Preliminary Study of Coatings and Impregnants for Upgrading Frost-Sensitive Aggregates

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Laboratory tests carried out to evaluate the potential of various types of coatings and impregnants as means of upgrading frost-sensitive aggregates are described. Although some of the materials tested would probably not be economical for commercial application, they were chosen to represent classes of materials that might include some that could be economically feasible. The impregnants tested were epoxy (diluted with a solvent), methyl methacrylate, boiled linseed oil, polyethylene glycol, and sulfur. The coatings included epoxy and linseed oil emulsion. Two aggregates were used that were known to be highly frost-susceptible, but for different reasons. One was a chert river gravel of moderately high absorption but very low permeability. Its mode of failure under freezing and thawing conditions is internal fracturing and dilation of the aggregate particles as a result of the development of excessive hydraulic pressures. This aggregate was treated by impregnation because of the high capillary potentials offered by the small pore sizes. The second aggregate was a vesicular, highly porous andesite with very large pore sizes. Its primary mode of failure in concrete under freezing conditions involves the expulsion of water to the paste phase ahead of the advancing frost zone. Because of the large absorptive capacity of this aggregate, an attempt was made to block the pores by using external surface coatings. Triplicate sets of specimens were used in the freeze-thaw tests for each aggregate-treatment combination in accordance with ASTM C682. Both coatings and all impregnants except sulfur were found to render the test aggregates non-frost-susceptible. Specimens containing untreated (control) aggregates failed early in freeze-thaw tests.

Deterioration of portland cement concrete (PCC) as a result of freezing and thawing can occur because of deficiencies in either the paste phase or the aggregate phase. The paste phase can readily be protected by incorporating a suitable entrained air-void system.

The coarse-aggregate fraction can be damaged by freezing and thawing (with resultant damage to the concrete) in two ways or in a combination of these ways (1,2). When the aggregates have moderate to high porosity and low permeability, the hydraulic pressures developed in the rock pores ahead of the advancing ice front during freezing can be of sufficient magnitude to cause fracturing of the aggregate. When the aggregates have high porosity and high permeability, the pressures developed by water being forced out of the rock during freezing can break the paste-aggregate bond and disrupt the surrounding paste phase.

In view of the nature of the destructive mechanisms involved, it was considered highly likely that frost-sensitive aggregates could be benefited by a process of drying followed by sealing of the penetrable pores. Indeed, this approach has been suggested by others (3,4).

For aggregates that have small pore sizes (low permeabilities), this could probably best be accomplished by impregnation, since the high capillary potential of these aggregates will aid in the upgrading process. Aggregates having large pore volumes and sizes (high permeabilities) would probably be more amenable to coating treatments. Chert is an example of the first type and vesicular lava (andesite) an example of the second. In the case of vesicular lava, impregnation might not even be economically practical because of large porosities (as high as 40 percent), but blocking of the pores at the surface with a viscous coating material might be feasible.

TEST AGGREGATES

One aggregate source was selected to represent each of the two failure modes (and, therefore, treatment processes) described above. The criteria used to select the two aggregate sources were (a) the frost susceptibility of the aggregate (the most deleterious aggregates received the highest priority), (b) the geographic area of the country (aggregates from areas that lack high-quality aggregates were preferred), (c) the degree of documentation of the performance of the aggregate (those aggregates on which the most documentation was available were favored), and (d) the availability of the aggregate.

Although the rationale behind the selection criteria is generally obvious, it should be mentioned that the decision to seek out the most deleterious aggregates for this test program was based on the premise that, if such aggregates could be upgraded, there should be no problem in upgrading less seriously affected aggregates. There are two difficulties in using "marginal" aggregates: The laboratory tests frequently lack sufficient discrimination to be of much use in showing improvements in marginal aggregates, and the applicability of a solution to a problem involving a marginal aggregate is much less general (i.e., the solution works for the aggregate tested and for better ones, but it may not work for worse aggregates).

Twenty-nine sources of documented frost-susceptible aggregates were identified in a detailed literature review. Based on the selection criteria given above, a chert gravel from Kentucky was selected for the impregnation tests and a hornblende andesite gravel from California for the coating tests.

According to the results of tests in previous research (5,6), concrete specimens containing these aggregates in the saturated state failed in only one or two freezing and thawing cycles in the laboratory (ASTM C671). The chert gravel was reported to display very low permeability (10^{-9}-10^{-5} darcys, averaging 10^{-6}) and moderate effective porosity (3-12 percent, averaging 8 percent). The andesite gravel reportedly had permeability values that averaged 1000 times that of the chert (10^{-4}-10^{-2} darcys, averaging 10^{-3}). In addition, the porosity of the andesite was appreciably greater than that of the chert (12-43 percent, averaging 28 percent). Petrographic descriptions of the two selected test aggregates, as abstracted from the references, are given below.

Chert River Gravel

Chert river gravel is an angular, yellow-brown and white, massive to laminated chert consisting mostly of microcrystalline quartz [average grain size 0.006 mm (0.0002 in) in diameter] and 0-20 percent fossil fragments composed of quartz grains with an average diameter of 0.06 mm (0.002 in). Most samples contain banded iron-stained chalcedony, which occurs as irregularly shaped masses as large as 1 cm (0.39 in) diameter or as interstitial cement, both commonly with quartz overgrowths. Most samples have a smooth weathered coating of limonite and chalcedony-rich chert. All contain as much as 9 percent dolomolds [average diameter of 0.5 mm (0.019

in)], most of which are disseminated but commonly occur in continuous layers. Many of these rhomb-shaped cavities are filled or partly filled with limonite. All samples, especially the chalcedony, contain numerous microfractures that appear to be incipient desiccation fractures. Many of the dolomolds are interconnected by these microfractures. The specific gravity of individual particles varies inversely with the percentage of dolomolds present.

Hornblende Andesite Gravel

Hornblende andesite gravel is somewhat variable in color, ranging from pale gray through dark gray, purplish gray, and grayish black. The structure varies from highly vesicular (pumiceous), crumbly, brittle, and fractured to massive and structureless. Occasionally, flow banding is exhibited and the aggregate is generally rich in hornblende phenocrysts, sometimes oxidized. Particle shapes vary from angular or subangular for the massive material to subrounded or rounded for the more vesicular material.

TREATMENT MATERIALS

The treatment materials chosen for coating or impregnating the test aggregates were selected to represent classes of potential coatings and impregnants. Given the preliminary nature of this study, no attempt was made to optimize the selection of treatment materials within the various classes with regard to economic or practical feasibility. However, a high-volume waste material (sulfur) was included as an impregnant in view of its obvious economic advantages. As a coating material, epoxy was selected to represent the most durable polymer type of material that would be expected, in general, to seal pore entrances on the aggregate particles. Linseed oil emulsion was selected to represent a class of coating materials that is intended to coat pore surfaces and reduce pore moisture by increasing the contact angle. The impregnants chosen and the classifications that they represent are as follows:

1. Boiled linseed oil--self-polymerized (oxidation) polymeric material,
2. Epoxy (diluted with organic solvent to reduce viscosity)--condensation-type polymeric material,
3. Methyl methacrylate--addition-type polymer,
4. Polyethylene glycol--water-miscible polymer of high molecular weight, and
5. Sulfur--a large-volume waste material.

Preliminary tests were carried out with some of the treatment materials to examine the effects of the treatments on the water-absorption properties of the aggregates. The results are given below:

Treatment	Reduction in Water Absorption (%)	
	Chert	Andesite
Epoxy coating		86
Linseed oil emulsion coating		60
Epoxy impregnation	76	
Methyl methacrylate impregnation	59	
Boiled linseed oil impregnation	46	

Tests were also carried out to determine the uptake of most of the treatment materials by the test aggregates. These results were as follows:

Treatment	Treatment Uptake (percent by weight)	
	Chert	Andesite
Epoxy coating		8.9
Linseed oil emulsion coating		3.8
Epoxy impregnation	2.8	
Methyl methacrylate impregnation	2.4	
Boiled linseed oil impregnation	1.0	
Polyethylene glycol impregnation	6.2	
Sulfur impregnation	4.0	

EXPERIMENTAL PROCEDURES

The test aggregates were graded into four size groups and recombined to provide a standard gradation, as given below (1 mm = 0.039 in):

Sieve Size Range (mm)	Percent by Weight
19.0-25.0	25
12.5-19.0	25
9.5-12.5	25
4.75-9.5	25

Specific gravities, absorptions, and dry rodded unit weights were determined for each aggregate, before and after treatment, to provide mixture design data. Details on the treatment techniques are given in Table 1.

The portland cement used in this research came from a single production lot of low-alkali (0.51 percent Na_2O equivalent) type 1 cement. The fine aggregate was a high-quality alluvial quartz sand. The concrete mixtures were proportioned to give slumps in the range of 5-7.5 cm (2-3 in), air contents of 5 ± 1 percent, and a water-cement ratio of 0.45 by weight.

The specimens used in this test were cylinders 7.5 cm (3 in) in diameter by 15 cm (6 in) high that had cast-in-place gage studs at each end. Each aggregate-treatment combination was represented by three replicate specimens. Specimens that contained untreated aggregates were included for control purposes.

The freezing and thawing tests were carried out in accordance with ASTM C671 and C682, except for the following:

1. Aggregate conditioning prior to mixing consisted of soaking for 24 h.
2. Specimen conditioning consisted of soaking in water at 1.7°C (35°F) for three weeks after the prescribed curing period (1 day in molds, 13 days in limewater).
3. Freeze-thaw cycling was continued for each specimen until either the dilation exceeded 25 μm (1000 μin) or 12 cycles were completed, whichever came first (test cycle once every two weeks).

Changes in specimen length during freezing were monitored by using linear variable differential transformers, and the expansions (dilations) that occurred were measured later from recorder charts. It can be shown that a dilation of approximately 75 microstrain represents the elastic limit of a typical PCC (6). For the 15-cm (6-in) long specimens used in this research, this represents a critical dilation of about 11.25 μm (440 μin). Therefore, it has been assumed here that dilation values that exceed this critical value are indicative of failure of the concrete as a result of freezing and thawing action.

RESULTS

The test results for the chert aggregate are summarized in Figure 1. Each curve shown represents the average for the three specimens tested for each treatment material. As usual in this test, the dilation values below the critical dilation tended to be somewhat erratic but, once the critical dilation was exceeded, subsequent dilation values tended to increase exponentially. It is evident from Figure 1 that all of the impregnants except sulfur (which failed in the 11th cycle) were effective in preventing freezing and thawing damage. It should be noted that the control (untreated) specimens failed early in the test (third cycle).

Table 1. Coating and impregnation techniques.

Treatment	Formulation of Treatment Material	Treatment Technique[a]
Epoxy coating	DGEBA, TETA (50 percent stoichiometric)	Aggregate removed from the oven, placed in epoxy mixture for 5 min while still hot, then placed on screens to polymerize
Linseed oil emulsion coating	100 percent LOE	Aggregate placed in LOE for 5 min (room temperature), then placed on screen for curing (7 days)
Epoxy impregnation	DGEBA:TETA: xylene (100:14.1:25 parts by weight)	Aggregate soaked in epoxy mixture for 1.75-2 h; treated aggregate spread out on screen for room-temperature polymerization
Methyl methacrylate impregnation	MMA:TMPTMA:AZO (100:10:0.5 parts by weight)	Aggregate soaked in MMA mixture for 4 h at room temperature; treated aggregate placed in 75°C hot-water bath for 24 h to polymerize MMA system
Boiled linseed oil impregnation	100 percent BLO	Hot aggregate (105°C) soaked in BLO; aggregate allowed to cool to room temperature during impregnation (24 h); treated aggregate placed in 105°C forced-draft oven for about 24 h to accelerate polymerization of BLO
Polyethylene glycol impregnation	100 percent PEG	Aggregate placed in 60°C oven for 24 h while soaking in PEG, then placed on screen to cool
Sulfur impregnation	95 percent sulfur, 5 percent tar	Aggregates heated to 121°C placed in 121°C sulfur mixture for 30 min, then placed on screen to cool

Notes: t°C = (t°F - 32)/1.8.
DGEBA = diglycidyl ether of bisphenol A, TETA = triethylenetetramine, LOE = linseed oil emulsion, MMA = methyl-methacrylate, TMPTA = trimethylol propane trimethacrylate, 2,2'-azobisisobutyronitrile (AZO), BLO = boiled linseed oil, PEG = polyethylene glycol.
[a]All aggregates were oven dried in a forced-draft oven at 105°C prior to receiving treatments.

Figure 1. Dilations in freezing and thawing cycles for concrete containing chert aggregate.

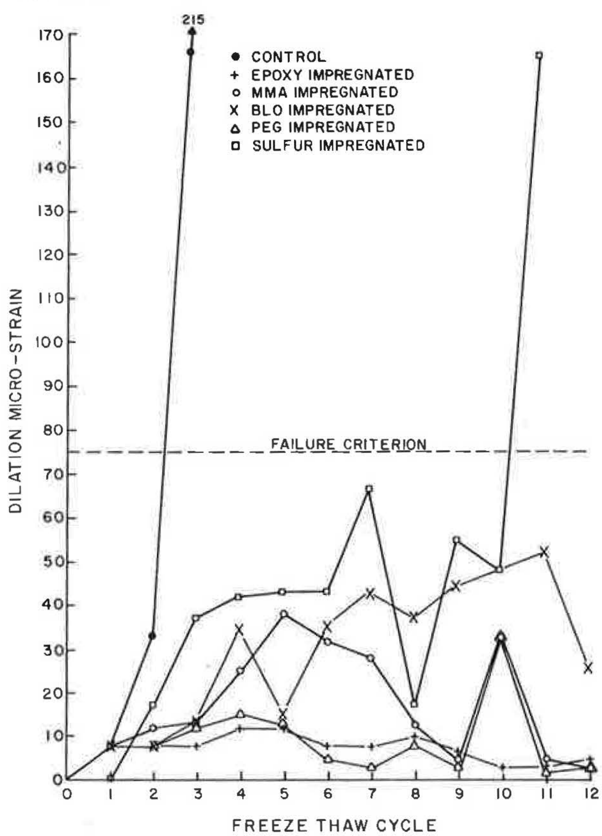
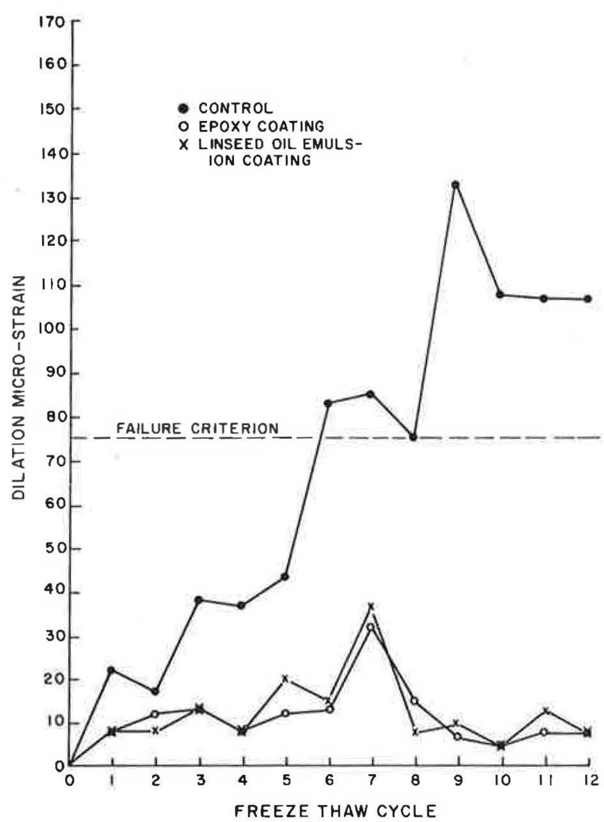


Figure 2. Dilations in freezing and thawing cycles for concrete containing andesite aggregate.



The test results for the andesite aggregate are shown in Figure 2. Again, each curve shown represents the average of three specimens. The tests demonstrate that both coating treatments tested were effective in preventing freezing and thawing damage. The control (untreated) specimen failed in six cycles of freezing and thawing.

CONCLUSIONS

Epoxy and linseed oil emulsion coatings and epoxy, methyl methacrylate, boiled linseed oil, and polyethylene glycol impregnants were all found to be eminently successful in upgrading highly frost-susceptible aggregates. Even sulfur, the only impregnant that did not meet the failure criterion, improved the frost resistance of these aggregates by a factor of five. The high degree of success in preventing freeze-thaw damage is considered to be a matter of no small consequence. Frost susceptibility is generally considered to be the most common characteristic limiting the use of aggregate materials in PCC. In addition, the results suggest that there exists a wide range of coating or impregnation materials that are capable of eliminating the frost susceptibility of aggregates.

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State of the Art in Use of Nuclear Density Gages on Portland Cement Concrete

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Proper consolidation during construction improves all of the important properties of portland cement concrete. The state of the art in state highway department use of commercially available, static nuclear density gages to monitor concrete consolidation is discussed based on the results of a 1977 Federal Highway Administration survey of state highway departments. The survey showed rapidly growing use of the gages for controlling the density of thin bridge-deck overlays, particularly of low-water-cement-ratio (0.32), low-slump (Iowa) concretes. Some use during construction of full-depth bridge decks and of pavements was also reported. A discussion of the problems involved in the use of nuclear gages—e.g., the effect of reinforcing steel on gage response and the selection of appropriate density standards—is also included.

Late in 1977, the Federal Highway Administration (FHWA) surveyed state highway departments regarding their use of nuclear gages to monitor the in-place density of fresh (plastic) portland cement concrete (PCC). This paper summarizes the responses to the survey and discusses various procedures and recommendations for future research. Portions of individual state responses are given in Table 1.

CURRENT STATE USE OF NUCLEAR GAGES

Eleven states reported extensive use of nuclear gages at the time of the survey, and another 16 reported current evaluation studies or plans for such studies in the immediate future. The responses showed that attention is being focused on use of the gages to control the density of thin bridge-deck overlays, particularly of concretes with low water-cement (w/c) ratio and low slump (Iowa concretes). Some use of nuclear procedures during construction of full-depth bridge decks and of pavements was also reported.

CONSTRUCTION OF BRIDGE-DECK OVERLAYS

Mode of Gage Operation

The first step in the development of a procedure for monitoring overlay densities is the choice of the operating mode for the nuclear gage--either direct transmission or backscatter. The states that use the gages extensively on thin overlays are divided almost evenly between the two modes. Neither method is clearly superior, and each shows certain disadvantages.

Direct Transmission

In direct transmission, the probe containing the radioisotope source is immersed in the concrete. The commercial gages normally allow the probe to be inserted to set depths that vary from 50 to as much as 300 mm (2-12 in). Use of the 50-mm setting on