DEVELOPMENT OF EARLY-AGE IMPERMEABILITY
IN HIGH-PERFORMANCE CONCRETES

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ABSTRACT

Durability of concrete is largely determined by its resistance to the transport of water and ionic solutions. The volume and connectivity of the capillary porosity control the rate of ingress of these fluids. HPC is used in aggressive environments to obtain extended service life and one of the major issues is ensuring adequate curing to develop the expected "impermeability". In this study, early-age permeability, pore structure and compressive strength were measured. In addition, as a way of more easily obtaining early-age data, electrical conductivity measurements were performed between 1 and 28 days of age. Both the volume and connectivity of the pore structure as well as the conductivity of the pore solution influence conductivity. To separate out the effects of the changes in pore solution from the development of the pore structure, the conductivity of the pore solutions was measured independently. The changes in concrete conductivity over time were found to be related to permeability, porosity and strength measurements. As expected, a marked decrease in transport properties after the first few days indicates a reduction in volume and connectivity of the capillary pore system. Different concrete mixtures undergo these changes at different rates, and using conductivity methods it appears possible to develop appropriate equivalent curing methods for various concretes.

Keywords: Permeability, High-Performance Concrete, Curing, Porosity, Transport Properties, Early Age.

INTRODUCTION

The durability benefits of high performance concrete (HPC) are widely recognized. Low water to cement ratios and the addition of supplementary cementing materials lead to concrete that, when properly compacted and cured, could be described as "impermeable". However, there has been some resistance to use of high performance concrete for bridges and parking decks in part because of concerns with additional curing required to both prevent plastic and drying cracking and also to develop sufficient chloride penetration resistance.

High performance concrete's "impermeability" occurs due to low capillary porosity. The volume and connectivity of the capillary porosity control the rate of ingress of deleterious fluids. In the course of hydration of HPC, the capillary pores become disconnected and the permeability is controlled by the "gel pores". Flow through the material, if uncracked, can become so slow as to render it negligible during the service life of the structure.

Concrete bridge decks, pavements and structures are built to meet state and provincial code requirements for durability. The minimum requirements for water to cementing materials ratio, entrained air and curing were established to a large extent based on the work of Powers and colleagues at the Portland Cement Association approximately 50 years ago. Perhaps one of their most referenced works[1] provides the time required for cement pastes of various water to cement ratios to obtain a discontinuous capillary pore system. In fact, the curing requirements in many current specifications can be traced back to their research. It must be remembered that their permeability experiments were performed on small, mature, leached cement paste samples. Further the apparatus used was not capable of providing either the high pressure required or the precise measurement of low flows necessary to evaluate low permeability mixtures. Their "conclusion about continuity or lack of continuity of capillaries rests on conformity or lack of conformity of the data to an equation for the permeability based on the assumption that resistance to flow through a granular body is determined by viscous drags on the individual particles composing that body"[1]. In other words, if the flow followed Stoke's Law. The time required to achieve discontinuity was based on the degree of hydration of cement under standard laboratory conditions.

Since that time, changes in the chemistry of cement and its particle size distribution as well as the use of mineral and chemical admixtures have changed the nature of concrete. The aim of this research project was to determine the minimum curing required to develop low permeability HPC. Evaluating the duration of curing required to achieve low permeability would aid in making decisions on alternate concrete materials and mix proportions for specific applications.

MATERIALS, CASTING AND TESTING METHODS

The ACI 318 code lists a range of exposure classifications for concrete exposed to harsh conditions. The maximum water to cementing materials ratio permissible for exposure to

chloride is 0.40. Similarly, in Canada, for current CSA Standard A23.1, the most severe is class C-1, exposure to both chlorides and cyclic freezing and thawing, which also has a maximum of 0.40 and a minimum 28-day strength of 35MPa. Given that these exposure conditions represent the greatest challenge to durability concerns, this water to cementing materials ratio represents a main focal point of the research. The authors realize that this water to cementing materials ratio does not meet the conventional definition of high performance concrete, but it does represent a large segment of bridges and pavements constructed. This paper presents data from a larger research project underway that will include more high performance concrete mixtures.

Six concrete mixtures form the basis of this paper. The mixtures consist of one 0.30 water to cementing materials ratio and five 0.40 w/cm ratio mixtures; three of the five containing replacement levels of supplementary cementing materials typically encountered in practice, and two with varying water content at the same water to cement ratio. The mixture proportions are given in Table 1. Cementing materials consisted of low-alkali ASTM Type I Portland cement (PC) with Bogue composition of 57.4% C3S, 15.6% C2S, 8.5% C3A, 7.9% C4AF and the same cement blended with approximately 7% silica fume (SF) from the Lafarge Woodstock (Ontario) plant; a CSA Class CI fly ash (FA) from Columbia Unit #1 with 17.5% CaO; and a Grade 80 ground granulated blast furnace slag (SG) from the Lafarge Hamilton (Ontario) plant. The fine aggregate, a local glacial sand, had a density of 2700 kg/m^3, an absorption of 0.8%, and a fineness modulus of 2.56. A crushed 10mm limestone with a density of 2670 kg/m^3 and absorption of 1.76% was used as the coarse aggregate. Concrete mixtures included an ASTM Type A water-reducer and a naphthalene sulfonate-based superplasticizer were used to obtain workable mixtures.

Table 1: Mixture Proportions

Mixture Designation	0.30 PC	0.40 PC (135)	0.40 PC (170)	0.40 35%SG	0.40 20%FA	0.40 7%SF
W/CM	0.30	0.40	0.40	0.40	0.40	0.40
Water (kg/m^3)	150	135	170	150	150	150
Cement (kg/m^3)	500	338	425	281	300	375*
Silica Fume (kg/m^3)	0	0	0	0	0	*
Slag (kg/m^3)	0	0	0	94	0	0
Fly Ash (kg/m^3)	0	0	0	0	75	0
Sand (kg/m^3)	679	859	692	782	767	784
Stone (kg/m^3)	1100	1100	1100	1100	1100	1100

*CSA Type 10SF Blended cement with ~8% silica fume

A 20-litre pan mixer was used to mix successive batches of a particular concrete mixture to yield sufficient quantity of concrete (and mortar) for all tests performed. Concrete was cast into 100mm by 200mm cylinders. Mortar recovered from fresh concrete passing a 5mm sieve similar to ASTM C403 was used for mercury intrusion porosimetry. The mortar was cast into 30mm by 45mm cylinders. Concrete and mortar cylinders were removed from the molds 18 to 24 hours after casting and stored in lime-saturated water until testing.

Similar cement paste mixtures were cast for pore solution analysis and determination of degree of hydration. The pastes were mixed in a Waring blender. Paste samples were cast in 50mm by 100mm molds, sealed and then rotated to prevent segregation and bleeding. The paste specimens were stored in the sealed cylinders in 100% humidity environment until testing.

Testing was performed on the specimens at 1, 2, 3, 7, 14, 21 and 28 days after casting. Permeability was continuously monitored in a low-pressure cell similar to that described by Hearn and Mills[2]. Driving pressure was approximately 0.4 MPa. The confining pressure was provided by silicon rubber rings encased in a metal ring on the circumferential surface that were compressed by a screw jack to the top and bottom plates. Pistons with linear voltage displacement transducers were used to measure the movement of the inflow and outflow pistons to micrometer accuracy. Conductivity was measured using equipment normally employed in ASTM C1202. The device provided by Germann instruments allows the user to select the voltage applied across the specimen. Due to high conductivity at early ages, the current measured after passing 30 volts through the sample for 15 minutes was used to calculate conductivity. Compressive strength testing was conducted as per ASTM C39. Mortar specimens for mercury intrusion porosimetry were crushed using a mortar and pestle. Particles passing the 2.5mm sieve and retained on the 1.25mm sieve were kept for analysis. Particles were immersed in propanol for a minimum of 24 hours. Solvent replacement was followed by drying in a 50°C vacuum oven for a minimum of 24 hours. A Quantachrome Autoscan 60 capable of maximum pressure of 415 MPa was used for mercury intrusion. The contact angle was assumed to be 140º. The determination of total porosity and threshold diameter are shown schematically in Figure 1.
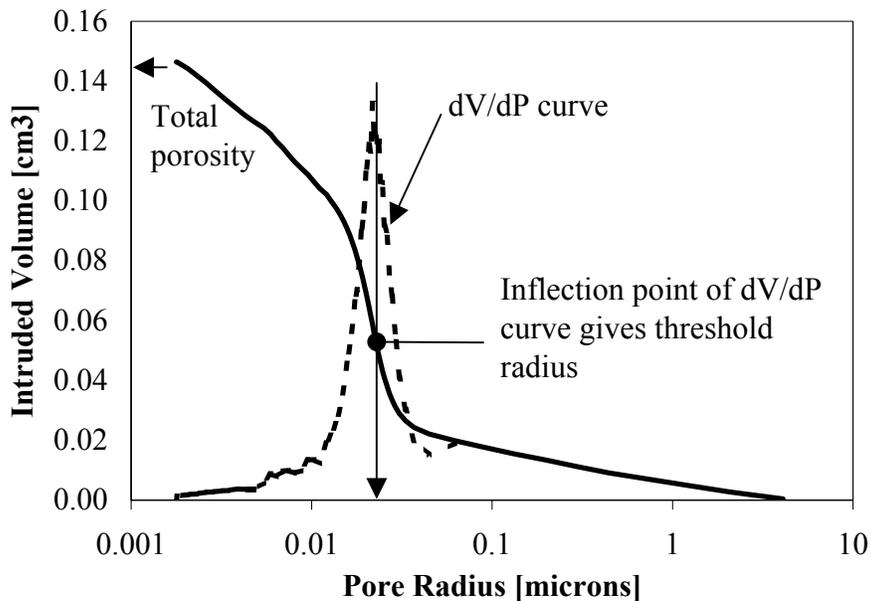

Figure 1: Determination of Total Porosity and Threshold Diameter for MIP curve

Pore solution analysis of paste used an expression device similar to that of Barneyback and Diamond[3]. Hydroxyl ion concentrations were determined by automatic potentiometric titration against sulphuric acid. Flame photometry was used to determine concentrations of sodium and potassium. Non-evaporable water content was determined from portions of the paste cylinders cast for pore solution analysis. Specimens of approximately 30-50 grams were dried at 105°C for a minimum of 24 hours. The specimens were placed in a furnace at 1050°C for 3 hours. The dried masses were determined after cooling to room temperature in a desiccator. The loss on ignition of the dry materials was measured to correctly determine the degree of hydration. Samples were sealed prior to drying to avoid carbonation.

RESULTS

CONCRETE TEST RESULTS

Saturated water permeability measurements are known to be difficult to obtain. In this project, duplicate samples yielded permeability differences as much as an order of magnitude for a particular day. Due to hydration of early age concrete, steady state flow through the sample was unobtainable. Permeability was calculated using Darcy's law using the inflow rate; results are shown in Table 2. For some specimens, the piston reached its minimal position rendering permeability calculations unobtainable on certain days. In the case of the 0.40 PC mixture with the higher water content, there was considerable slip-stick of O-rings causing the inflow pistons not to move freely. The authors are currently correcting problems associated with friction and will retest these sample mixtures. Neglecting variability in some measurements, the results show that permeability generally decreases with age. However, the 0.30 PC mixture appeared to remain approximately constant after 3 days, the 0.40 PC (135) mixture after 7 days. Permeability in the mixtures containing supplementary cementing materials continued to reduce for a longer period.

Table 2: Water Permeability Results [10^{-12} m/s]

Days	0.30 PC	0.40 PC (135)	0.40 PC (170)	0.40 35%SG	0.40 20%FA	0.40 7%SF
1	n.d.	150	n.d.	600	61	20
2	n.d.	23	n.d.	140	30	9.0
3	5.0	8.0	n.d.	59	8.0	5.6
7	5.3	0.9	n.d.	9.8	7.0	2.1
14	6.0	n.d.	n.d.	2.3	1.0	1.0
21	5.4	1.0	n.d.	1.0	2.0	0.6
28	n.d.	n.d.	n.d.	0.5	1.0	0.4

n.d. – no data

The calculated conductivity is the average of two samples. The samples were 50mm slices of 100mm diameter cylinders. The same two samples were tested at all ages. The samples were

kept in the apparatus for the first three days and then returned to lime water between subsequent measurements. Sodium hydroxide solution (0.3M) was used in both chambers of the ASTM C1202 apparatus. The solution was selected to approximate pore solution as well as to minimize leaching. The use of sodium chloride would change conductivity over time due to the difference in conductivity of chloride and hydroxyl ions. The current measured after passing 30 volts through the sample for 15 minutes was used to calculate bulk conductivity. The term bulk conductivity is used to clarify that the current flows through the composite sample (the hydrated solid material and the pore solution). Results are presented in Table 3. Conductivity decreases with time for all concrete mixtures. The influence of water to cementing materials ratio, supplementary cementing materials and water content can be seen in the results. The 0.30 ordinary Portland mixture initially had lower conductivity than all other mixtures, but over time, the mixtures with supplementary materials developed lower conductivity.

Table 3: Bulk Conductivity Results [uS/cm]

Days	0.30 PC	0.40 PC (135)	0.40 PC (170)	0.40 35%SG	0.40 20%FA	0.40 7%SF
1	364	549	547	712	820	600
2	286	407	423	574	479	338
3	265	353	379	478	421	321
7	213	271	317	211	272	131
14	183	233	249	110	201	70
21	152	211	200	82	154	37
28	141	185	236	53	128	29

Compressive strength data is an average of two 100mm by 200mm cylinders for each day. Results are shown in Table 4. Although strength is not necessarily a good indicator of durability, strength was measured to determine the correlation to porosity and transport properties. Some concrete mixtures experienced set delay due to admixture usage to obtain workability and this is evidenced by low one-day strengths.

Table 4: Compressive strength results [MPa]

Days	0.30 PC	0.40 PC (135)	0.40 PC (170)	0.40 35%SG	0.40 20%FA	0.40 7%SF
1	33.2	20.6	25.2	10.3*	7.8*	14.0*
2	48.1	33.6	37.2	23.1	26.7	41.1
3	54.1	37.9	40.3	26.4	31.7	47.1
7	60.1	48.8	48.0	44.3	43.1	58.4
14	64.2	47.4	52.6	56.8	48.6	67.2
21	64.6	54.2	54.4	58.7	50.6	73.4
28	67.1	56.1	50.8	63.9	56.7	83.1

* low 1-day strength due to set retardation resulting from water reducer

MORTAR TEST RESULTS

Total porosity, as determined by mercury intrusion porosimetry, is shown in Table 5. Threshold radii was determined as the maximum of the derivative of the volume versus mercury intrusion porosity curve, results are shown in Table 6.

Table 5: Total Porosity by Mercury Intrusion [%]

Days	0.30 PC	0.40 PC (135)	0.40 PC (170)	0.40 35%SG	0.40 20%FA	0.40 7%SF
1	14.5	12.3	13.8	14.7	13.5	14.0
2	12.9	10.4	12.0	13.8	11.3	12.5
3	10.0	9.8	11.0	12.6	10.9	12.1
7	9.4	7.4	10.2	n.d.	9.3	8.4
14	8.2	7.9	7.3	8.3	7.3	8.5
21	7.5	8.6	9.6	8.0	7.6	7.9
28	8.5	6.0	9.3	7.1	6.8	7.5

n.d. – no data

Table 6: Threshold Pore Radius [um]

Days	0.30 PC	0.40 PC (135)	0.40 PC (170)	0.40 35%SG	0.40 20%FA	0.40 7%SF
1	0.022	0.028	0.030	0.145	0.131	0.039
2	0.018	0.020	0.021	0.039	0.036	0.025
3	0.014	0.022	0.019	0.026	0.028	0.015
7	0.016	0.017	0.021	n.d.	0.023	0.015
14	0.015	0.019	0.013	0.014	0.018	0.019
21	0.015	0.021	0.014	0.016	0.013	0.015
28	0.018	0.011	0.011	0.020	0.015	0.013

n.d. – no data

PASTE TEST RESULTS

To separate out the effects of the changes in pore solution from the development of the pore structure, the conductivity of the pore solutions was determined independently. Pore solution expressed from paste samples was centrifuged to separate solids prior to testing. To determine hydroxyl concentration, from 0.3 to 1 mL of solution was required for titration against standard sulphuric acid titration. After 7 days, the 0.30 PC mixture would not yield necessary quantities of pore solution for analysis. As shown in Table 7, the hydroxyl concentrations increased during the first 28 days after casting in all but the silica fume mixture. Silica fume reacts rapidly with calcium and alkali hydroxides to form secondary calcium silicate hydrates.

Table 7: Pore Solution Analysis [OH]$^-$ mol/L

Mixture	0.30 PC	0.40 PC	0.40 35%SG	0.40 20%FA	0.40 7%SF
1	0.652	0.310	0.242	0.275	0.322
2	0.693	0.354	0.307	0.349	0.293
3	0.709	0.374	0.295	0.344	0.327
7	0.796	0.390	0.279	0.451	0.266
14	n.d.	0.442	0.413	0.498	0.230
21	n.d.	0.485	0.404	n.d.	0.190
28	n.d.	0.451	0.447	n.d.	n.d.

n.d. – no data

Tables 8 and 9 show concentrations of potassium and sodium ions respectively. In some cases, there was a sufficient quantity to determine hydroxyl concentration but not to determine potassium and sodium concentrations. The sum of the sodium and potassium ion concentrations are within 10% of the hydroxyl ion concentration except in a few cases where measurement error is likely, indicating there are likely insignificant quantities of other ions in the pore solution contributing to conductivity.

Table 8: Pore Solution Analysis [K]$^+$ mol/l

Mixture	0.30 PC	0.40 PC	0.40 35%SG	0.40 20%FA	0.40 7%SF
1	0.465	0.250	0.246	0.197	0.228
2	0.525	0.284	0.221	0.242	0.258
3	0.558	0.302	0.222	0.247	0.255
7	n.d.	0.313	0.241	0.287	0.212
14	n.d.	0.377	0.319	n.d.	0.204
21	n.d.	0.407	0.272	n.d.	0.164
28	n.d.	n.d.	0.279	n.d.	n.d.

n.d. – no data

Table 9: Pore solution Analysis [Na]$^+$ mol/L

Mixture	0.30 PC	0.40 PC	0.40 35%SG	0.40 20%FA	0.40 7%SF
1	0.220	0.057	0.069	0.069	0.087
2	0.250	0.069	0.070	0.098	0.102
3	0.262	0.076	0.076	0.098	0.102
7	n.d.	0.079	0.091	0.130	0.089
14	n.d.	0.096	0.127	n.d.	0.094
21	n.d.	0.105	0.109	n.d.	0.084
28	n.d.	n.d.	0.118	n.d.	n.d.

n.d. – no data

The method of Snyder et. al.[4] was used to determine the conductivity of the pore solution from the concentrations of hydroxyl, potassium and sodium ions. The results are presented in Table 10.

Table 10: Pore Solution Conductivity [mS/cm]

Days	0.30 PC	0.40 PC (135)	0.40 PC (170)	0.40 35%SG	0.40 20%FA	0.40 7%SF
1	137	67	67	57	60	69
2	139	76	76	66	74	66
3	143	80	80	64	74	71
7	n.d.	83	83	63	92	60
14	n.d.	94	94	88	n.d.	54
21	n.d.	102	102	84	n.d.	45
28	n.d.	n.d.	n.d.	91	n.d.	n.d.

n.d. – no data

The degree of hydration was calculated by:

$$\alpha = \frac{W_n}{W_n^o} * 100 \tag{1}$$

$$W_n = \frac{W_{105}}{W_{1050}}(1-L)-1 \tag{2}$$

where:

α – Degree of hydration, %
W_n – Non-evaporable water, g/g cement
W_n^o – Non-evaporable water content of fully hydrated cement, g/g cement
W_{105}, W_{1050} – Mass of paste at 105°C and 1050°C, g
L – ignition loss of the original material, g/g material.

The amount of non-evaporable water at full hydration, W_n^o, used here was determined by Hooton[5] (W_n^o: Portland cement = 0.252, 5% silica fume = 0.244, 30% slag = 0.265). For lack of information, 0.252 was used the 20% fly ash paste. The results are shown in Table 11.

Table 11: Degree of Hydration of Paste

Mixture	0.30 PC	0.40 PC	0.40 35%SG	0.40 20%FA	0.40 7%SF
1	0.443	0.412	0.372	0.406	0.502
2	0.505	0.479	0.438	0.512	0.551
3	0.531	0.499	0.526	0.516	0.576
7	0.523	0.573	0.475	0.539	0.699
14	0.568	0.571	0.573	0.576	0.626
21	n.d.	0.589	0.597	n.d.	0.693
28	0.503	0.607	0.684	n.d.	n.d.

n.d. – no data

DISCUSSION

Rapid chloride permeability (ASTM C1202) is often used in performance-based specifications for concrete bridges and parking decks. A maximum charge is permitted when tested at a specified age (usually 28 days, but in some cases 56 days [6]). The problem occurs in determining the curing required for a particular concrete mixture to achieve the allowable limit at the specified age. The standard rapid chloride test is difficult to perform at early ages. Often the current or temperature reaches the maximum tolerated by the equipment at some time during the test, commonly stopping prior to the six-hour test period. However, the short-term conductivity test can be used to estimate the standard 6-hour coulomb values. In Figure 2, the bulk conductivity values from Table 3 are shown. The current measured is used to calculate the equivalent six-hour charge, assuming the current was to remain constant during the test. This assumption holds if negligible heating occurs during the test, which is the normal case for high performance concrete (at least at 28 days). Standard ASTM C1202 tests were also performed at 28 days, the values determined agree with those calculated from the current measured at 15 minutes. The calculated charge and the measured charge are shown in Table 12.
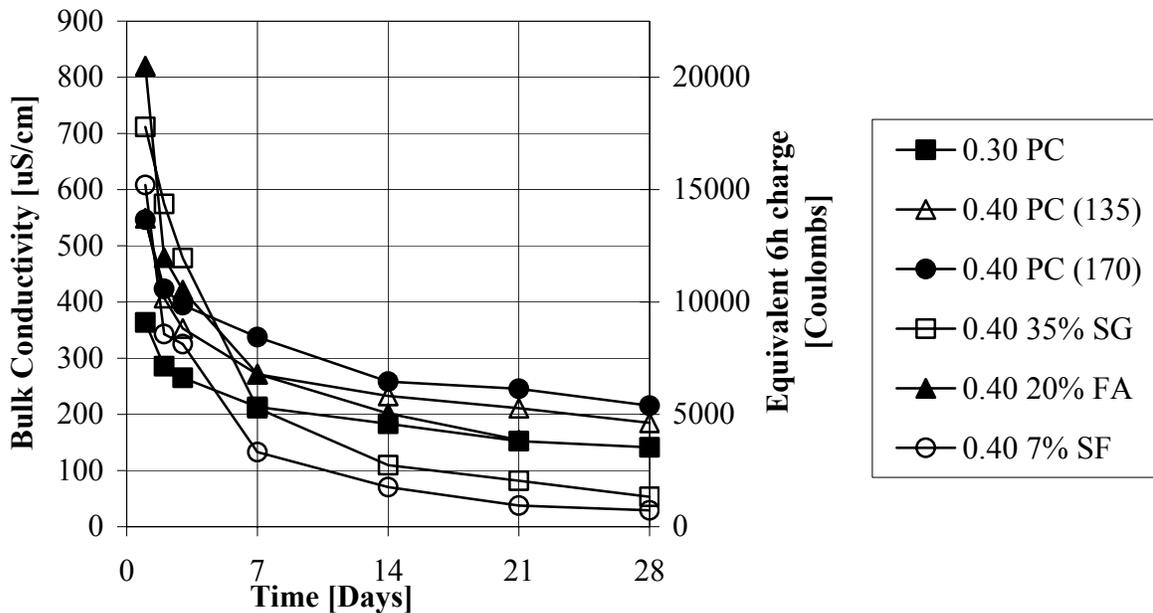
Figure 2: Bulk Conductivity vs. Time

Table 12: Calculated and measured charge at 28 days age

	0.30 PC	0.40 PC (135)	0.40 PC (170)	0.40 35%SG	0.40 20%FA	0.40 7%SF
Measured charge	3610	4770	6080	1040	3870	n.d.
Calculated charge	3580	4690	5980	1350	3250	730

n.d. – no data

If the maximum allowable charge passed were 1000 coulombs, only the silica fume mixture would be permissible, at 1500 coulombs both the slag and silica fume mixtures would pass. But HPC mixtures with lower water to cementing materials ratios and containing supplementary materials would typically be less than 1000 coulombs.

The specimens in this paper were immersed in lime-saturated water for 28 days. Although there are no measurements of conductivity with moist curing terminated at some time during the 28 days, it is expected that the conductivity would increase. Figure 3 highlights the importance of curing on transport properties, such as conductivity. The authors are currently studying the effect of curing on transport properties and will present data from this work in the future.
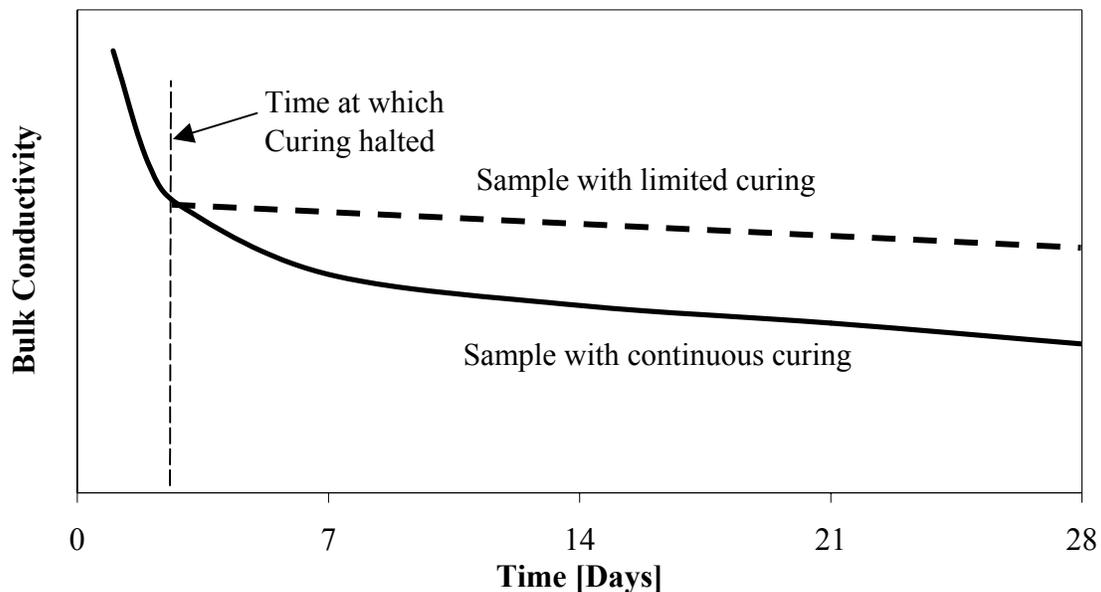
Figure 3: Schematic of the effect of curing on conductivity

Conductivity was used in this project as one method to determine the development of "impermeable" concrete. As cement hydrates, the volume and connectivity of the capillary pores decrease. The decreased porosity would lead to decreased conductivity with time. However, the concentration of ions in the pore solution increases with time, increasing conductivity. The conductivity of pore solution is approximately three orders of magnitude higher than the bulk conductivity. Normalized conductivity is the ratio of the bulk conductivity to the pore solution conductivity. (It is equivalent to the reciprocal of the formation factor.) The normalized conductivity values with time calculated using the values in Tables 3 and 10 are shown in Figure 4. The normalized conductivity decreases with time showing that the effect of decreasing porosity overwhelms the effect of increasing pore solution conductivity. The concrete mixtures with supplementary cementing materials initially have greater normalized conductivity than the 0.40 Portland mixtures, but the

normalized conductivity becomes less for these mixtures between three to seven days. The mixtures with insufficient pore solution (for determination of pore solution conductivity) have for the most part reached terminal hydration. The normalized conductivity is expected to be remain relatively stable from the last data point shown here.
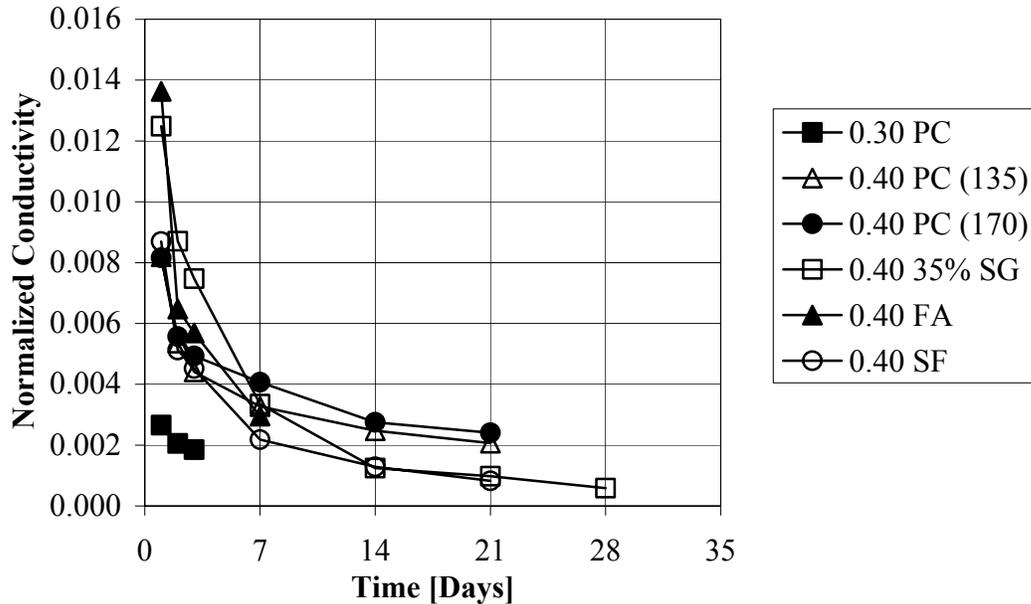

Figure 4: Normalized Conductivity vs. Time

In Figure 5, the relationship between water permeability and normalized conductivity is compared. Given that saturated water permeability results are variable at best, the relationship is fairly good. This seems to indicate that conductivity measurements could be used as a surrogate for permeability. Figure 6 shows that a similar linear relationship exists between water permeability and bulk conductivity suggesting that pore solution conductivity is a second order effect. The collection of pore solution and the measurement of ion concentrations are beyond the capabilities of most laboratories. The good correlation of permeability with bulk conductivity is encouraging in developing an easy-to-measure, quick test method that would gauge durability.
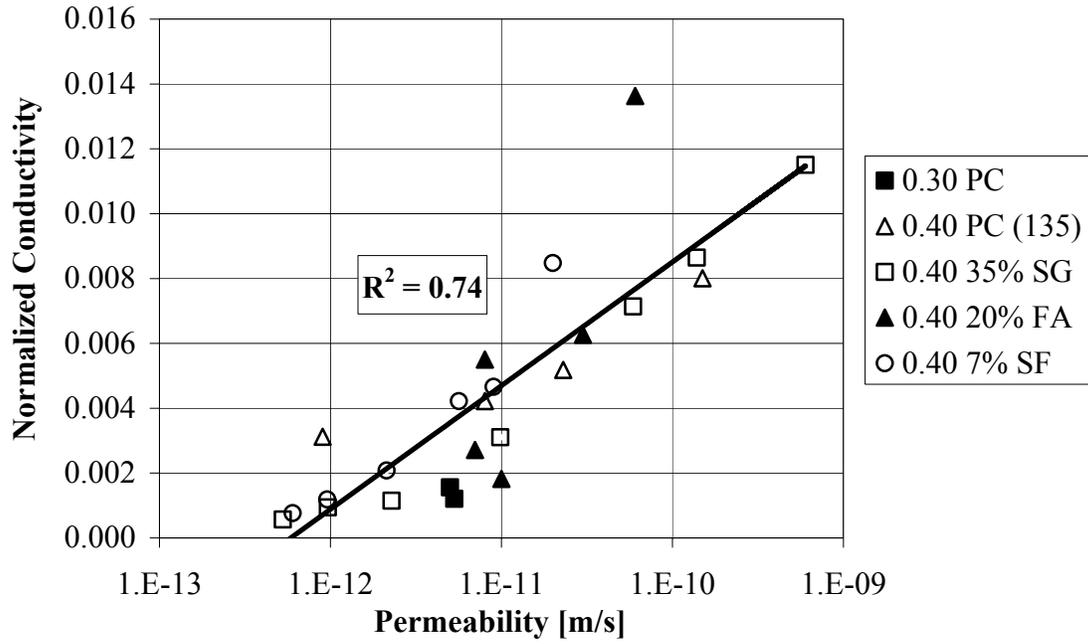
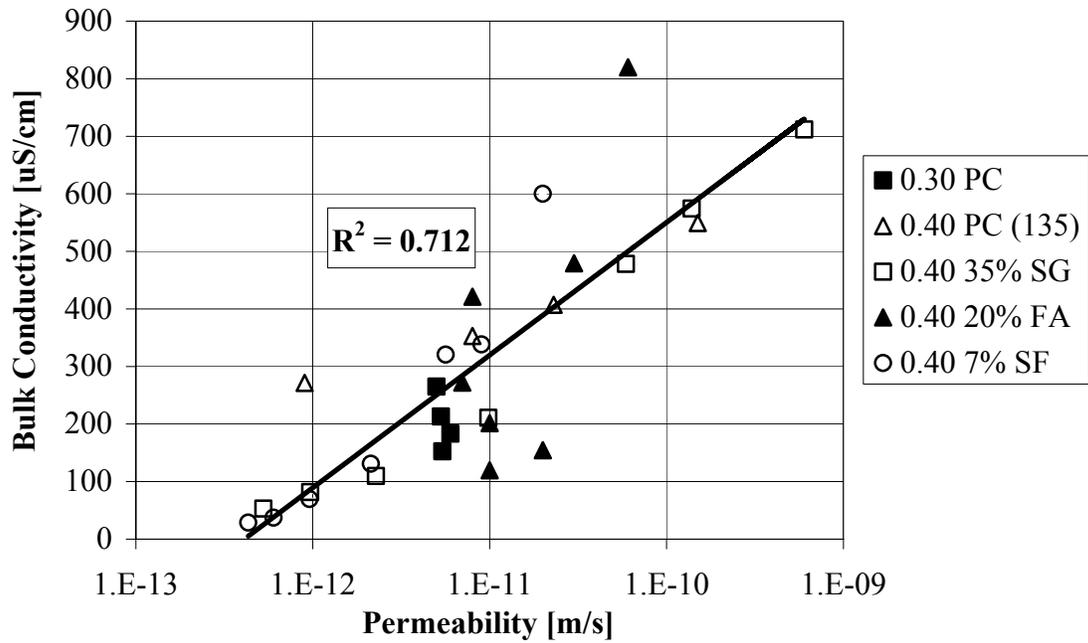Figure 5: Water Permeability vs. Normalized Conductivity

Figure 6: Water Permeability vs. Bulk Conductivity

To further investigate the relationship between bulk conductivity and other measured properties, correlations are given in the following figures. Figure 7 shows that there is a good linear relationship between bulk conductivity and compressive strength when there is continuous moist curing. This relationship is not expected to hold when curing is interrupted or terminated. In normal practice, cylinders for compressive strength are moist cured until testing. Tests for compressive strength or transport properties on these specimens are not likely to correspond to the properties developed for the concrete in the structure, which would not have been cured for the same duration. Durability decreases when curing is not continued for an adequate period. The decreased durability would manifest itself in increased conductivity such as that shown in Figure 2, as well as other transport properties.
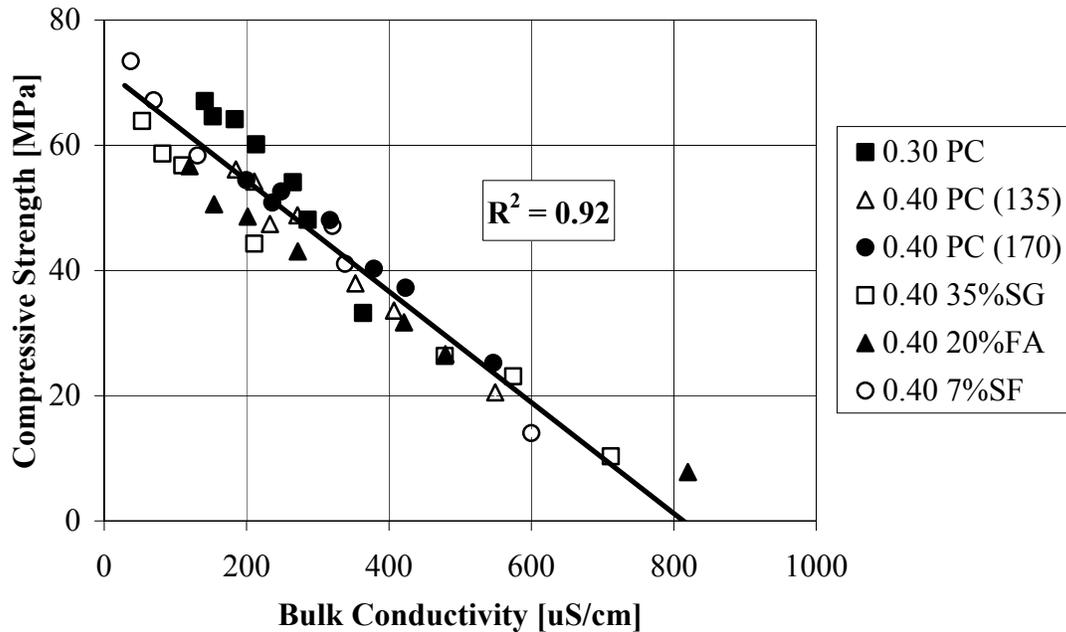

Figure 7: Bulk Conductivity vs. Compressive Strength at various ages
(for wet cured specimens only)

Figure 8 shows that a good linear relationship exists between bulk conductivity and total porosity. Recall that the total porosity and threshold diameter were measured by mercury intrusion porosimetry on mortar sieved from fresh concrete. While the porosity measured differs from that of concrete, if the coarse aggregate is thought of as non-porous inclusions, the porosity would be similar. Figure 9 shows a polynomial relationship for threshold radius and bulk conductivity. Permeability is known to be related a power of to the diameter; a similar trend seems to fit the data in this paper.
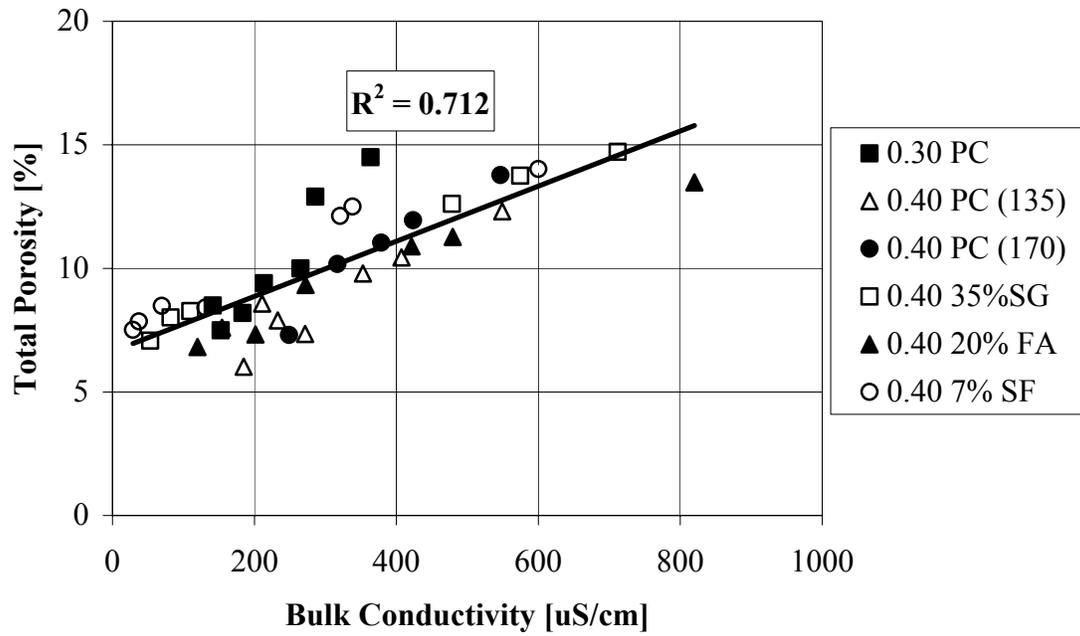
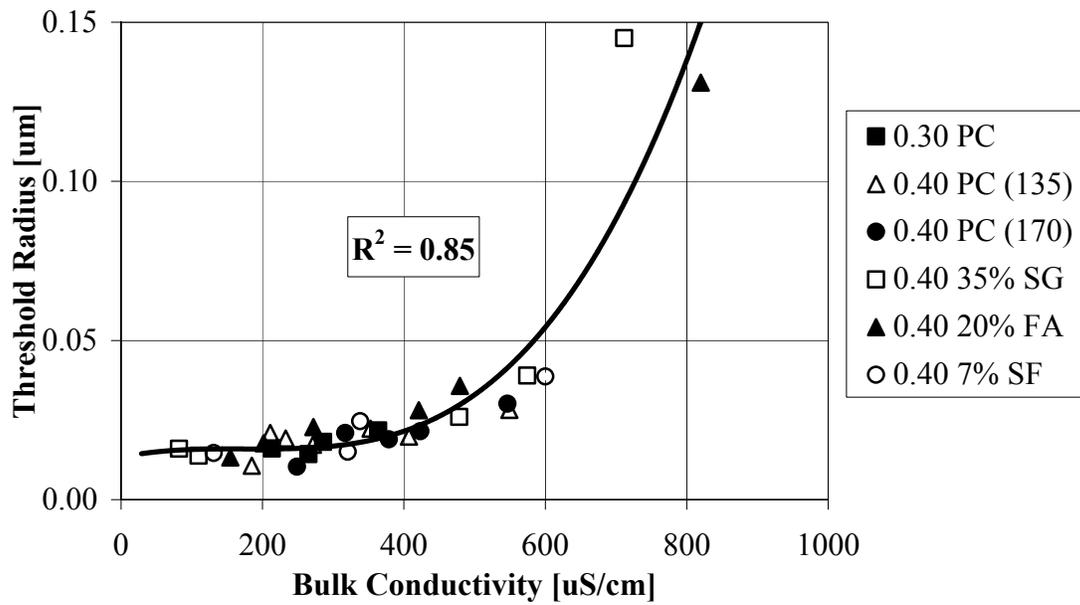Figure 8: Bulk Conductivity vs. Total Porosity

Figure 9: Bulk Conductivity vs. Threshold Radius

CONCLUSIONS

The changes in concrete conductivity over time were found to be related to permeability, porosity and strength measurements. As expected, a marked decrease in transport properties after the first few days indicates a reduction in volume and connectivity of the capillary pore system. Early-age water permeability correlates well to conductivity. This indicates that conductivity can be used as an easier indicator test. Time for conductivity to reduce to a value equivalent to permeability equal to 10^{-12} m/s varies with the concrete mixture. Low water to cement ratio and addition of supplementary materials (especially silica fume) shorten that time. Testing of high performance concretes is in progress with the intent to develop curing specifications for various concretes based on simple tests such as bulk conductivity measurements.

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