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PARTICULATE ADMIXTURE FOR ENHANCED FREEZE-THAW RESISTANCE OF CONCRETE

by G. G. Litvan and P. J. Sereda

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PARTICULATE ADMIXTURE FOR ENHANCED
FREEZE-THAW RESISTANCE OF CONCRETE

by

G.G. Litvan and P.J. Sereda
Research Officer and Head, Materials Section
Division of Building Research,
National Research Council of Canada, Ottawa Canada.

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ABSTRACT

Incorporation of porous particles with at least 30 per cent total porosity and pore diameters, mainly between 0.3 and 2 microns, added to the plastic mix were found to improve significantly the freeze-thaw resistance of hydrated neat cement paste and concrete. Particles of commercially fired clay bricks, diatomaceous earth, and fly ash (sintered and agglomerated) were tested. The concentration required for the achievement of a given level of frost resistance depends on the physical characteristics of the material. Typically, granules of a particular brick (0.5 ± 0.08 mm in size, 36 per cent total porosity) in 16 wt per cent concentration allowed 0.5 w/c cement paste specimen to endure 1260 freeze-thaw cycles without injury. Incorporation of porous particles as admixtures eliminates the problems due to the instability and strength reducing effect of conventionally entrained air bubbles.

On a trouvé que des particules poreuses incorporées à au moins 30% de porosité totale et de diamètres des pores, surtout entre 0.3 et 2 microns ajoutés au mélange plastique, améliorent significativement la résistance au gel et au dégel des pâtes de ciment et de béton purs hydratés. Les particules des briques d'argile cuites commercialement, la terre à diatomacées, et la cendre volante (calcinée et agglomérée) ont été mises à l'essai. La concentration nécessaire pour la réalisation d'un niveau donné de résistance au gel dépend des caractéristiques physiques du matériau. D'une manière typique, les granules d'une brique particulière (0.5 ± 0.08 mm de grosseur, porosité totale de 36%) d'une concentration du poids de 16% a permis à un spécimen de pâte de ciment ayant un teneur en eau de 0.5 d'endurer 1260 cycles de gel et de dégel sans avarie. En incorporant les particules poreuses en tant qu'adjuvants on élimine les problèmes causés par l'instabilité et l'effet de réduction de résistance des bulles d'air entraînées d'une manière conventionnelle.

The method of air entrainment by the incorporation of small air bubbles in the plastic concrete mix has proven to be eminently successful in improving the freeze-thaw durability of concrete. The beneficial effect of the method on hardened paste depends on two factors: the total air content and the spacing between the air entrained voids. Controlling these two factors within narrow limits during the mixing, transportation, placement, and finishing operations is a most difficult task because the air content is affected by numerous factors such as mix composition, temperature, and the time taken for the mixing and finishing operations. The situation is made worse by the lack of a test for determining void spacing in the plastic mix. Only after hardening can the spacing be measured, and then by a cumbersome and lengthy procedure, at a time when corrective measures are no longer possible. Furthermore, incorporation of air weakens the concrete significantly, a serious drawback. In fact, the amount of air for frost protection is restricted usually to 6 per cent in order to avoid excessive reduction of mechanical properties.

All these difficulties can be overcome when the voids necessary for durability are provided by the addition of porous particulate substances to the plastic mix. In this manner voids can be provided in the required number, size and distribution by adding an appropriate amount of a suitable material.

The possible use of this concept was tested and the results of preliminary experiments are reported in this communication.

Experimental

Cement mixes were prepared in accordance with ASTM Standard Method C-305. Freezing and thawing test was performed in an automatically operated apparatus according to "Procedure B" of the Standard ASTM Method C-666. Performance was evaluated by monitoring the changes of the fundamental transverse frequency of concrete (ASTM C-215) or by measuring the residual length changes (1).

Porosity and pore size distribution were determined with an AMINCO 60,000 lb/in² porosimeter. Compressive strength was assessed in conformity with ASTM C-39 Standard Method. Entrained air and spacing of voids were evaluated by the linear traverse method in conformity with ASTM C-457 Standard Method.

Materials

Type I cement, graded sand and limestone aggregate (0.6 to 1.2 cm) were used for the fabrication of test specimens. As porous additives, sized particles of commercially fired crushed bricks, designated A & B, two types of diatomaceous earth, originating from California and B.C., and fly ash in spherically agglomerated and sintered forms were used. The air entraining agent was Darex.

Results

Series I. The freeze-thaw resistance, expressed as the number of cycles resulting in 0.2 per cent residual length change of 2.5- by 2.5- by 37.5-cm neat cement (w/c ratio 0.5) specimens containing crushed brick particles (type B), is shown in Figure 1. Testing was performed after 28 days of curing. The results are plotted as a function of the average distance between the particle edges, henceforth referred to as spacing. Control sample containing no additive expanded 0.2 per cent after 13 cycles.

Spacing can be readily calculated from a knowledge of the size and concentration of the added particles. The mesh sizes utilized and the corresponding average particle diameters are given in Table 1. The spacing for various concentrations, assuming spherical shape, are shown in Figure 2. It is evident that the spacing can be reduced by increasing the concentration or by reducing particle size because at a given weight per cent the number of particles increases with decreasing size.

Series II. The objective of the second series of experiments was to obtain confirmation of the results of the first series and investigate the effect of another type of brick particle and diatomaceous earth.

TABLE 1

Average Particle Diameter in the Utilized Fractions

TYLER MESH		DIAM. mm
RANGE	AVG	
8-12	10	2.00
12-16	14	1.41
16-20	18	1.00
20-30	25	0.71
30-40	35	0.50
40-50	45	0.35
50-100	75	0.22
16-30	23	0.89
30-50	40	0.45

Figure 2. Spacing (distance between the edges of particles) as function of particle diameter for various concentrations.

Figure 1. Freeze-thaw resistance, expressed as number of cycles required to produce 0.2% residual expansion, as function of spacing, of neat cement samples.

TABLE 2

Number of Freeze-Thaw Cycles Endured by Neat Cement Specimens, Containing Porous Particles, Before Breakdown. Calculated Spacings, in mm, are given in Parenthesis. The w/c = 0.5.

ADDITIVE						
Type	Conc. % by wt.	SIZE mm				
		.15 - .30	.30 - .42	.42 - .59	.59 - .83	.83 - 1.17
B R I C K "A"	9		(0.42) 95, 400	(0.60) 54, 100	(0.86) 130, 130	(1.24) 127, 475
	16	(0.18) >1260	(0.29) 650, 1250	(0.42) >1260, >1260	(0.58) >1260, >1260	(0.84) >1260, >1260
	28			(0.26) >1260, >1260	(0.36) >1000	
B R I C K "B"	9			(0.60) 75, 111	(0.86) 35, 39	
	16	(0.12) 41, 33	(0.24) 115, 67	(0.42) 131, 145	(0.58) 323, 399	(0.84) 48, 59
	28			(0.26) >1260, >1260	(0.36) 115, >1260	
D E I A A R T T H	2.6			650, >1260	105, 119	
	3.6					26, 37
S A N D	16			21		28, 44
NEAT CEMENT PASTE				48, 53		

In contrast to the first series where the 0.2 per cent expansion was taken as criterion for the test, in the second series the actual number of cycles needed to deteriorate the specimens to such an extent that they had to be removed from the automatic freeze-thaw machine, were recorded and are given in Table 2. A different basis of comparison was necessary because in a consider-

TABLE 3

Number of Freeze-Thaw Cycles Endured by Neat Cement Samples, Containing Fly Ash Particles, Before Breakdown

w/c ratio	FLY ASH					
	Type	Conc. % by wt	SIZE mm			
			.149 - .30	.30 - .42	.42 - .59	.59 - .84
.05	agglom.	9				25
		16			42	23
	sintered	9		18	30	30
		16			26	26, 77
	plain	0	25, 26			
.07	agglom.	9	13		20	13
		16	17	13		13, 22
	sintered	9			20	13
		16		18	13	13, 15
	plain	0	13			

able number of cases the sought value could not be ascertained owing to the lack of continuous length change data. It must be noted that in every case where the samples endured more than 1260 cycles, no residual length change greater than 0.01 per cent was observed.

Porous fly ash particles made by spherical agglomeration and sintering were also used as additive in neat cement specimens and the number of freeze-thaw cycles endured by them before breakdown are given in Table 3.

For experiments with concrete, bars (7.5 by 7.5 by 30 cm) were fabricated with crushed brick (type A) or diatomaceous earth as additive. The basic concrete mix had a cement:aggregate:sand ratio of 1:2.75:2.25 and a water:cement ratio of 0.58. In Table 4 the performance of concrete bars containing diatomaceous earth particles .84 to .30 mm in size and approximately 4.4 per cent by weight in concentration is compared to that of bars made of the same mix but without the additive. Frost resistance was evaluated on the basis of residual length changes, $\Delta l/l$, and decrease of the relative modulus of elasticity, E/E_0 , during repeated freezing and thawing.

The residual length changes of concrete bars containing brick particles (Type A, 0.3 to 0.8 mm) in various concentrations are given in Table 5 and compared with those of air-entrained concrete (1 ml/1520 g cement). A discrepancy appears to exist inasmuch as specimens containing brick particles in 10 per cent concentration showed less frost resistance than the sample containing 5 per cent brick particles. As durability is directly proportional to the quality of entrained air, and thus to particle concentration, the sample containing 5 per cent brick is expected to be the most susceptible to freeze-thaw damage. Values of Table 6 are consistent with the stated concentrations and, therefore, the relatively poor performance of the specimen with 10 per cent brick has to be ascribed to experimental error in fabrication. This view is supported by the variation of slump which was 3.5 in. in the case of the

TABLE 4

Residual Fractional Length Changes, $\Delta l/l$, and
Relative Dynamic Modulus of Elasticity, E/E_0
of Concrete Specimens With and Without Diatomaceous
Earth Additives, During Repeated Freezing and
Thawing Cycles.

Number of cycles	Diatomaceous earth			
	with	without	with	without
	$\Delta l/l$, %		E/E_0	
0	0.00, 0.00	0.00, 0.00	1.00, 1.00	1.00, 1.00
10	0.00, 0.00	0.00, 0.00	0.95, 0.95	0.97, 0.97
25	0.00, 0.00	0.00, 0.00	0.97, 0.97	0.97, 0.97
100	0.01, 0.01	0.00, 0.03	0.99, 0.99	0.92, 0.92
200	0.03, 0.03	0.09, 0.12	0.98, 0.97	0.38, 0.38
300	0.04, 0.04	0.39, 0.45	0.97, 0.97	
430	0.06, 0.07	1.63, 1.31		

TABLE 5

Residual Length Changes of Concrete Specimens Containing Type "A" Brick Particles. Concentrations are Given in Weight Per Cent of Cement Content

Sample	$\Delta l/l, \%$		
	50 cycles	160 cycles	360 cycles
Plain	0.117, 0.103	0.702, 0.478	1.908, 1.202
5% brick added	0.009, 0.006	0.015, 0.018	0.026, 0.025
10% brick added	0.011, 0.010	0.108, 0.034	0.218, 0.158
20% brick added	0.003, 0.005	0.012, 0.015	0.022, 0.022
air entrained	0.007, 0.005	0.010, 0.009	0.025, 0.019

TABLE 6

The Air and Brick Contents, and Spacing Factors of Specimens Containing Type "A" Brick Particles. Brick Concentrations are Given in Weight Per Cent of Cement Content and the Spacing Factors in Inches

Sample	Air Content		Brick Content	
	Vol %	Spacing Factor	%	Spacing Factor
Plain	2.25	0.046	0.00	∞
Air entrained	5.34	0.013	0.00	∞
5% brick	1.92	0.033	1.16	0.030
10% brick	1.62	0.029	1.91	0.022
20% brick	2.49	0.033	3.69	0.012

TABLE 7

Compressive Strength of Plain and Air Entrained Concrete Compared with that of Concrete Containing Type "A" Brick Particles. Concentrations are Given in Weight Per Cent of Cement Content

Sample	Compressive strength kPa (lb/sq in.)	
Plain	24,408 (3540)	25,925 (3760)
5% brick added	26,821 (3890)	27,235 (3950)
10% brick added	27,304 (3960)	27,717 (4020)
20% brick added	28,269 (4100)	27,304 (3960)
Air entrained	24,063 (3490)	21,236 (3080)

10 per cent brick sample and 2.5 in. in the others. Because of the small quantities available, the results of the slump test are suspect, too, but if correct the higher slump could explain the irregular behaviour. The compressive strength of cylinders made from the same mixes are listed in Table 7.

The pore size distribution and total porosity of the various additives is summarized in the histograms of Figure 3.

Discussion

The results of Tables 2 and 4 attest to the effectiveness of the porous particles in enhancing the freeze-thaw durability of cement paste; while the mortar paste was destroyed after 21 freeze-thaw cycles, cement containing 16 per cent brick particles endured 1260 cycles without injury.

In practice, the obvious aim is to provide adequate durability with least amount of brick. At a given brick concentration freeze-thaw

Figure 3 Pore size distribution of additives used in experiments

resistance can be enhanced by using smaller particles, thus achieving smaller spacing. Particle size cannot be reduced, however, beyond the stage at which the internal pore volume of the particle becomes insufficiently large to protect the area surrounding it. The limiting particle size in case of bricks appears to be between 0.4 and 0.8 mm.

The two types of brick protect the concrete to a different degree, type "A" being more effective than type "B". Apparently the larger porosity of brick A (36 per cent vs 16 per cent) and its particular pore size distribution (Figure 3) is advantageous.

None of the fly ash conglomerates proved to be beneficial, presumably because of unfavourable pore size distribution (Figure 3). The results of the present experiments, however, must not be interpreted as proof of the inability of fly ash based particles to act as a protective agent against freeze-thaw damage. It may be assumed that if particles with the desired porosity and pore size distribution can be fabricated, the product will be an effective admixture.

Diatomaceous earth has the required total porosity and pore size distribution and when mixed in concrete or cement paste causes a significant improvement in the freeze-thaw resistance. It has to be noted, however, that diatomaceous earth particles originating from certain deposits, while effective from a frost resistance point of view, cause "map cracking," suspected to be caused by alkali-silica reaction.

The compressive strength values of Table 7 reveal that the incorporation of brick particles, in contrast to air entrainment, increases compressive strength, an important consideration for certain applications.

The described method of rendering cement and concrete frost resistant is described in Canadian Patent Application 241,032.

Conclusion

Incorporation of suitable porous particles in the plastic mix increases the freeze-thaw resistance of hardened concrete without the serious shortcomings of the conventional air entrainment method (instability of bubbles and strength reduction).

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