Concrete Pavement Mixture Design and Analysis (MDA):

Evaluation of the Fresh and Hardened Properties of Concrete Mixtures Containing Hydrophilic and Hydrophobic Types of Permeability-Reducing Admixtures to Develop a Standard Testing Protocol



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16. Abstract

Concrete durability may be considered as the ability to maintain serviceability over the design life without significant deterioration, and is generally a direct function of the mixture permeability. Therefore, reducing permeability will improve the potential durability of a given mixture and, in turn, improve the serviceability and longevity of the structure. Given the importance of this property, engineers often look for methods that can decrease permeability. One approach is to add chemical compounds known as integral waterproofing admixtures or permeability-reducing admixtures, which help fill and block capillary pores in the paste.

Currently, there are no standard approaches to evaluate the effectiveness of permeability-reducing admixtures or to compare different products in the US. A review of manufacturers' data sheets shows that a wide range of test methods have been used, and rarely are the same tests used on more than one product.

This study investigated the fresh and hardened properties of mixtures containing commercially available hydrophilic and hydrophobic types of permeability-reducing admixtures. The aim was to develop a standard test protocol that would help owners, engineers, and specifiers compare different products and to evaluate their effects on concrete mixtures that may be exposed to hydrostatic or non-hydrostatic pressure.

In this experimental program, 11 concrete mixtures were prepared with a fixed water-to-cement ratio and cement content. One plain mixture was prepared as a reference, 5 mixtures were prepared using the recommended dosage of the different permeability-reducing admixtures, and 5 mixtures were prepared using double the recommended dosage. Slump, air content, setting time, compressive and flexural strength, shrinkage, and durability indicating tests including electrical resistivity, rapid chloride penetration, air permeability, permeable voids, and sorptivity tests were conducted at various ages. The data are presented and recommendations for a testing protocol are provided.

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EVALUATION OF THE FRESH AND HARDENED PROPERTIES OF CONCRETE MIXTURES CONTAINING PERMEABILITY-REDUCING ADMIXTURES TO DEVELOP A STANDARD TESTING PROTOCOL

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INTRODUCTION

Long-term concrete performance is strongly influenced by concrete durability (Aitcin 2003). Concrete durability may be considered as the ability to maintain serviceability over its design life without significant deterioration. Potential durability may be considered a direct function of the mixture permeability because it controls the rate of penetration of aggressive chemicals and the movement of water during heating or freezing (Day 2006). Therefore, reducing permeability will improve the potential durability of a given mixture and in turn, improve the serviceability and longevity of the structure (Lane et al. 2010, Viles 2010). Given the importance of this property, engineers often look for methods that can reduce the permeability of concrete.

It is not possible to make concrete completely waterproof; however, permeability can be minimized with different methods. Permeability can be reduced by reducing cracks, avoiding bleeding channels, reducing the number of percolated (capillary) pores in the paste system, and improving the interfacial transition zone between paste and aggregates (Lamond and Pielert 2006). A common way to reduce permeability is by decreasing the water-to-cementitious materials ratio (w/cm) (Hamami et al. 2012); however, depending on the workability requirements of different applications, there may be limitations because an adequate w/cm is required to ensure achieving a fully hydrated, easily placed, and properly consolidated end-product. It is frequently reported (Yurdakul et al. 2014, Liu et al. 2012, Bagheri and Zanganeh 2012, Megat Johari et al. 2011) that incorporating supplementary cementitious materials (SCM), such as fly ash, slag cement, and silica fume, can also decrease permeability. However, SCMs were not used in this study because they were outside the intent of the work.

The permeability of concrete can also be further reduced by including materials into the mixture that help to fill the capillary pores and so reduce their size and connectivity. These products are marketed as "integral waterproofing admixtures" or "permeability-reducing admixtures" (Dao et al. 2010, Ramachandran 1995). The effectiveness of these admixtures depends on their dosage and chemical interactions with the cement paste matrix (ACI 212.3R 2010).

Water can penetrate concrete either due to capillary absorption or hydrostatic pressure; therefore, depending on their method of reducing the water ingress, these admixtures are subcategorized as follows:

- Permeability-reducing admixtures for concrete exposed to non-hydrostatic conditions (PRAN)
- Permeability-reducing admixtures for concrete exposed to hydrostatic conditions (PRAH)

Permeability-reducing admixtures can be in powder, liquid, or suspension form. ACI 212.3R (2010) classifies these products in three categories:

• Hydrophobic chemicals: These materials are based on soaps and long-chain fatty acid derivatives, vegetable oils, and petroleum. They provide a water-repellent lining to the pores, although the pores remain physically open. They are known to perform well under non-

- hydrostatic conditions because they are effective in reducing capillary absorption and chloride penetration (ACI 212.3R 2010, Ramachandran 1995). These admixtures are subcategorized as PRAN.
- Finely divided solids: These materials include inert and chemically active fillers (such as talc, bentonite, or clay). They reduce permeability under non-hydrostatic conditions by filling up voids and physically restrict the water penetration through the pores. These admixtures are subcategorized as PRAN.
- Crystalline materials: These materials are hydrophilic and consist of active chemicals that react with water and cement particles in the concrete to block the pores. They can reduce permeability under hydrostatic pressure. These admixtures are subcategorized as PRAH.

Although some researchers question the effectiveness of these admixtures in the long term (Day 2006), these admixtures claim to provide concrete with lower permeability, lower water absorption, slower ingress of aggressive elements, reduced drying shrinkage, and the self-healing of minor cracks (Viles 2010, Munn et al. 2003, Ramachandran 1995). Like all additives, these materials may have side effects on other properties of the mixture, such as extended setting time and reduced strength.

Permeability-reducing admixtures have been commonly used in water retaining structures, bridge decks, foundation walls, sewage works, tunnels, and pavements where deicing salt usage is extensive (Viles 2010). At present, there are no standard approaches to evaluate the efficiency of these products in reducing permeability or to compare different products in the US. This may be partially due to the wide range of uses they are applied to and the different mechanisms by which they work. The European Standard EN 934-2 (2000) evaluates the effectiveness of these admixtures on permeability by only testing the capillary absorption. However, depending on the formulation, different admixtures may show different behaviors under the various permeability measuring test methods.

A review of data sheets provided by various manufacturers shows that a wide range of test methods (e.g., CRD C48-92, DIN 1048, BS EN 12390, ASTM C1202, ASTM C1556, and ASTM C1585) have been used to evaluate the effectiveness of these admixtures.

These test methods can be broadly grouped into different forms:

- Sorption: A measure of capillary suction, which is directly controlled by size and connectivity of capillary pores (e.g. BS 1881-122 2011). This approach is extremely sensitive to the moisture state of the sample and may yield different results if tests are conducted on formed or finished surfaces compared to a sawn interior surface. Pore blockers generally tend to perform well in these tests.
- Fluid penetration, either by ponding or under pressure (e.g. DIN 1048, 1991): Again, the test results are sensitive to the starting moisture state of the specimen. Crystalline materials tend to perform well under this test.
- Resistivity: Electrical current will tend to pass more readily through fluids than solids and is therefore a reasonable indirect indicator of connectivity of the pores. Samples should be saturated prior to testing. Permeability-reducing admixtures that have a high ionic content

tend to yield false poor results because the ions help transfer current, even if the pore sizes are small.

There is therefore no ideal single test for the range of products available on the market, and selections and evaluations should take into account the form of the product being tested, the environment to which it is exposed, and the purpose for its use. Therefore, a standard test protocol is needed that recommends various permeability measuring test methods (and limits), as well as other fresh and hardened properties that are important for a particular application.

This study investigated the fresh and hardened properties of mixtures containing a selection of commercially available permeability-reducing admixtures. The aim was to develop a standard testing protocol that would help engineers, owners, and specifiers to compare different products and evaluate their effects on concrete mixtures. 5 permeability-reducing admixtures from different manufacturers were selected that covered the range of types available (2 of them representing PRAN and 3 of them representing PRAH type).

In this program, 11 concrete mixtures were prepared with a fixed water-to-cement ratio (w/c) and cement content. One mixture was prepared as a reference, 5 mixtures were prepared using the recommended dosage of the different permeability-reducing admixtures, and 5 mixtures were prepared using double the recommended dosage for each product. Slump, air content, setting time, compressive and flexural strength, shrinkage, and durability indicating tests including electrical resistivity, rapid chloride penetration, air permeability, permeable voids, and sorptivity tests were conducted at various ages.

MATERIALS AND METHODS

Materials

A single batch of each of the following commercially available materials was obtained:

- ASTM C150 Type I portland cement
- 1-in. nominal maximum size crushed limestone
- No 4 nominal maximum aggregate size river sand

The chemical composition of the cement is presented in Table 1.

Table 1. Chemical composition of ASTM C150 Type I portland cement, percentage by mass

Chemical composition	ASTM C150 Type I portland cement
Silicon dioxide (SiO ₂)	20.22
Aluminum oxide (Al ₂ O ₃)	4.43
Ferric oxide (Fe ₂ O ₃)	3.19
Calcium oxide (CaO)	62.71
Magnesium oxide (MgO)	3.51
Sulfur trioxide (SO ₃)	3.24
Potassium oxide (K ₂ O)	0.69
Sodium oxide (Na ₂ O)	0.08
Equivalent alkalis (NaEq)	0.54

After reviewing 13 commercially available admixtures produced by different manufacturers, 5 permeability-reducing admixtures from various manufacturers were selected that covered the range of types available. Among these 5 products, two of them represent PRAN and three of them represent PRAH type permeability-reducing admixtures. In the interest of confidentiality, the products are only referred to as "A", "B", "C", "D", and "E" in the remainder of this report.

Mix Design

Permeability-reducing admixtures are intended to be used in well-proportioned concrete mixtures with a w/cm of 0.45 or lower (ACI 212.3R 2010). In this study, a fixed cement content, aggregate system, and w/c was selected for consistency.

The full test program included 11 mixtures with a constant w/c of 0.45 and a fixed cement content of 564 lb/yd³. One mixture was prepared as reference (designated as "0"), 5 mixtures

were prepared using the manufacturers' recommended dosage (labeled "R") of the different products, and 5 mixtures were prepared using double the recommended dosage (labeled "2R").

The proportioning recommendations and mixing specifications of each product were reviewed. The selected cement content and w/c were within the acceptable range for four of the products. One admixture recommended using higher cementitious content and lower w/c than the selected values due to its water-reducing effect. However, the team chose to use a constant set of values for uniformity of the data. In order to control the magnitude of the matrix and to prevent any potential incompatibilities between different materials, water-reducing admixtures, air-entraining admixtures, or SCMs were not used in this study.

Specimens and Testing

For each mixture, 16 4×8 in. concrete cylinders and 4 3x4x16 in. prisms were prepared and stored in the fog room in accordance with ASTM C192 until testing. Cylinders were cut into slices for the permeability related testing. Data provided are an average of at least two tests from slices taken at various depths through the cylinder. The finished surface was not tested.

The test matrix is provided in Table 2.

Table 2. Test matrix

Fresh properties	resh properties Method		Age (days)
Slump/Slump flow	ASTM C143/ASTM C1611	1	-
Air content	ASTM C231	1	-
Setting time	ASTM C403	1	-
Hardened properties	Method	No. of specimens	Age (days)
Compressive strength	ASTM C39	3 per age	7, 28
Flexural strength	ASTM C78	4 per age	56
Electrical resistivity	AASHTO TP 95-11	3 per age	7, 28
Rapid chloride penetration	ASTM C1202	3 per age	7, 28
Air permeability	Univ. of Cape Town Method	3 per age	7, 28
Permeable voids	ASTM C642	3 per age	7, 28
Sorptivity	ASTM C1585	3 per age	7, 28
Shrinkage	ASTM C157	4 per age	28

RESULTS AND DISCUSSION

Workability

Workability is of interest primarily to contractors, because it affects how they may place the concrete. A slump test was conducted in accordance with ASTM C 143 to assess the effect of the addition of permeability-reducing admixtures on workability. Test results are presented in Figure 1. Based on ASTM C143, the acceptable range of two tests by the same operator on the same material is 1.07 in. of a mix having 3.4 in. slump. Based on this recommendation, the acceptable variation range of the slump value obtained from the reference mix was calculated and is presented as lines in Figure 1.

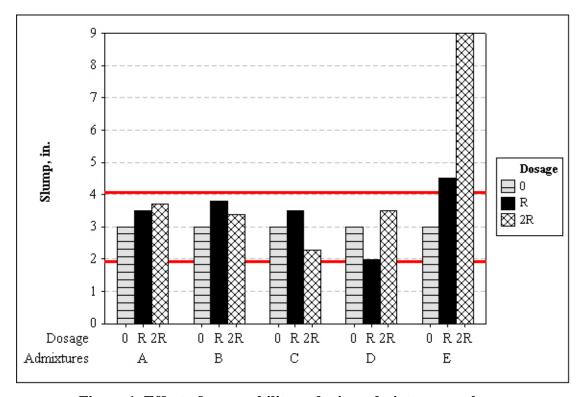


Figure 1. Effect of permeability-reducing admixtures on slump

Test results show that the addition of permeability-reducing admixtures mostly did not affect the workability because the slump of the products "A", "B", "C", and "D" were within the acceptable variation limits of the slump of the reference mix. However, as expected, admixture "E" at the recommended dosage increased the workability by about 50% compared to the reference mix. Further increasing the dosage of admixture "E" resulted in a significant increase in slump. If the mixtures were adjusted to constant slump by removing water, then lower permeability values may be expected.

The permeability-reducing admixtures are mainly added into concrete for their effect on reducing and blocking the passage of water. While the admixtures make concrete more watertight, it

would be undesirable for an admixture to have unexpected side effects such as on the workability. Therefore, it is recommended that mix water should be reduced to balance the amount of water in the admixture in order to maintain a constant w/cm.

Permeability-reducing admixtures should also indicate whether they influence workability more than the scatter of the test method when compared to the reference mix. If the permeability-reducing admixtures improve the slump, then a lower design w/cm may be selected to ensure that achieving the specified slump is achieved. Mix proportioning should aim to ensure that the workability is appropriate for the construction system planned. If the permeability-reducing admixtures decrease the slump, appropriate types and dosages of water-reducing admixtures may be selected to overcome this issue. The interaction between permeability-reducing admixtures and other types of chemical admixtures should be checked to ensure incompatibility when used together.

Air Content

The need for a controlled air void system is driven by the desire to provide the concrete with the ability to resist freezing and thawing cycles by providing locations that freezing and expanding water can move into without exerting internal pressure on the system. As such, an effective air void system is a critical component to durable concrete exposed to cold environments.

Some project specifications allow air content to be in the range of 1% lower and 2% higher than the target air content (Kosmatka et al. 2002), and these limits are shown as lines in Figure 2 along with the test results.

The test results conducted in accordance with ASTM C 231 show that the air content of the mixtures containing permeability-reducing admixtures are mostly within the range of -1% and +2% of the reference mix. However, the air content of the mix "B" at the recommended dosage was 4% higher than the reference mixture, which is greater than the allowable increased air content of 3.5% in non–air-entrained concrete required in ASTM C494. The cause of this variation is unknown.

Although having higher air content may be desirable where freezing and thawing is a concern, higher air content may adversely affect the strength (Kosmatka et al. 2002). Therefore, it is recommended that mixtures incorporating PRAN or PRAH permeability-reducing admixtures should indicate if they affect the air content by more than -1% or +2% of the reference mixture. If an admixture influences the air content by more than this range, the supplier should be asked to show that the influence in air content is not detrimental to the air void system. When needed, adjustment to the air-entraining or air-detraining admixture dosage shall be made to meet specification requirements. Mix proportioning and trial batches should demonstrate that acceptable air void systems can be provided.

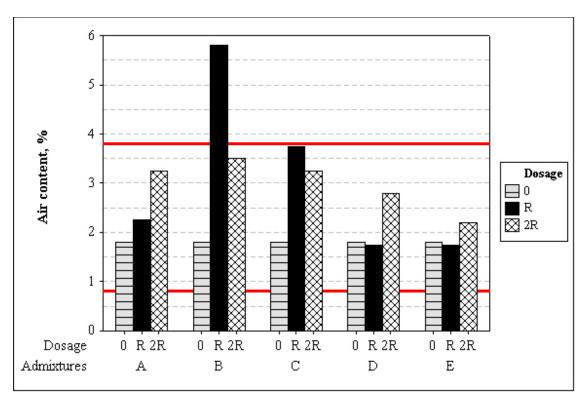


Figure 2. Effect of permeability-reducing admixtures on air content

Setting Time

Setting of concrete can be defined as the onset of rigidity in fresh concrete. Initial set indicates the time limit at which fresh concrete can no longer be handled and placed, while final set indicates the onset of the development of mechanical strength due to hardening (Brooks et al. 2000). Setting is important to the contractor because it influences when finishing and sawing activities should take place. Tests were conducted in accordance with ASTM C403.

The effect of the permeability-reducing admixtures on initial and final setting time is plotted in Figure 3 and Figure 4, respectively, along with the limits given in ASTM C494 for Type S Special Admixtures. Although the mixture proportions were not in accordance with ASTM C494, the limits therein appear to be a reasonable starting point for evaluation.

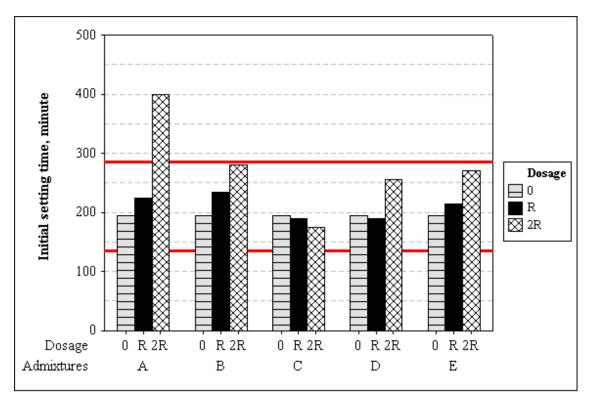


Figure 3. Effect of permeability-reducing admixtures on initial set time

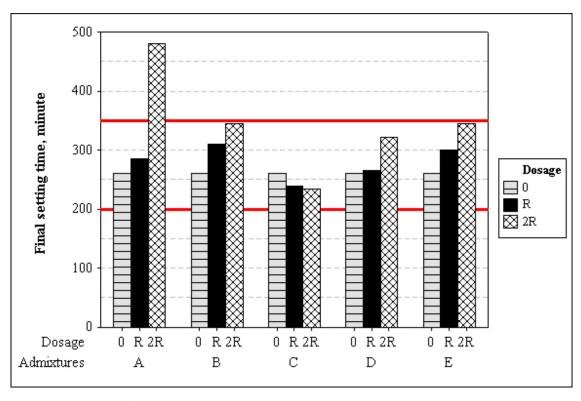


Figure 4. Effect of permeability-reducing admixtures on final set time

The permeability-reducing admixtures tested did not significantly affect the initial and final setting time at the recommended dosages. However, when the selected dosage was higher than the recommended dosage, as in one case, the setting time was significantly affected and resulted in set retardation.

To ensure adequate time for placing, consolidating, and finishing, it is recommended that the effect of permeability-reducing admixtures on setting time should be noted by the manufacturer. If needed, set retarding or accelerating admixtures shall be used to meet specification requirements.

Strength

Many current specifications are dependent largely on the compressive strength of a mixture. This is historically because strength is relatively easy to measure and is an important component of structural performance. However, correlation between strength and potential durability are poor. The effects of the admixtures on 7-day and 28-day compressive strength (ASTM C39) and 56-day flexural strength (ASTM C78) are illustrated in Figure 5, Figure 6, and Figure 7, respectively.

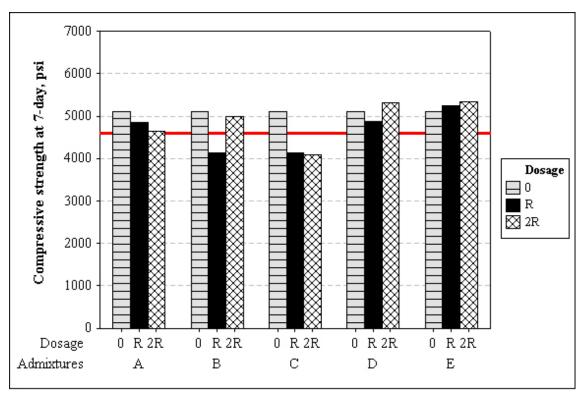


Figure 5. Effect of permeability-reducing admixtures on 7-day compressive strength

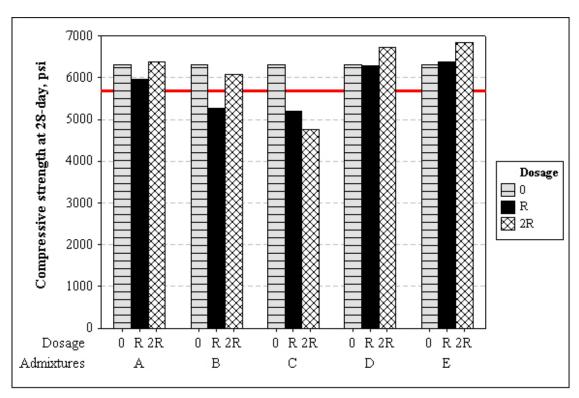


Figure 6. Effect of permeability-reducing admixtures on 28-day compressive strength

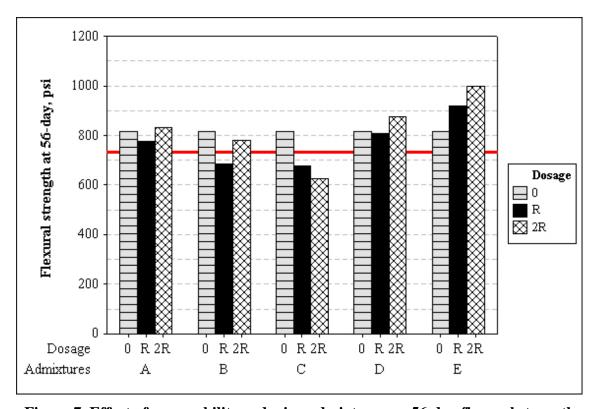


Figure 7. Effect of permeability-reducing admixtures on 56-day flexural strength

ASTM C494 requires that mixtures with special admixtures should not have less than 90% of the strength of the reference mix at the same age. According to this specification, admixtures "B" and "C" did not meet this criterion at the recommended dosages at 7, 28, and 56 days. The reduction of strength in these products may be due to the increased air content of these mixes as shown in Fig. 2.

Improving the strength is not the aim of using permeability-reducing admixtures, and strength is not an adequate indicator of concrete durability (Ballim and Alexander 2005). However, because it is considered to be among the performance criteria, the strength of the mixtures containing PRAN or PRAH permeability-reducing admixtures should not be less than 90% of the reference mix at 28 days, or design guidance should be provided on how to achieve specified strengths for a given product and mixture.

Electrical Resistivity

As discussed above, concrete electrical resistivity is the ability of concrete to oppose the movement of electrons (Smith et al. 2004) and is an indication of pore connectivity. The higher the electrical resistivity, the lower the permeability.

The effects of permeability-reducing admixtures on 7-day and 28-day electrical resistivity were tested (AASHTO TP 95-11) and are illustrated in Figure 8 and Figure 9, respectively. As expected, the resistivity of all the mixtures improved as the testing age increased from 7 days to 28 days. However, the relative performance differences between the reference mix and the mixes containing admixture at 28 days were similar to those at 7 days. The addition of permeability-reducing admixtures increased the electrical resistivity by approximately 7% to 30% compared to the reference mix at the recommended dosages. However, given the objective of using the permeability-reducing admixtures, it is suggested that mixtures incorporating PRAN or PRAH admixtures should provide a minimum of 10% higher electrical resistivity compared to the reference mix at 28 days unless the chemical makeup of the product can be demonstrated to lead to a false low result.

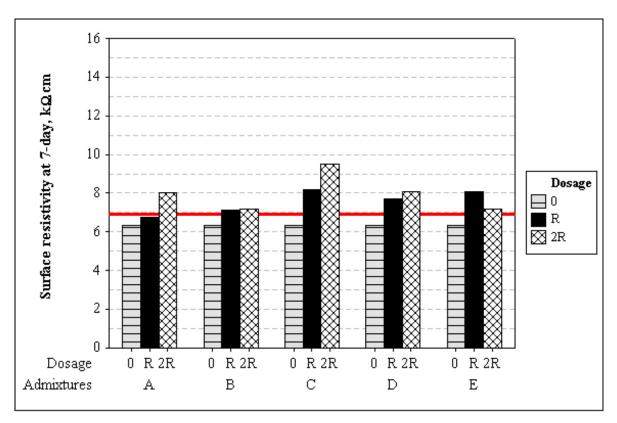


Figure 8. Effect of permeability-reducing admixtures on 7-day electrical surface resistivity

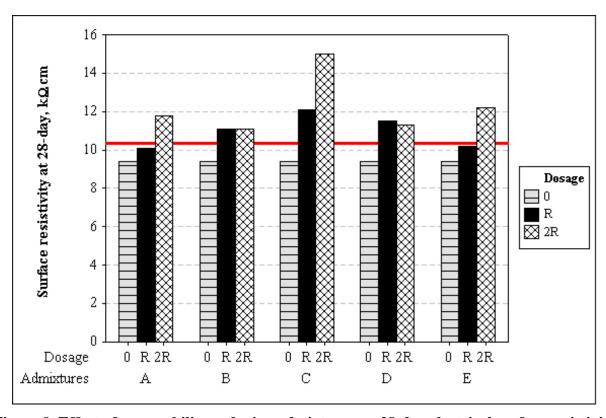


Figure 9. Effect of permeability-reducing admixtures on 28-day electrical surface resistivity

Rapid Chloride Penetration

The rapid chloride test method (ASTM C 1202) measures the current that passes through a saturated concrete specimen under high DC voltage and exposed to a chloride solution on one face. Like resistivity, it is an indirect indicator of the permeability of the system. The effects of permeability-reducing admixtures on 7-day and 28-day rapid chloride penetration (RCP) are illustrated in Figure 10 and Figure 11, respectively. Considering that two samples may differ by 42% according to ASTM C1202, the test results are all within the range of scatter. Due to this large scatter, and because some products will be skewed by this method because of ionic movement, this test method may not be suitable to evaluate the effectiveness of these admixtures on permeability. However, if the test used, the chloride penetration of the mixtures containing PRAN or PRAH types of permeability-reducing admixtures should be 10% lower than that of the reference mixture at 28 days. The recommended limit is shown as a line.

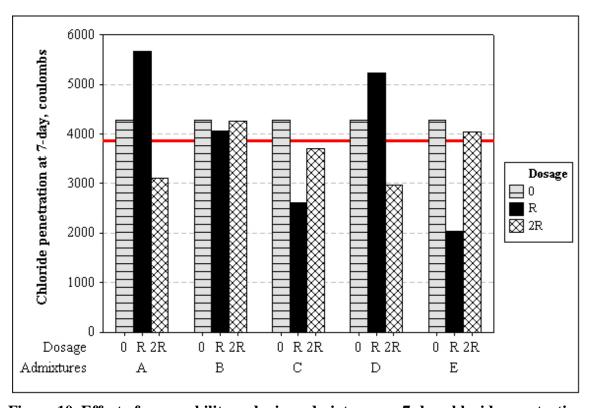


Figure 10. Effect of permeability-reducing admixtures on 7-day chloride penetration

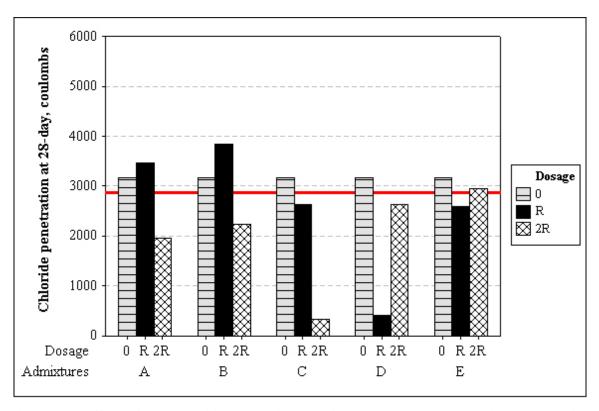


Figure 11. Effect of permeability-reducing admixtures on 28-day chloride penetration

Air Permeability

Measurement of permeability is a direct means of assessing the ability of the system to resist passage of fluids through the matrix. Samples have to be dried before they are tested. Air permeability tests were conducted in accordance with the University of Cape Town Method (Alexander et al. 1999), except that dry air was used instead of oxygen as the test gas. The 7-day and 28-day test results are presented in Figure 12 and Figure 13, respectively.

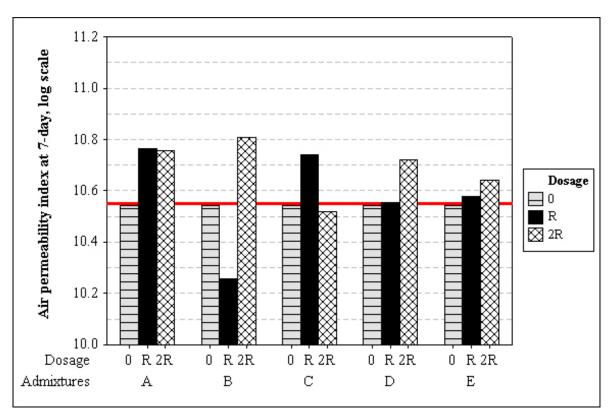


Figure 12. Effect of permeability-reducing admixtures on 7-day air permeability index

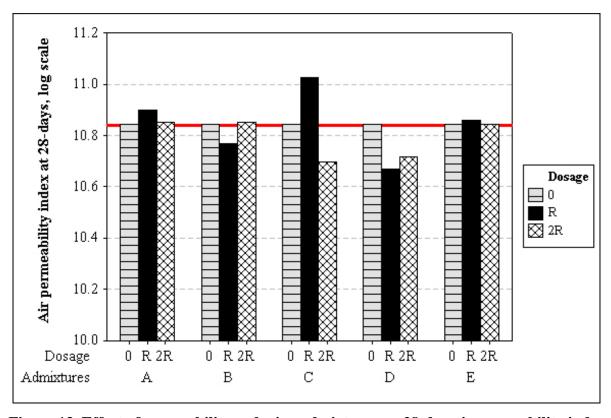


Figure 13. Effect of permeability-reducing admixtures on 28-day air permeability index

Air permeability index (API) is the negative log of the D'Arcy coefficient of permeability (m/s) and uses a log scale (Buenfeld and Okundi 2000). A higher air permeability index indicates lower permeability (Dinku and Reinhardt 1997). As reported by Alexander and Beushausen (2010), the interpretation shown in Table 3 can be applied to the results.

Table 3. Interpretation of API data

Result	Interpretation
API >10.0	Excellent
9.5< API< to 10.0	Good
9.0< API to <9.5	Poor
API < 9.0	Very poor

Based on the provided classification, all the mixtures (including the reference) may be considered "excellent" with an API value higher than 10. The coefficient of variation of this test, by single operator, is stated to be 1.4% (Stanish et al. 2006). The API of the mixes containing permeability-reducing admixtures was similar to the values obtained from the reference mix. Therefore, it is recommended that the API values of mixtures containing PRAN or PRAH admixtures should be equal to or greater than the API of the reference mixture at 28 days. The recommended limit is shown as a line.

Permeable Voids

Absorption is another parameter that indicates concrete permeability (Richardson 2010) (ASTM C642). The effect of permeability-reducing admixtures on 7-day and 28-day permeable voids is illustrated in Figure 14 and Figure 15, respectively.

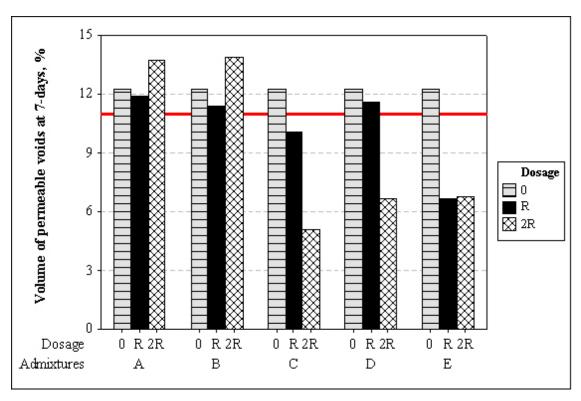


Figure 14. Effect of permeability-reducing admixtures on 7-day volume of permeable pore space voids

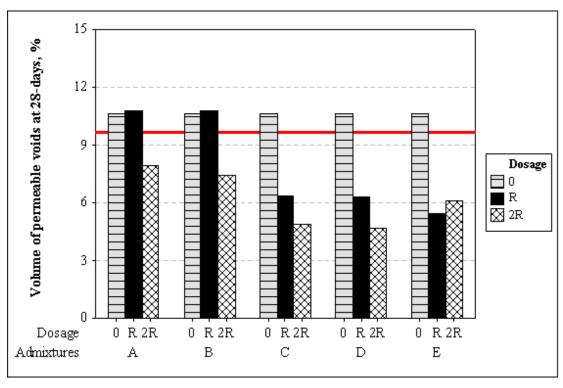


Figure 15. Effect of permeability-reducing admixtures on 28-day volume of permeable pore space voids

Admixtures "A" and "B" did not improve the water absorption at the recommended dosage at both 7 and 28 days. However, increasing the admixture dosage helped decrease the permeable voids at 28 days. On the other hand, due to the different chemistry and reactivity, admixtures "C", "D", and "E" provided significantly less absorption compared to the reference mixture and thus improved the impermeability, especially at later ages. The results of this test showed that the mixes containing the PRAH type admixture did not significantly contribute to decreasing the permeable pore voids volume. Therefore, this test may be considered unsuitable to make a fair judgment on the mixes containing PRAH admixtures. It is recommended that mixtures containing PRAN admixtures should have a minimum of 10% lower permeable pore voids volume than the reference mixture at 28 days. The recommended limit is shown as a line.

Sorptivity

Sorptivity is also an indicator of concrete durability because it measures the rate of absorption by capillary suction. This test is suitable for evaluating the effects of curing on a surface in addition to assessing the quality of the concrete mixture.

The effect of permeability-reducing admixtures on 7-day and 28-day secondary sorptivity is illustrated in Figure 16 and Figure 17, respectively. As the test results show, an overall trend is that the addition of the permeability-reducing admixtures provided lower sorptivity than the reference mixture, as desired. Especially important is the fact that admixture "C" significantly decreased (up to 50%) the rate of absorption at the recommended dosage at both early and later ages.

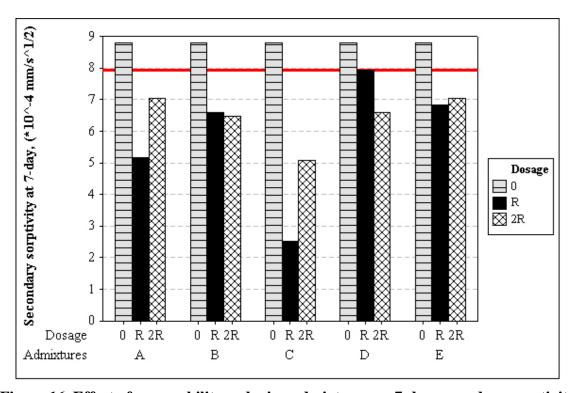


Figure 16. Effect of permeability-reducing admixtures on 7-day secondary sorptivity

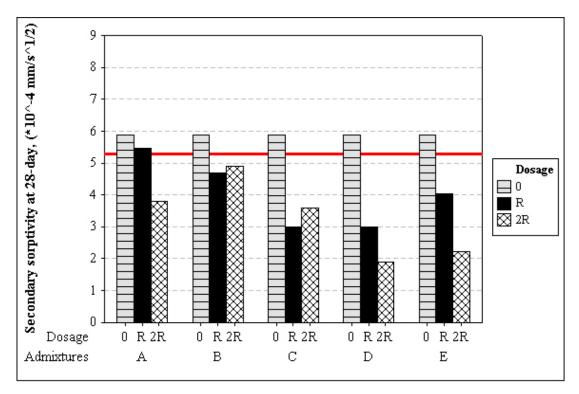


Figure 17. Effect of permeability-reducing admixtures on 28-day secondary sorptivity

This test method may be more suitable for PRAN than PRAH types of admixtures since it does not apply hydrostatic pressure. It is recommended that mixtures containing PRAN admixtures should provide a minimum of 10% lower initial and final sorptivity than the reference mixture at 28 days. The recommended limit is shown as a line.

Shrinkage

Drying shrinkage of a mixture is related to the rate of loss of water in the mixture to the environment or in hydration and has direct impacts on the risk of cracking in the long term. It is unlikely to be directly impacted by permeability-reducing admixtures, but side effects should be kept to a minimum. The 28-day shrinkage test results are presented in Figure 18. According to the limit in ASTM C494, when the length change of the reference mix is less than 0.03% (as in this study), the addition of permeability-reducing admixtures should not shrink more than 0.01% of the reference mixture. All the products were within the acceptable limit (shown as a line).

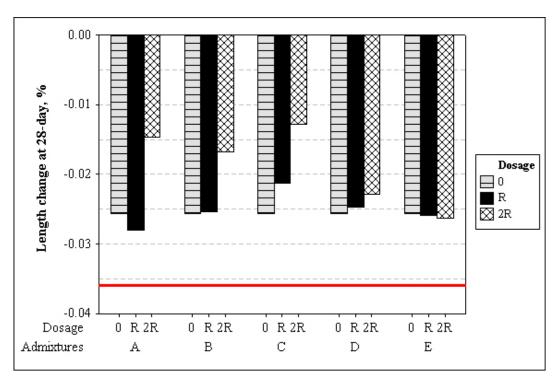


Figure 18. Effect of permeability-reducing admixtures on 28-day length change

It is recommended that the increase of the length change of mixtures containing permeability-reducing admixtures over the reference mixture should not be more than 0.01% at 28 days (moist curing for 14 days and then drying for 14 days).

Discussion on the European Standard EN 934-2 to Evaluate Permeability-Reducing Admixtures

The requirements for water resisting admixtures specified by the European Standard EN 934-2 (2000) are presented in Table 4. Although these requirements do not cover as wide a range of properties as this study, the proposed recommended limits and the EN 934-2 specified requirements for a given property are generally similar. However, the capillary absorption test in EN 934-2 is conducted on mortars, whereas in this study tests were conducted on concrete. The capillary absorption is related to water transportation in the cement paste and particularly in the aggregate-paste interface. In this study, the inclusion of the coarse aggregates likely resulted in the increased porosity of the interfacial transition zone (ITZ) (Scrivener and Nemati 1996). It is therefore not surprising that the requirement provided for the capillary absorption in EN 934-2 is different than the proposed requirement in this study.

Table 4. Specific requirements for water resisting admixtures (at equal consistence or equal w/c ratio*) adapted from EN 934-2 (2000)

Property	Reference mortar/concrete	Test method	Requirements
Capillary absorption	EN 480-1 mortar	EN 480-5	Tested for 7 days after 7 days curing: test mix 50% m/m of control mix. Tested for 28 days after 90 days curing: test mix ≤ 60% of m/m of control mix.
Compressive strength	EN 480-1 concrete mix I	prEN 12390-3	At 28 days: test mix \geq 85% of control mix.
Air content in fresh concrete	EN 480-1 concrete mix I	prEN 12350-7	Test mix ≤ 2% V/V above control mix unless otherwise stated by the manufacturer.

^{*} All tests shall be performed either at equal consistence or equal w/c ratio

Summary

The limits recommended from this work are summarized and presented in Table 5. These limits are set to ensure that mixtures containing permeability-reducing admixtures (exposed to either hydrostatic or non-hydrostatic pressure) can be demonstrated to provide fluid penetration benefits with minimal, or reported, side effects on other critical performance parameters.

Slump, air content, and setting time are not required to match a reference mix. However, design guidance should be provided to account for changes in these properties if they occur.

 ${\bf Table~5.~Recommended~limits~for~concretes~containing~permeability-reducing~admixtures}$

	Property	Test method	Recommended limit
nt .	Surface resistivity	AASHTO TP 95-11	Mixes incorporating permeability-reducing admixtures should provide a minimum of 10% higher electrical resistivity than the reference mix unless it can be demonstrated that the chemical makeup of the admixtures results in a false low result.
Permeability enhancement	Chloride penetration ASTM C1202	If required, chloride penetration of mixtures containing permeability-reducing admixtures should be 10% less than that of the reference mix.	
bility enl	Air permeability	Univ. of Cape Town	The API of mixtures containing permeability-reducing admixtures should have an API index value that is equal to or greater than that of the reference mixture.
Permea	Permeable voids*	ASTM C642	The permeable pore space voids volume of mixtures containing PRAN permeability-reducing admixtures should be a minimum of 10% lower than that of the reference mixture.
	Sorptivity*	ASTM C1585	The sorptivity of mixtures containing PRAN permeability-reducing admixtures should be a minimum of 10% lower than that of the reference mix.
	Workability	ASTM C143	The effect of permeability-reducing admixtures on slump should be noted by the manufacturer to allow adjustments in mix proportioning for desired workability.
properties	Air content and air void system	ASTM C231	The effect of permeability-reducing admixtures on air content and air void system should be noted by the manufacturer to allow adjustments in mix proportioning. When needed, adjustment to the air-entraining admixture or air-detraining admixture dosage shall be made to meet specification requirements.
Other prop	Setting time	ASTM C403	The effect of permeability-reducing admixtures on setting time should be noted by the manufacturer to allow adjustments in mix proportioning. If needed, set retarding or accelerating admixtures shall be used to meet specification requirements.
	Strength	ASTM C39 & C78	The strength of mixtures containing permeability- reducing admixtures should not be less than 90% of the strength of the reference mix or design guidance should be provided on how to achieve specified strengths.
	Shrinkage	ASTM C157	The increase of the length change of mixtures containing permeability-reducing admixtures over the reference mixture should not be more than 0.01%.

RECOMMENDATIONS

Based on the data collected and following some of the recommendations of ASTM C494 and EN-934-2, a testing protocol for various fresh and hardened concrete properties was developed to evaluate the effectiveness and performance of hydrophilic and hydrophobic types of permeability-reducing admixtures. Limits were set to ensure that the durability indicating properties exhibit higher performance than the mixtures with no permeability-reducing admixture while other properties are not adversely affected or are at least compensated for.

The selection of the type of permeability-reducing admixture depends on the environment to which it is exposed and the purpose for its use. If the structure will be exposed to hydrostatic pressure, a PRAH type admixture may be suitable for the purpose. However, if the expected water penetration will be through capillary absorption, then a PRAN type admixture may be sufficient.

Comparative tests should be conducted using a reference mixture without product at a w/cm of 0.45 and a similar test mixture containing the product at the selected dosage. Adjustments should be made to match air content of the reference mixes while keeping w/cm constant. Recommended limits are given in Table 5.

REFERENCES

- AASHTO TP 95-11. (2011). "Standard method of test for surface resistivity indication of concrete's ability to resist chloride ion penetration." American Association of State and Highway Transportation Officials.
- ACI 212.3R-10. (2010). "Report on chemical admixtures for concrete." American Concrete Institute Committee 212, Farmington Hills, MI.
- Aitcin P. C. (2003). "The durability characteristics of high performance concrete: A review." *Cement and Concrete Composites*, 25(4-5), 409–420.
- Alexander, M. G., and Beushausen, H. (2010). "Durability design of reinforced concrete structures." Concrete Technology for Structural Engineers Workshop, South Africa.
- Alexander, M. G., Mackechnie, J. R., and Ballim, Y. (1999). "Guide to the use of durability indexes for achieving durability in concrete structures." Research Monograph 2, University of Cape Town and University of the Witwatersrand, South Africa.
- ASTM C1202. (2012). "Standard test method for electrical indication of concrete's ability to resist chloride ion penetration." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C143. (2012). "Standard test method for slump of hydraulic-cement concrete." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C150. (2012). "Standard specification for portland cement." ASTM Committee C01, ASTM International, West Conshohocken, PA.
- ASTM C1556. (2011). "Standard test method for determining the apparent chloride diffusion coefficient of cementitious mixtures by bulk diffusion." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C157. (2008). "Standard test method for length change of hardened hydraulic-cement mortar and concrete." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C1585. (2013). "Standard test method for measurement of rate of absorption of water by hydraulic-cement concretes." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C1611. (2014). "Standard test method for slump flow of self-consolidating concrete." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C192. (2014). "Standard practice for making and curing concrete test specimens in the laboratory." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C231. (2014). "Standard test method for air content of freshly mixed concrete by the pressure method." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C39. (2014). "Standard test method for compressive strength of cylindrical concrete specimens." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C403. (2008). "Standard test method for time of setting of concrete mixtures by penetration resistance." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C494. (2013). "Standard specification for chemical admixtures for concrete." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- ASTM C642. (2013). "Standard test method for density, absorption, and voids in hardened concrete." ASTM Committee C09, ASTM International, West Conshohocken, PA.

- ASTM C78. (2010). "Standard test method for flexural strength of concrete (using simple beam with third-point loading)." ASTM Committee C09, ASTM International, West Conshohocken, PA.
- Bagheri, A. R., and Zanganeh, H. (2012). "Comparison of rapid tests for evaluation of chloride resistance of concretes with supplementary cementitious materials." *J. Mater. Civ. Eng.*, 10.1061/(ASCE)MT.1943-5533.0000485, 1175–1182.
- Ballim, Y., and Alexander, M. G. (2005). "Towards a performance-based specification for concrete durability." African Concrete Code Symposium, South Africa.
- Brooks, J. J., Megat Johari, M. A., and Mazloom, M. (2000). "Effect of admixtures on the setting times of high-strength concrete." *Cement and Concrete Composites*, 22(4), 293–301.
- BS 1881-122. (2011). "Testing concrete. Method for determination of water absorption." British Standard, UK.
- BS EN 12390. (2012). "Testing hardened concrete. Shape, dimensions and other requirements for specimens and moulds." British-Adopted European Standard.
- Buenfeld, N., and Okundi, E. (2000). "Effect of cement content on transport in concrete." *Magazine of Concrete Research*, 52(1), 73-76.
- CRD-C 48-92. (1992). "Standard test method for water permeability of concrete." U.S. Army Corps of Engineers.
- Day, K. W. (2006). "Concrete mix design, quality control and specification." 3rd Ed., Taylor and Francis, NY.
- Dao, V. T. H., Dux, P. F., Morris, P. H., and Carse, A. H. (2010). "Performance of permeability-reducing admixtures in marine concrete structures." *ACI Materials Journal*, 107(3), 291-296.
- DIN 1048. (1991). "Testing concrete; testing of hardened concrete." Deutsches Institut Fur Normung E.V. (German National Standard), Germany.
- Dinku, A., and Reinhardt, H. (1997). "Gas permeability coefficient of cover concrete as a performance control." *Materials and Structures*, 30(7), 387–393.
- EN 934-2. (2000). "Admixtures for concrete, mortar and grout, Part 2: Concrete admixtures, definitions and requirements." European Committee for Standardization, Brussels.
- Hamami, A. A., Turcry, Ph., and Ait-Mokhtar, A. (2012). "Influence of mix proportions on microstructure and gas permeability of cement pastes and mortars." *Cement and Concrete Research*, 42(2), 490–498.
- Kosmatka, S. H., Kerkhoff, B., and Panarese, W. C. (2008). "Design and control of concrete mixtures." 14th Ed., Portland Cement Association, Skokie, IL.
- Lamond, J. F., and Pielert, J. H. (2006). "Chapter 1, Introduction." In *Significance of Tests and Properties of Concrete and Concrete Making Materials*. Lamond, J. F., and Pielert, J. H. (eds.), ASTM International STP 169D, PA.
- Lane, S. D., Detwiler, R. J., and Hooton, R. D. (2010), "Testing transport properties in concrete: Assessing key indicators of durability." *Concrete International*, 32(11), 33–38.
- Liu, R., Durham, S. A., Rens, K. L., and Ramaswami, A. (2012). "Optimization of cementitious material content for sustainable concrete mixtures." *J. Mater. Civ. Eng.*, 24(6), 745–753.
- Megat Johari, M. A., Brooks, J. J., Kabir, S., and Rivard, P. (2011). "Influence of supplementary cementitious materials on engineering properties of high strength concrete." *Constr. Build. Mater.*, 25(5), 2639–2648.

- Munn, R. L.; Kao, G.; and Chang, Z. T. (2003), "Performance and compatibility of permeability reducing and other chemical admixtures in Australian concretes," *Seventh CANMET/ACI International Conference on Superplasticizers and Other Chemical Admixtures in Concrete*, SP-217, V. M. Malhotra (ed.), American Concrete Institute, Farmington Hills, MI, 361–379.
- Ramachandran, V. S. (1995). *Concrete Admixtures Handbook Properties, Science, and Technology*. 2nd Ed., Noyes Publications, NJ.
- Richardson, A. (2010). "Chapter 5, Durability performance tests." In *Concrete Durability: A Practical Guide to the Design of Durable Concrete Structures*. Marios Soutsos (ed.), Thomas Thelford, London, UK.
- Scrivener, K. L., and Nemati, K. M. (1996). "The percolation of pore space in the cement paste/aggregate interfacial zone of concrete." *Cement and Concrete Research*, 26(1), 35–40.
- Smith, K. M., Schokker, A. J., and Tikalsky, P. J. (2004). "Performance of supplementary cementitious materials in concrete resistivity and corrosion monitoring evaluations." *ACI Materials Journal*, 101(5), 385–390.
- Stanish, K., Alexander, M., and Ballim, Y. (2006). "Assessing the repeatability and reproducibility values of South African durability index tests." *Journal of the South African Institution of Civil Engineering*, 48(2), 10–17.
- Viles, R., (2010). "Chapter 7, Chemical admixtures." In *Concrete Durability: A Practical Guide to the Design of Durable Concrete Structures*. Marios Soutsos (ed.), Thomas Thelford, London, UK.
- Yurdakul, E., Taylor, P., Ceylan, H., and Bektas, F. (2014). "Effect of water-to-binder ratio, air content, and type of cementitious materials on fresh and hardened properties of binary and ternary blended concrete." *J. Mater. Civ. Eng.*, 26(6), 04014002.