How to Make Concrete that will be Immune to the Effects of Freezing and Thawing

by B. Mather

Synopsis: Concrete will be immune to the effects of freezing and thawing if (1) it is not in an environment where freezing and thawing take place so as to cause freezable water in the concrete to freeze, (2) when freezing takes place there are no pores in the concrete large enough to hold freezable water (i.e., no capillary cavities), (3) during freezing of freezable water, the pores containing freezable water are never more than 91 percent filled, i.e., not critically saturated, (4) during freezing of freezable water the pores containing freezable water are more than 91 percent full, the paste has an air-void system with an air bubble located not more than 0.2 mm (0.008 in.) from anywhere ($\bar{L} \leq 0.2$ mm), sound aggregate, and moderate maturity. Sound aggregate is aggregate that does not contain significant amounts of accessible capillary pore space that is likely to be critically saturated when freezing occurs. The way to establish that such is the case, is to subject properly air-entrained, properly mature concrete, made with the aggregate in question, to an appropriate laboratory freezing-and-thawing test such as ASTM C 666 Procedure A. Moderate maturity means that the originally mixing water-filled space has been reduced by cement hydration so that the remaining capillary porosity that can hold freezable water is a small enough fractional volume of the paste so that the expansion of the water on freezing can be accommodated by the air-void system. Such maturity was shown by Klieger in 1956 to have been attained when the compressive strength reaches about 4,000 psi.

Keywords: age; aggregates; air-entrained concretes; air entrainment; capillarity; compressive strength; concretes; freeze-thaw durability; porosity; saturation; soundness; voids; water

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INTRODUCTION

Most of the concrete that has ever been made in the world was made and used in environmental circumstances in which the issue of whether or not it would be adversely affected by freezing and thawing was irrelevant since the environmental exposure either did not involve freezing and thawing or it could be assumed with confidence that, when the concrete froze, it would not be critically saturated. It is not necessary to know how to make concrete that will be immune to the effects of freezing and thawing if the concrete that one is interested in making will never be exposed to freezing and thawing when in a critically saturated condition.

Concrete that is used in environmental situations in which it is exposed to freezing and thawing while critically saturated and which is unaffected by such exposure has been made for probably as long as concrete has been made. Such inadvertently or unintentionally frost-resistant concrete has been made using aggregates that were frost resistant, with cement paste that was air entrained, or was concrete that, in spite of becoming critically saturated, had capillary porosity sufficiently low that the 9 percent expansion of the freezable water could be accommodated by the elastic and creep strain capacity of the concrete without rupture.

So far as I can tell from such literature as I have examined, up until about 50 or 60 years ago most people concerned with resistance of concrete to freezing and thawing simply used the rule of trying to figure out what materials and proportions and construction practices had been used previously with success in a comparable exposure and attempted to duplicate or at least simulate those same materials and proportions and practices in the new concrete; without understanding, in any particular detail, why those aggregates, a cement of that composition, and a concrete produced in the way a particular concrete was produced, yielded "durable" concrete.

This symposium honors Paul Klieger. With that in mind, I looked at the index of the first 227 Bulletins of the Research Department of Portland Cement Association Research and Development Laboratories, published in July 1969, and I observed that 18 Bulletins are listed in the author index following the entry "Klieger, Paul." I then looked at the titles and abstracts of

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each of these, beginning with Bulletin 24 in 1949, which deals with the effect of entrained air on a number of properties of concrete including freezing-and-thawing resistance, all the way down to Bulletin 218 in 1967 which deals with laboratory studies of blended cements for their effects on properties of concrete including freezing and thawing. So far as I could tell, 15 of the 18 Bulletins listed were concerned wholly or in part with freezing and thawing. As I shall emphasize later, it was Klieger's 1956 Highway Research Bulletin No. 150 paper that became PCA Research Bulletin 82 (Klieger, 1956) that was called "Curing Requirements for Scale Resistance of Concrete" that contributed for the first time, at least that I ever took in, the third critical element to the recipe on how to make concrete that will be immune to the effects of freezing and thawing.

In the development of this topic, I shall state the conclusion first and then discuss some of the backup and derivation.

RECIPE FOR CONCRETE THAT WILL BE IMMUNE TO THE EFFECTS OF FREEZING AND THAWING

There are two ways of stating the recipe, depending upon how much information the audience being addressed is presumed to be qualified to assimilate. If the audience is not presumed to be able to assimilate much information and should not be given the opportunity to exercise much judgment, then the recipe might properly take the form of saying "Follow the guidance and specification requirements stated in appropriate applicable guides and specifications." What this will then come down to is that the recipe will require that the cementitious materials, the aggregates, the mixing water, and the admixtures will be required to be tested to determine compliance with the appropriate options of the applicable ASTM or other governing standards, that the mixtures shall be proportioned according to the applicable portions of the ACI <u>Manual of Concrete Practice</u> (ACI 211.1) and the materials will be handled, batched, conveyed, mixed, placed, compacted, finished, and cured, all also in accordance with applicable documents in the ACI <u>Manual of Concrete Practice</u>. One of the critical features of this recipe will be that someone must decide whether or not it is practical to have job-site testing for air content of fresh concrete. If so, under normal conditions, the cementitious materials used will be nonair-entraining and the air-void system in the cement paste in the concrete will be obtained by the use of an air-entraining admixture, the appropriateness of the dosage of which will be confirmed by job-site testing for air content of fresh concrete. If such tests are not practical and air-entraining cement is available, then air-entraining cement meeting ASTM C 150 for Type IA, Type IIA, Type IIIA, or ASTM C 595 for Type I(SM)-A, Type IS-A, Type I(PM)-A or Type IP-A, Type IS-A(MS) or Type IP-A(MS) will be specified. The

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aggregates will be required to meet ASTM C 33 which will cause only frost-resistant aggregate to be used and the mixing water and the admixtures will be in accordance with appropriate specifications to ensure that they contribute what they need to contribute and do not interfere with the achievement of the appropriate levels of relevant properties produced by the interaction of everything else that is going on.

Under the second alternative, where the audience is perceived to be qualified to understand technical considerations and exercise some level of judgment, the recipe for concrete that will be immune to the effects of freezing and thawing may take a somewhat different form. Instead of discussing whether one shall use air-entraining cement or nonair-entraining cement and an air-entraining admixture, the recipe may instead require that the paste portion of the concrete shall contain an air-void system characterized by a bubble-spacing factor ($\bar{L}$) not greater than 0.008 in. (0.2 mm) (ACI 201, 212); which is to say simply that regardless of how it is achieved, the cement paste in the hardened concrete shall contain an air bubble not farther away than 0.008 in. (0.2 mm) from anywhere.

Two comments are appropriate at this point. First, this requirement will, under most conditions, be met if air-entraining cement meeting the relevant ASTM cement specifications or an air-entraining admixture meeting ASTM C 260 is used at a dosage sufficient to meet the air content of fresh concrete requirements stated in the documents prepared by ACI Committees 201 and 212 in the ACI Manual. The second comment is that the 0.008 in. (0.2 mm) spacing factor is the maximum for concrete of a particular level of permeability through which the freezing isotherm is moving at a given rate as a function of the thermal properties of the concrete and the temperature difference between the near surface concrete and the surroundings. What this means is that, assuming the hydraulic pressure mechanism for frost damage, if the depth to which the temperature drops and the speed with which it drops is great enough or the permeability of the concrete is low enough, then the bubbles may need to be closer together if they are to serve their intended function. If the thermal gradient is steeper, the freezing isotherm will move into the concrete more rapidly and if the concrete is less permeable the excess water will move through it more slowly. One further consideration is that most processes by which the permeability of concrete can be materially reduced are also processes by which its porosity is concomitantly significantly reduced. Therefore, all other things being equal, if it is harder for the water to get through the concrete there will also usually be substantially less water going through it as a function of whatever mechanism is presumed to be causing water to move or need to move in connection with the phenomenon of freezing. Finally there is the point that the liquid in a capillary pore is a solution of given concentration before any of it freezes. When some of it freezes the solution concentration of the rest increases and it has a lower freezing

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point thus it will only freeze when a lower isotherm comes by.

The issue of aggregates may very well be dealt with, not simply by requiring with compliance of ASTM C 33, but rather by requiring that the aggregates under consideration be combined into a concrete mixture using either a standard air-entrained cement paste or the cement paste such as that likely to be used in the construction under consideration. Once the concrete has been proportioned, mixed, formed into specimens, finished, and cured, any of several testing procedures may be employed. Perhaps the most widely used is ASTM C 666 Procedure A, which involves subjecting prismatic specimens to laboratory freezing and thawing while the specimen is immersed in water or ice and determining, at selected intervals, the degree to which such treatment has affected some measurable property of the concrete. The most widely used property is fundamental resonant frequency of vibration from which dynamic Young's modulus of elasticity may be calculated. However, alternatively or additionally, the specimens may be tested for pulse velocity, length change, or change in mass, or they may be sacrificed periodically to measure flexural strength or tensile strength or compressive strength in comparison with companion specimens moist cured to the same age. In addition to ASTM C 666, there is also ASTM C 671, the method for critical dilation of concrete specimens and its companion ASTM C 682, the practice for evaluation of frost resistance of coarse aggregate in air-entrained concrete by critical dilation. This pair of methods, derived from recommendations originated by T. C. Powers (1955) and used very effectively by Bailey Tremper in the California DOT (Tremper and Spellman, 1961), and later advocated strongly by Alan D. Buck (Buck, 1976) at the Waterways Experiment Station, permits the measurement of the time required for a specimen of given initial moisture condition to achieve both critical saturation and, consequently, susceptibility to damage by freezing and thawing if the concrete is intrinsically not frost resistant either by reason of containing nonfrost-resistant aggregate or a paste of inadequate air-void system or a fractional volume of freezable water greater than can be accommodated by the air-void system which is a consequence of inadequate maturity at the time of freezing. Finally, there is the procedure ASTM C 672 for scaling resistance of surfaces exposed to deicing chemicals, by the use of which one can measure this specific manifestation of lack of immunity to freezing and thawing. ASTM C 672 is a procedure to the development and use of which Paul Klieger made probably greater contributions than were made by anyone else (Klieger, 1955; 1956; Klieger and Perenchio, 1963; Verbeck and Klieger, 1956). The mechanism by which a particle of porous unsound aggregate generates internal force sufficient to produce a popout if frozen while critically saturated has been elegantly described by Bache and Isen (1968). The procedure for recognizing such particles has been most thoroughly studied by Larson and Cady (1969).

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Finally, after one has dealt with the air-void system in the paste and the quality of the aggregate, there is the third critical requirement regarding concrete that is to be immune to frost action and that is maturity and it is this requirement that we owe primarily to Paul. In his 1956 Highway Research Board paper, which became PCA Bulletin 82 (Klieger, 1956), one of his conclusions was "the development of a certain level of strength has merit, as an index to the amount of curing required for air-entrained concrete prior to permitting the use of deicers." What he found was that for a number of different concretes made with Types I, II, and III cement and, in the case of the Type I and Type II with and without 2 percent calcium chloride, cured at 73, 40, and 25° F, that it took anywhere from 7 days to more than 60 days of moist curing to develop satisfactory resistance to the form of freezing and thawing damage called "deicer scaling." He then reported the compressive strengths of these concretes and they ranged in general from somewhat over 3,000 to somewhat over 4,000 psi. To be on the safe side, I have recommended that one not allow repeated cycles of freezing and thawing of critically saturated concrete to occur prior to the concrete having achieved a strength of 4,000 psi. As I mentioned, Paul said this in 1956. It took awhile for some of us, especially me, to take it in.

In 1962 I presented a paper at the Highway Research Board on effects of duration of moist curing on freezing and thawing of concrete as measured by ASTM C 666 Procedure A (Mather, 1962). In it results were given of quite a lot of tests on quite a number of different concretes made with and without entrained air at water-cement ratios of 0.5 and 0.8 by mass, subjected to freezing and thawing after 14 and 180 days of moist curing at 73° F. It was clear from these data that nonair-entrained concrete, even at 0.5 water-cement ratio, cured for 180 days, which developed strengths of up to 8,100 psi, never got a durability factor greater than 10. On the other hand, air-entrained concrete of 0.8 water-cement ratio cured only 14 days with compressive strengths under 4,000 psi showed durability factors rarely as high as 60. Lower water-cement ratio (0.5) concrete showed durability factors in excess of 60 even at 14 days because the volume of originally water-filled space was initially much less than in the 0.8 W/C concrete and it was sufficiently reduced by cement hydration so that when that space was filled with freezable water its fractional volume was not greater than that which could be accommodated by the air-void system. Since these data are not readily accessible I present them here. The concretes were made using five portland cements, with and without an air-entraining admixture used to give an air content of 6.0 ± 0.5 percent. The aggregate was 3/4-in. nominal maximum size crushed limestone. Mixtures were made at 0.5 and 0.8 W/C by mass. Three rounds were made using a batch on each of three days for each mixture.

Figure 1 (table 1) compares 20 concretes, 10 made at 0.5 water-cement ratio, 10 made at 0.8 water-cement ratio; 10 with entrained air to give an air content of 6.0 ± 0.5 percent and 10

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nonair-entrained; made using the five portland cements; two Type I cements (one of high- and one of low-alkali content), and one each of Types II, III, and IV. As might have been expected, the Type III cement concrete showed the least, and the Type IV cement concrete the greatest increase in strength between 28 and 180 days. The nonair-entrained concretes all showed very little resistance to freezing and thawing; but an apparent trend toward a slight increase in durability factor with increasing age and increasing strength is suggested for those concretes with strengths of 7,000 psi or more. This increase only brings the durability factor up to 10.

The Type III cement concrete showed a reduction of durability factor between 14 and 180 days at both water-cement ratios, and the Type I (low-alkali) cement concrete showed a reduction at the 0.8 water-cement ratio.

Figure 2 (see table 2) shows the behavior of concretes of both water-cement ratios, made with Type II cement, as progressive replacement of the cement, by solid volume, with fly ash is made in three stages. For both water-cement ratios at both ages there is a progressive reduction of both strength and durability factor as the proportion of cement replaced by fly ash increases.

Figure 3 (table 2) shows similar data for ground granulated iron blast-furnace slag.

Figure 4 (table 3) shows the behavior of concretes of both water-cement ratios made with Type IV cement, as the cement is replaced by selected percentages by solid volume of each of six materials. All concretes made with Type IV cement show an increase in both strength and durability factor with age.

Figure 5 (table 3) shows similar relations for concretes made with the Type I (low-alkali) cement. At the 0.5 water-cement ratio all show an increase in strength and durability factor with age. At the 0.8 water-cement ratio the concrete made with Type I (low-alkali) cement, as noted in Fig. 1, shows a decrease in durability factor with age; such a decrease was also shown when this cement was replaced either by 12 percent uncalcined diatomite, 30 percent calcined shale, 35 percent pumicite, or 50 percent slag; with 45 percent fly ash replacement, there was essentially no change in durability factor with age, and with 35 percent natural cement the durability factor showed a sharp increase with age.

Figure 6 (table 4) compares data on 26 mixtures, all made with Type II cement, to which no pozzolan or special cement was added; 13 at the 0.5, and 13 at the 0.8 water-cement ratio. One mixture at each water-cement ratio is that shown in Fig. 1, and contains entrained air but no other chemical admixtures; all the other mixtures contain other chemical admixtures, eight each contain calcium lignosulfonate, calcium chloride, and triethanolamine.

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Figure 7 shows on one graph all the data on air-entrained concrete given in Fig. 1 through 6. Two heavy lines have been drawn indicating an envelope within which essentially all the data points fall. It is suggested, therefore, that for concrete made using the aggregate under study, the compressive strength developed is related to the durability factor within these limits regardless of the age of the concrete (14 to 180 days for durability factor; 28 to 180 days for strength), the nominal water-cement ratio (0.5 to 0.8), the presence of chemical or mineral admixture; or in essence what Klieger showed in 1956, that if you want frost resistance concrete--for example, concrete with a durability factor over 50--you need air-entrained concrete which has no higher a fractional volume of freezable water than that of concrete having matured so as to develop a compressive strength of about 4,000 psi.

One can do some interesting arithmetic if one were to assume a value of the fractional volume of freezable water, consider the 9 percent expansion of this volume, when the water is converted to ice, and the volume of entrained air required to take up that much increase in volume of the freezable water. If one were to assume that there is slightly more than 10 percent freezable water in a given concrete, it would follow that there is 1 percent or so of increase in volume and ideally only 1 percent by volume of entrained air would accommodate this if the air were used with maximum efficiency and were nearly completely compressed. With a 2 percent air content, the air content of each air void would only need to be compressed to one-half of its original volume and the volume increase would be accommodated. However, in fact, one needs 9 percent fractional volume of air in the mortar fraction of concrete in order to achieve successful protection against frost action. This was first emphasized by Klieger in his paper that became PCA Bulletin 77 (Klieger, 1956A). The conventional assumption is that this is because a lot of the air bubbles are a lot bigger than they need to be. If they were all of the optimum size and optimum distribution, the total air content would be quite a lot less and the penalty in strength of 5 percent per 1 percent of air could be proportionally reduced.

WHY THE RECIPE WORKS

In the preceding portions of this paper, I have discussed different forms of the recipe for making concrete that will be immune to frost action as a function of the sophistication of the audience to which the recipe is being presented. There is, however, a third audience that not only wants the recipe presented in a form that permits the exercise of judgment through the possession of understanding, but also wants technical and scientific explanations of how and why all these things work. The principal contributors to the understanding of how air entrainment works in doing good things for the resistance of concrete to

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freezing and thawing have been T. C. Powers (1949, 1954), Richard Helmuth (Powers and Helmuth, 1953), and Bob Philleo (1986). Contributions by each of them are cited in the list of references. The principal contributors to the understanding of the role played by aggregate properties include also T. C. Powers (1955) but especially George Verbeck (Verbeck and Landgren, 1960). Indeed, the subject of resistance of natural aggregate materials, sand, gravel, and crushed stone, to freezing and thawing has a very long history going back at least to 1818 when Brard in France (DeThury, 1829) developed the prototype of what is now ASTM C 88, the standard test for soundness of aggregate by the use of sodium sulfate or magnesium sulfate. This widely misunderstood and generally reviled procedure has been used to assess the durability of building stone and was spoken of quite favorably by Schaffer (1932). It is not always realized that it was always the intent of this test to simulate the expansion of water being converted to ice on freezing by the use of the mechanism of precipitating hydrated sulfate solids in pores in the rock, dehydrating or partially dehydrating these sulfates by oven drying and causing the internal expansion that simulates ice formation by the rehydration of the dehydrated or partially dehydrated sulfates when the specimen is re-immersed in the sulfate solution. ACI Committee 201 has, in the last few years, given attention to the relationship of this mechanism and the deterioration of concrete associated with the movement of sulfate solutions into concrete by capillary action, the subsequent precipitation of salt by dehydration and damage due to rehydration. In this case, the test that was to simulate freezing and thawing also reproduces a completely unrelated phenomenon which may cause destruction of concrete by internal expansion in regions where freezing cannot occur.

CONCLUSION

Concrete will be immune to the effects of freezing and thawing even if critically saturated with water if it is made using sound aggregate, has a proper air-void system, and has matured so as to have developed a compressive strength of about 4,000 psi.

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C 88 Test Method for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate

C 150 Specification for Portland Cement

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C 260 Specification for Air-Entraining Admixtures for Concrete

C 457 Practice for Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete

C 595 Specification for Blended Hydraulic Cements

C 666 Test Method for Resistance of Concrete to Rapid Freezing and Thawing

C 671 Test Method for Critical Dilation of Concrete Specimens Subjected to Freezing

C 672 Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals

C 682 Practice for Evaluation of Frost Resistance of Coarse Aggregates in Air-Entrained Concrete by Critical Dilation Procedures

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