. 1985. Comparison of Aggregate Pore Characteristics as Measured by Mercury Intrusion Porosimeter and Iowa Pore Index Tests. ACI Materials Journal, Vol. 82, No. 4, pp. 453–458.
- Stark, D. 1976. *Characteristics and Utilization of Coarse Aggregates Associated with D-Cracking*. Report RD047.01P. Portland Cement Association, Skokie, IL.
- Thompson, S. R., M. P. Olsen, and B. J. Dempsey. 1980. *D-Cracking in Portland Cement Concrete Pavements*. RSynthesis eport UILI-ENG-80-2014. Illinois Cooperative Highway and Transportation Research Program, University of Illinois, Urbana, IL.
- Verbeck, G. and R. Landgren. 1960. Influence of Physical Characteristics of Aggregates on Frost Resistance of Concrete. *Proceedings of the American Society for Testing Materials*, Vol. 60, pp. 1063–1079.