Freeze-Thaw and Scaling Tests on Silicone-Treated Concrete

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Laboratory tests of freeze-thaw durability and scaling resistance of concretes treated with silicone water repellents indicated that the silicone gave no appreciable benefit to air-entrained concrete; a slight improvement was noted in the durability and scaling resistance of silicone-treated non-air-entrained concrete.

• IN 1957, the Ontario Department of Highways started using a sprayed silicone water repellent as a protective coating on new structures, both on exposed surfaces and on deck sections which were subsequently covered with asphalt. The use of silicones was introduced after the reported success with these materials on the New York Thruway (1) where it was found that the silicone-treated concrete had greater resistance to salt scaling and improved light reflectance.

When used as a protective coating, silicone is in the form of a resin derived from alkylchlorosilanes and dissolved in a hydrocarbon solvent, or, alternatively, a water-soluble sodium siliconate may be used. In either case, the liquid is brushed or sprayed onto the surface, the silicone reacts with the surface, and the solvent evaporates leaving a water-repellent surface.

This reaction produces a surface that is water repellent but not sealed against water penetration. Because there were very few data available to indicate what degree of protection from freeze-thaw and scaling action might be expected, the Materials and Research Division undertook a series of laboratory tests to compare the freeze-thaw and scaling resistance of silicone-treated and untreated concrete. Several different testing and curing procedures were used in an attempt to simulate different field conditions, but the effectiveness of silicone in preventing moisture penetration beneath asphalts was not investigated.

The tests described in this paper were started in 1958 and completed early in 1960.

SILICONES TESTED

No attempt was made to evaluate individual commercial silicone products. Two materials were used in the tests: (a) the silicone resin type in a hydrocarbon solvent and (b) a water-soluble sodium siliconate. The silicone resin in hydrocarbon solvent (silicone 1) according to the manufacturer's data sheet contains 5 to 8 percent undiluted silicone resin. The water-soluble sodium siliconate (silicone 2), supplied concentrated, contains according to the manufacturer's data sheet 30 percent total solids and 20.5 percent silicone.

The percentage of solids in the samples was 4.8 percent for silicone 1 and 32.8 percent for silicone 2.

Silicone 1 was applied as supplied by the manufacturer. Silicone 2 was diluted 1 part to 12.4 parts of water by volume, giving a 2.8 percent solids solution assuming 30 percent solids in the concentrate, or 3.1 percent solids taking 32.8 percent solids in the concentrate.

The materials were applied by brush according to the manufacturer's instructions, sufficient material being brushed onto the surface of each specimen to give a coverage

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TABLE 1

CONCRETE STONE AND SAND SPECIFICATIONS

Stone		Sand	
Sieve Size	Cumulative % Passing	Sieve Size	Cumulative % Passing
1-in.	100	No. 4	100
¾-in.	90-100	No. 8	80-100
⅝-in.	65-90	No. 14	50-85
⅜-in.	20-55	No. 28	25-60
No. 4	0-10	No. 48	5-30
		No. 100	0-10
		No. 200	0-3

TABLE 2

CURING PROCEDURES

Curing Procedure	Moist Curing (days)	Dry Curing (days)	Time of Silicone Application (days)
1	28	-	28
2	21	14	35
3	35	-	28
4	21	21	35

of approximately 180 sq ft per imperial gallon. In all cases, this was more solution than could be readily absorbed by the surface, although dry surfaces absorbed the solution more readily than wet ones.

FREEZE-THAW TESTS

Materials Tested

Normal portland cement was used. Two combinations of aggregate from Ontario were used: (a) crushed gravel (Brighton) and natural sand (Caledon), and (b) crushed rock (Hagersville) and natural sand (Paris). The air-entraining agent was neutralized Vinsol resin. Results of routine tests on these materials are given in the Appendix. Both silicone 1 and 2 were used.

Fabrication of Specimens

The aggregates were graded to meet the Department's specifications for ¾-in. concrete stone and concrete sand (Table 1). All mixes were proportioned to produce concrete containing approximately 6 bags of cement per cu yd of concrete (based on Canadian bag of cement = 87.5 lb). The sand contents were varied slightly according to the air content anticipated and the water contents were adjusted to give 3 ± ½-in. slump in all cases.

Nine 3- by 4- by 16-in. beams were made from each mix: three to receive no silicone treatment (treatment A), three to be coated with silicone 1 (treatment B), and three to be coated with silicone 2 (treatment C). Each group of three beams was compacted by vibrating for 15 sec with an external vibrator. The beams were removed from the molds 24 hr after placing.

Curing

Four different curing procedures were used (Table 2) involving different periods of moist curing at 72 F and 100 percent relative humidity, and air drying in the laboratory. Testing commenced immediately on completion of the curing period indicated.

Testing Procedure

Two test procedures were used: (a) freezing and thawing in water, and (b) freezing in air and thawing in water. The beams were subjected to freezing and thawing in an automatic freeze-thaw machine, giving 8 cycles of freezing and thawing per day between 0 F and 40 F. The procedures used were substantially in agreement with ASTM methods C290-57T, "Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water," and C291-57T, "Tentative Method of Test for Resistance of Concrete Specimen to Rapid Freezing in Air and Thawing in Water."

From time to time (usually about every 24 cycles) the beams were removed from the freeze-thaw machine during a thawing cycle and were tested for change in weight, length, and relative dynamic modulus of elasticity. Testing continued for 300 cycles or until the beams disintegrated.

TABLE 3

FREEZE-THAW MIXES AND DURABILITY FACTORS

MIX NO.	DATE MADE	BATCH PROPORTIONS: CEMENT	C.A.	F.A.	WATER	A.E.A.	TEST RESULTS: SLUMP	AIR CONTENT	CEMENT FACTOR	W/C RATIO	CURING PROCEDURE	TESTING PROCEDURE	SILICONE APPLIED	SILICONE TREATMENT	DURABILITY FACTOR
—	d.m.y.	lb.	lb.	lb.	lb.	c.c.	inch	%	bags/cu.yd.	—	—	—	days	—	—
10	3.3.58	29.1	105.4	76.4	15.0	—	$3\frac{1}{2}$	—	N.T.	0.49	1	1	—	A	N.T.
													28	B	N.T.
													28	C	N.T.
11	6.3.58	29.1	105.4	76.4	15.0	—	3	—	N.T	0.49	2	1	—	A	N.T
													35	B	N.T
													35	C	N.T
12	6.3.58	29.1	105.4	71.5	13.6	5.5	$2\frac{7}{8}$	5.0	6.03	0.44	1	1	—	A	N.T
													28	B	N.T
													28	C	N.T
13	10.3.58	29.1	105.4	71.5	13.6	5.5	$2\frac{1}{2}$	5.0	6.12	0.44	2	1	—	A	N.T
													35	B	N.T
													35	C	N.T
14	19.12.58	29.1	105.4	76.4	15.7	—	$3\frac{1}{4}$	—	6.00	0.51	1	2	—	A	V.L
													28	B	V.L
													28	C	V.L
15	12.12.58	29.1	105.4	76.4	15.0	—	$2\frac{7}{8}$	—	5.95	0.49	2	2	—	A	~ 10
													35	B	~ 12
													35	C	~ 20
16	19.12.58	29.1	105.4	71.5	15.7	5.5	3	5.0	5.96	0.51	1	2	—	A	96.9
													28	B	97.5
													28	C	98.8
17	12.12.58	29.1	105.4	71.5	14.8	5.5	3	5.0	6.00	0.48	2	2	—	A	96.3
													35	B	97.4
													35	C	98.6
30	4.3.59	29.1	105.4	74.0	14.9	2.5	3	4.0	6.00	0.49	3	2	—	A	98.6
													28	B	98.7
													28	C	99.8
31	4.3.59	29.1	105.4	74.0	14.9	2.5	3	3.9	6.00	0.49	4	2	—	A	102.5
													35	B	102.5
													35	C	102.7
32	18.8.59	29.1	105.4	76.4	15.0	—	$2\frac{5}{8}$	—	5.95	0.49	3	2	—	A	~20
													28	B	~40
													28	C	~30
33	18.8.59	29.1	105.4	76.4	15.1	—	$2\frac{3}{4}$	—	5.87	0.49	4	2	—	A	64.4
													35	B	~ 37
													35	C	~35
34	20.8.59	29.1	105.4	71.5	14.6	5.8	$3\frac{1}{2}$	6.2	5.90	0.48	3	2	—	A	100.3
													28	B	99.9
													28	C	99.2
35	25.8.59	29.1	105.4	71.5	14.7	5.8	$3\frac{1}{4}$	6.0	5.90	0.48	4	2	—	A	101.7
													35	B	100.8
													35	C	101.6
36	25.8.59	29.1	105.4	76.4	15.6	—	$2\frac{3}{4}$	—	5.90	0.51	3	1	—	A	~ 5
													28	B	~15
													28	C	~ 5
37	28.8.59	29.1	105.4	76.4	15.7	—	$3\frac{1}{2}$	—	5.90	0.51	4	1	—	A	~16
													35	B	~15
													35	C	~20
38	2.10.59	29.1	105.4	74.0	16.1	2.2	$3\frac{1}{8}$	3.0	6.00	0.53	3	1	—	A	85.4
													28	B	87.0
													28	C	80.8
39	2.10.59	29.1	105.4	74.0	16.0	2.2	$3\frac{1}{4}$	3.2	6.00	0.53	4	1	—	A	77.5
													35	B	86.7
													35	C	84.6
40	2.10.59	29.1	105.4	71.5	15.0	6.3	3	4.5	6.00	0.49	3	1	—	A	96.9
													28	B	93.2
													28	C	96.2
41	2.10.59	29.1	105.4	71.5	15.1	5.8	3	4.5	6.00	0.49	4	1	—	A	76.5
													35	B	77.3
													35	C	79.0
42	23.10.59	29.1	93.5	79.1	16.4	5.4	$2\frac{7}{8}$	4.2	6.00	0.54	4	2	—	A	64.4
													35	B	77.3
													35	C	77.5
43	23.10.59	29.1	93.5	79.1	15.9	5.4	3	4.2	6.05	0.52	3	2	—	A	22
													28	B	25
													28	C	30.5

NOTES:

1. Aggregates.
Mixes Nos. 10-17 & 30-41
C.A. Brighton.
F.A. Caledon.
Mixes Nos. 42 & 43
C.A. Hagersville
F.A. Paris.

2. W/C ratio approximate. Absorption of aggregates assumed to be 0.4 %

3. Curing Procedures
(1) 28 days moist
(2) 21 days moist + 14 days dry.
(3) 35 days moist.
(4) 21 days moist + 21 days dry.

4. Testing Procedures.
(1) Freeze and Thaw in Water.
(2) Freeze in Air and Thaw in Water.

5. Silicone Treatment.
(A) None
(B) Silicone 1
(C) Silicone 2

6. Durability Factor.

$$D.F. = \frac{PN}{M}$$

where P = relative dynamic modulus of elasticity at N cycles, percent.
N = number of cycles at which P = 60 or 300, whichever occurs first.
M = 300 cycles.

7. N.T. = Not Tested.
V.L. = Very low.

Tests and Results

Details of all freeze-thaw tests are shown in Table 3. In the first group of mixes (Nos. 10-17), curing procedures 1 and 2 were used in combination with the two testing procedures for air-entrained and non-air-entrained concretes. The second group of mixes (Nos. 30-41) was similar to the first group except that curing procedures 3 and

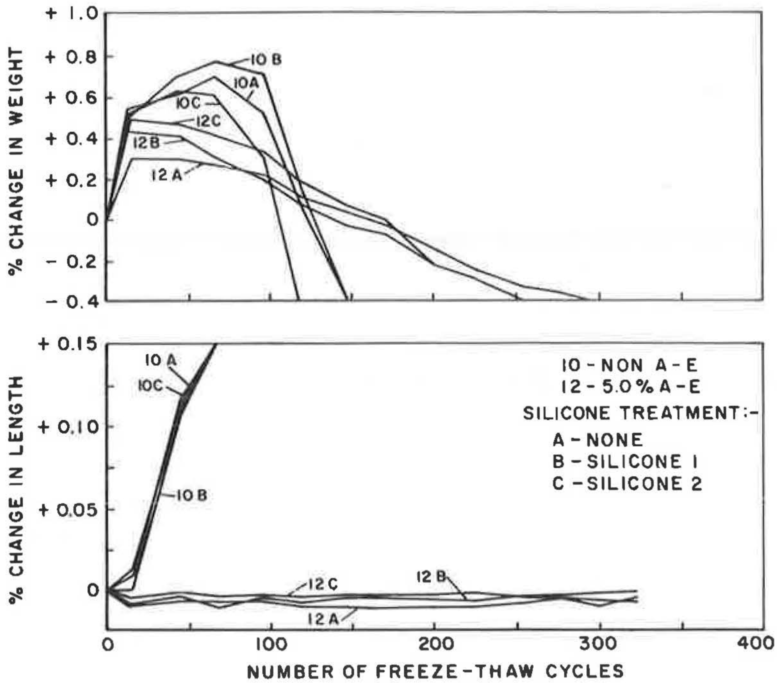


Figure 1. Effect of silicone treatment on freeze-thaw durability of air-entrained and non-air-entrained concrete, 28 days moist, freeze and thaw in water.

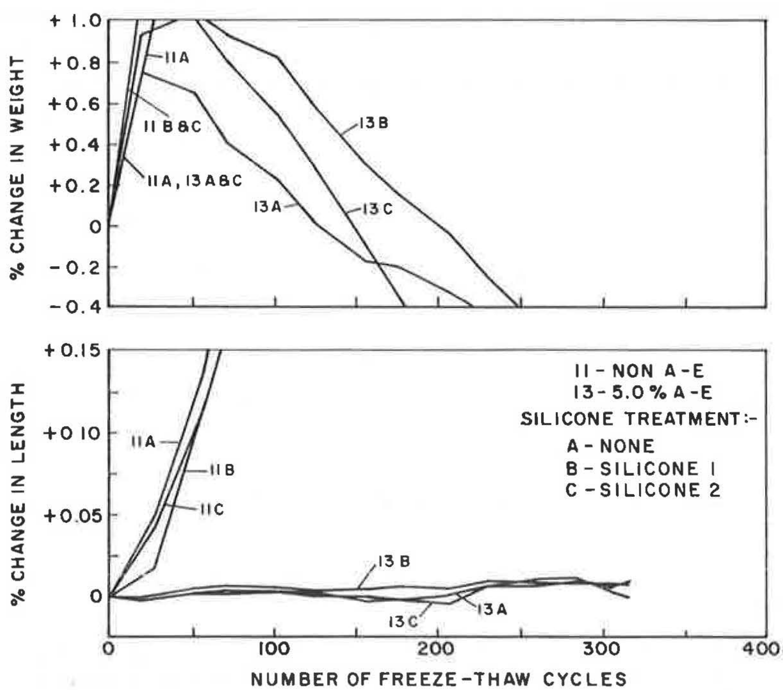


Figure 2. Effect of silicone treatment on freeze-thaw durability of air-entrained and non-air-entrained concrete, 21 days moist + 14 days dry, freeze and thaw in water.

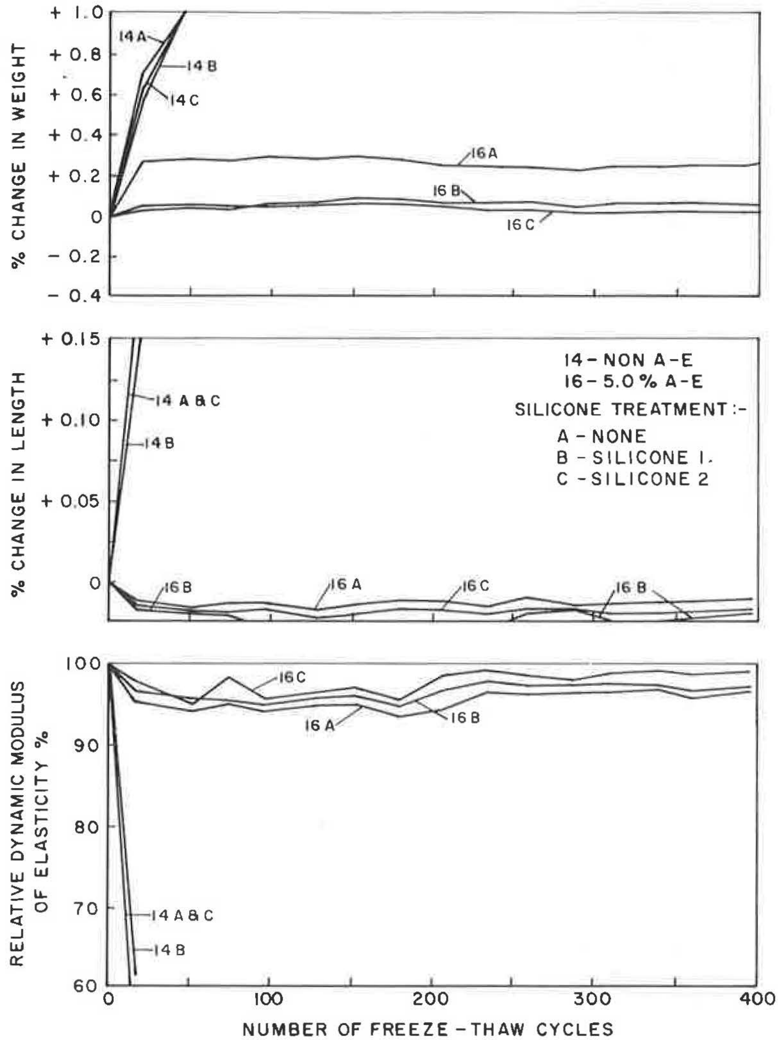


Figure 3. Effect of silicone treatment on freeze-thaw durability of air-entrained and non-air-entrained concrete, 28 days moist, freeze in air and thaw in water.

4 were used, thus allowing a longer period for the silicone to cure; an additional set of mixes with less entrained air was included in this series (Nos. 30, 31, 38, and 39). Mixes 42 and 43 were made with a coarse aggregate which contained a high proportion of chert, to determine whether silicone treatment would improve the freeze-thaw resistance of this material.

The results of tests for change in weight, length, and relative dynamic modulus of elasticity are shown in Figures 1 through 12 and average durability factors are given in Table 3.

SCALING TESTS

Materials Tested

Normal portland cement was used. The aggregate consisted of crushed gravel from Brighton, Ont., and natural sand from Paris, Ont. The admixture was neutralized Vinsol resin. Results of routine tests on these materials are given in the Appendix. Also, silicones 1 and 2 were both used.

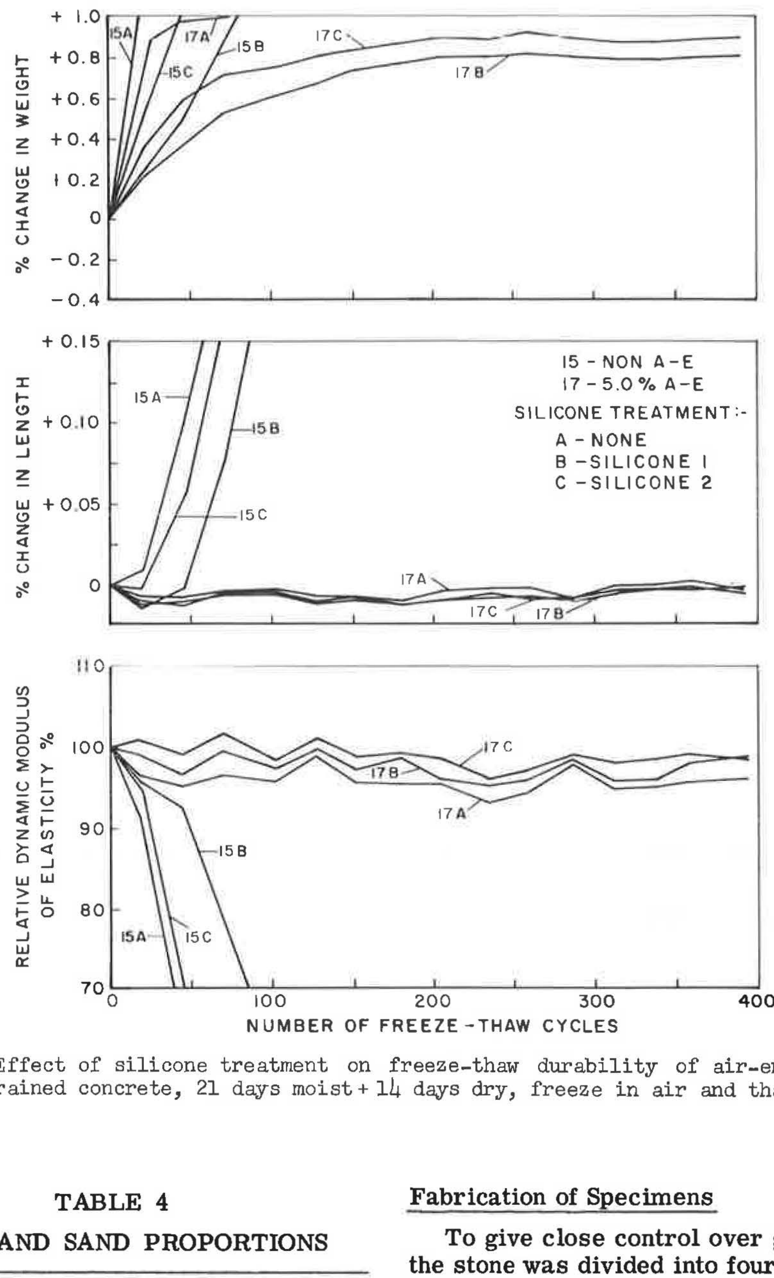


Figure 4. Effect of silicone treatment on freeze-thaw durability of air-entrained and non-air-entrained concrete, 21 days moist + 14 days dry, freeze in air and thaw in water.

TABLE 4

STONE AND SAND PROPORTIONS

Stone		Sand	
Sieve Size	Cumulative % Passing	Sieve Size	Cumulative % Passing
1-in.	100	No. 4	100
¾-in.	90	No. 14	65
½-in.	60	No. 28	40
⅜-in.	30	No. 48	15
No. 4	0	No. 100	5

Fabrication of Specimens

To give close control over grading, the stone was divided into four sizes and the sand into five, and recombined in the porportions given in Table 4.

All mixes were proportioned to produce concrete containing approximately 6 bags of cement per cu yd of concrete (based on Canadian bag of cement = 87.5 lb). The sand contents were varied slightly according to the air content anticipated, and the water contents were adjusted to give 3 ± ½-in. slump in all cases.

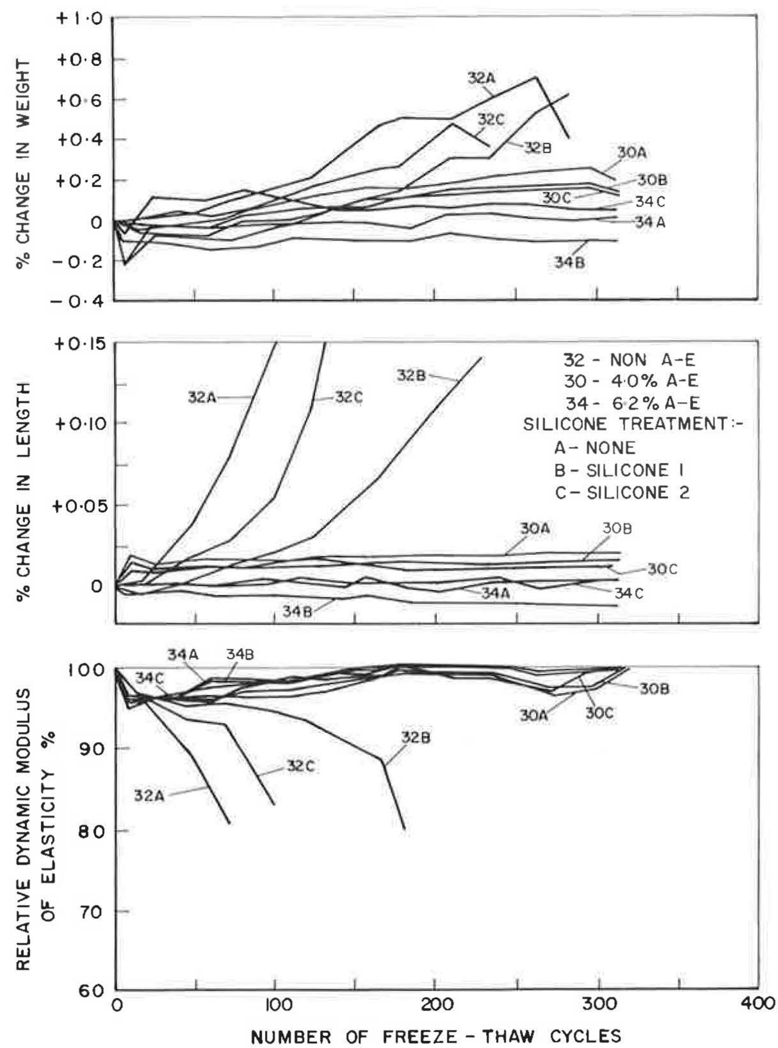


Figure 5. Effect of silicone treatment on freeze-thaw durability of air-entrained and non-air-entrained concrete, 35 days moist, freeze in air and thaw in water.

Six slabs, each approximately 15 by 6 by 3 in., were made from each mix. The slabs were compacted by vibrating for 5 sec with an external vibrator. The surface was then struck off level with three passes of a wood float. After 2 hr, the surface of the slab was given a rough texture by making three passes with a stiff bristle brush. If any moisture was brought to the surface, the brushing was repeated at 3 hr.

After 24 hr of curing, the slabs were removed from the moist room and a mortar dike about ¾-in. high was cast around the edge of each slab. It was found that an air-entrained mortar made with pass 14 sand, bonded to the slab with an epoxy resin bonding agent, gave the most satisfactory results. After the dikes had been cast, the slabs were returned to the curing room.

Curing

All slabs were cured at 72 F and 100 percent relative humidity for 14 days, followed by 14 days dry curing in the laboratory at 72 F. Where required, the silicone coat

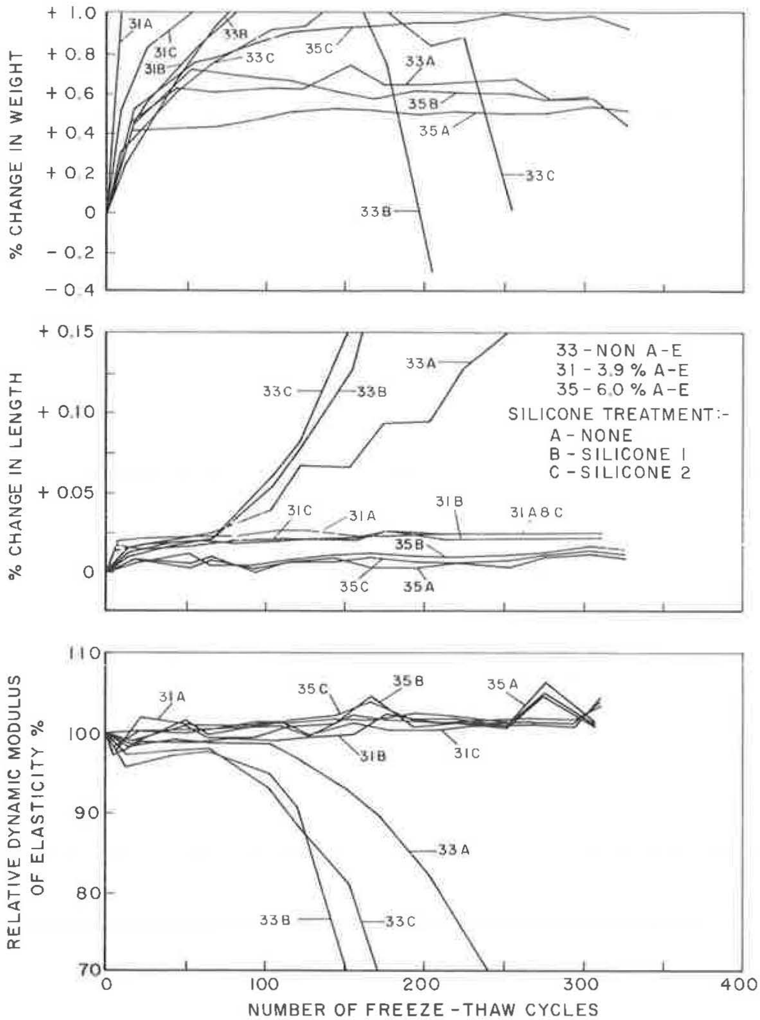


Figure 6. Effect of silicone treatment on freeze-thaw durability of air-entrained and non-air-entrained concrete, 21 days moist + 21 days dry, freeze in air and thaw in water.

was applied at 28 days, after which two different procedures were followed: (a) slabs were stored dry in the laboratory for 4 days, followed by 3 days covered to a depth of ¼ in. with water, and (b) slabs were stored dry in the laboratory for 7 days.

Testing Procedure

The test procedures used were based on those developed by Verbeck and Klieger (2). Two curing procedures were used and two testing methods, resulting in four different types of procedures.

In a similar testing procedure, Verbeck and Klieger found that the most severe conditions were obtained using 2 to 3 percent sodium chloride solution (by weight). Therefore, in this study a 3 percent sodium chloride solution was selected for use in all cases to give a severe test, and no attempt was made to study the effect of different solution strengths or of other de-icing agents.

Testing of all slabs began at an age of 35 days. Two test procedures were used:

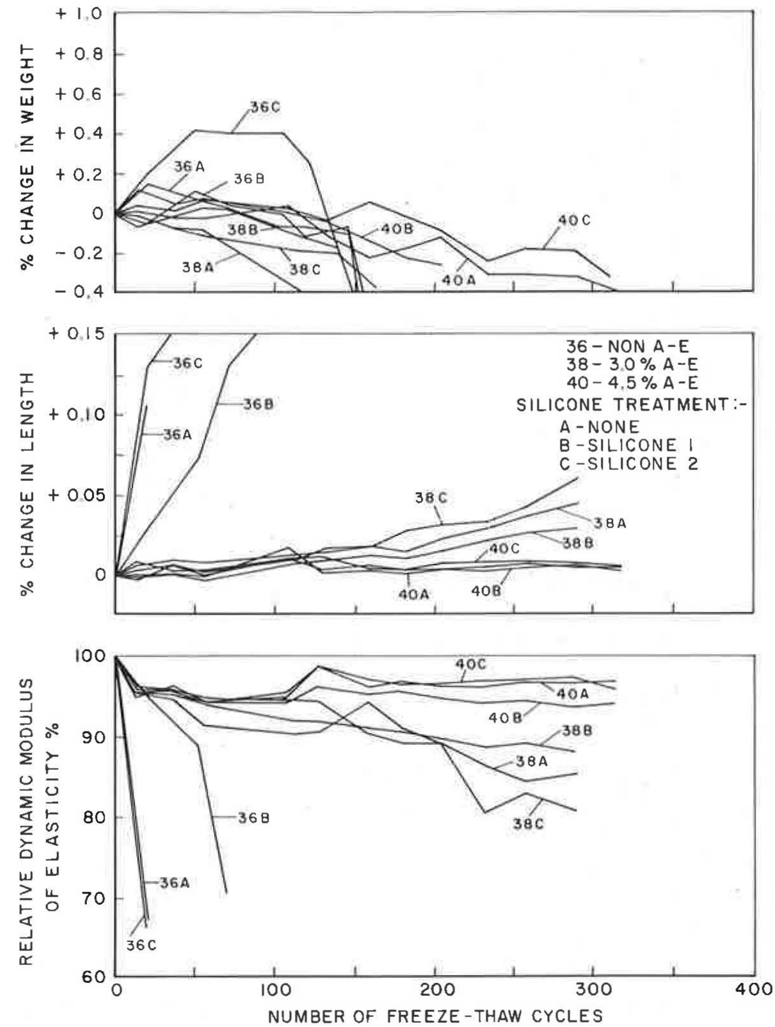


Figure 7. Effect of silicone treatment on freeze-thaw durability of air-entrained and non-air-entrained concrete, 35 days moist, freeze and thaw in water.

1. Freezing with de-ice solution on slab. At an age of 35 days the slabs were placed in a cold room at -10 F with a 3 percent sodium chloride solution covering the surface to a depth of 1/4 in. At 9 AM on the following morning, the slabs were brought into the laboratory at 72 F and allowed to thaw until 4 PM at which time they were again placed in the cold room with the de-ice solution still on the surface. The procedure was repeated each day, the slab being rinsed and the de-ice solution replaced with fresh solution twice a week. The slabs were left in the freeze portion of the cycle over the weekends.

2. This method was the same as the preceding except that the thaw solution was placed on the slab after each freezing cycle and removed before the next freezing cycle. Thus the slab was frozen with the surface only slightly damp.

The slabs were examined periodically and given a rating from 0 to 5 according to the degree of surface scaling, by comparison with standard rated slabs prepared for the purpose. These ratings would probably approximate Verbeck and Klieger's (2) ratings: 0 = no scaling, 1 = very slight scaling, 2 = slight to moderate scaling, 3 =

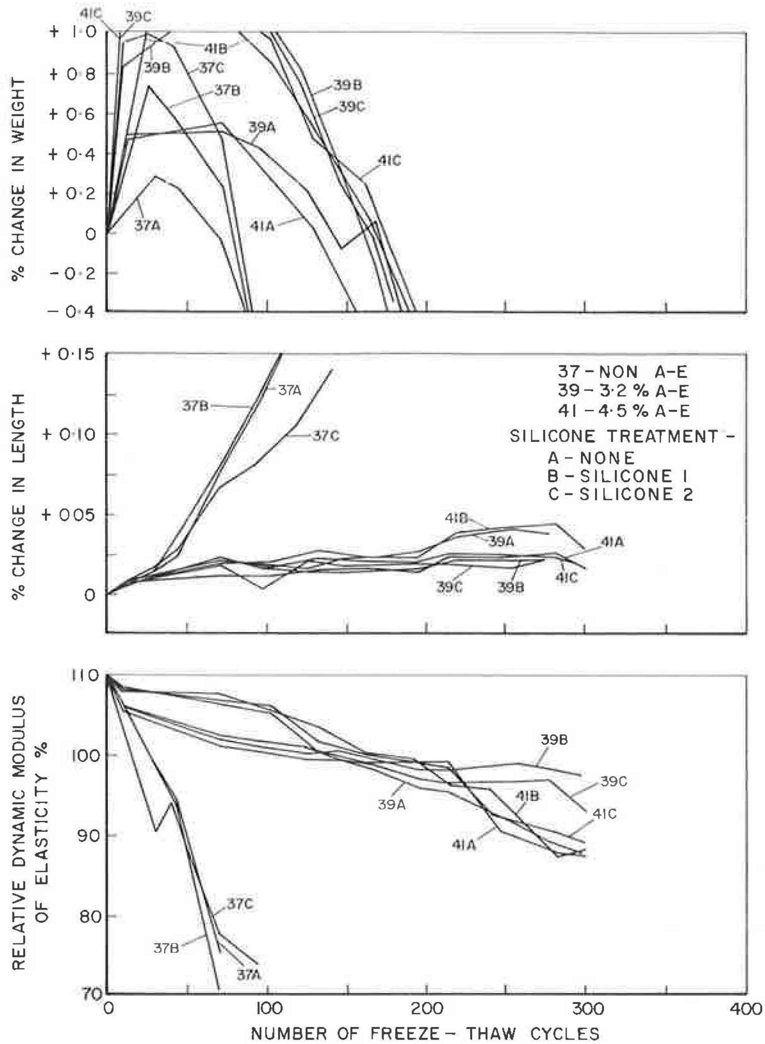


Figure 8. Effect of silicone treatment on freeze-thaw durability of air-entrained and non-air-entrained concrete, 21 days moist + 21 days dry, freeze and thaw in water.

moderate scaling, 4 = moderate to severe scaling, and 5 = severe scaling. The standard slabs are shown in Figure 13.

On some occasions, unsatisfactory dikes made it necessary to remove slabs from testing for a short period to make repairs. Two slabs were prepared for each condition of test and the average rating was plotted against the number of cycles of the scaling test.

Tests and Results

Details of the scaling tests are given in Table 5. For the first group of mixes (Nos. 22 to 25), curing procedure 1 was used, mixes 22 and 23 being non-air-entrained concrete and mixes 24 and 25 being air-entrained. Mixes 26 to 29 were repetitions of mixes 22 to 25, except for the different curing procedure. Mixes 46 and 47 were air-entrained but had a lower air content than the other air-entrained mixes, and were treated similarly to mixes 26 to 29.

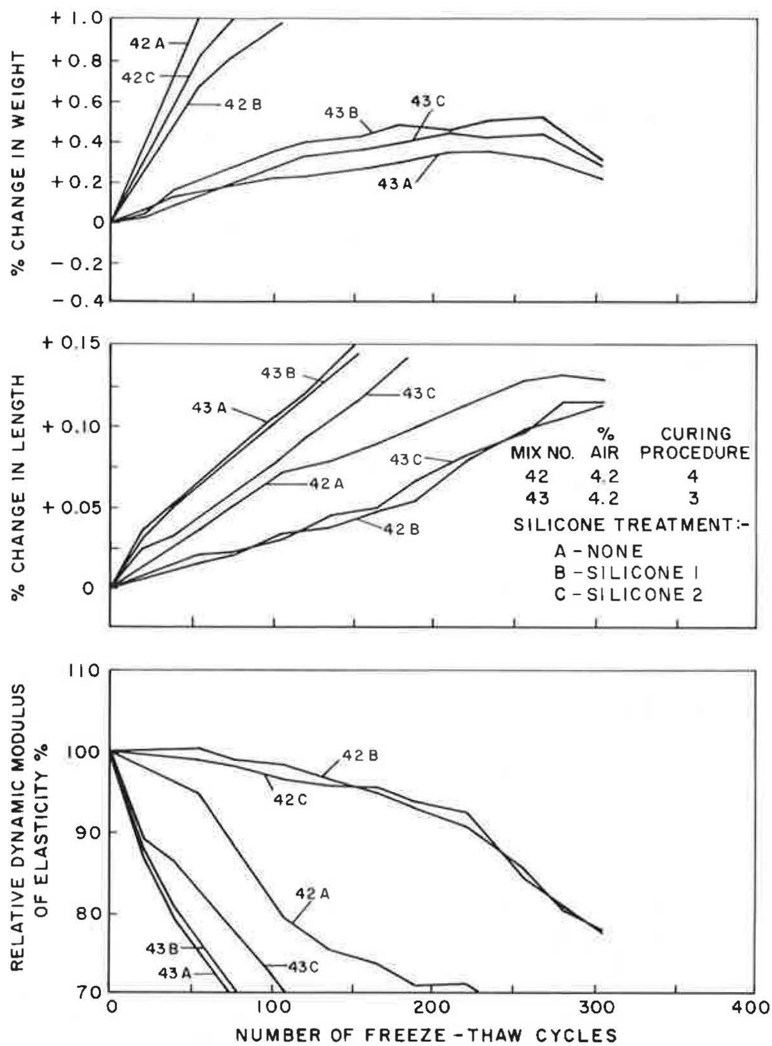


Figure 9. Effect of silicone treatment on freeze-thaw durability of air-entrained concrete using coarse aggregate of poor durability, freeze in air and thaw in water.

Results of the tests are given in Table 5 and plots of them are shown in Figures 14 and 15.

ANALYSIS

Freeze-Thaw Tests

The results of the freeze-thaw tests are given in Table 3 and Figures 1 to 12. In considering the effect of the silicone solution on freeze-thaw durability, it is advantageous to consider first the effect of the different curing and testing procedures on the durability of untreated non-air-entrained and air-entrained concrete. Results of tests on untreated concrete are compared in Figures 10 to 12.

Figure 10 shows the effect of different curing procedures for concrete tested by freezing in air and thawing in water. The non-air-entrained concrete subjected to a period of dry curing (mix 33A) shows much greater durability than similar concrete that was continuously moist cured before testing (mix 32A). Air-entrained concretes

TABLE 5

SCALING MIXES AND RESULTS OF SCALING TESTS

MIX NO.	DATE MADE (1959)	BATCH PROPORTIONS: CEMENT	STONE	SAND	WATER	A. E. A.	TEST RESULTS: SLUMP	AIR CONTENT	CEMENT FACTOR	W/C RATIO	CURING PROCEDURE	TEST PROCEDURE	SILICONE TREATMENT	NUMERICAL SCALE RATING AT INDICATED NUMBER OF CYCLES (Bracketed figures are Nº of cycles at which a rating 5 was reached.): 5	15	25	50	75	100	150
	d m	lb.	lb.	lb.	lb.	c. c.	inch.	%	bags/cu.yd.	—	—	—	—	—	—	—	—	—	—	—
22	24.2	27.2	97.3	71.1	14.0	–	$2\frac{5}{8}$	—	6.01	0.49	1	1	A	1.5	2.75	3.75	(30)	–	–	–
													B	0	1.5	2.25	3.25	3.75	4.0	4.75
													C	1.25	1.75	2.75	3.25	3.75	4.25	(124)
23	24.2	27.2	97.3	71.1	14.0	–	$2\frac{5}{8}$	–	6.01	0.49	1	2	A	1.0	1.5	1.5	1.5	1.5	1.5	1.75
													B	0	0	0.25	0.5	0.5	0.5	1.0
													C	0	0	0.25	0.5	0.75	0.75	1.25
24	24.2	27.2	97.3	66.8	13.0	5.0	3	5.2	6.07	0.45	1	1	A	1.0	1.5	1.5	1.5	1.75	2.5	2.5
													B	0	0.75	1.25	1.75	2.75	2.75	2.75
													C	0.5	1.25	1.25	1.25	2.0	2.25	2.5
25	24.2	27.2	97.3	66.8	13.0	5.0	3	5.7	6.04	0.45	1	2	A	0	0	0	0	0.5	0.5	0.5
													B	0	0	0	0	0.5	0.5	0.5
													C	0	0	0.25	0.25	0.5	0.5	0.5
26	9.3	27.2	97.3	71.1	14.3	–	$3\frac{1}{8}$	–	5.99	0.50	2	1	A	1.5	2.75	3.75	4.5	(63)	–	–
													B	0.5	1.5	1.75	2.5	3.0	3.5	4.75
													C	1.0	1.75	2.0	3.5	3.75	4.25	(126)
27	9.3	27.2	97.3	71.1	14.5	–	$3\frac{1}{2}$	–	6.00	0.51	2	2	A	0	0	0	0.5	0.5	0.5	0.75
													B	0	0	0	0.5	0.5	0.5	0.75
													C	0	0	0.25	0.25	0.5	0.5	0.5
28	9.3	27.2	97.3	66.8	13.5	5.0	$3\frac{1}{8}$	5.9	6.00	0.47	2	1	A	1.0	1.5	1.5	1.5	2.0	2.5	3.5
													B	0	0.75	0.75	1.5	2.0	2.5	3.0
													C	0.75	1.5	1.5	2.0	2.0	2.5	3.5
29	9.3	27.2	97.3	66.8	13.6	5.0	$3\frac{1}{4}$	6.1	5.98	0.47	2	2	A	0	0	0	0.5	1.25	1.25	1.5
													B	0	0	0	0.5	1.0	1.0	1.25
													C	0	0	0	0.25	0.5	0.5	0.5
46	21.4	27.2	97.3	68.8	13.8	2.0	$3\frac{1}{8}$	4.1	6.06	0.48	2	1	A	0	0.5	1.0	1.25	1.5	2.0	3.0
													B	0	0.25	0.5	1.0	1.5	2.0	3.0
													C	0	0.5	0.5	1.0	1.5	2.0	3.0
47	21.4	27.2	97.3	68.8	13.8	2.0	$3\frac{1}{8}$	4.2	6.05	0.48	2	2	A	0	0.5	0.5	0.5	0.5	0.5	0.75
													B	0	0	0	0	0	0	0
													C	0	0	0	0	0	0	0.25

(mixes 35A and 34A) are much less affected by the different curing procedures, but of course they show much greater durability than non-air-entrained concrete.

The rapid gain in weight of the dry cured specimens in the early stages of freezing and thawing is due to absorption of water by the dry concrete and not to deterioration caused by freezing and thawing. Because of the difficulty of interpreting the part played by absorption, the change in weight of the specimens is considered the least reliable of the three methods of measuring durability.

Figures 11 and 12 show the effect of different testing procedures on concretes cured by procedures 3 and 4, respectively. In both cases, the much more severe effect of test procedure 1 is demonstrated. This is noticeable in the case of air-entrained concrete as well as plain concrete, although the low durability of mix 41A as compared with mix 35A is probably due partly to the lower air content.

The effect of silicone treatment on the freeze-thaw durability of normal concretes is shown in Figures 1 to 8. Each combination of the different curing and testing procedures is shown in a separate figure.

In general, the results demonstrate the close similarity between the freeze-thaw durability of silicone-treated and untreated concrete. In many cases, there is a slight but quite definite improvement in the durability of the silicone-treated non-air-entrained concrete, but this is much less noticeable with air-entrained concrete; for example, mix 15 vs 17 in Figure 4, and mix 32 vs 34 in Figure 5.

The different curing and testing procedures resulted in no significant difference in the effectiveness of the silicone coating, although the results indicated a possible slight advantage (for the silicone) in a period of dry curing (curing procedures 2 and 4) and in the use of the less severe test procedure of freezing in air and thawing in water. Similarly, the use of low air contents in air-entrained concrete (3 to 4%) did not increase the beneficial effect of the silicone to any appreciable extent; in fact, this con-

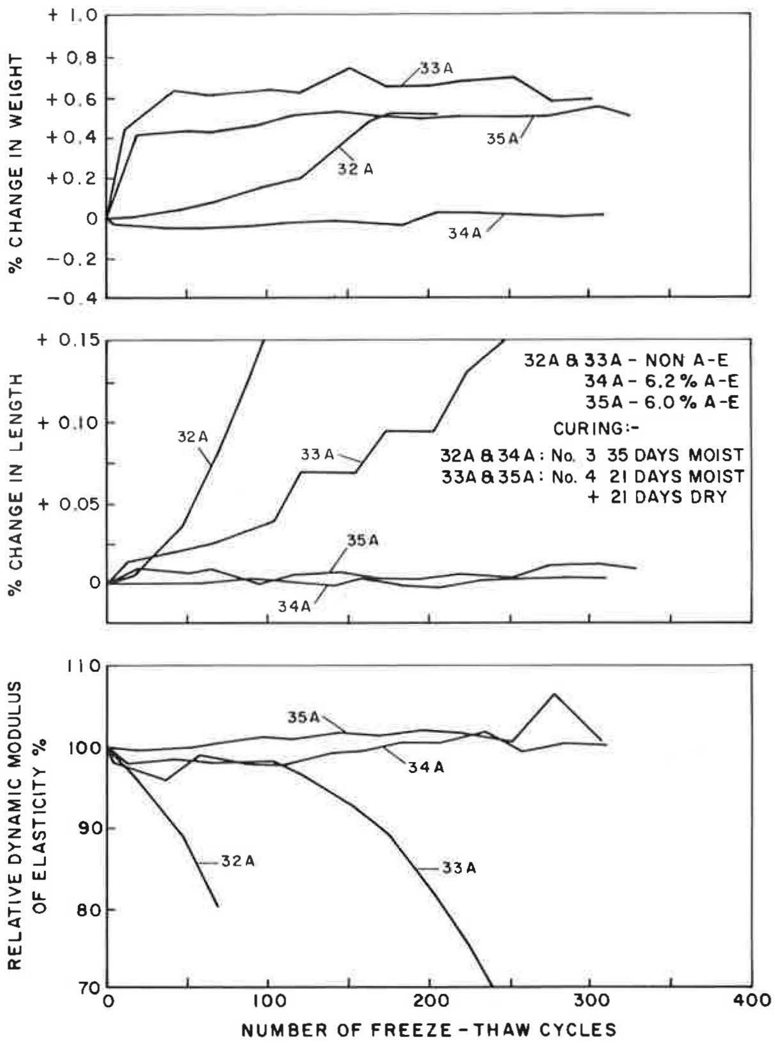


Figure 10. Effect of different curing procedures on untreated air-entrained and non-air-entrained concrete, freeze in air and thaw in water.

crete was only slightly less resistant to freezing and thawing than normal air-entrained concrete tested under similar conditions (mixes 30, 31, 38, and 39 in Figs. 5, 6, 7, and 8, respectively).

In one case (mix 33, Fig. 6) untreated non-air-entrained concrete showed markedly better durability than silicone-treated specimens of the same concrete. In this case, the concrete was cured moist for 21 days followed by 21 days dry curing (curing procedure 4) and tested by freezing in air and thawing in water (testing procedure 2). No explanation can be found for this behavior.

To demonstrate the difference that may be expected between untreated specimens and specimens that have been treated with silicones, beams from mixes 15 and 17 after completion of freeze-thaw testing are shown in Figures 16 and 17 with supplemental data given in Table 6.

A short series of tests were made with specimens made with a coarse aggregate containing a high percentage of chert which normally breaks down very rapidly when subjected to freeze-thaw testing. The results of these tests are shown in Figure 9.

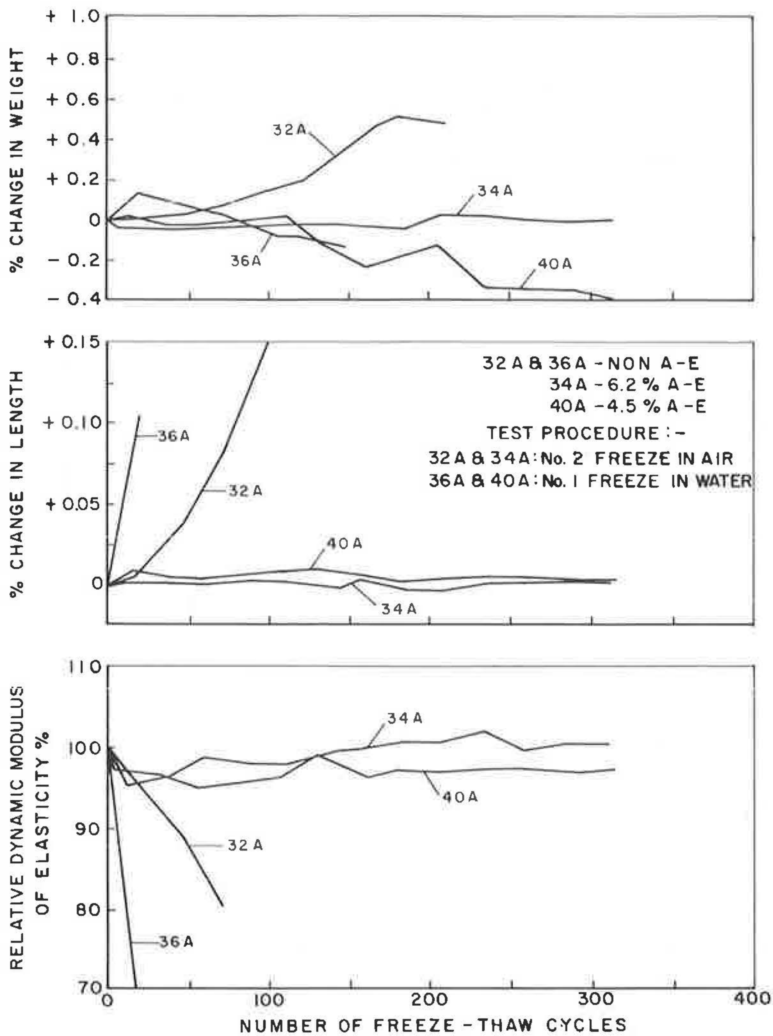


Figure 11. Effect of different testing procedures on untreated air-entrained and non-air-entrained concrete, 35 days moist.

All specimens moist cured for 35 days before testing gave similar results, with the silicone only having a slight beneficial effect. The silicone treatment gave a substantial improvement in the durability of specimens cured moist for 21 days followed by 21 days followed by 21 days of dry curing before beginning the test, the durability factor having increased by about 13 percent from 64.4 to 77.3 and 77.5 percent for the two different treatments. It appears that the silicone was relatively successful in preventing the penetration of water through the durable surface formed by the air-entrained mortar, but the improvement is not sufficient to warrant field use of this material, because experience has shown that the durability factor of over 90 percent is normally required for concrete to perform well under field conditions.

Scaling Tests

The results of the scaling tests are shown in Table 5 and in Figures 14 and 15. As with the freeze-thaw tests, a number of different curing and testing procedures were

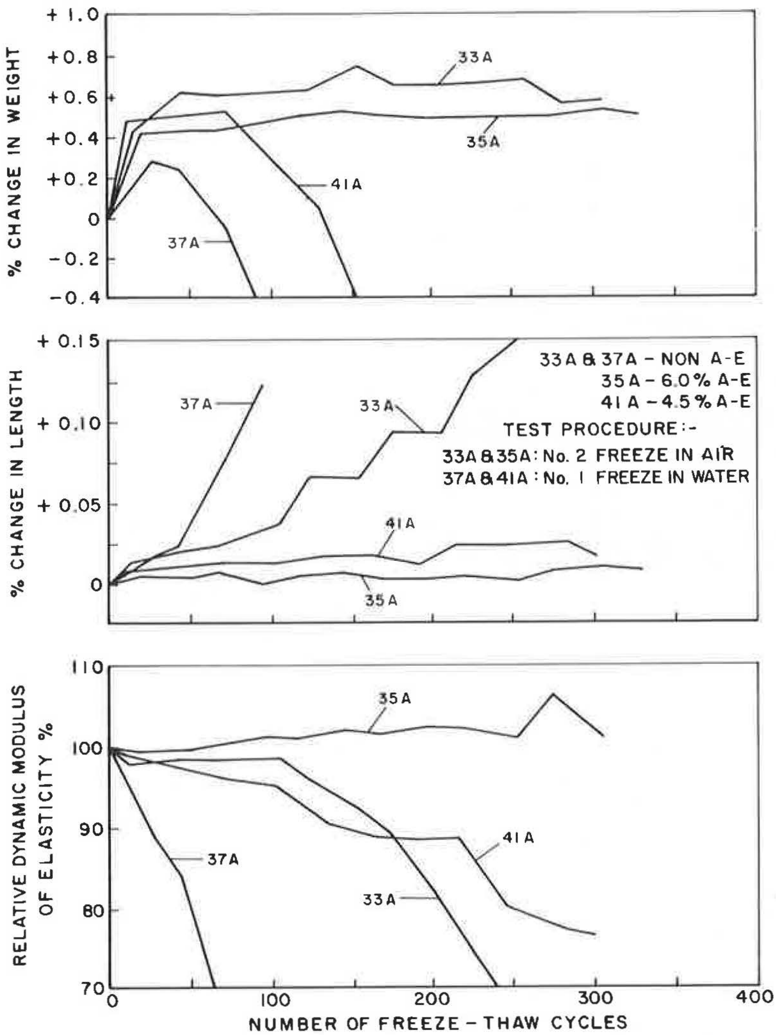


Figure 12. Effect of different testing procedures on untreated air-entrained and non-air-entrained concrete, 21 days moist + 21 days dry.

used. The slight difference in the curing procedures had little effect on the result except that the slabs that had been covered with water for three days before starting to test tended to scale more rapidly in the early stages. The two different testing procedures used had a great effect on the rate of scaling. Testing procedure 1 (freezing with the salt solution on the surface of the slab) gave the most severe condition. Scaling was much less rapid when the salt solution was removed before freezing (testing procedure 2); it may be assumed that this procedure gave a large proportion of the moisture in the surface time to evaporate before freezing started. This effect is in general agreement with the findings of Verbeck and Klieger (2).

The figures show that in most conditions the silicone coat provides only a slight beneficial effect or none at all. However, with non-air-entrained concrete subjected to the severe conditions of testing procedure 1, there was a distinct improvement in scaling resistance of silicone-coated concrete compared with untreated specimens of the same concrete. This was confirmed for both curing procedures, though the rate of scaling was more rapid when the slab had been covered with water for three days bef

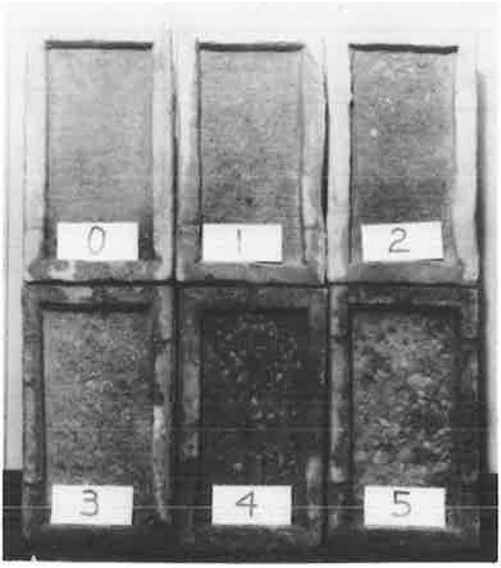


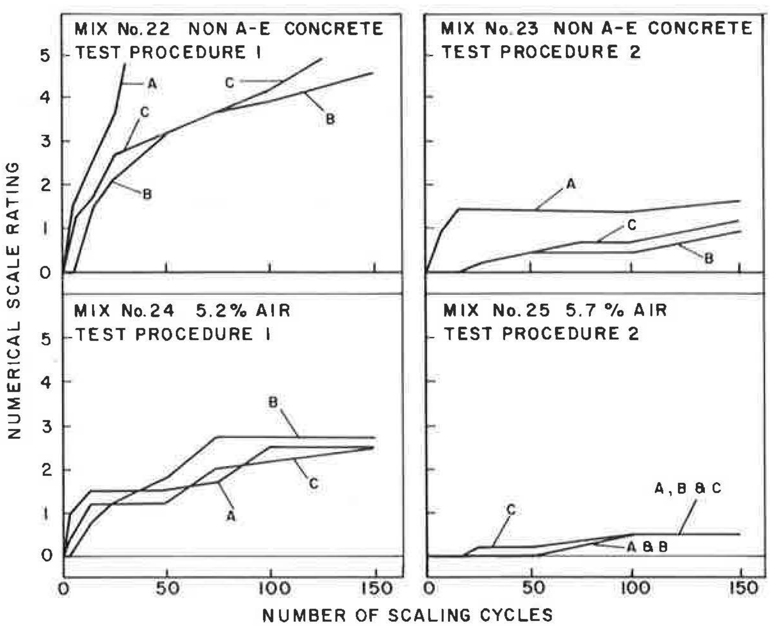
Figure 13. Standard slabs rated from 0 to 5.

TABLE 6

SILICONE TREATMENT AND NUMBER OF FREEZE-THAW CYCLES FOR MIXES 15 AND 17

Mix	Silicone Treatment	No. of Freeze-Thaw Cycles
15A	None	151
15B	1	181
15C	2	151
17A	None	495
17B	1	495
17C	2	495

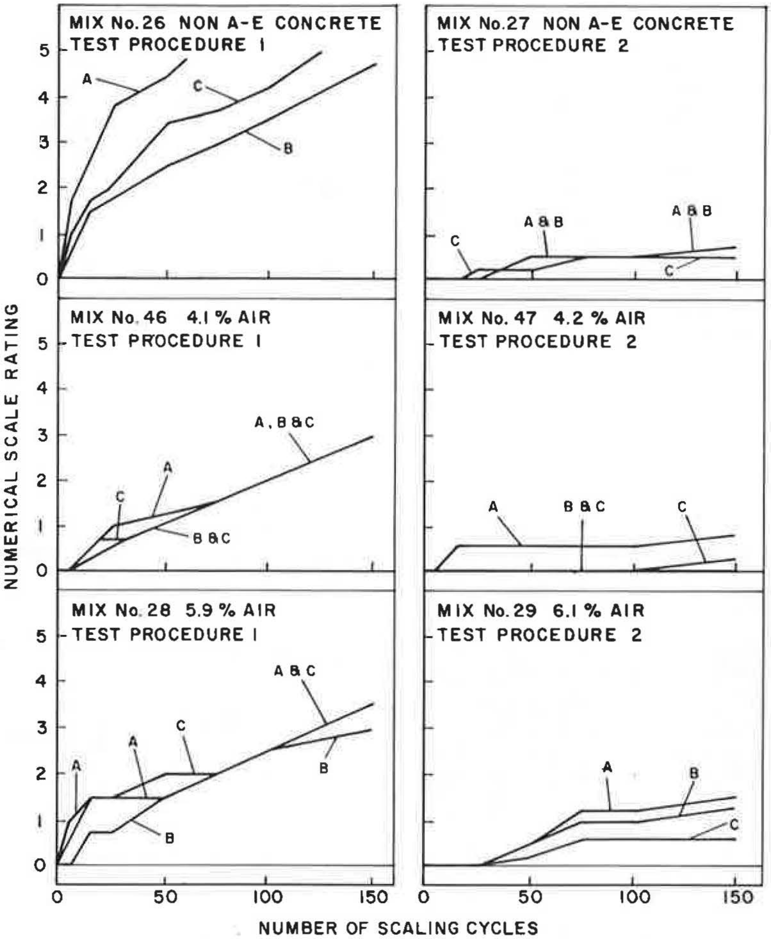
starting to test. This, together with the less severe scaling resulting from testing procedure 2, indicates the advantages of allowing a drying period after the completion of curing, before applying de-icing



NOTES: SILICONE TREATMENT AT 28 DAYS
A - NO TREATMENT
B - SILICONE I
C - SILICONE 2

TEST PROCEDURE I - FREEZING WITH DE-ICE SOLUTION ON SLAB.
TEST PROCEDURE 2 - FREEZING WITH SLAB SURFACE DAMP.

Figure 14. Results of scaling tests, 14 days moist + 18 days dry + 3 days water covered.



NOTE: FOR DETAILS OF SILICONE TREATMENT AND TEST PROCEDURES SEE FIG. 14

Figure 15. Results of scaling tests, 14 days moist + 21 days dry.

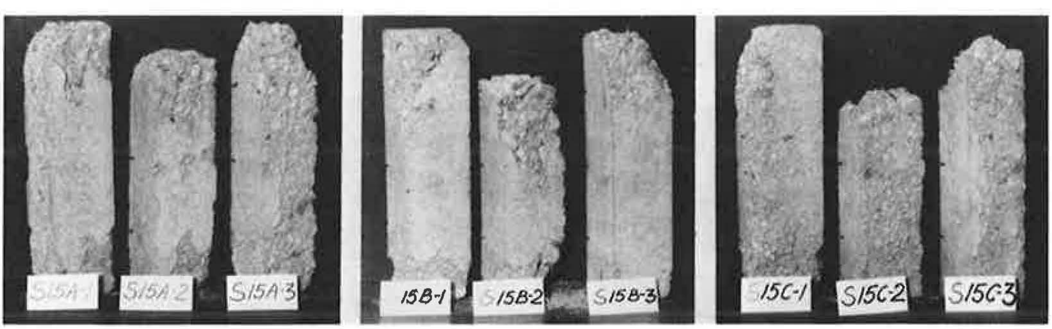


Figure 16. Non-air-entrained specimens (mix 15) after testing, 21 days moist + 14 days dry, freeze in air and thaw in water.

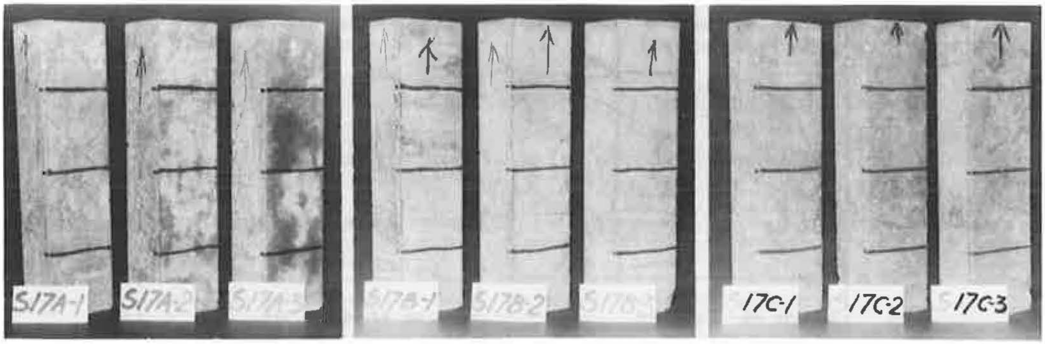


Figure 17. Five percent air-entrained specimens (mix 17) after testing, 21 days moist + 14 days dry, freeze in air and thaw in water.

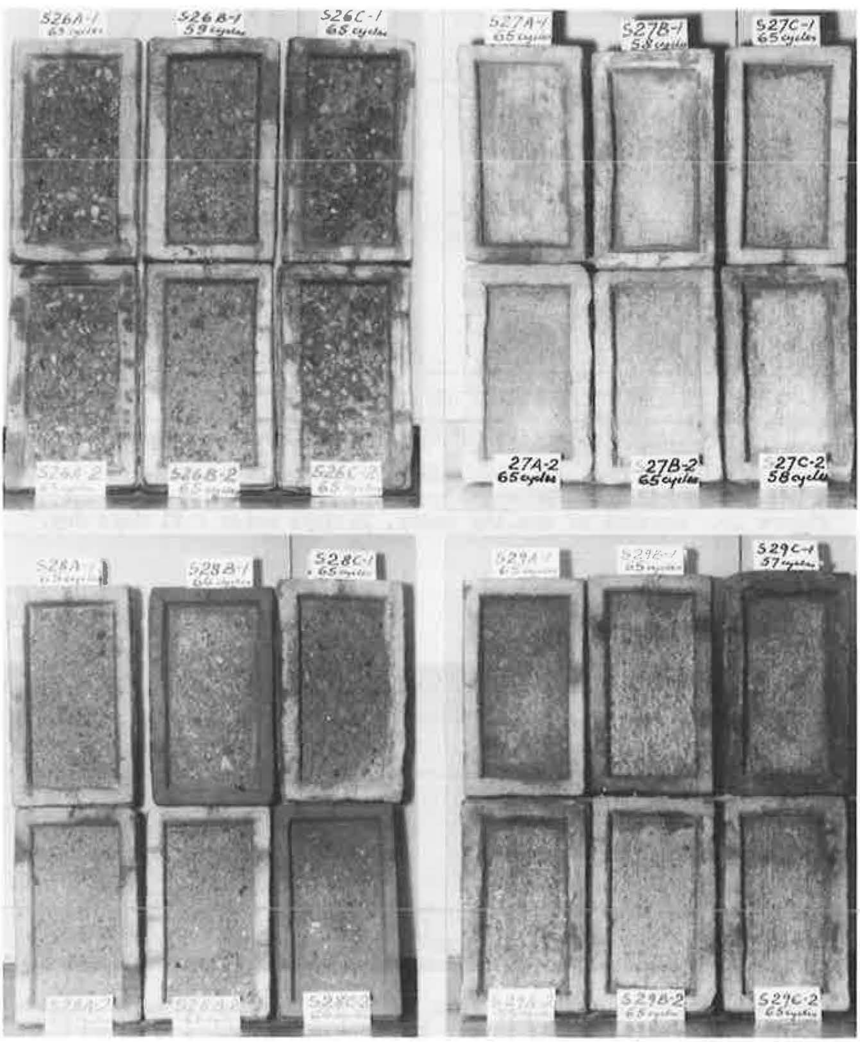


Figure 18. Specimens from mixes 26 through 29 after testing.

salts. Results of scaling tests on mixes 46 and 47 (Fig. 15) indicate that the effectiveness of the silicone coat is not increased when the concrete has a lower than normal air content (4.1 and 4.2%).

Figure 18 shows slabs from mixes 26, 27, 28, and 29 which had been subjected to between 55 and 65 cycles of the scaling test. The more severe scaling of the slabs from mix 26 is noteworthy, and the difference between the silicone-treated slabs (26B and 26C) and the untreated slabs (26A) can be seen. At this time, slabs 26A had reached a scale rating of 5.

The silicone coat on non-air-entrained concrete formed a relatively resistant surface crust, but once this crust had been broken through, severe scaling would spread rapidly across the whole slab. The early stages of this condition can be seen on slabs 26B-1 and 26B-2 on which there are a few deeply-scaled spots. On slabs 26C-1 and 26C-2, the condition is further advanced, much of the surface being deeply scaled.

CONCLUSIONS

Laboratory tests have the advantage of giving a direct comparison of differently-treated specimens under closely-controlled conditions which cannot be duplicated in the field, but the question arises as to the extent to which the test procedures adopted for the laboratory tests actually simulate conditions that may occur in the field. Therefore, it is obvious that the results of laboratory tests represent the performance of the materials under the conditions of test only; however, it is usually possible to interpret test results in the light of previous field experience. For instance, Klieger (3) states that, for the particular test procedure he used, extended laboratory experience shows that a scale rating of 1 at 100 cycles of test indicates excellent resistance to surface scaling under field conditions. For the more severe conditions of scale testing procedure 1, a scale rating of 2.5 at 100 cycles may be taken to indicate excellent performance under field conditions. Similarly, in the freeze-thaw tests a durability factor greater than 90 will in general indicate excellent resistance to freezing and thawing in the field.

It is apparent from the results that (a) adequate scaling and freeze-thaw resistance can be obtained by the use of proper air entrainment and (b) the use of silicone on non-air-entrained concrete does not increase its resistance to an acceptable level.

Smith (4) has reported Ontario's field experience with silicones, and concluded that they impart no lasting durability or scaling resistance to inadequately air-entrained concrete.

The results of the laboratory tests taken in conjunction with field experience indicate that the general use of silicone as a protective coating for concrete cannot be justified.

ACKNOWLEDGMENTS

This work has been carried out by the staff of the Materials and Research Division of the Department of Highways, Ontario, under the supervision of A. Rutka, Materials and Research Engineer, and J. Casey, Principal Testing Engineer.

The testing program was initiated by P. Smith, Senior Materials Engineer (Concrete), who together with J. Ryell and B. Chojnacki, made valuable contributions to the work.

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Appendix

ROUTINE TESTS ON MATERIALS

Cement

In all tests, the cement used was normal portland cement supplied from a plant in Ontario. Routine tests on samples of this cement were made in accordance with CSA Specification A5-1951, except that determinations of sodium and potassium oxides were made in accordance with ASTM C114-58T. Results are given in Table 7.

TABLE 7

PHYSICAL AND CHEMICAL ANALYSIS OF CEMENT USED

Property	Sample			
Date Sampled	12 Nov. '58	23 Feb. '59	22 Apr. '59	6 July '59
Normal consistency (%)	24.5	23.5	24.0	24.0
Fineness (ret. on No. 200)(%)	3.6	4.6	2.0	2.0
Autoclave expansion (%)	0.03	0.04	0.05	0.25
Setting time (Vicat):				
Initial (hr:min)	2:45	3:25	2:40	2:45
Final (hr:min)	4:15	5:20	4:40	4:00
Compressive strength (psi):				
3 days	2,430	2,390	2,220	2,230
7 days	3,220	2,970	2,830	2,880
28 days	4,630	4,180	4,350	4,150
Loss on ignition (%)	1.56	-	-	1.77
Insoluble residue (%)	0.16	-	0.20	0.15
Sulfur trioxide (%)	2.16	-	2.37	2.25
Magnesia M_gO (%)	3.05	-	3.49	3.18
K_2O (%)	-	-	0.74	0.48
Na_2O (%)	-	-	0.64	0.48

Coarse Aggregate

Two ¾-in. coarse aggregates were used from Brighton, Ont., (crushed gravel) and Hagersville, Ont., (crushed rock). Average results of tests on the coarse aggregates are given in Table 8.

TABLE 8

PROPERTIES OF AGGREGATES USED

Aggregate Type	Source	Bulk Spec. Gravity[1]	24-Hr Absorp. (% by wt.)
Coarse	Brighton	0.70	0.4
	Hagersville	2.63	1.4
Fine	Caledon	2.66	0.9
	Paris	2.69	1.2

[1]Saturated surface dry.

Petrographic analysis of the aggregates showed that the Brighton gravel contained approximately 70 percent limestone, 15 percent igneous rock, and 15 percent miscellaneous metamorphics and other materials. The Hagersville material contained approximately 52 percent chert, 42 percent limestone and 6 percent sandstone and shale.

Fine Aggregate

Two natural aggregates were used from Caledon and Paris, Ont. Average results of tests on the fine aggregates are given in Table 8.