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Proportioning for performance-based concrete pavement mixtures

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Proportioning for performance-based concrete pavement mixtures

by

Ezgi Yurdakul

A dissertation submitted to the graduate faculty
in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

Major: Civil Engineering (Civil Engineering Materials)

Program of Study Committee:
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2013

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Ezgi Yurdakul

ABSTRACT

The work presented in this dissertation involves an effort to develop a mix proportioning tool that can be used to determine the required type and amount of concrete components in a mixture based on the desired fresh and hardened concrete properties.

Concrete performance is affected by the quantity and quality of the paste, and aggregate systems. Therefore, this study analyzed the effect of binder systems with different types and content; the paste quality; and size, shape, texture and gradation of different aggregate systems on various fresh and hardened concrete properties.

In this experimental program, a total of 178 mixtures were prepared with 7 different gradation systems, 12 binder systems, 25 binder contents, 6 different water-to-binder ratio (w/b), and 3 different nominal air content.

Fresh properties of slump, air content, air-void system, setting time, unit weight, and temperature were tested. Hardened properties of compressive strength, rapid chloride penetration, surface resistivity, air permeability, and shrinkage were tested at various ages. However, to develop such a tool, this study overall focused on the assessment of workability, compressive strength, and durability as these three properties are commonly used as indicators of concrete performance. Durability was assessed by testing the rapid chloride penetration, and surface resistivity at 28-days.

As part of this study, an artificial neural network (ANN) approach has been used for concrete mix proportion design to analyze the complexity between concrete properties and concrete components.

Results have shown that development of a performance-based mix proportioning tool is possible for mixtures when aggregate gradation is not varied. Development of a mix proportioning tool with addition of the various aggregates systems generally was not as successful due to the increased variability of the mix design parameters such as size, shape, texture, and gradation of aggregates. The proposed mix proportioning tool is promising and achievable in terms of predicting the values of the tested properties based on the mix design variables. Although the proposed mix proportioning tool is not completely ready for prime time, the findings of this study can be implemented in real time when this approach is used with a larger data set.

CHAPTER 1. INTRODUCTION

The aim of this study is to develop a mix proportioning tool that can be used to determine the required type and amount of concrete components in a mixture based on the desired fresh and hardened concrete properties.

Concrete performance is known to be affected by the quantity and quality of the paste, and aggregate systems. Therefore, to develop such a tool, the work was analyzed under 3 different studies and then the results were combined:

- The effect of paste quantity on concrete performance: The fresh and hardened concrete properties are affected by paste quantity. However, this effect may be positive or negative depending on the property. For example, when the other parameters are kept constant, increasing the paste content improves the workability. Thus, high paste content may be seen desirable in terms of workability. However, on the other hand, higher the paste content larger the shrinkage (Popovics 1990). Therefore, paste content should be kept as minimum as possible due to its negative effect on shrinkage. Due to the fact that properties are affected differently by the paste quantity, the study presented in Chapter 2 investigated the paste quantity that is required to achieve the desired workability while keeping the negative effect on shrinkage, durability, strength at minimum. Rather than simply assessing the properties based on the paste quantity, this study focused on the paste-to-voids volume ratio. The paste-to-voids volume ratio was preferred to be analyzed because in concrete mixes, enough cement

paste should be provided that not only fills the voids between aggregates but also covers the aggregates and separates them to reduce the inter-particle friction between aggregates.

- The effect of paste quality on concrete performance: Concrete performance is likely to be compromised if there is insufficient amount of paste to fill all of the voids between the aggregate particles, as mentioned above. However, once sufficient paste is provided, the quality of the paste dominates the trends. Therefore, after determining the paste quantity required to achieve the desired workability and finishability (based on the results of the study presented in Chapter 2), the paste content was fixed, accordingly. Later, a variety of mix characteristics were further varied to determine the effect of paste quality on concrete performance. Chapter 3 presents the details of this study in which the effect of water-to-binder ratio (w/b), air content, and type of cementitious materials were analyzed on fresh and hardened properties of binary and ternary blended concrete mixtures in pavements.

• The effect of aggregate systems on overall concrete performance: Aggregates constitute 60 % to 75 % of the total volume of concrete; therefore their selection is very important in the development of a mix proportioning tool process. Gradation, shape, porosity, and surface texture of aggregates affect the concrete properties. For example, spherical, well rounded with smooth surfaced aggregates increase workability whereas angular, elongated, rough surfaced aggregates decrease workability and cause segregation. The volume of voids

between fully compacted aggregates is a key factor determining the paste volume requirements (Koehler and Fowler, 2006). Therefore, throughout this study, instead of considering voids of the fine, intermediate, and coarse aggregates separately, the voids between the combined aggregates are determined. In the process of developing a mix proportioning tool, Chapter 4 includes the findings of a sub-study in which various aggregate systems with different shape, texture, size and aggregate gradations were evaluated as a combined data set.

Background

Terminology

The terminology used in this dissertation was obtained from American Concrete Institute (ACI) Concrete Terminology standard (2013).

Specification

In this dissertation, specification refers to “an explicit set of requirements to be satisfied by a material, product, system, or service” (ACI 2010). In the concrete construction industry, following are the primary types of specifications:

- Materials specifications - provide minimum requirements for components and properties
- Construction specifications - form part of the contract between owner and contractor

Mix Proportioning

In this dissertation, mix proportioning refers to the process of determining the quantities of concrete components required to achieve the specified concrete properties.

Prescriptive-Based Specifications

According to ACI (2010), prescriptive-based specifications focus on the properties of:

- concrete components
- mix proportions
- batching, mixing, and transport of fresh concrete
- a range of construction operations from placing to curing.

Prescriptive-based specifications rely on observed or implied relationships between the specified and desired properties, although the desired end-product performance may or may not be described (ACI 2010).

Performance-Based Specifications

Performance-based specifications define the required results, the criteria to evaluate performance, and verification methods without requirements for how the results are to be obtained (ACI 2010). Performance-based specifications promote better use of materials because they focus on concrete properties (e.g. workability, durability, and strength) needed to achieve service life capacity and longevity (Bickley et al. 2006; Lobo et al. 2006; Day 2006; Braselton and Blair 2004; Taylor 2004).

Moving Toward Performance-Based Specifications

Prescriptive-based specifications are often based on empirical relationships (Lobo et al. 2006). In addition, these specifications are often overly conservative, which may lead to higher costs and negative results (Lobo et al. 2006). Therefore, a shift from prescriptive specifications to performance-based specifications is needed since

performance-based specifications address requirements for mechanical and functional properties of the concrete.

Mix Proportioning

According to Kosmatka et al. (2008), a properly proportioned concrete should have the following properties:

- Acceptable workability of the concrete in fresh state
- Durability, strength, and uniformity of the hardened concrete
- Economy

A critical aim in proportioning a concrete mixture is to ensure that “it fits for the purpose for which it is intended and for the expected life during which it is to remain in service” (Neville 2000) while using available materials at minimum cost.

Selecting Mix Characteristics

Prior to the mix proportioning, the selection of mix characteristics (e.g. cement, water-to-binder ratio, and aggregates) should be done for durable concrete structures (Van Dam et al. 2006). The mix characteristics are determined based on the following factors (Kosmatka et al. 2008):

- Exposure conditions (e.g. sulfate attack)
- Intended use of the concrete
- The size and shape of building elements
- The physical properties of the concrete (e.g. strength and durability)

Challenges

Specifying the Minimum Cement Content

In the United States of America, many state Department of Transportation (DOT) agencies specify a minimum cement content between 500 and 600 pcy as presented in Table 1 (Rudy 2009, Pavement Interactive 2007). For example, Washington State DOT (2002) specifies the minimum binder content to be 565 pcy with Class F fly ash at up to 25% replacement level. Virginia State DOT (2011) states the minimum binder content to be 585 pcy for A3 general portland cement concrete (PCC) pavements. Furthermore, Minnesota State DOT (2009) requires the minimum binder content of 530 pcy for Grade A concrete for PCC pavements. However, these binder contents are often specified conservatively and may exceed the amount needed for the desired strength and durability.

**Table 1. Minimum cement content specifications for slip-form paving mixtures.
(After Rudy 2009)**

State	Minimum cement content for plain concrete (lb/yd³)
Illinois (ILDOT, 2007)	565
Indiana (INDOT, 2008)	564
Iowa (Iowa DOT, 2008)	573
Kansas (KDOT, 2007)	602
Michigan (MDOT, 2003)	564
Minnesota (MnDOT, 2008)	530
Missouri (MoDOT, 2008)	560
New York (NYSDOT, 2008)	605
Ohio (ODOT, 2008)	600
Pennsylvania (PennDOT, 2009)	587
Virginia (VDOT, 2007)	564
Wisconsin (WisDOT, 2008)	565

The Usage of Excessive Amount of Cement Content in Prescriptive-Based Methods

One challenging issue with the current mix proportioning methods is that they may promote using higher amount of some materials than needed to achieve the desired strength and durability (Grove and Taylor 2012, Lee et al. 2009, Shilstone and Shilstone 2002). This excessive amount should be minimized to prevent its negative impact on costs and environment because:

- cement is the most expensive component in concrete
- each ton of portland cement clinker production emits approximately 1 ton of carbon dioxide (Mehta, 1999)
- cement production emits approximately 5% of global carbon dioxide (CO₂) and 5% of global energy consumption (Hendriks et al. 2004)

In addition to the environmental effects, the high cement content will cause the concrete to become sticky as well as have shrinkage and cracking problems (Dhir et al. 2004). Therefore, cement content should be balanced to achieve performance while minimizing risk of these problems.

Current Mix Proportioning Methods

Most mix proportioning methods that are being currently used were developed years ago when the water-reducing admixtures and supplementary cementitious materials were not commonly used (Grove and Taylor 2012). For example, one of the most commonly used mix proportioning guidelines, ACI 211, has not been significantly changed since 1991. However, technology has been improving day-by-day in concrete industry. For example, in today's world, it is possible to produce concrete with zero

slump that is workable; or have a concrete that can reach to a compressive strength of 4000 psi at 4-hours (Grove and Taylor 2012). Despite these advancements, the basic rules of concrete technology have not changed, and the mix proportioning methods have not adapted to these advancements and become more prospective-based.

Motivation

The motivation behind developing a mix proportioning tool is that the current mix proportioning methods are predominantly prescriptive-based, thus limiting the choice of materials, and may promote using higher amounts of some materials than needed which results in increased cost. The construction industry has been moving from prescriptive towards performance-based specifications. Therefore, a performance-based mix proportioning method needs to be developed which can predict the required amount and type of materials to fulfill the desired concrete properties for a given project specification.

Research Goal and Objective

As part of the transition between prescriptive-to-performance based specifications, this study aimed to develop a mix proportioning tool that can be used to determine the required concrete components' amount (e.g. the amount of cement content) based on the desired fresh and hardened concrete properties.

To develop such a tool, this research study overall focused on the assessment of:

- workability,
- compressive strength, and

- durability

These three properties are used as indicators of concrete performance; therefore a similar level of attention was paid while evaluating each of these properties. Durability was assessed by testing the rapid chloride penetration, and surface resistivity at 28-days.

The primary target of applying this mix proportioning tool was for concrete pavements therefore the mix design of the mixtures and desired fresh and hardened properties were determined based on the requirements of the pavement.

Significance of the Research

It is well documented in the literature (Wassermann et al. 2009, Buenfeld and Okundi 1998) that concrete performance and tested properties are compared based on the parameter of “cementitious content”. Although it is technically true that cementitious content affects both fresh and hardened concrete properties, it does not go beyond being a theoretical assessment because this parameter does not consider the amount, type, size, shape, texture, and gradation of the aggregate systems. Therefore, due to the given limitation, using the parameter of “cementitious content” is not sufficient to fully understand the overall concrete behavior since it is critical to consider the interaction between paste and aggregate systems.

Therefore, a new parameter is needed that integrates the required amount of paste based on the aggregate system. This study applies a new concept by using the parameter of “Paste-to-voids volume ratio ($V_{\text{paste}}/V_{\text{voids}}$)”. The paste-to-voids volume ratio ($V_{\text{paste}}/V_{\text{voids}}$) was calculated by calculating the paste volume of concrete mixtures and dividing that value by the volume of voids between the combined consolidated

aggregates. The paste volume was calculated by adding up the volume of water, the cementitious materials, and the measured air in the system. The voids refer to the space between the compacted combined aggregates that was determined by following a similar procedure as the ASTM C29.

Although the idea of relating performance of a mixture to paste volume (for a given aggregate system) was initially used to assess the self-consolidating concrete (Koehler and Fowler 2007), this dissertation presents the studies which apply the concept of “ $V_{\text{paste}}/V_{\text{voids}}$ ” for the first time in concrete pavements. Using this new concept ($V_{\text{paste}}/V_{\text{voids}}$) provides essential information to engineering practice, and may be more accurate than the parameter of “cementitious content” because $V_{\text{paste}}/V_{\text{voids}}$ considers how much paste is needed to:

- coat the aggregate particles
- fill the space (voids) between the combined aggregate systems
- separate the aggregate particles to provide the desired workability

Generally, depending on the project specifications, various performance criteria are required to be achieved. While workability is a property that is commonly ensured to be achieved due to its aid during the placement, the selection of hardened properties will vary depending on project requirements. Until recent years, mix proportioning has been commonly done based on the fact that strength has a higher priority than durability criteria. As distinct from this approach, in this study, similar level of attention was paid on workability, durability, and strength while determining the effect of $V_{\text{paste}}/V_{\text{voids}}$ on concrete performance.

Dissertation Organization

This dissertation is divided into six chapters. Chapter 1 provides an introduction and background information to this dissertation.

The main findings and results are presented in Chapters 2, 3 and 4. Each chapter comprises a paper that has been either published, submitted for publication, or ready for submission to peer reviewed journals. The papers are ordered in the thesis as follows:

- Chapter 2:

Yurdakul E, Taylor P. C., Ceylan H., and Bektas F. *Effects of Paste-to-Voids Volume Ratio on the Performance of Concrete Mixtures*. Accepted for publication in Journal of Materials in Civil Engineering (ASCE).

Chapter 2 presents a study that was conducted to investigate the minimum paste volume required with an appropriate water-to-cementitious ratio (w/cm) to achieve required workability, durability, and strength requirements of concrete mixtures for pavements.

- Chapter 3:

Yurdakul E, Taylor P. C., Ceylan H., and Bektas F. *Effect of Water-to-Binder Ratio, Air Content, and Type of Cementitious Materials on Fresh and Hardened Properties of Binary and Ternary Blended Concrete*. Submitted for publication in Journal of Materials in Civil Engineering (ASCE).

Chapter 3 presents a study that was conducted to develop data to help quantify how changes in the air content, water-to-binder ratio (w/b), and type and replacement

level of supplementary cementitious materials (SCMs) influence the properties of binary and ternary concrete mixtures.

- Chapter 4:

Yurdakul E, Taylor P. C., and Ceylan H. *Development of a Performance-Based Mix Proportioning Tool Using the Artificial Neural Network Modeling*. Will be submitted to Journal of Materials in Civil Engineering (ASCE).

Chapter 4 presents a study that was conducted to develop a performance-based mix proportioning tool that predicts the required quantities of locally available materials for the desired fresh and hardened concrete properties. Artificial-neural network modeling was used to establish the relationship between the variables and the tested properties.

Finally, Chapter 5 presents the major findings and conclusions of the study and Chapter 6 provides recommendations for future research.

In addition to the research presented in the main part of this dissertation, additional research studies were conducted during the PhD study. Appendices present the conference papers that include the findings of these research studies:

- Appendix A:

Yurdakul E, Taylor P. C., and Ceylan H. *The Application of X-Ray Fluorescence to Assess Mix Concrete Proportions*. Proceedings of the 10th International Conference on Concrete Pavements, International Society for Concrete Pavements, Quebec, Canada, July 8-12, 2012.

- Appendix B:

Yurdakul E, Taylor P. C., Ceylan H., and Bektas F. *Minimizing Cementitious Content for Performance and Sustainability in Rigid Pavements*. Proceedings of the International Concrete Sustainability Conference, National Ready-Mixed Concrete Association, Boston, USA, August 9-11, 2011.

- Appendix C:

Yurdakul E, Taylor P. C., Ceylan H., and Bektas F. *Development of a Protocol to Assess Integral Waterproofing Admixtures*. Proceedings of the International Conference on Long-Life Concrete Pavements, Federal Highway Administration, Seattle, USA, September 18-21, 2012.

- Appendix D:

Yurdakul E, Taylor P. C., Ceylan H., and Bektas F. *Preliminary Investigation on Determining the Minimum Cement Content in Rigid Pavements*. Presented in the 90th Annual Transportation Research Board (TRB) Meeting, Washington DC, USA, January 23-27, 2011.

- Appendix E:

Yurdakul E, Taylor P. C., Ceylan H., and Bektas F. *Investigating the Minimum Binder Content Requirements by Using Class F Fly Ash in Concrete Pavements*. Presented in the International Conference on Construction, Architecture and Engineering, Athens, Greece, June 20-23, 2011.

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CHAPTER 2. EFFECT OF PASTE-TO-VOIDS VOLUME RATIO ON THE PERFORMANCE OF CONCRETE MIXTURES

A paper accepted for publication in Journal of Materials in Civil Engineering (ASCE)

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Abstract

The purpose of this study is to investigate the minimum paste volume required with an appropriate water-to-cementitious ratio (w/cm) to achieve required workability, strength, and durability requirements of concrete mixtures for pavements. In this experimental program, 64 concrete mixtures with varying w/cm, cementitious content and binder type were prepared and tested. The fine aggregate-to-total aggregate ratio was held constant for all the mixtures. Fresh and hardened concrete properties of the mixtures were determined at various ages.

Test results have shown that approximately 1.5 times more paste by volume is required than voids between the aggregates to achieve a minimum performance in concrete for pavements. For a given w/cm, strength is independent of cementitious

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content after a critical value is provided. When w/cm is constant, increasing paste content increased chloride penetrability and air permeability.

CE Database subject headings: Concrete pavements; Optimization; Concrete tests; Durability; Compressive strength; Permeability; Fly ash; Slag.

Introduction

A critical aim in proportioning a concrete mixture is to ensure that “it fits for the purpose for which it is intended and for the expected life during which it is to remain in service” (Neville 2000) while using available materials at minimum cost.

Good concrete is controlled through materials selection, mix proportioning, and workmanship (e.g. concrete must be properly mixed, transported to the job site, placed, finished and followed by proper curing) (PCA 1975). In general, workability and strength are the properties most often assessed (Bickley et al. 2010; PCA 1975).

However, the concrete properties of interest are not limited to these and other properties such as air content, setting time, shrinkage may be assessed (Bickley et al. 2010). In addition, the mix proportions should be optimized for economy and sustainability (PCA 1975).

This paper discusses an approach to achieve a quality concrete mixture with minimum impact whilst meeting specifications. Currently, construction specifications are predominantly prescriptive with some performance characteristics (Taylor 2004); however, the construction industry has been moving from prescriptive towards performance-based (P2P) specifications (Bickley et al. 2010; Lobo et al. 2006; Day 2006; Taylor 2004).

Performance-based specifications promote better use of materials because they focus on concrete properties (e.g. workability, strength, and durability) needed to achieve service life capacity and longevity (Bickley et al. 2010; Lobo et al. 2006; Day 2006; Braselton and Blair 2004; Taylor 2004). The Federal Highway Administration (FHWA) has charted a roadmap to performance-based specifications (Bickley et al. 2010). Taylor (2004) has postulated nine steps in the transition from P2P and stated that some of the essential requirements may be initially prescriptive, but in time would become performance criteria. As part of the transition, this study analyzed the effect of paste-to-voids volume ratio on concrete properties in order to reduce dependence on prescriptive minimum cement content based specifications.

In concrete mixes, enough cement paste should be provided that not only fills the voids between aggregates but also covers the aggregates and separates them to reduce the inter-particle friction between aggregates when the mixture is in the fresh state (Kosmatka et al. 2008; Koehler and Fowler 2007; Hu and Wang 2007; Ferraris and Gaidis 1992; Kennedy 1940). This is known as “excess paste theory” (Kennedy 1940). However, studies (Wassermann et al. 2009; Dhir et al. 2004; Kennedy 1940) have shown that in some cases, specified cement contents are higher than required. This high cementitious content will increase shrinkage and permeability, and thus decrease the durability of concrete. In addition, increasing cement content also has a negative impact on the environment due to the high energy consumption and CO₂ emission of cement production (Liu et al. 2012; Wassermann et al. 2009). It is likely possible to reduce this amount without compromising workability, strength, and durability (Kennedy 1940).

The purpose of this study is to investigate the critical minimum paste requirement for performance by analyzing the effect of paste-to-voids volume ratio on concrete properties.

Material and Methods

Materials

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

- ASTM C150 Type I ordinary portland cement (OPC)
- ASTM C618 Class F fly ash
- ASTM C618 Class C fly ash
- ASTM C989 Grade 120 slag cement
- 25.4-mm nominal maximum size crushed limestone
- 4.75-mm nominal maximum size concrete sand
- ASTM C494 Type F polycarboxylate based high range water reducer (HRWR)

The chemical composition of the cementitious materials is presented in Table 1.

Combined Aggregate Gradation

The void percentage of the combined aggregates was kept constant as 19.8% for all the mixtures to remove the aggregate grading as a variable from the experimental matrix. This was selected by assessing the 0.45 power chart (Bureau of Public Roads 1962), Shilstone workability factor chart and specific surface of the aggregates.

The fine aggregate-to-total aggregate ratios of 0.45, 0.42 and 0.39 provided the best mixtures in FHWA 0.45 power curve, Shilstone workability factor chart and

specific surface charts, respectively (Fig. 1a-c). Based on the average of these numbers, the fine aggregate-to-total aggregate ratio was selected as 0.42.

The appropriateness of the selected ratio of 0.42 was checked by plotting the data in an ASTM C33 plot (Fig. 1d) and a “Haystack” plot (Fig. 1e). The Haystack plot did not present an ideal combination, but was the best combination that could be achieved with the materials available. While not ideal, this type of gradation is common in many construction sites, and is therefore an appropriate combination for this research study.

The unit weight and volume of voids in the combined aggregate were measured in accordance with ASTM C29. The overall unit weight of the combined aggregates was 2098 kg/m^3 .

The specific gravity and absorption of the coarse and fine aggregates were determined using ASTM C127 and ASTM C128, respectively. The saturated surface dry (SSD) specific gravity and the absorption values of the coarse aggregates were 2.67 and 1.0%, respectively. The SSD specific gravity and the absorption values of the fine aggregates were 2.62 and 1.1%, respectively.

Mix Design

In concrete mixes for pavements, the cementitious content generally ranges between 300 and 360 kg/m^3 (Pavement Interactive 2007). Typical water cementitious ratios for concrete paving materials are between 0.40 and 0.50 (Van Dam et al. 2000). The range selected in this study included the extremes at both ends (i.e. 240 and 415 kg/m^3) to show their effects on concrete performance. Thus, a total of 64 mixtures were prepared as follows:

- 4 cementitious contents – 240, 300, 360 and 415 kg/m³ (numbers were converted from English units to SI units and rounded up to the nearest 5 kg/m³. The selected cementitious content range ranges between 400, 500, 600 and 700 lb/yd³)
- 4 w/cm – 0.35, 0.40, 0.45 and 0.50
- 4 cementitious materials systems –Type I OPC, 20% Class F fly ash, 20% Class C fly ash and 40% slag cement

The fine aggregate-to-total aggregate ratio was selected as 0.42 based on data from the combined aggregate gradation charts as discussed in the combined aggregate gradation section.

A polycarboxylate based high range water reducing admixture was used in the lean mixtures to increase workability. No air-entraining agent was added to the mixtures.

Mix Proportions

The mix proportions for the 64 mixtures are given in Table 2. These numbers represent the equivalent values of the mix proportions that were originally designed in English units and converted to the SI units.

Specimens and Testing

For each mixture, fifteen 100x200-mm. concrete cylinders were cast in accordance with ASTM C31 and stored in the fog room until testing in accordance with ASTM C192. The tests conducted are given in Table 3.

Although slump, setting time and air content tests were not control parameters, they were measured to evaluate the effect of the variables on concrete behavior (Table 4).

When the slump was more than 200-mm, the slump flow was determined. At early ages, RCP samples tended to boil, therefore tests were only conducted at 28 and 90 days. The air permeability test was also conducted on 2 specimens per mixture at 1, 3, 28 and 90 days using the University of Cape Town Air Permeability Method (Alexander et al. 1999).

Results and Discussion

Much of the data is presented in figures in which the horizontal axis is the volume of paste divided by the volume of voids in the aggregate system. This is because the properties of the mixture are governed by the paste volume and the paste quality. If there is insufficient paste to fill all of the voids between the aggregate particles, then performance is likely to be compromised. Once adequate paste is obtained to fill the voids and separate the aggregate particles, then the quality of paste (not the quantity) dominates the trends, as predicted by the excess paste theory (Kennedy 1940). The aim of this study was to investigate where this transition occurs.

Workability

The effect of the paste-to-voids volume ratio on workability is presented in Fig. 2. It can be seen that a minimum paste content is needed to make a workable mixture.

For the plain mixtures, a minimum of 1.5 times more paste is required than the voids between the aggregate particles to achieve a workable mix. Below this number, even a high dosage of HRWR did not contribute to improved workability. In fact, those mixtures containing insufficient paste content exhibited honeycombing and were difficult to mix, consolidate and finish. A slump of 50-mm was achieved in mixtures containing lower paste contents compared to the plain mixtures (Fig. 2). The increased workability with the addition of SCM is consistent with the literature (Johari et al. 2011; Johansen et al. 2006).

For mixtures containing SCM, the minimum paste volume should be approximately 1.25 times more than the voids volume between the aggregates. This result is not surprising, because the spherical morphology of the fly ash particles would be expected to reduce the inter-particle friction in the mixtures.

As shown in Fig. 3 (left cylinder), mixtures with 240 kg/m^3 of cementitious content at w/cm of 0.35 did not have adequate paste content to fill the voids between the aggregates. These mixtures were harsh and not workable, and could not be well-consolidated. The lack of consolidation, in turn, affected the hardened properties. The workability of these mixtures could not be improved with the use of water reducing admixtures. These findings were expected, but the mixtures were deliberately made to verify the assumption.

Compressive Strength

The results are presented in Fig. 4. It can be seen that strength increased with increasing paste content, up to a limit. Once a certain paste content was reached; strength became independent of paste content.

The data in Fig. 5 also supports the results discussed above as increasing paste content increased strength up to a point, after which strength was not improved by further increasing the paste content. After a maximum strength was achieved, increasing paste content slightly decreased the compressive strength likely due to not all of the cementitious materials participating in the pozzolanic reaction (Liu et al. 2012).

As shown in Fig.5 there appears to be a trend that above a given amount, increasing cementitious content does not lead to marked increases in 28-day strength. For OPC and class C fly ash this amount is about 300 kg/m^3 while for class F fly ash and slag cement this amount is about 350 kg/m^3 . It is likely that the differences in performance between these systems at this age are due to the differences in hydration rates of the binders (Johari et al. 2011; Johansen et al. 2006).

The plots also indicate that increasing w/cm decreases strength for all mixtures (Wassermann et al. 2009; Mindess et al. 2003; Popovics 1990; Abrams 1920). For a given cementitious content, other than the mixtures with 300 kg/m^3 of cementitious material, increasing w/cm decreased the compressive strength. However, it should be noted that the strength and w/cm relationship applies only to workable mixes (Kennedy 1940).

Fig. 6 shows that increasing paste does not improve strength above a certain value. For the aggregates used in this study, the critical values of $V_{\text{paste}}/V_{\text{voids}}$ are about 150% for mixtures containing OPC and C fly ash and about 175% for slag cement and F fly ash mixtures.

Fig. 7 shows the effect of minimum paste volume on cement efficiency. The cement efficiency was calculated by calculating the strength per unit mass of cement in a mixture. The trends presented in Fig. 7 support the findings discussed above that after reaching the paste volume that is required for the mix to reach a plateau, increasing paste volume does not provide any benefits in terms of compressive strength. The peak efficiencies are higher for the mixtures containing OPC and C fly ash, and lower for the mixes with F fly ash and slag cement. As stated before, this efficiency difference is most likely related to the chemistry and fineness and their effects on hydration rates. It is also notable that the effect of w/cm on efficiency is relatively small. Decreasing efficiency with increasing paste content indicates that the cost and sustainability of mixtures with high paste content is not optimized.

Rapid Chloride Penetration

RCP test was conducted at 28 and 90-days because the low cementitious content samples exhibited such high conductivity that no data could be obtained at earlier ages. This is likely because of the pore structure (Bagheri and Zanganeh 2012) caused by the inadequate paste content that could not fill the voids between the aggregate particles (Fig. 8).

The effect of paste volume on rapid chloride penetration (RCP) is presented in Fig. 9. Hydration at later ages helped to fill some of the capillary voids and reduce penetrability. Increasing the paste volume increased the chloride penetrability which is consistent with the literature (Arachchige 2008). This can be explained by the differences between aggregate and paste. In general, aggregates are likely to be denser than cement paste (especially at early ages) and have a lower permeability than cement paste, so concretes with low paste content tend to have lower permeability, despite the introduction of the more porous interfacial transition zones (Scrivener and Nematı 1996).

The data presented in Fig. 10 supports the findings discussed above: in all cases chloride penetration increased with increasing paste volume. The mixtures containing slag cement and C fly ash exhibited less penetration compared to the plain mixes at 28 days. At 28 days, mixtures with Class F ash exhibited higher penetrability than plain concrete, likely due to its initially slower hydration rate. However, at 90 days, plain concrete showed higher penetrability than mixtures with Class F fly. This result is not surprising because increasing the testing age of the mixes incorporating SCM reduces the porosity of the concretes as a result of the continued pozzolanic reaction (Bagheri and Zanganeh 2012; Liu et al. 2012). The reduction in penetration of concrete containing SCM may also be due to their contribution to improve the interfacial transition zone (ITZ) between the cement paste and aggregates (Toutanji 2004). Decreasing w/cm also decreased penetrability, most notably in the plain cement mixtures.

Air Permeability

Air permeability index is the negative log of the Darcy coefficient of permeability (m/s) and uses a log scale (Buenfeld and Okundi 2000). Therefore, higher air permeability index (API) indicates higher impermeability. As reported by Alexander and Beushausen (2010) the following interpretation can be applied to the results:

- API > 10.0 - Excellent
- $9.5 < \text{API} < 10.0$ – Good
- $9.0 < \text{API} < 9.5$ – Poor and
- $\text{API} < 9.0$ – Very poor

Similar to the RCP tests, data could not be obtained from concrete mixtures with 300 kg/m^3 of binder content due to the high porosity caused by the inadequate paste content.

In Fig. 11, increasing paste volume above certain value decreased permeability. When $V_{\text{paste}}/V_{\text{void}}$ was increased from 100 to 150%, air permeability decreased likely because mixtures with low cementitious content had macro porosity. This result is consistent with the findings in the literature that compaction plays a more critical role than concrete microstructure on air permeability (Buenfeld and Okundi 2000). Once the required paste content was provided, increasing paste content increased permeability because air tends to penetrate through the relatively porous paste faster through aggregates. Above a given $V_{\text{paste}}/V_{\text{void}}$ value all of the samples may be classified as “excellent” at 90 days.

However, 90-day data is better than those in the 28 day set. Air permeability decreases as concrete age increases because cement hydration continues over time and the pore sizes get smaller, thus concrete becomes less permeable. The observed results are consistent with the findings reported in the literature (Alexander et al. 2007; Dinku and Reinhardt 1997).

In Fig. 12, there is some benefit with decreasing w/cm, while the type of binder does not appear to have a significant effect at 28-days.

Minimum Paste Volume Requirement

A paste thickness index (PTI) was calculated. The specific surface area of the aggregate system was calculated for the combined gradation assuming spherical particles. This with the mass of aggregates gave a total surface area of the aggregates in each mix, which divided by the volume of paste yielded an average paste thickness. Calculations were based on the assumption that all aggregate particles in the selected combined aggregate system were spherical and that all were coated by a layer of constant paste thickness. Neither of these assumptions is correct, but the approach provides a reasonable point of comparison. It was found that there was a linear relationship between the PTI and the $V_{\text{paste}}/V_{\text{void}}$ ratio; therefore there were no changes to the conclusions.

The PTI values ranged from 100 to 250 μm . This is of interest because the interfacial transition zone (ITZ) is typically reported to be from 40 to 50 thick μm (Prokopski and Halbiniak 2000) which is a significant fraction of the nominal paste

thickness. If about $\frac{1}{3}$ to $\frac{1}{2}$ of the paste is functionally ITZ, then approaches that modify the quality of the ITZ will have a marked effect on system performance.

In general, two different trends were observed. In all cases, very low paste contents led to poor performance of the mixture. Above a certain threshold, performance was either constant or deteriorated slightly. Deterioration in performance was generally observed in permeability tests with increasing paste, which is to be expected because permeability of paste is likely to be lower than that of aggregate (Scrivener and Nematî 1996). Other parameters such as strength were generally constant with increasing paste.

The threshold varied between tests and between cementitious systems (Table 5). Values are not provided for RCP because it was found that the ability to consolidate the samples was dominant, and as soon as sufficient paste was provided to achieve this, performance fell off with increasing paste content.

In broad terms then, about 1.5 to 2 times more paste by volume is required than the space between the combined aggregate particles. This is to fill all the space and to line all of the aggregate particles, providing lubrication in mixture in its fresh state when it is being transported, and to glue the particles together in the hardened state. This is applicable for the single aggregate system tested here. It is possible that these numbers may change for different aggregate forms (river gravel as opposed to crushed limestone) and gradations.

Conclusions

The following conclusions, which are limited to the materials used in this study, can be drawn:

1. Approximately 1.5 times more paste by volume is required than voids between the aggregates to obtain a minimum workability. Below this threshold value, the use of water-reducing admixtures provides little benefit.
2. For a given w/cm, increasing cementitious content does not significantly improve compressive strength once the critical minimum is reached. The critical value is about twice the voids content of the aggregate system.
3. For a given w/cm, increasing paste content increases chloride penetrability and air permeability.
4. The results suggest the addition of SCM would help improve workability, long-term strength, and durability in portland cement concrete (PCC) pavements.

These results suggest that the strength and durability of concrete could be improved by optimizing the mixture proportions. However, further research is needed to investigate the different aggregate gradation systems on concrete performance.

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Table 1. Chemical composition of cementitious materials, percentage by mass

Chemical composition	OPC	F ash	C ash	Slag
Silicon dioxide (SiO ₂)	20.22	49.71	36.71	37.20
Aluminum oxide (Al ₂ O ₃)	4.43	15.29	19.42	9.48
Ferric oxide (Fe ₂ O ₃)	3.19	7.16	6.03	0.47
Calcium oxide (CaO)	62.71	15.66	25.15	40.10
Magnesium oxide (MgO)	3.51	5.29	4.77	10.99
Sulfur trioxide (SO ₃)	3.24	0.87	1.97	1.11
Potassium oxide (K ₂ O)	0.69	2.17	0.46	0.41
Sodium oxide (Na ₂ O)	0.08	1.73	1.64	0.26
Equivalent alkalis (NaEq)	0.54	3.16	1.94	0.53

Table 2. Mix proportions

No	OPC (kg/m ³)	F ash (kg/m ³)	C ash (kg/m ³)	Slag (kg/m ³)	Binder (kg/m ³)	Water (kg/m ³)	HRWR (ml/100 kg)	w/cm	Sand (kg/m ³)	Stone (kg/m ³)	Vpaste/ Vvoid
1	237				237	83	2258	0.35	911	1258	110
2	190	47			237	83	2258	0.35	907	1258	112
3	190		47		237	83	2258	0.35	908	1254	111
4	142			95	237	83	2258	0.35	908	1254	112
5	297				297	104	1519	0.35	867	1197	141
6	237	60			297	104	1519	0.35	861	1190	145
7	237		60		297	104	1519	0.35	864	1192	143
8	178			119	297	104	1355	0.35	863	1192	144
9	356				356	125	889	0.35	823	1136	176
10	285	71			356	125	889	0.35	817	1128	181
11	285		71		356	125	889	0.35	819	1131	179
12	214			142	356	125	889	0.35	818	1130	179
13	415				415	145	527	0.35	779	1076	213
14	332	83			415	145	527	0.35	772	1066	220
15	332		83		415	145	527	0.35	775	1070	217
16	249			166	415	145	352	0.35	774	1068	218
17	237				237	95	1437	0.4	898	1239	119
18	190	47			237	95	1539	0.4	893	1233	122
19	190		47		237	95	1539	0.4	895	1236	120
20	142			95	237	95	1283	0.4	895	1235	121
21	297				297	119	1314	0.4	850	1174	154
22	237	60			297	119	1232	0.4	845	1167	158
23	237		60		297	119	1232	0.4	847	1170	156

Table 2. Mix proportions (cont.)

No	OPC (kg/m ³)	F ash (kg/m ³)	C ash (kg/m ³)	Slag (kg/m ³)	Binder (kg/m ³)	Water (kg/m ³)	HRWR (ml/100 kg)	w/cm	Sand (kg/m ³)	Stone (kg/m ³)	Vpaste/ Vvoid
24	178			119	297	119	1232	0.4	847	1169	157
25	356				356	142	376	0.4	803	1109	192
26	285	71			356	142	376	0.4	797	1101	197
27	285		71		356	142	0	0.4	800	1104	195
28	214			142	356	142	376	0.4	799	1103	195
29	415				415	166	235	0.4	756	1044	235
30	332	83			415	166	235	0.4	749	1034	242
31	332		83		415	166	59	0.4	752	1038	240
32	249			166	415	166	235	0.4	751	1037	240
33	237				237	107	667	0.45	884	1221	128
34	190	47			237	107	667	0.45	880	1216	131
35	190		47		237	107	718	0.45	882	1218	130
36	142			95	237	107	667	0.45	882	1217	130
37	297				297	133	452	0.45	834	1152	166
38	237	60			297	133	493	0.45	829	1144	170
39	237		60		297	133	493	0.45	831	1147	168
40	178			119	297	133	493	0.45	830	1146	169
41	356				356	160	0	0.45	783	1082	209
42	285	71			356	160	0	0.45	777	1073	215
43	285		71		356	160	0	0.45	780	1077	213
44	214			142	356	160	0	0.45	779	1076	213
45	415				415	187	0	0.45	733	1012	259
46	332	83			415	187	0	0.45	726	1002	266

Table 2. Mix proportions (cont.)

No	OPC (kg/m ³)	F ash (kg/m ³)	C ash (kg/m ³)	Slag (kg/m ³)	Binder (kg/m ³)	Water (kg/m ³)	HRWR (ml/100 kg)	w/cm	Sand (kg/m ³)	Stone (kg/m ³)	Vpaste/ Vvoid
47	332		83		415	187	0	0.45	729	1006	263
48	249			166	415	187	0	0.45	728	1005	264
49	237				237	119	718	0.5	872	1203	138
50	190	47			237	119	718	0.5	867	1197	141
51	190		47		237	119	718	0.5	869	1200	140
52	142			95	237	119	718	0.5	868	1199	140
53	297				297	148	329	0.5	817	1128	180
54	237	60			297	148	329	0.5	812	1122	184
55	237		60		297	148	329	0.5	814	1124	182
56	178			119	297	148	329	0.5	814	1124	183
57	356				356	178	0	0.5	764	1054	228
58	285	71			356	178	0	0.5	758	1046	234
59	285		71		356	178	0	0.5	760	1050	232
60	214			142	356	178	0	0.5	759	1048	232
61	415				415	208	0	0.5	710	980	284
62	332	83			415	208	0	0.5	702	970	292
63	332		83		415	208	0	0.5	705	973	289
64	249			166	415	208	0	0.5	705	973	290

Table 3. Test matrix

Concrete properties	Method	No of specimens	Age (days)
Slump/Slump flow	ASTM C143/ASTM C1611	1	-
Air content	ASTM C231	1	-
Setting time	ASTM C403	1	-
Compressive strength	ASTM C39	2 per age	1, 3, 28, 90
Rapid chloride penetration (RCP)	ASTM C1202	2 per age	28, 90
Air permeability	University of Cape Town Method	2 per age	1, 3, 28, 90

Table 4. Fresh concrete properties

No	Slump	Slump	Air	Setting (min)	
	(mm)	flow (mm)	(%)	Initial	Final
1	0		2.8	290	415
2	0		4.5	330	500
3	5		3.5	395	620
4	0		2.2	N/A	N/A
5	0		1.5	200	300
6	50		4	340	530
7	40		3	395	510
8	5		2.3	355	505
9	50		1.8	205	265
10	70		3.3	265	370
11	145		3.3	340	450
12	75		3.2	265	370
13	40		1.8	160	240
14	75		1.5	205	290
15	110		2.2	260	340
16	65		2.2	230	335
17	0		3.5	210	340
18	0		2.8	330	590
19	40		4.8	265	365
20	50		2.8	350	530
21	90		3.5	290	375
22	75		2.3	235	330
23	85		4.5	325	420
24	120		3.5	260	440
25	65		2	200	285
26	75		2.5	330	465
27	50		2.5	255	350
28	75		2.3	225	415
29	115		2.2	205	275
30	230		1	260	370
31	105		1.5	290	370
32	65		2.3	240	365

Table 4. Fresh concrete properties (cont.)

No	Slump	Slump	Air	Setting (min)	
	(mm)	flow (mm)	(%)	Initial	Final
33	0		2.6	175	265
34	0		2	310	495
35	0		3.3	300	460
36	5		3	225	410
37	25		2.8	190	275
38	65		3	290	440
39	75		2.8	360	475
40	20		2.8	260	390
41	100		3.3	195	260
42	215		1.5	225	375
43	185		2.3	270	400
44	75		2.2	260	375
45	150		3.5	215	280
46		565	0.5	290	370
47		535	1.5	340	505
48	95		2	290	395
49	0		3.5	225	340
50	205		3.5	355	490
51	40		2.5	320	425
52	125		3	340	585
53	75		3	215	305
54	190		2.8	335	450
55	90		2.3	320	410
56	75		3.3	265	465
57	230		0.8	230	295
58	265		1.8	320	450
59	0		0.8	275	365
60	255		1.3	335	455
61		510	0.5	250	340
62		545	1.3	350	455
63		565	0.5	315	430
64	280		1	370	530

**Table 5. Critical minimum paste required for performance and expresses as
Vpaste/Vvoid percent**

Property	OPC	F ash	C ash	Slag
Workability	150	150	125	125
Compressive strength	150	175	150	175
Air permeability	175	175	125	200

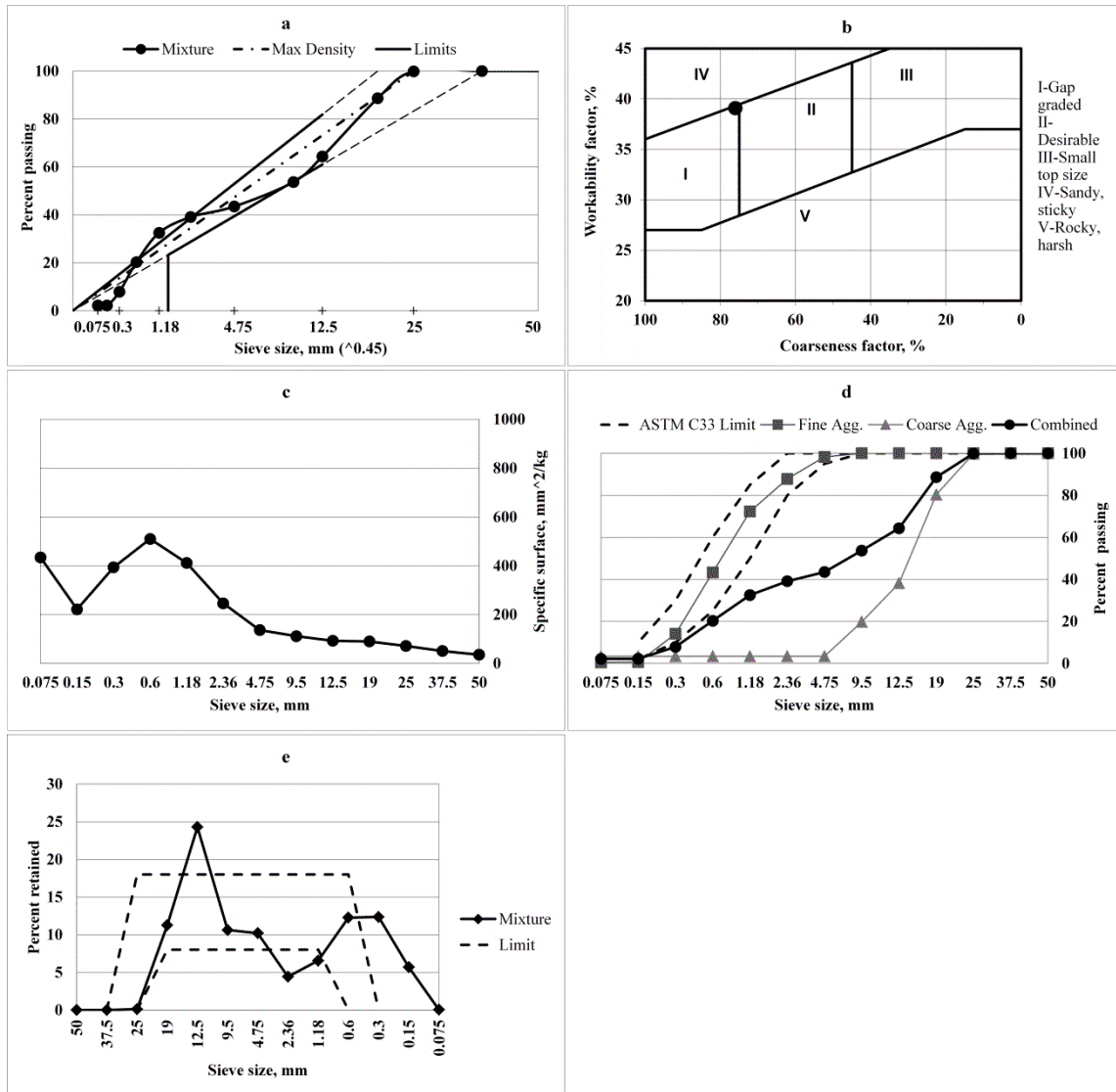


Fig. 1. Combined aggregate gradation curves.

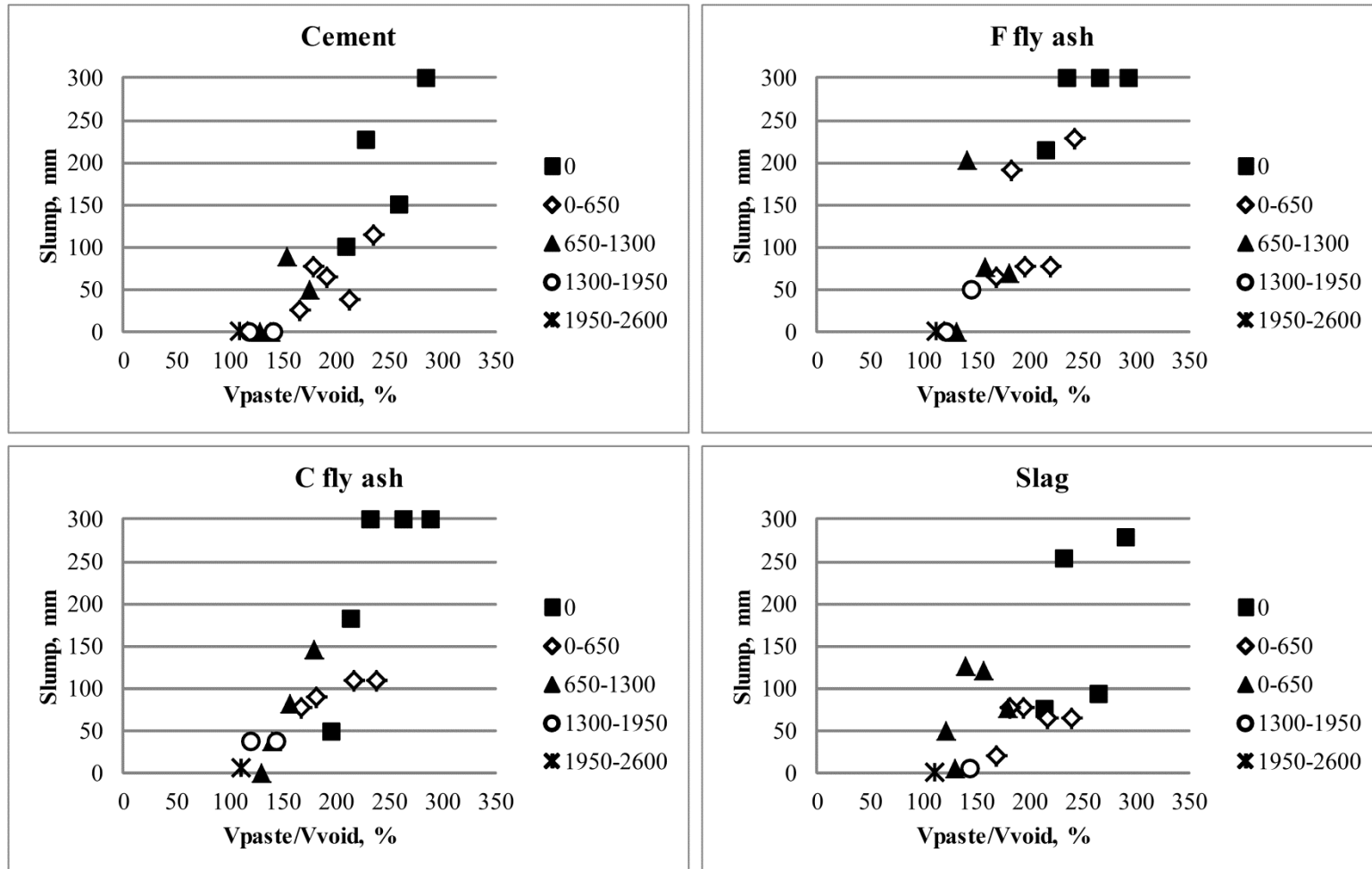


Fig. 2. Plots of slump vs. paste-to-voids volume ratio. The symbols reflect the water reducing admixture dosage in ml/100 kg of cementitious materials. (Mixtures in which slump flow were recorded are shown here as 300-mm slump).

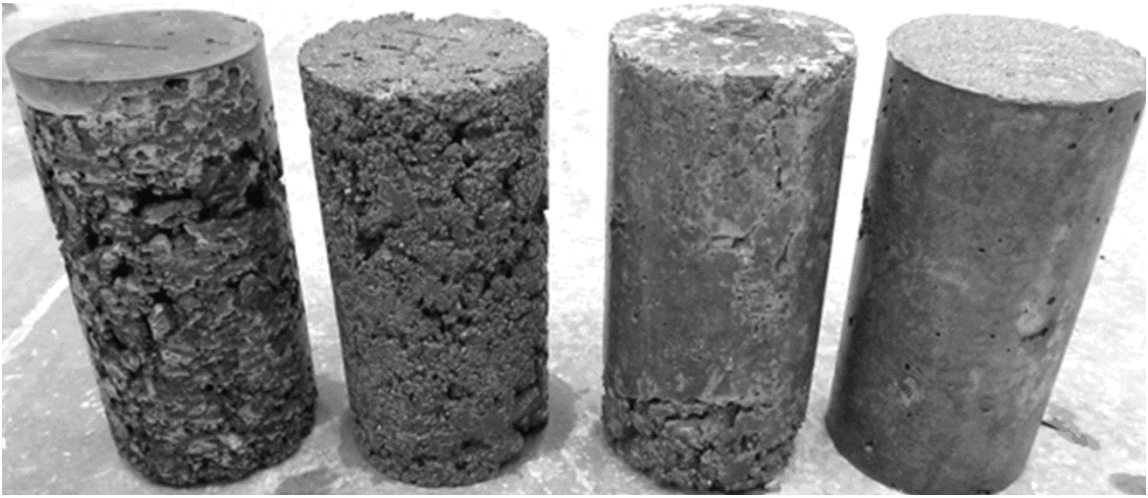


Fig. 3. Concrete mixes with 240 kg/m³ of OPC (from left to right, w/c of 0.35, 0.40, 0.45, and 0.50).

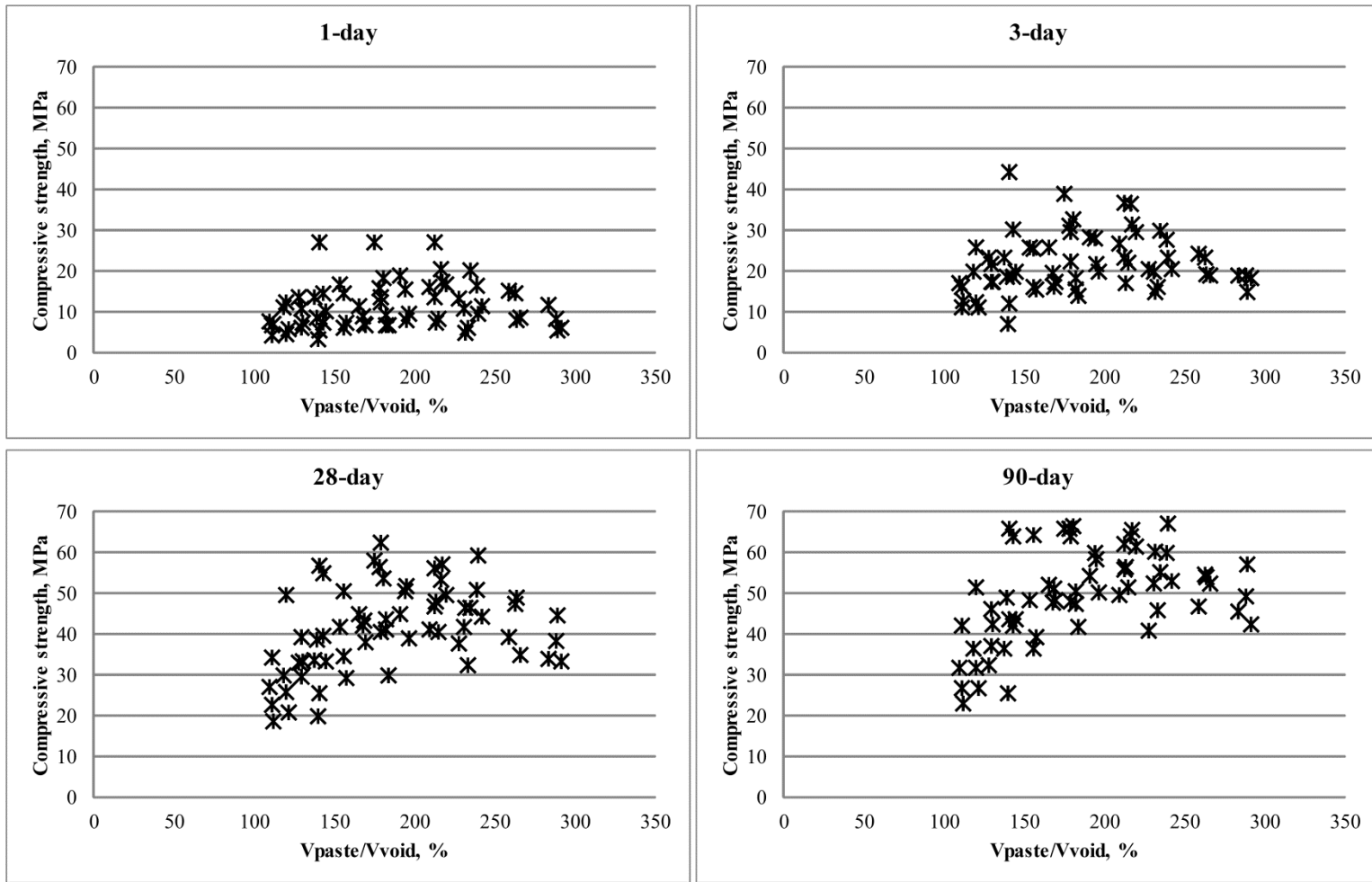


Fig. 4. Overview of the effect of Vpaste/Vvoid on strength for all mixtures at various ages.

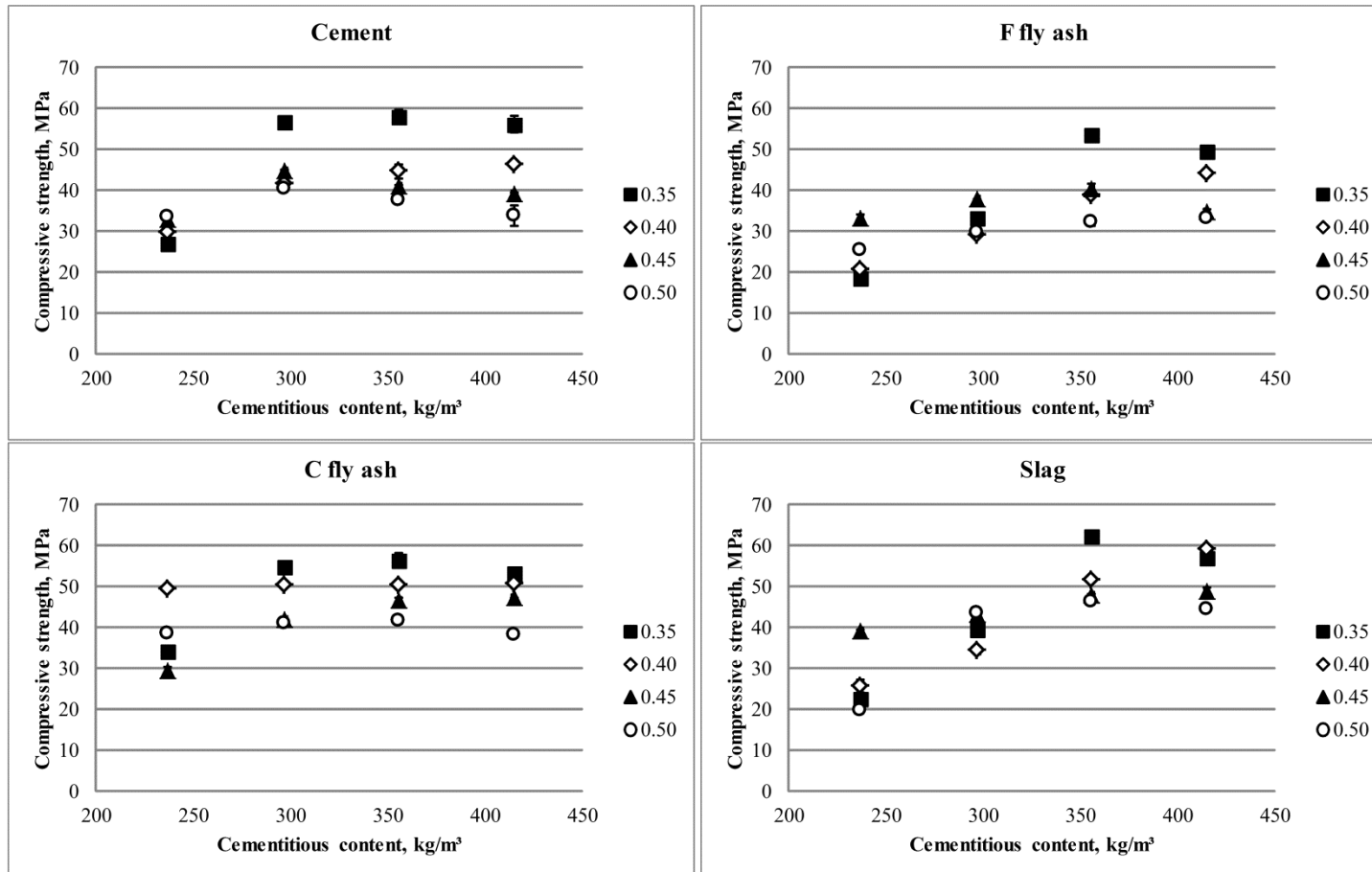


Fig. 5. Compressive strengths for different binder systems at 28 days as a function of cementitious content. Symbols reflect the w/cm of each mixture. The acceptable range of the strengths of two cylinders (4x8-in.) is 9 % (ASTM C39, 2005).

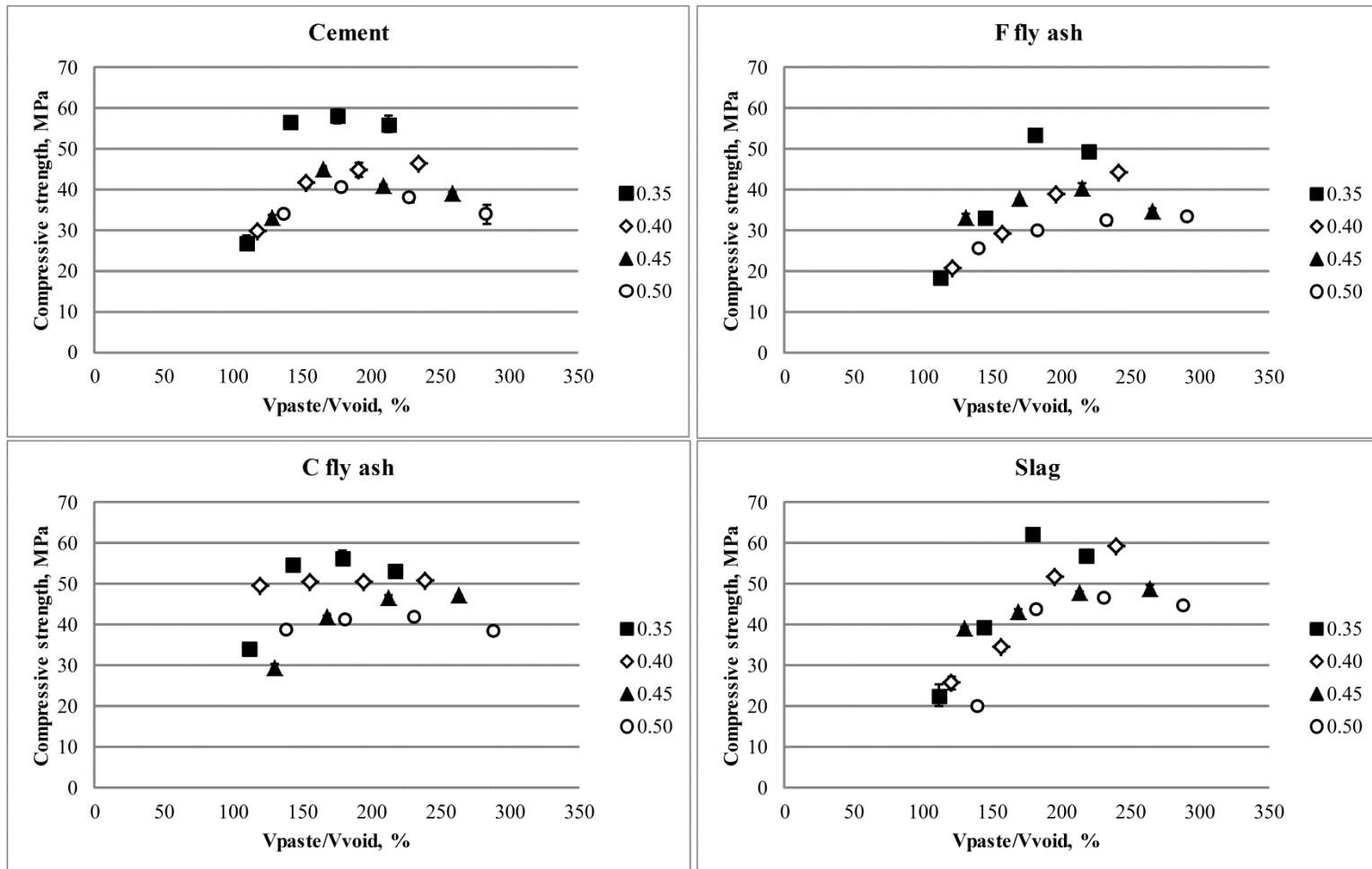


Fig. 6. Compressive strengths for different binder systems at 28 days as a function of Vpaste/Vvoid. Symbols reflect the w/cm of each mixture. The acceptable range of the strengths of two cylinders (4x8-in.) is 9 % (ASTM C39, 2005).

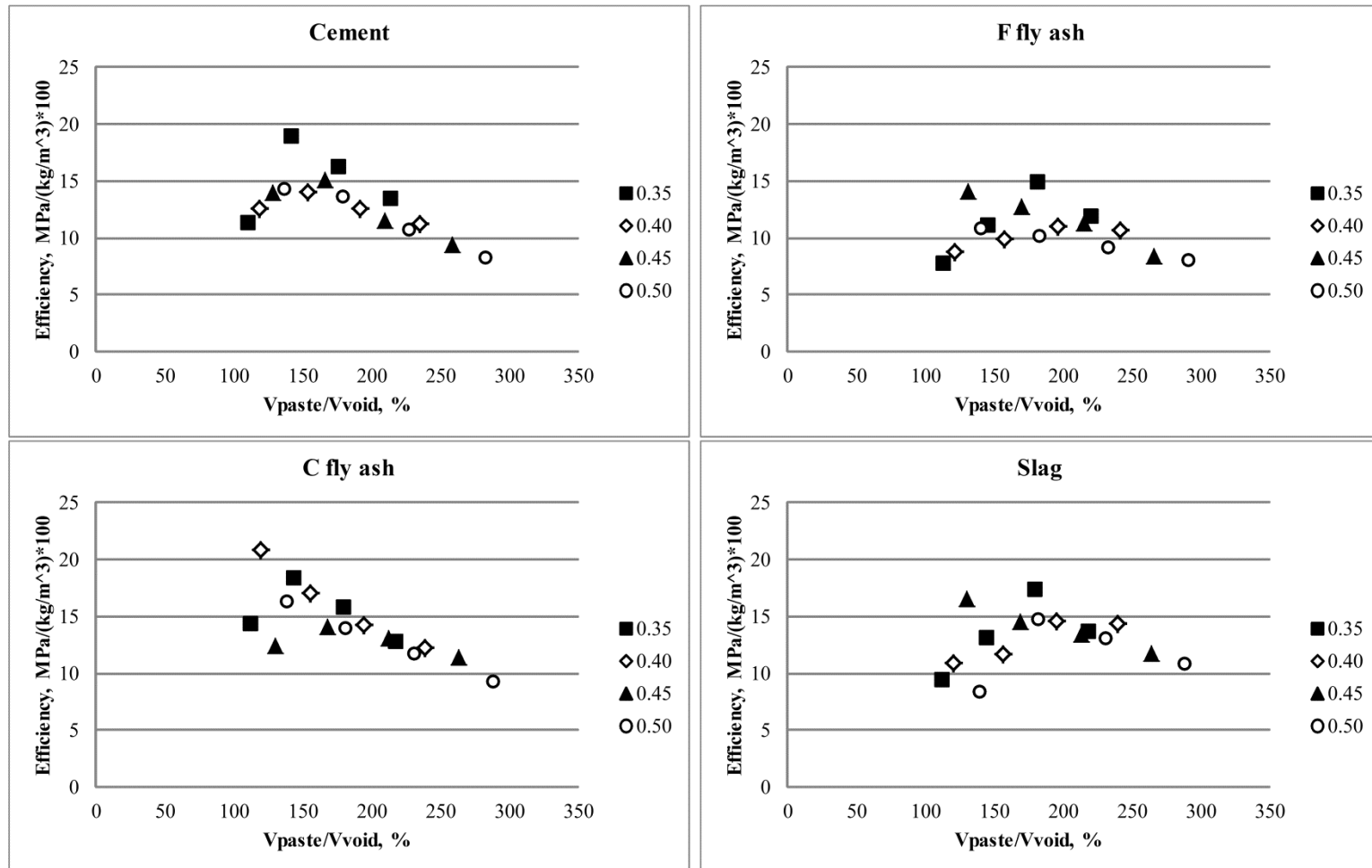


Fig. 7. Cementing efficiency of different binder systems at 28 days as a function of Vpaste/Vvoid. Symbols reflect the w/cm of each mixture.

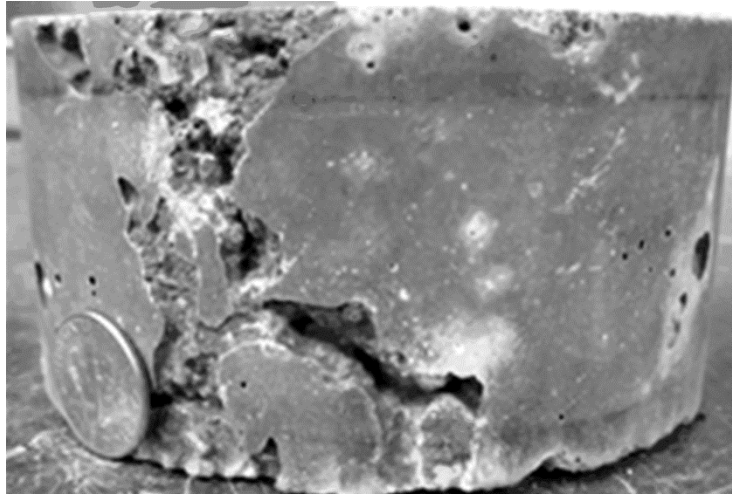


Fig. 8. Porosity of mix with 300 kg/m³ of cementitious content and 0.35 of w/cm.

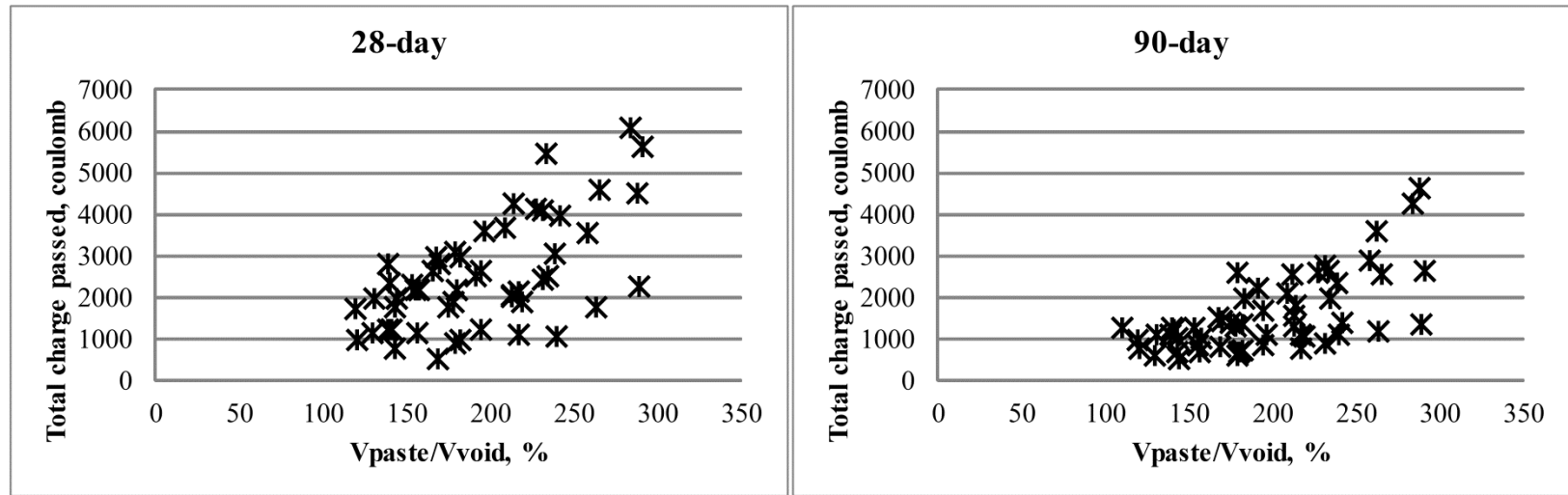


Fig. 9. Overview of the effect of V_{paste}/V_{void} on penetrability for mixtures at 28 and 90 days.

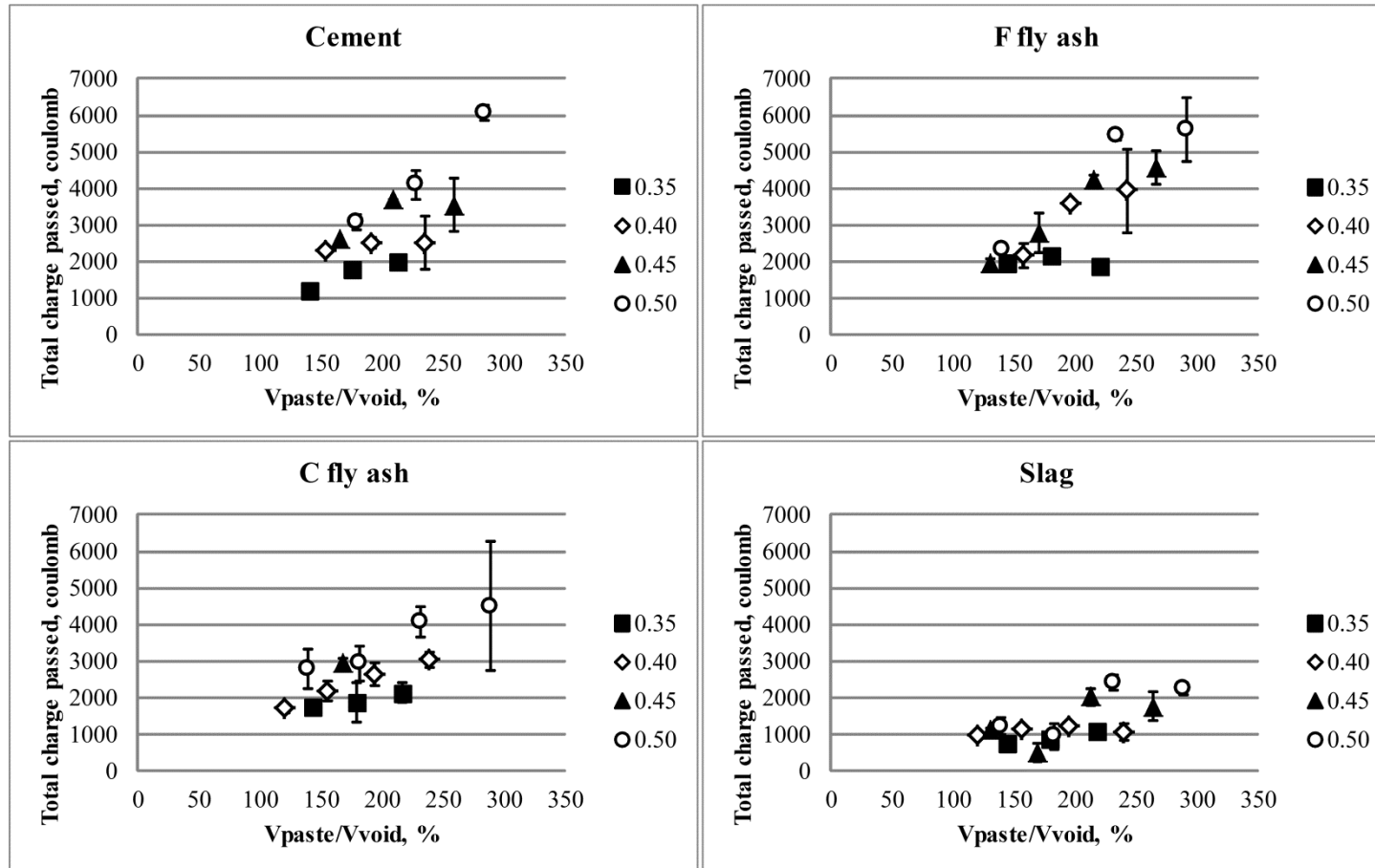


Fig. 10. Penetrability data for different binder systems at 28 days as a function of V_{paste}/V_{void} . Symbols reflect the w/cm of each mixture. The test results of different samples from the same batch should not differ by more than 42 %, as suggested by the ASTM C1202, 1997.

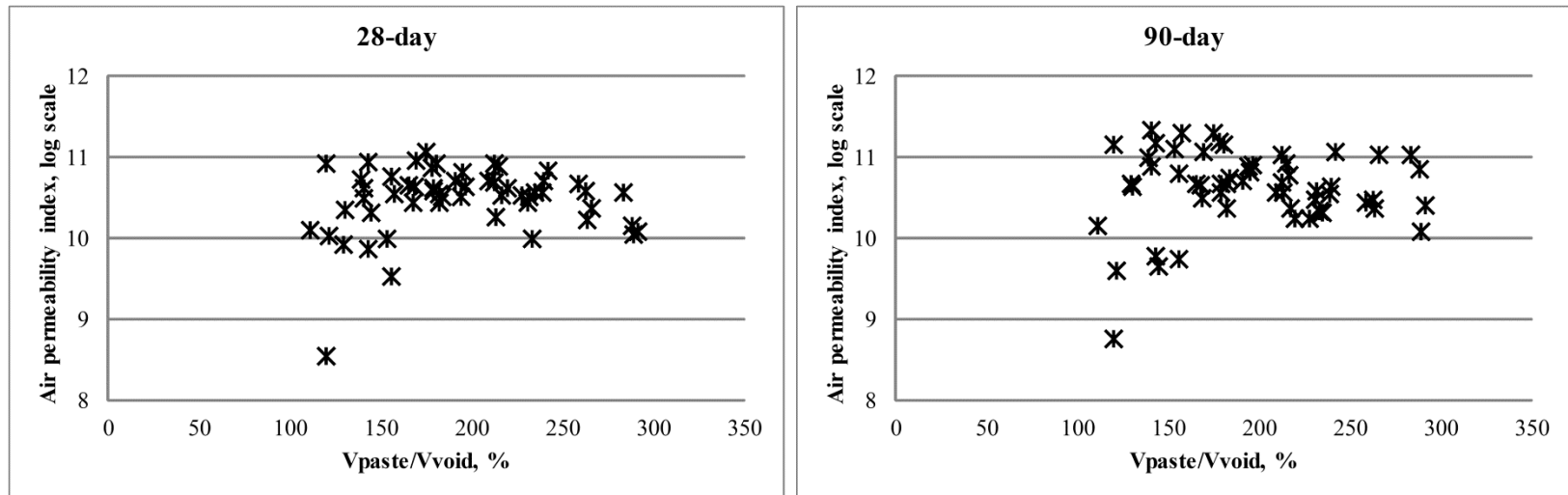


Fig. 11. Overview of the effect of Vpaste/Vvoid on API for mixtures at 28 and 90 days.

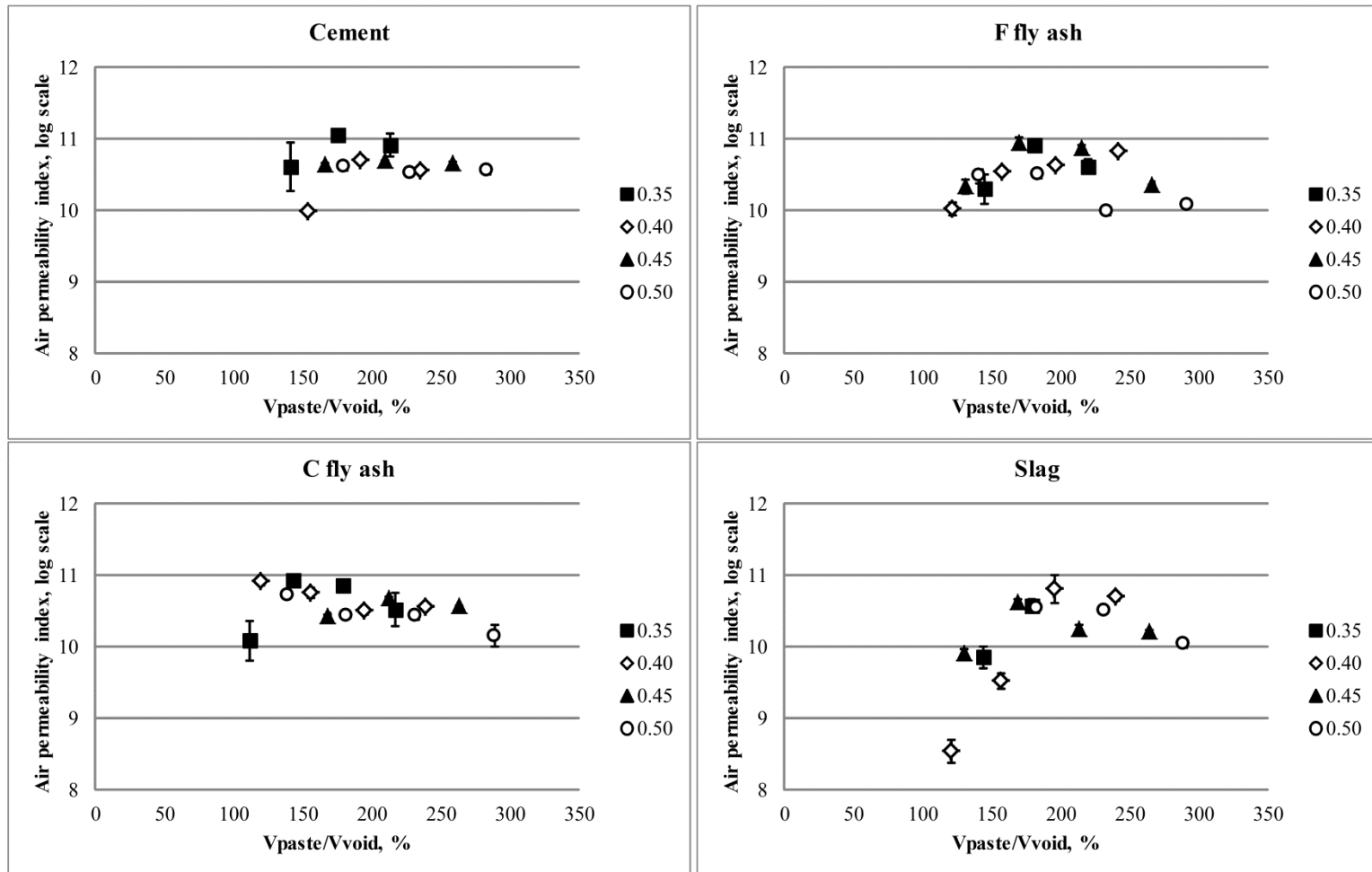


Fig. 12. API data for different binder systems at 28 days as a function of Vpaste/Vvoid. Symbols reflect the w/cm of each mixture. The precision of this test method is 1.8% (Stanish et al. 2006).

CHAPTER 3. EFFECT OF WATER-TO-BINDER RATIO, AIR CONTENT, AND TYPE OF CEMENTITIOUS MATERIALS ON FRESH AND HARDENED PROPERTIES OF BINARY AND TERNARY BLENDED CONCRETE

A paper submitted to Journal of Materials in Civil Engineering (ASCE)

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Abstract

The purpose of this study is to investigate the effect of water-to-binder ratio (w/b), air content, and type of cementitious materials on fresh and hardened properties of binary and ternary blended concrete mixtures in pavements. In this experimental program, a total matrix of 54 mixtures with w/b of 0.40 and 0.45; nominal air content of 2%, 4% and 8%; and three types of supplementary cementitious materials (SCMs) and one ordinary portland cement at different combinations was prepared. Binder systems included ordinary portland cement, binary mixtures with slag cement, Class F and C fly ash, and ternary mixtures containing a combination of slag cement and one type of fly ash.

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Workability, total air content, air-void system parameters (i.e., spacing factor and specific surface) in fresh concrete, setting time, compressive strength, surface resistivity, and shrinkage were determined. Test results showed that ternary mixtures followed the trends of their constituent materials. Binary and ternary mixtures containing Class C fly ash and slag cement exhibited higher compressive strength than the control mixture. The surface resistivity and shrinkage results of binary and ternary mixtures were equal or improved compared to the control mixture.

CE Database subject headings: Construction materials; Concrete; Portland cement; Slag; Fly ash; Durability; Compressive strength.

Introduction

Cement is the most expensive component in concrete. The production of cement emits approximately 5% of global carbon dioxide (CO₂) and consumes 5% of global energy (Hendriks et al. 2004). Therefore, using cement more efficiently will be beneficial in improving sustainability. Cement content can be reduced by replacing a portion of it with supplementary cementitious materials (SCMs), which are generally less expensive and more environmentally-friendly (Rupnow 2012, Udoeyo et al. 2012, Hariharan et al. 2011). In addition to these benefits, SCMs generally: 1) improve the workability of concrete and decrease the tendency to bleed and segregate by enhancing the packing density due to their lower specific gravity; 2) reduce pore size and the porosity of both cement matrix and the interfacial transition zone (ITZ), thereby increasing performance; and 3) increase ultimate durability in terms of decreasing permeability and increasing resistance to thermal cracking and alkali-aggregate

expansion as a result of the pozzolanic reaction (Liu et al. 2012, Bagheri and Zanganeh 2012, Megat Johari et al. 2011).

On the other hand, incorporating a single type of SCM (binary mixtures) may have negative side-effects such as extended setting time (Bleszynski et al. 2002, Khan et al. 2000). In such cases, a possible solution is to use a ternary mixture which is a combination of three cementitious materials that are blended to balance fresh properties, durability, strength, and economy (Schlorholtz 2004).

In addition to SCM, air content and water-to-binder ratio (w/b) also affect concrete properties. For example, as w/b decreases, the porosity of the paste decreases, and concrete becomes less permeable thereby resulting in increased strength and enhanced durability (Wassermann et al. 2009, Dhir et al. 2004). Therefore, ACI 302 (2004) recommends a w/b of no greater than 0.50 for concrete floors and slabs subjected to moderate and severe exposures to freezing and thawing (F-T), and a w/b of no greater than 0.45 for concrete subjected to deicing chemicals.

Having a good air-void system increases durability when concrete is subjected to the F-T conditions; and improves the workability and consistency of concrete mixtures by increasing the paste volume, for a given w/b (Kosmatka et al. 2008, Taylor et al. 2006). However, depending on its form, air (particularly the large voids) can adversely affect durability by increasing permeability (Kropp and Hilsdorf 1995); and reducing strength due to the increased porosity. A common rule of thumb is that for each 1% of air by volume, strength decreases approximately by 5% (Soutsos 2010). However, in

mixtures with low w/b and cement content, entrained air is believed to improve the strength by improving the ITZ (Mehta and Monteiro 2006).

Despite the published studies and increased interest in ternary blended concretes, limited information is available about the optimum balance of SCMs along with their interactivity between w/b and air content. The purpose of this study is to develop data to help quantify how changes in the air content, w/b, and type and replacement level of SCMs influence the properties of binary and ternary concrete mixtures.

Engineers and contractors are cautious about using ternary blended concrete mixtures due to the practical limitations and possible side effects such as low-early strength, increased plastic shrinkage cracking, and extended curing time. This paper aims to address those assumptions by providing comparative information regarding the effects of mix characteristics on fresh and hardened properties of plain concrete, binary, and ternary blended concrete mixtures used for pavements.

Material and Methods

Cementitious Materials

A single batch was obtained of each of ASTM C150 (2002) Type I ordinary portland cement, ASTM C618 (2003) Class F fly ash, ASTM C618 (2003) Class C fly ash and ASTM C989 (1999) Grade 120 slag cement. The chemical composition of the cementitious materials is presented in Table 1.

Aggregates

No.4 (4.75-mm) nominal maximum size river sand and 25.4-mm nominal maximum size crushed limestone were used in the study.

The void percentage of the combined aggregates was kept constant at 19.8% for all the mixtures to remove the aggregate grading as a variable from the experimental matrix. This was selected by assessing the FHWA 0.45 power curve (Bureau of Public Roads 1962), Shilstone workability factor chart and specific surface chart (Yurdakul 2010).

The fine aggregate-to-total aggregate ratios of 0.45, 0.42 and 0.39 provided the best mixtures in FHWA 0.45 power curve, Shilstone workability factor chart and specific surface charts, respectively (Fig. 1a-c). Based on the average of these numbers, the fine aggregate-to-total aggregate ratio was selected as 0.42. The appropriateness of the selected ratio of 0.42 was checked by plotting the data in an ASTM C33 (2003) plot (Fig. 1d) and a “Haystack” plot (Fig. 1e). The Haystack plot did not present an ideal combination, but was the best combination that could be achieved with the materials available. While not ideal, this type of gradation is common in many construction sites, and is therefore an appropriate combination for this research study.

The unit weight and volume of voids in the combined aggregate were measured in accordance with ASTM C29 (2003). The overall unit weight of the combined aggregates was 2098 kg/m³. The specific gravity and absorption of the coarse and fine aggregates were determined using ASTM C127 (2001) and ASTM C128 (2001), respectively. The saturated surface dry (SSD) specific gravity and the absorption values of the coarse aggregates were 2.67 and 0.93%, respectively. The SSD specific gravity and the absorption values of the fine aggregates were 2.62 and 1.07%, respectively.

Mix Design

In this experimental program, a total matrix of 54 mixtures was prepared. A fixed cementitious content of 356 kg/m³ was used for all mixtures. The fine aggregate-to-total aggregate ratio was selected as 0.42 based on data from the combined aggregate gradation charts as discussed in the aggregate section. The binder combination, air content, and w/b were selected as variables and are presented in Table 2.

ASTM C494 (2010) Type F polycarboxylate based high-range water-reducing admixture was used in the drier mixtures to improve workability. To achieve the target air content, a tall-oil based synthetic air entraining admixture was used.

Specimens and Testing

For each mixture, nine cylinders (100x200-mm) and three beams (76x76x279-mm) were prepared. Mixtures were prepared in accordance with ASTM C 192 (2002). Specimens were prepared in accordance with ASTM C31 (2003) and stored under plastic sheeting until the samples were demolded after 24 hours, and moist cured in a fog room at 100% relative humidity and 23 °C until tested. The tests conducted are given in Table 3.

Results and Discussion

Workability

Workability was investigated using the slump test. The results are depicted in Figure 2. According to Figure 2, when w/b was kept constant, increasing air content slightly increased workability. The effect of air content was very minor in binary

concretes containing Class C fly ash and slag cement. For a given w/b, increasing the replacement dosage of Class F fly ash increased the workability of binary mixtures. This result is not surprising because Class F fly ash particles would be expected to reduce the inter-particle friction in the mixtures, and improve workability (Kosmatka et al. 2008). The highest slump (280-mm) was obtained by binary mixtures having 15% and 30% of Class F fly ash at 8% nominal air content. In binary mixes, slag concrete had a higher water demand compared to the control concrete. The water demand of the ternary mixtures containing slag cement and Class F fly ash was found to be higher than that of the binary fly ash concrete, and lower than the binary slag concrete. The Class C fly ash mixtures were somewhat improved, while the ternary mixtures followed the trends of their constituent materials. For both binary and ternary mixtures, increasing w/b from 0.40 to 0.45 increased workability, as expected. However, there was little effect with changing SCM type at the lower w/b. This effect of SCMs was more marked in mixtures with higher water contents.

Air-Void System

The air-void analyzer (AVA) test was conducted to determine the air-void system of the fresh concrete mixes. The effect of the nominal air content, w/b and SCM dosage on the air-void system is presented in Figure 3.

The commonly accepted rule of thumb for a good air-void system requires a spacing factor less than 0.2-mm measured using ASTM C457 (1998). Recent studies (Wang et al. 2009, Wang 2008) have shown that concrete with a spacing factor of less than 0.3-mm when measured by AVA would be equivalent and acceptable for F-T

durability. Therefore, the AVA test results were evaluated according to the acceptance criterion of the spacing factor of <0.3 -mm. Based on this criterion, Figure 3 shows that mixes designed to have a 2% of air content do not meet the requirement. In addition, some of the mixes having a nominal air content of 4% had a spacing factor less than 0.3-mm. This is in agreement with the experience that 4% total air traditionally exhibits marginal performance in the field. Furthermore, excluding the control mixture, the mixes having a nominal air content of 8% had a spacing factor less than 0.3-mm, as desired.

Little difference is seen in respect to the effect of w/b on relationship between spacing factor and nominal air content, except perhaps more scatter is seen in the higher w/b system. The effects of the inclusion of SCMs on the air-void system of binary and ternary mixtures could not be established as there was no obvious trend.

Setting Time

The final setting times were recorded and presented in Figure 4. The addition of both Class C and Class F fly ashes increased the setting time compared to the control mixture, likely as a result of their dilution effect due to the partial substitution of cement with a less reactive material (Fajun et al.1985). However, among all the mixtures, binary concretes containing Class C fly ash exhibited the highest retarding effect. The increase in time of mixtures with Class C fly ash is greater than the increases caused by the Class F fly ash likely due to the lower hydration rate of Class C fly ash.

On the other hand, the addition of slag cement resulted in similar setting times to the control mixture. This result is consistent with the literature (Tikalsky et al. 2011). The ternary mixtures also slightly retarded the setting time consistent with the effects of

their ingredients. Increasing the nominal air content and w/b did not play a significant role on affecting the setting time.

Compressive Strength

The 28-day strength data is presented in Figure 5. Increasing the air content decreased the compressive strength, as expected. According to the test results, a strength loss of about 3.5% occurred per unit air increase across the full spectrum of materials tested. This is consistent with the literature (Soutsos 2010).

As expected, increasing w/b from 0.40 to 0.45 decreased compressive strength for similar systems.

The binary mixtures containing Class C fly ash exhibited similar strengths to the control mixtures at 28-day. However, due to the slow pozzolanic reactivity of Class F fly ash, the binary mixtures containing Class F fly ash exhibited lower 28-day compressive strength compared to the control mixtures. However, increasing the fly ash replacement dosage from 15% to 30% did not significantly affect the compressive strength. For a given w/b and nominal air content, increasing the slag cement replacement dosage increased the 28-day compressive strength (Hooton 2000). Ternary mixtures containing Class F fly ash and slag cement exhibited higher strength than the binary mixtures with Class F fly ash, due to the contribution made by slag cement. Ternary mixtures containing Class C fly ash and slag cement resulted in higher compressive strength than the ternary mixtures with Class F fly ash and slag cement. Ternary mixtures overall exhibited slightly higher strength than the control mixtures.

Surface Resistivity

Concrete surface resistivity is the ability of concrete to oppose the movement of electrons (Smith et al. 2004). The higher the electrical surface resistivity, the more difficult it is for electrons to travel through the concrete, thus the lower the permeability.

The 28-day surface resistivity data is presented in Figure 6. Concrete mixtures overall benefited from the addition of SCMs because their 28-day surface resistivity results were equal or higher than the control mixture, depending on the type and amount of the SCMs used. For a given w/b, increasing the replacement dosage of Class F fly ash increased the resistivity of binary mixtures, while binary mixtures with Class C fly ash exhibited similar results as control mixtures. Binary mixtures with slag cement exhibited the highest resistivity amongst all the tested mixtures. For a given w/b, increasing the replacement dosage of slag cement further increased the resistivity of binary mixtures. This is likely due to the effect of slag cement on increasing the density of the microstructure and blocking capillary pores with secondary hydration products which results in reducing the porosity of the ITZ, thus decreasing the overall permeability compared to the control mixture (Wee et al. 2000; Hooton 2000; Bijen 1996). Ternary mixtures with Class F fly ash and slag cement showed higher resistivity than the ternary mixtures with Class C fly ash and slag cement. This is due to the Class F fly ash being more pozzolanic than Class C fly ash with a greater portion of the ash being in the glass phase (Rupnow 2012).

Increasing nominal air content slightly increased the surface resistivity (more significant in binary mixtures with slag cement), likely due to air bubbles having a

higher resistivity than the paste around them, and causing longer duration for the electrons to travel from one surface to another, thereby exhibiting a higher surface resistivity. Increasing w/b from 0.40 to 0.45 overall decreased the surface resistivity slightly, likely as a result of the increased capillary porosity.

Shrinkage

The 28-day shrinkage results are presented in Figure 7. Both binary and ternary mixture results showed shrinkage results comparable or less than the control mixtures. This result indicates that ternary mixtures will be no more prone to shrinkage cracking compared to the control mixtures.

When one parameter is kept constant, increasing w/b or air content slightly increased the shrinkage due to the increased paste volume. This result is expected since shrinkage is affected by the volume of cement paste in concrete (Hale et al. 2008, Hooton 2000). Since the paste volume was not significantly different for all, the shrinkage for all mixtures would not have been expected to be dramatically different (Hale et al. 2008). Therefore, changing the SCM type and/or dosage did not significantly affect the shrinkage of binary and ternary mixtures.

Conclusions and Recommendations

The effect of mix characteristics (w/b, SCM, and air content) was investigated on fresh and hardened properties of both binary and ternary mixtures. Test results have shown that the ternary blended mixtures overall improved the concrete performance by improving the workability, strength, and durability; therefore are applicable. Ternary mixtures overall performed in accordance with their ingredients; however the degree of

improvement that they contribute varies based on the selected dosage and type of SCMs.

The following conclusions are made based on the results:

- When w/b was kept constant, increasing air content slightly increased workability.
- The ternary mixtures slightly retarded the setting time consistent with the effects of their ingredients.
- As expected, the compressive strength is strongly influenced by the w/b and air content.
- Binary and ternary mixtures containing Class C fly ash and slag cement exhibited higher compressive strength than the control mixture.
- Increasing w/b slightly decreased the surface resistivity.
- The surface resistivity results of binary and ternary mixtures were equal or higher than the control mixture.
- Surface resistivity is improved by the inclusion of SCMs, especially with Class F fly ash and slag cement.
- The shrinkage of binary and ternary mixture were comparable or less than the control mixture. Changing the SCM type and/or dosage did not significantly affect the shrinkage of binary and ternary mixtures.

Further research is needed to investigate the different aggregate gradation systems on fresh and hardened properties of binary and ternary mixtures. Additional tests should be conducted to determine the effect of mix characteristics on the carbonation rate, bleeding, and freeze-thaw in ternary concrete.

Acknowledgments

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Table 1. Chemical composition of cementitious materials, percentage by mass

Oxides	Ordinary portland cement	Class F fly ash	Class C fly ash	Slag cement
Silicon dioxide (SiO_2)	20.13	52.10	36.70	37.60
Aluminum oxide (Al_2O_3)	4.39	16.00	20.10	9.53
Ferric oxide (Fe_2O_3)	3.09	6.41	6.82	0.44
Calcium oxide (CaO)	62.82	14.10	23.30	40.20
Magnesium oxide (MgO)	2.88	4.75	4.92	11.00
Sulfur trioxide (SO_3)	3.20	0.59	1.88	1.14
Potassium oxide (K_2O)	0.57	2.36	0.48	0.44
Sodium oxide (Na_2O)	0.10	1.72	1.62	0.45
Loss on ignition	2.55	0.09	0.25	0.00

Table 2. Variables

w/b	0.40
	0.45
Air content (%)	2
	4
	8
Binder combinations	100% portland cement (P)
	15% Class F fly ash (F) and 85% P
	15% Class C fly ash (C) and 85% P
	20% slag cement (SL) and 80% P
	30% F and 70% P
	30% C and 70% P
	40% SL and 60% P
	20% F, 20% SL, and 60% P
	20% C, 20% SL, and 60% P

Table 3. Test matrix

Fresh Property	Method	# of Tests	Age (days)
Slump	ASTM C143	1	-
Air content	ASTM C231	1	-
Air void analysis (AVA)	ASTM C457	2	-
Setting time	ASTM C403	1	-
Hardened Property	Method	# of Specimens	Age (days)
Compressive strength	ASTM C39	3	28
Surface resistivity	Wenner Probe	3	28
Shrinkage	ASTM C157	3	28

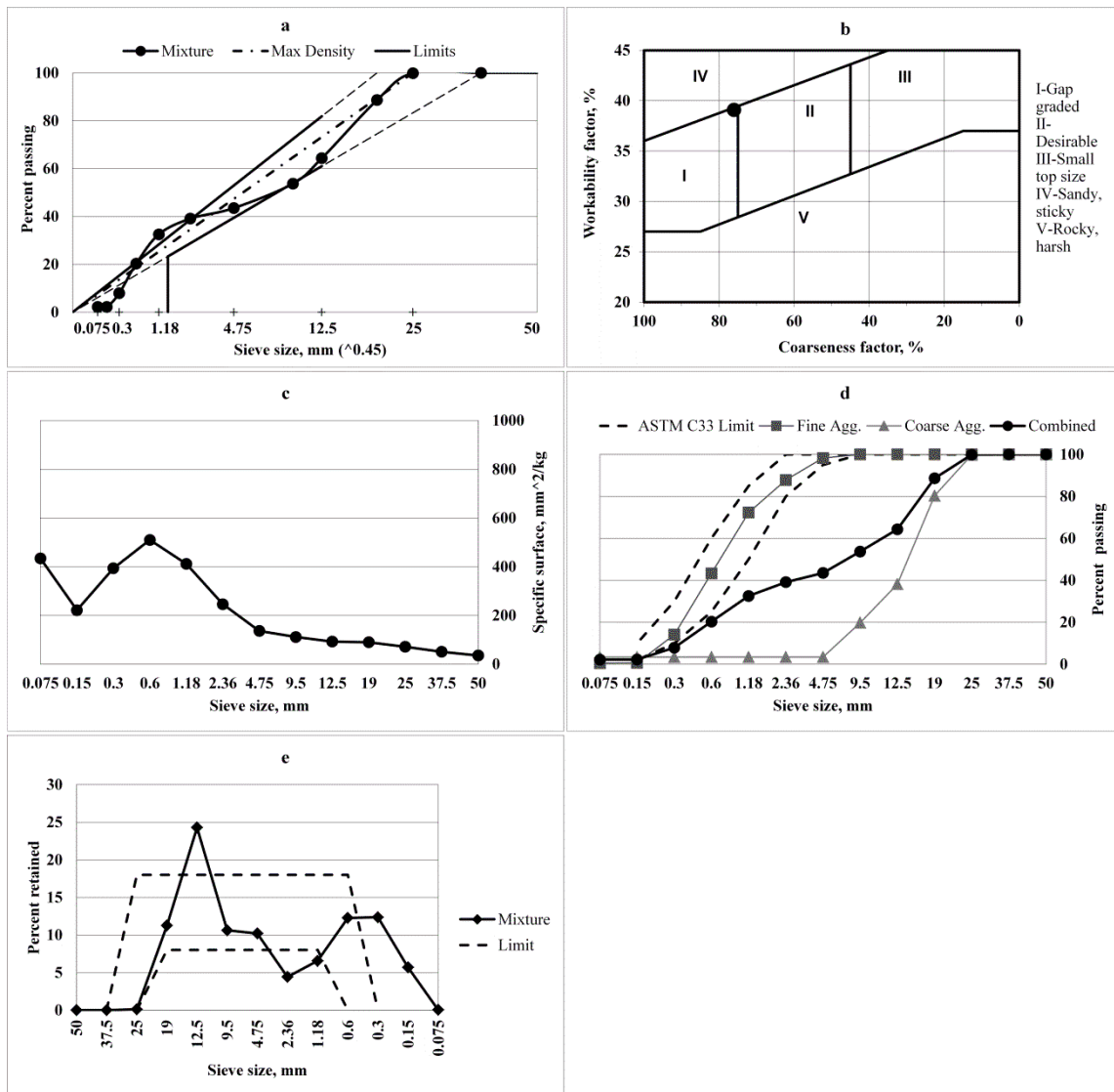


Fig. 1. Combined aggregate gradation curves.

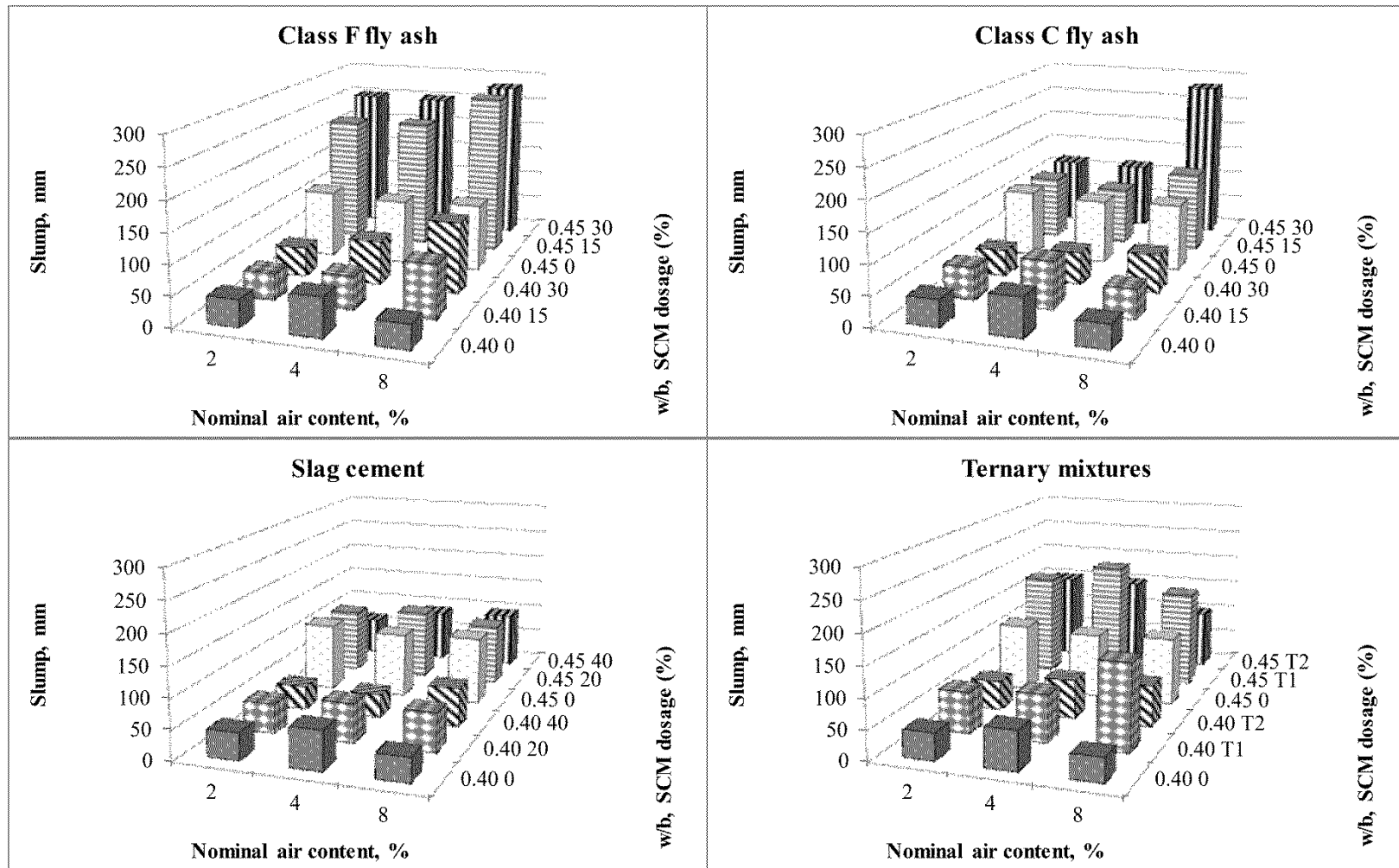


Fig. 2. The effect of w/b, SCM dosage, and nominal air content on slump.

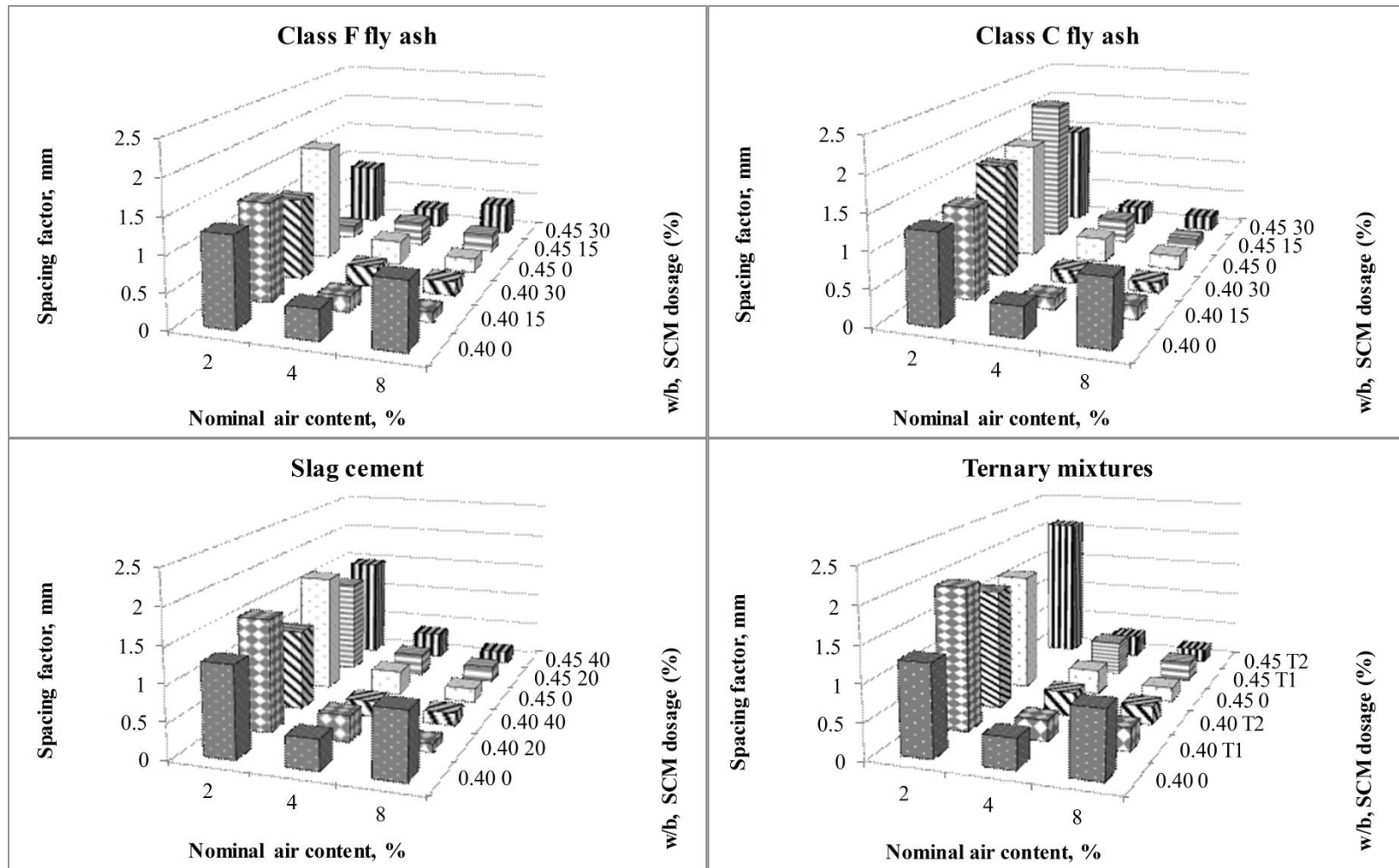


Fig. 3. Plots of spacing factor vs. w/b, SCM dosage, and nominal air content.

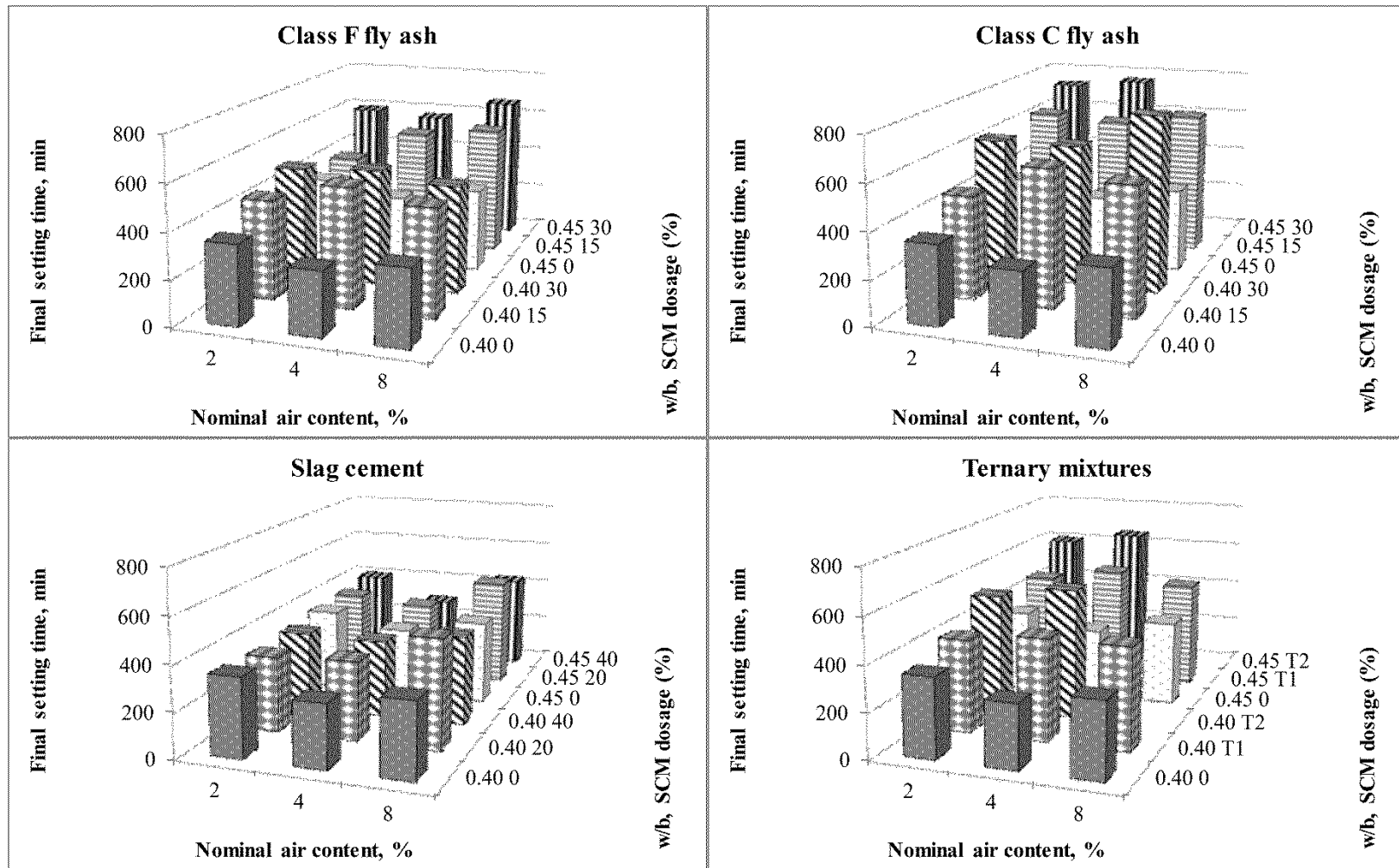


Fig. 4. The effect of w/b, SCM dosage, and nominal air content on final setting time.

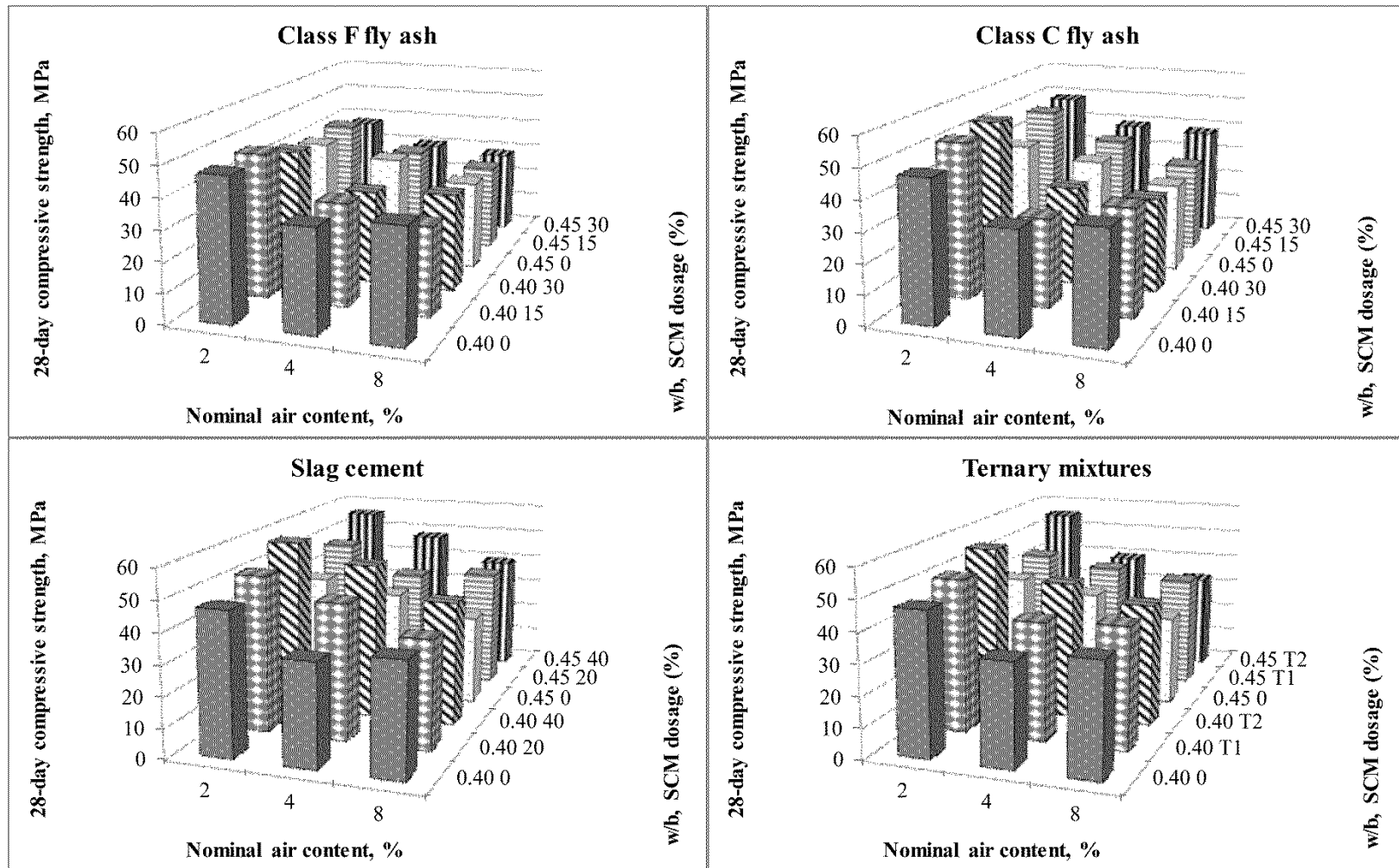


Fig. 5. The effect of w/b, SCM dosage, and nominal air content on 28-day compressive strength.

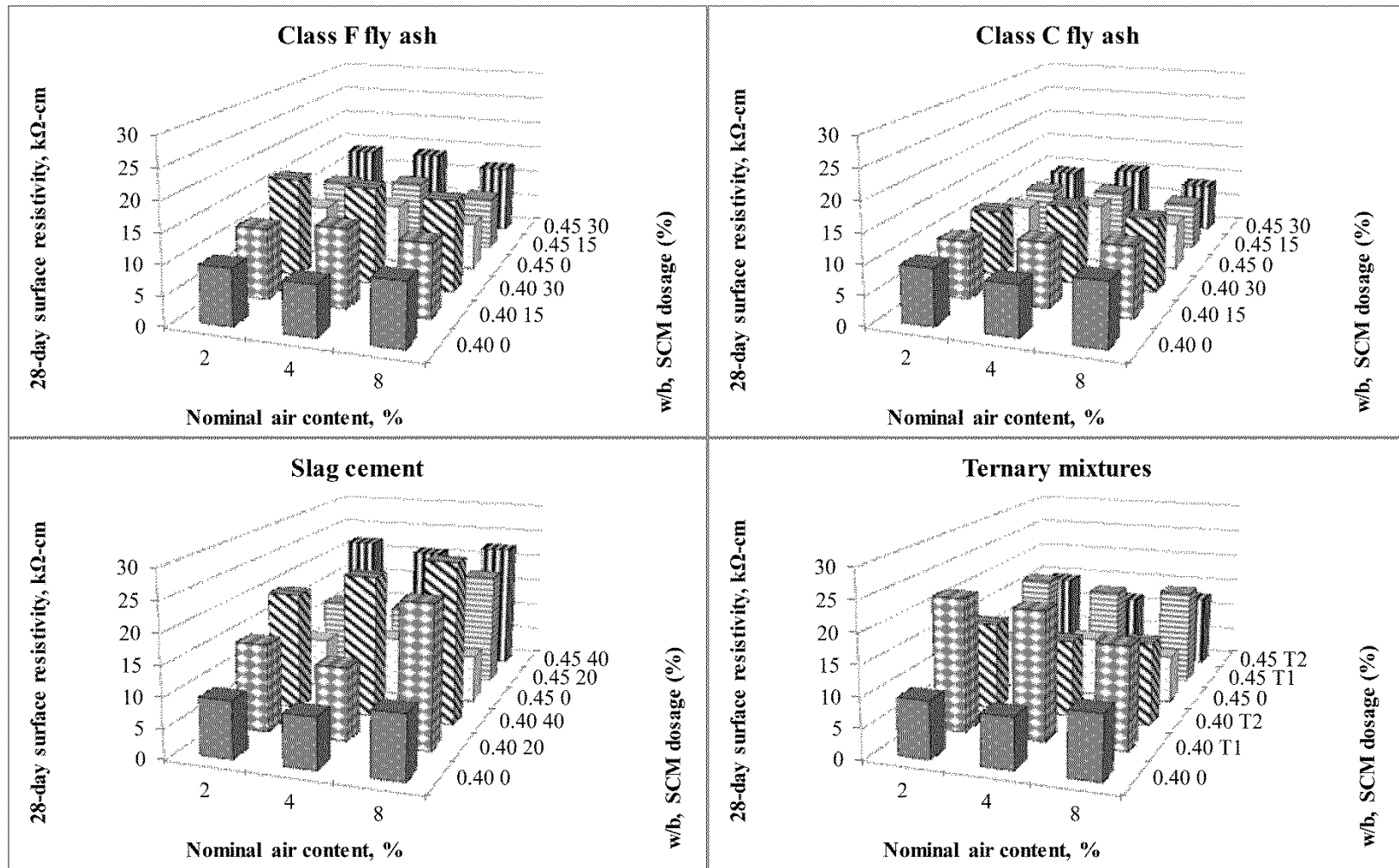


Fig. 6. The effect of w/b, SCM dosage, and nominal air content on 28-day surface resistivity.

CHAPTER 4. DEVELOPMENT OF A PERFORMANCE-BASED MIX PROPORTIONING TOOL USING THE ARTIFICIAL NEURAL NETWORK MODELING

A paper to be submitted to Journal of Materials in Civil Engineering (ASCE)

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Abstract

The purpose of this study is to develop a performance-based mix proportioning tool that predicts the required quantities of locally available materials for the desired fresh and hardened concrete properties. Artificial-neural network modeling was used to establish the relationship between the variables and the tested properties. In this experimental program, mixtures were obtained from four different projects that were prepared and tested in three laboratories to verify the applicability of this tool with different variables and conditions. A total matrix of 178 mixtures with a wide range of binder content; water-to-binder ratio (w/b); various size, shape, texture and gradation of aggregate systems; and a nominal air content of 2%, 4% and 8% were prepared.

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Binder systems included ordinary portland cement, binary mixtures with slag cement, Class F and C fly ash, and ternary mixtures containing a combination of slag cement and one type of fly ash. The correlations between the actual and predicted values of paste-to-voids volume ratio, workability, compressive strength, rapid chloride penetration, and surface resistivity at 28-day were analyzed. Based on the performed analysis, the proposed mix proportioning tool is promising and achievable in terms of predicting the values of the tested properties based on the mix design variables, or predicting the amount and type of materials needed to achieve the desired concrete properties. Therefore, the findings of this study can be implemented in real time, however this approach should be used with a larger data set to be beneficial for performance-based specifications universally.

CE Database subject headings: Neural networks; Network analysis; Specifications; Concrete; Durability; Compressive strength.

Introduction

Concrete is a heterogeneous and complex material with the interactions between its components. It is well documented (Yurdakul et al. 2012, Hu and Wang 2011, Ashraf and Noor 2011, Wassermann et al. 2009, Kim et al. 2005, Jamkar and Rao 2004) that overall concrete performance is affected by the mix design variables such as quantity and type of the cementitious materials, the quality of the paste, and the shape, texture, gradation and size of aggregates. Each mix design variable has an impact on fresh and hardened concrete properties. For example, increasing water-to-binder ratio (w/b) increases the workability, whilst adversely affecting concrete strength due to the

increased capillary porosity (Popovics 1990, Kennedy 1940, Abrams 1920). In addition to the effect of each mix design variable on concrete performance, the interactions between these variables also affect the concrete properties which make concrete behavior even more complicated to analyze. Therefore, to understand the overall concrete behavior in-depth, the effect of every component and their interactions with each other should be taken into consideration. Concrete mix proportioning has to be a well-thought process that requires “balance” in terms of selecting the type and quantity of concrete components properly. However, mix proportioning is commonly done by engineers who follow a similar “recipe” based on a previously produced concrete rather than adjusting the quantity of materials based on the needs of the mixture and locally available materials (Lee et al. 2009, Ji et al. 2006).

Another challenging issue with the current mix proportioning methods is that they are predominantly prescriptive-based, thus limit the choice of materials, and may promote using higher amount of some materials than needed which results in increased cost (Yurdakul et al. 2012, Grove and Taylor 2012, Lee et al. 2009, Shilstone and Shilstone 2002). The construction industry has been moving from prescriptive towards performance-based specifications. Therefore, a performance-based mix proportioning method needs to be developed which can predict the required amount and type of materials to fulfill the desired concrete properties for a given project specification. The proposed method should be user-friendly, easy to apply in practice, and flexible in terms of providing a wide range of type and amount of material selection.

To analyze the complexity between concrete properties and concrete components, an artificial neural network (ANN) approach has been used for concrete mix proportion design (Mohammed et al. 2012, Agrawal and Sharma 2010, Lee et al. 2009, Lee and Yoon 2009, Yeh 2007, Ji et al. 2006, Kim et al. 2005). ANN modeling can analyze the complex interactions between input and output variables in a system even when the nature of these interactions is unknown. Using ANN modeling reduces cost, labor, and time because the conventional mix proportioning methods require trial mixtures and expensive time-consuming experiments to ensure that the produced mixture satisfies the required performance specifications (Ji et al. 2006).

The objective of this study is to develop a universally or nationally applicable performance-based mix proportioning tool that predicts the required quantities of locally available materials for the desired fresh and hardened concrete properties. This tool was developed by analyzing the statistical relationships between the selected mix characteristics and their correspondent effects on tested properties. Artificial-neural network modeling was used to establish the relationship between the variables and the tested properties. To verify the applicability of this tool on different projects in various locations, this paper uses data from 4 projects:

- Project A: This study was conducted by researchers at Iowa State University to investigate the effects of the paste quantity at a given water-to-binder ratio (w/b) with a fixed combined aggregate gradation on fresh and hardened concrete properties (Taylor et al. 2012-a).

- Project B: This study was conducted by Iowa State University to investigate the effects of the paste quality with a fixed combined aggregate gradation on fresh and hardened concrete properties (Taylor et al .2012-b).
- Project C: This study was conducted by the National Ready-Mixed Concrete Association, USA to investigate the effects of the paste quantity on fresh and hardened concrete properties. This project used the same mix design principle as Project A but expanded the available data set by further-varying the w/b, binder content, and selecting a different fixed combined aggregate gradation than the one used in Project A. Ordinary portland cement, used in Project A, was also used in Project C.
- Project D: This study was conducted by Oklahoma State University to investigate the effect of aggregate size, shape, texture, and gradation on fresh and hardened concrete properties while using a fixed binder type, binder content and w/b (Ley et al. 2012).

Material and Methods

Materials

Table 1 presents the materials that were used in each project.

Table 2 presents the chemical compositions of the cementitious materials that were used in each project.

Aggregate Systems

Table 3 presents the aggregate properties used for each project.

Project A and B

The void percentage of the combined aggregates was kept constant at 19.8% for all the mixtures to remove the aggregate grading as a variable from the experimental matrix (Yurdakul et al. 2012, Taylor et al. 2012-a, Taylor et al. 2012-b). The fine aggregate-to-total aggregate ratio of 0.42 was selected by assessing the FHWA 0.45 power curve (Bureau of Public Roads 1962) and Shilstone workability factor chart (Fig. 1a-b). The appropriateness of the selected ratio of 0.42 was checked by plotting the system in an ASTM C33 plot (Fig. 1c) and a “Haystack” plot (Fig. 1d).

Project C

The same approach as Projects A and B was followed to determine the fine aggregate-to-total aggregate ratio and the void content of the combined aggregate system of Project C. The void percentage of the combined aggregates was kept constant at 25.5% for all the mixtures. The fine aggregate-to-total aggregate ratio was selected as 0.39.

Project D

This study used a fixed binder type, binder content, and w/b whilst varying the aggregate size, aggregate type, and aggregate gradation. Each aggregate combination was tested using five different gradations: 1) center of the Shilstone chart; 2) bottom center of the Shilstone chart; 3) minimum voids contents as determined by the Toufar method within Compass (The Transtec Group 2004); 4) close to the power 45 line; and 5) mix with 60% of the largest aggregate size and 40% of the fine aggregate size (Ley et al. 2012).

Paste-to-Voids Volume Ratio ($V_{\text{paste}}/V_{\text{voids}}$)

The paste-to-voids volume ratio ($V_{\text{paste}}/V_{\text{voids}}$) was calculated by calculating the paste volume of concrete mixtures and dividing that value by the volume of voids between the combined consolidated aggregates.

The void of the combined aggregate system was determined following a similar procedure as the ASTM C29. The difference between the ASTM C 29 and the procedure followed in this study is that ASTM C29 calculates the void content for a single aggregate type (either for fine or coarse aggregate individually) whereas this study applied the same principle provided in ASTM C29 on combined aggregate system based on the fine aggregate-to-total aggregate ratio that was selected. The rest of the testing and void content calculations were conducted following the rodding procedure provided in the ASTM C29.

The paste volume was calculated by adding up the volume of water, the cementitious materials, and the measured air in the system. The cementitious materials volume was determined by dividing the cementitious materials content by the corresponding specific gravity for each type of cementitious material. The volume of the air used in the calculation was obtained from the air content test performed in accordance with the ASTM C231.

Variables

Table 4 presents the variables of the combined study.

Specimens and Testing

For each mixture, 4x8-in. concrete cylinders were cast in accordance with ASTM C31 and stored in the fog room until testing in accordance with ASTM C192. The tests conducted are given in Table 5.

Artificial Neural Network (ANN) Modeling

Two commercially available software packages were used for artificial neural network modeling. Software A is capable of providing an equation to present how to obtain the desired properties using the selected inputs which is necessary to establish a mix proportioning model. However, Software A is not able to provide the training, testing, validation, and overall prediction charts all together. Therefore, Software B was used to demonstrate the predictions for the overall data set. The obtained predictions based on using these two software packages were compared, and the results were found to be well correlated.

Back-propagation learning algorithm was adopted in this study. In back-propagation neural networks, the mathematical relationships between the various variables are not specified. Instead, they learn from the examples fed to them. The data was collected from 178 mixtures that were prepared in three different laboratories. Prior to exporting the data to the ANN software, the data was randomly ordered. Among this randomly ordered data set, for both software packages, 70% of the data was chosen for ‘training’, 15% of the data was chosen for ‘validation’ and 15% of the data was chosen for ‘testing’. The selected inputs for each predicted parameter slightly varied due to the

each concrete property being affected by different mix design variables as shown in Table 6.

After a number of trials, the best network architecture and parameters that minimize the root mean square (RMS) error of the testing data were selected as follows:

Software A:

- Validation method = Random Holdback
- Holdback Proportion = 0.3333
- Number of hidden layers = 1
- Number of hidden units = 3
- Learning rate = 0.1
- Penalty method = Squared

Software B:

- Data division = Random
- Training = Levenberg-Marquardt back propagation
- Performance = Mean squared error
- Derivative = Default
- Number of hidden neurons = 10

Results and Discussion

Prediction of Paste-to-Voids Volume Ratio ($V_{\text{paste}}/V_{\text{voids}}$)

The data of Project A was initially used to predict the $V_{\text{paste}}/V_{\text{voids}}$ ratio. The data from only one project (64 mixtures) was selected deliberately to minimize the variability that may occur due to the different aggregate systems, lab environment, and operators of other projects.

Software A was initially used to obtain the equations leading the prediction of the $V_{\text{paste}}/V_{\text{voids}}$ ratio by using the selected inputs shown in Table 6. The correlation between the predicted and actual values is presented in Fig. 2. The regression line fitted data well and the coefficient of determination (R^2) was determined as 1.0. The coefficient of determination of training was 1.0, and the coefficient of determination of validation was 0.999. The trends are presented in Fig. 3. This result shows that the applied ANN modeling is accurate to predict the $V_{\text{paste}}/V_{\text{voids}}$ based on the selected inputs using the data of Project A.

The verification of the selected method was done by manually selecting randomized 36 data points as ‘training’ and 18 data points as ‘validation’ out of 64 mixtures of Project A. The neural network modeling was run based on those randomly selected 54 mixtures. The obtained results are presented in Fig. 4. The coefficient of determination (R^2) of training and validation was 1.0.

The ‘testing’ was conducted by applying this equation on the rest of the data (remaining 10 mixtures) that was not selected to predict the $V_{\text{paste}}/V_{\text{voids}}$ of the mixtures of the Project A in this model. The correlation between the actual

V_{paste}/V_{voids} and the predicted values by using the equation (testing set) is presented in Fig. 5. The coefficient of determination (R^2) of testing was obtained as 1.0.

The presented trend in Fig. 5 shows that the predictions are accurate; and thus the obtained equation can be used for mixes having similar gradation systems to predict their correspondent V_{paste}/V_{voids}. The calculation of V_{paste}/V_{voids} requires determining the voids volume of the combined aggregate systems and predicting this parameter by using such a model will reduce the time-consuming experiments, thus lower the labor and cost of the projects. The concrete properties are governed by the paste volume and the paste quality (Yurdakul et al. 2012). Therefore, using the parameter of V_{paste}/V_{voids} would be more beneficial rather than using the cementitious content in performance-based specifications.

Software B was used to present the predictions for the data points of Project A as shown in Fig. 6. Similar to the predictions obtained by using Software A, the correlation coefficient (R) for the overall data set of Project A was 1.0 ($R^2=1.0$).

After verifying the accuracy of the developed ANN model, Software B was used to conduct an analysis by using the data of Project A and Project B (118 data points in total) to predict the V_{paste}/V_{voids}. The variables between these projects are presented in Table 4. The mixtures of these two projects were both prepared in the same lab by the same operators. The inputs selected to predict the V_{paste}/V_{voids} were cement content, F fly ash content, C fly ash content, slag cement content, w/b, and the tested properties namely slump, air content, and 28-day compressive strength values. The correlation between the predicted and actual values is presented in Fig. 7. The regression line fitted

data well and the correlation coefficient (R) was determined as 0.999 ($R^2=0.998$) which shows a high accuracy of the used model.

Evaluation of Minimum Vpaste/Vvoids Requirements for Concrete Properties

As part of the Project A, the minimum critical Vpaste/Vvoids requirements for various concrete properties were calculated for the selected aggregate gradation system (Table 7).

In broad terms, approximately 1.5 to 2 times more paste by volume is required than the space between the combined aggregate particles. This is to fill all the space and to line all of the aggregate particles with paste, providing lubrication in mixture in its fresh state, and to glue the particles together in the hardened state. Although the provided critical Vpaste/Vvoids values are applicable only for the selected single aggregate system tested in Projects A and B, the provided values may be used as a guideline for determining the required minimum Vpaste/Vvoids needed for the desired concrete properties. Therefore, the applicability of the selected mix proportioning can be verified by initially predicting the Vpaste/Vvoids based on the selected design variables and the desired concrete properties, and then comparing these values with the minimum Vpaste/Vvoids requirements in Table 7.

To determine the applicability of this method on systems having different gradation systems, Software B was used to conduct an analysis by using the combined data of the four projects. The variables between these projects are presented in Table 4. Same inputs (cement content, F fly ash content, C fly ash content, slag cement content, w/b, and tested values of slump, air content, and 28-day compressive strength) were

selected as the previously conducted prediction. The relationship between the predicted and actual Vpaste/Vvoids values is presented in Fig. 8.

The correlation coefficient was determined as 0.971 ($R^2=0.943$). Although, the accuracy of predicting the Vpaste/Vvoids is still high, it can be noted that the coefficient of determination (R^2) was decreased from 0.998 to 0.943 (as compared to the predictions obtained based on Projects A and B) due to the addition of mixtures with different aggregate size, type, shape, and gradation systems. The accuracy was also affected due to the mixtures being conducted at three different laboratories by different operators.

In addition to its capability to provide equations of the predictions using the selected inputs, Software A also provides a prediction profiler to show how each input affects the selected output. Fig. 9 shows an example of a prediction profiler to demonstrate the trends between inputs (selected mix design variables and output (Vpaste/Vvoids)).

Prediction of Slump

Slump was predicted by using the combined data set of Projects A, B, C and D. Fig. 10 presents the results of the predictions for each project that were obtained according to Software A. The coefficient of determination (R^2) of 0.885, 0.844, 0.901, and 0.906 were obtained from Projects A, B, C and D, respectively. The obtained coefficient of determination values seem to be affected in regards with the increased variability of the projects. The overall predictions seem to be close to the actual tested values. Fig. 11 presents the prediction results of the combined data set of four projects by using Software B. The obtained correlation coefficient (R) for the combined study

was 0.906 ($R^2=0.821$). The predictions varied mostly ± 3 -in. from the actual tested value. Considering the ASTM C143 allows 0.82-in. to 1.50-in. depending on the obtained slump range from the multi-laboratory testing, the obtained predictions seem reasonable.

Prediction of Strength

The strength of concrete is controlled by the mix proportioning of concrete components. Air content and w/b are two chief factors that adversely affect the concrete strength. Therefore, these inputs were included to adequately to predict the 28-day compressive strength of the mixtures using both of the ANN model software packages.

Software A analyzed the each project data set separately by using the same inputs. The coefficient of determination (R^2) of the model for Projects A, B, C and D were 0.900, 0.905, 0.962, and 0.750 accordingly. The results are presented in Fig. 12. As the variability of the mix proportioning decreased (such as Project C), the accuracy of the predictions increased. However, when the variability increased especially due to the different size, shape, texture and gradation of the aggregates in Project D, the accuracy of the predictions decreased significantly. This result shows the sensitivity of the applied technique. Software B analyzed the combined data set from four projects. The correlation coefficient (R) was obtained as 0.907 ($R^2=0.823$). The results are presented in Fig. 13. Overall, despite of the high variation in the mixtures, the predictions were close to the actual strength values that were determined through lab experiments.

Prediction of Durability

In this study, the durability of concrete is evaluated by testing the 28-day rapid chloride penetration and surface resistivity of the concrete mixtures.

Projects A and C conducted the rapid chloride penetration test to evaluate the effect of mix design variables on concrete durability. Therefore, an analysis was conducted using Software B on the combined data points of both Projects A and C. Fig. 14 shows that the correlation coefficient (R) was determined as 0.946 ($R^2=0.895$). Overall, considering the high precision allowed in the rapid chloride penetration testing, it can be stated that the predictions were close to the actual tested values.

Project B conducted the surface resistivity test using the Wenner Probe to evaluate the effect of mix design variables on concrete durability. Fig. 15 shows that the correlation coefficient (R) was determined as 0.973 ($R^2=0.947$). Therefore, the overall predicted values were found to be close to the actual tested 28-day surface resistivity values of the mixtures of the Project B.

Conclusions and Recommendations

Development of a performance-based mix proportioning tool was the objective of this study, to be achieved by investigating the relationships between various concrete components and properties. A statistical artificial neural network modeling software was used to account for the effect of all variables on each tested concrete property. The following conclusions are made based on the results:

- Portland cement concrete (PCC) is a complex and heterogeneous material. It is composed of aggregates, cement, and water. Supplementary cementitious materials (SCMs) and chemical admixtures may also be included. Each component has a complex physical and chemical variability. Changing the characteristics of these materials such as the amount, chemistry, and fineness of

cementitious materials; and shape, texture, and gradation of aggregate systems has a high impact on concrete properties. Given the fact that cement hydration is a chemical reaction, these complex variables also have interactions with each other which make concrete behavior even more complex to analyze. Therefore, to understand the overall concrete behavior in-depth, the effect of every component and their interactions with each other should be taken into consideration.

- Based on the performed analysis, predicted slump, strength, and durability were close to the actual tested property. The obtained coefficient of determination (R^2) ranged between 0.750 to 1.0, and the correlation coefficient (R) ranged between 0.906 to 1.0, depending on the complexity of the selected inputs.
- The addition of the aggregates (Project D) generally tended to decrease the precision of the predictions due to the increased variability of the mix design parameters such as size, shape, texture, and gradation of aggregates.

The proposed mix proportioning tool is promising and achievable in terms of predicting the values of the tested properties based on the mix design variables, or predicting the amount and type of materials needed to achieve the desired concrete properties. Therefore, the findings of this study can be implemented in real time although; the proposed mix proportioning tool is not completely ready for prime time. Considering that the cementitious materials and aggregate characteristics vary depending on the location along with the environmental conditions that the concrete may be exposed to (such as presence of sulfate and deicing chemicals), it is challenging to

propose a universally or nationally applicable statistical model that could be used on different projects in various locations. When a different aggregate system is selected, the model is required to be calibrated for a given set of materials. Therefore, the proposed model may be used in locations that have similar project specifications, climate and locally available materials as Iowa. While the method may be applicable worldwide, local models will have to be built around local materials.

Further research is recommended to investigate the effect of size, gradation, shape, and texture of aggregate systems as the available data used in this study is not wide enough to establish a reliable relationship using a statistical approach. Upon using this approach with a larger data set, the proposed model will be beneficial for performance-based specifications. If a model is to be useful, more detailed information of materials such as cement chemistry and fineness should be included in it to fully evaluate the effect of cementitious materials on tested properties. Additional tests such as early-age strength, air-void systems, and freeze-thaw should be conducted to determine the effect of concrete components on these properties. Different statistical models may be used to investigate the precision of the applied method.

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Table 1. Selected materials

Materials	Project A	Project B	Project C	Project D
ASTM C150 ordinary portland cement (OPC)	✓	✓	✓	✓
ASTM C618 Class F fly ash	✓	✓	✓	
ASTM C618 Class C fly ash	✓	✓		
ASTM C989 slag cement	✓	✓	✓	
1.5" nominal maximum size crushed limestone				✓
1.5" nominal maximum size river gravel				✓
1" nominal maximum size crushed limestone	✓	✓	✓	
3/4" nominal maximum size crushed limestone				✓
3/4" nominal maximum size river gravel				✓
3/8" nominal maximum size crushed limestone				✓
3/8" nominal maximum size river gravel				✓
No 4 nominal maximum size concrete sand	✓	✓	✓	✓
No 4 nominal maximum size manufactured sand				✓
ASTM C494 Type F polycarboxylate based high range water reducer (HRWR)	✓	✓	✓	
ASTM C494 lignosulfonate mid-range water reducer				✓
ASTM C260 tall-oil based air entraining admixture		✓		

Table 2. Chemical composition of the cementitious materials, percentage by mass

Oxides	Project A				Project B				Project C*		Project D
	OPC	F ash	C ash	Slag	OPC	F ash	C ash	Slag	OPC	F ash	OPC
SiO ₂	20.2	49.7	36.7	37.2	20.1	52.1	36.7	37.6	20.2	60.5	21.1
Al ₂ O ₃	4.4	15.3	19.4	9.5	4.4	16.0	20.1	9.5	4.4	29.1	4.7
Fe ₂ O ₃	3.2	7.2	6.0	0.5	3.1	6.4	6.8	0.4	3.2	2.9	2.6
CaO	62.7	15.7	25.2	40.1	62.8	14.1	23.3	40.2	62.7	0.7	62.1
MgO	3.5	5.3	4.8	11.0	2.9	4.8	4.9	11.0	3.5		2.4
SO ₃	3.2	0.9	2.0	1.1	3.2	0.6	1.9	1.1	3.2	0	3.2
K ₂ O	0.7	2.2	0.5	0.4	0.6	2.4	0.5	0.4	0.7	0.64	0.3
Na ₂ O	0.1	1.7	1.6	0.3	0.1	1.7	1.6	0.5	0.1	0.12	0.2

*The chemical composition data of slag cement used in Project C was not available

Table 3. Specific gravity and absorption properties of the selected aggregates

Project	Aggregate Size/Type	Specific Gravity (SSD)	Absorption (%)
Project A	1" Limestone	2.67	0.96
	River Sand	2.62	1.07
Project B	1" Limestone	2.67	0.93
	River Sand	2.62	1.07
Project C	1" Limestone	2.91	0.78
	River Sand	2.63	0.43
Project D	1.5" Limestone	2.74	0.45
	1.5" Gravel	2.64	1.55
	3/4" Limestone	2.70	0.66
	3/4" Gravel	2.65	1.26
	3/8" Limestone	2.72	0.58
	3/8" Gravel	2.62	1.95
	River Sand	2.65	0.55
	Manufactured Sand	2.63	0.70

Table 4. Variables of the combined study

Mix Characteristics		Project A	Project B	Project C	Project D
Binder Systems	Portland cement (P)	100% P	100% P	100% P	100% P
	Class F fly ash (F)	20% F	15% F 30% F	25% F	-
	Class C fly ash (C)	20% C	15% C 30% C	-	-
	Slag cement (SL)	40% SL	20% SL 40% SL	40% SL	-
	Ternary Systems	-	20 % F & 20 % SL 20 % C & 20 % SL	-	-
Binder content (pcy)		400 500 600 700	600	ranged between 417 and 720	470
w/b		0.35 0.40 0.45 0.50	0.40 0.45	0.40 0.47 0.55	0.45
Air content (%)		2	2 4 8	2	2
Coarse aggregate size (in)		1	1	1	3/8 3/4 1.5
Fine aggregate size (in)		4.75	4.75	4.75	4.75
Coarse aggregate type		Limestone	Limestone	Limestone	Limestone Gravel
Fine aggregate type		River sand	River sand	River sand	River sand Manufactured sand

Table 5. Test matrix

Concrete properties	Method	Age (days)
Slump/Slump flow	ASTM C143/ASTM C1611	-
Air content	ASTM C231	-
Compressive strength	ASTM C39	28
Rapid chloride penetration (RCP)	ASTM C1202	28
Surface resistivity	Wenner Probe	28

Table 6. Input variables

Inputs	Outputs				
	Vpaste/ Vvoids	Slump	Strength	Chloride penetration	Surface resistivity
Cement content	✓	✓	✓	✓	✓
Class F fly ash content	✓	✓	✓	✓	✓
Class C fly ash content	✓	✓	✓	✓	✓
Slag cement content	✓	✓	✓	✓	✓
Water content	✓	✓	✓	✓	✓
w/b	✓	✓	✓	✓	✓
WRA dosage		✓	✓	✓	✓
Air content		✓	✓	✓	✓
Fine aggregate content	✓				
Coarse aggregate content	✓				

Table 7. Critical minimum Vpaste/Vvoids, %

Property	OPC	F ash	C ash	Slag
Workability	150	150	125	125
Compressive strength	150	175	150	175
Air permeability	175	175	125	200

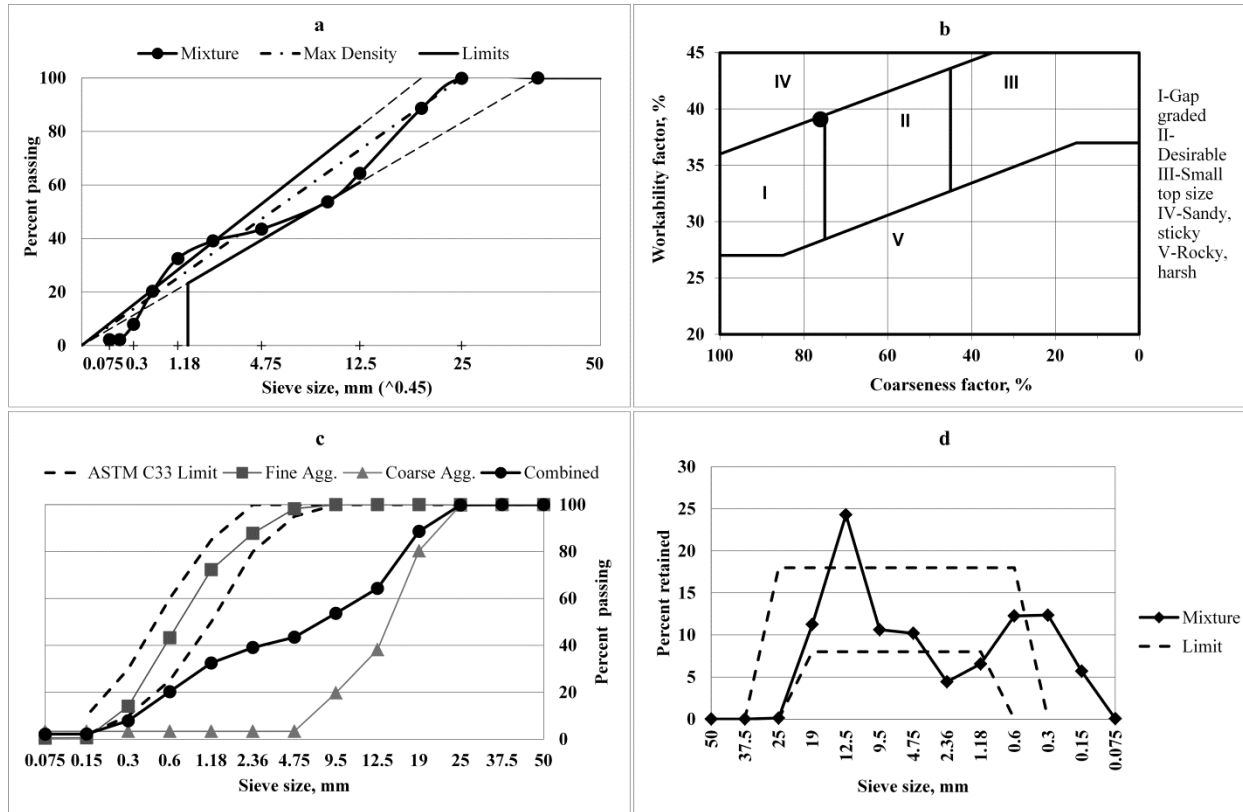


Fig. 1. Combined aggregate gradation curves.

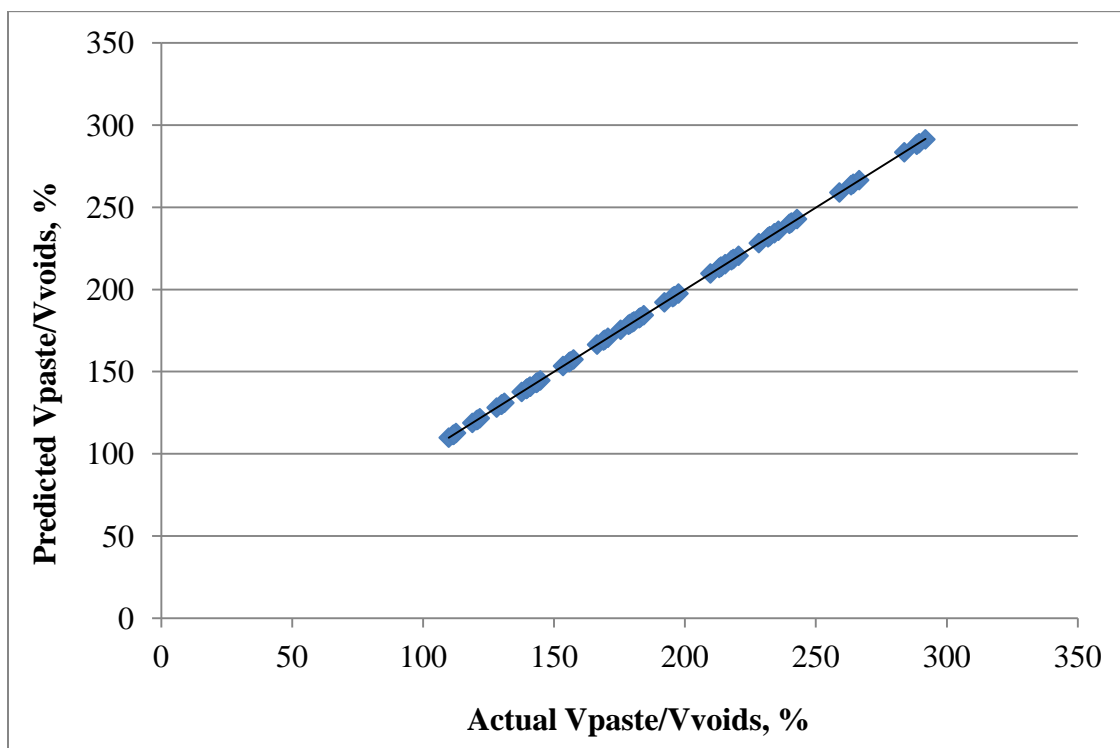


Fig. 2. Prediction of Vpaste/Vvoids based on 64 data points of Project A provided by Software A.

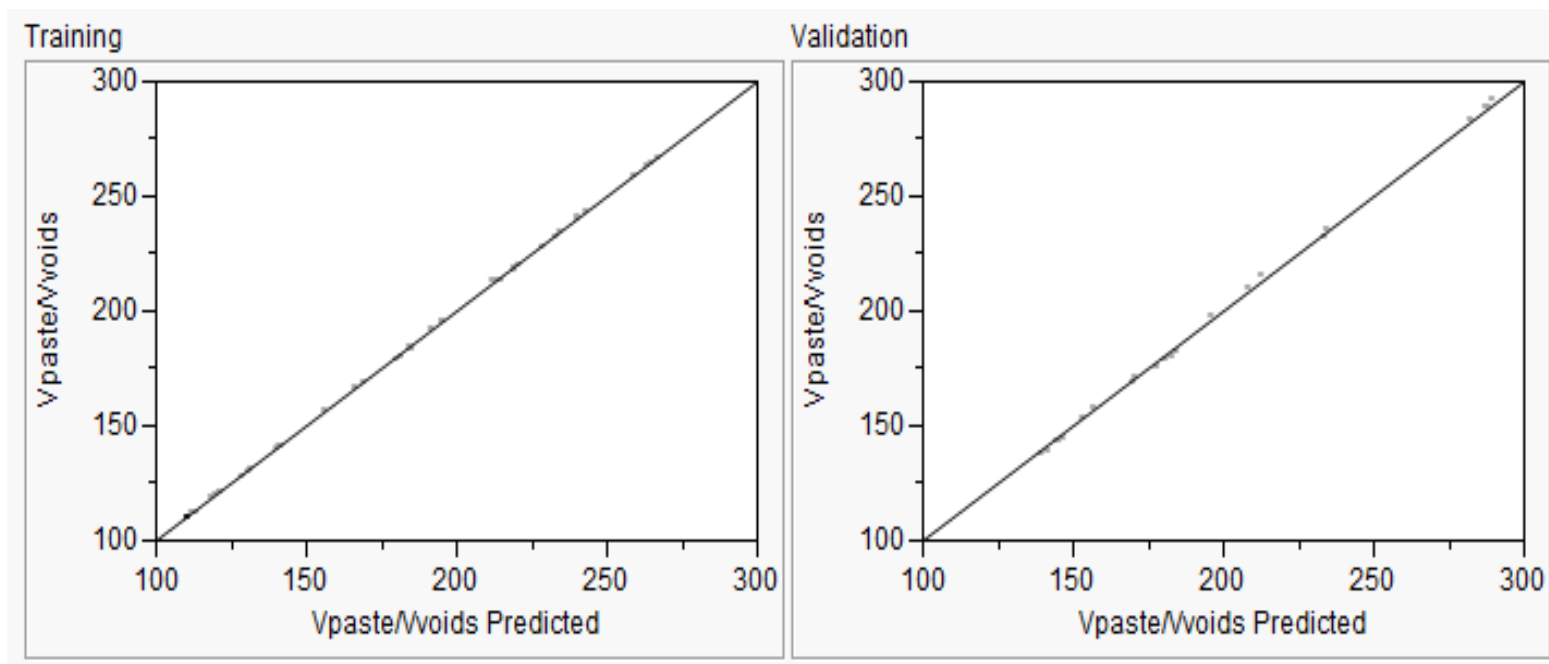


Fig. 3. Training and validation of the model based on 64 data points of Project A provided by Software A.

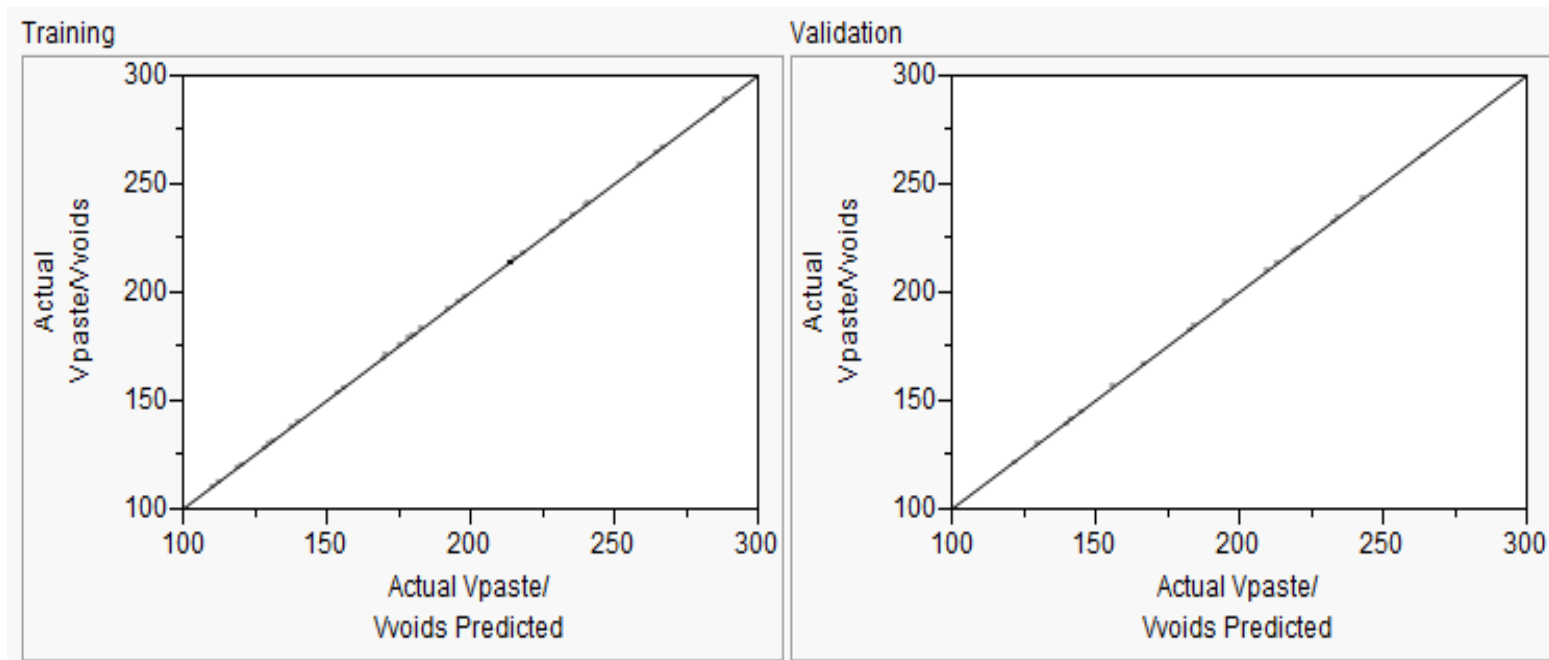


Fig. 4. Training and validation of the model based on 54 data points of Project A provided by Software A.

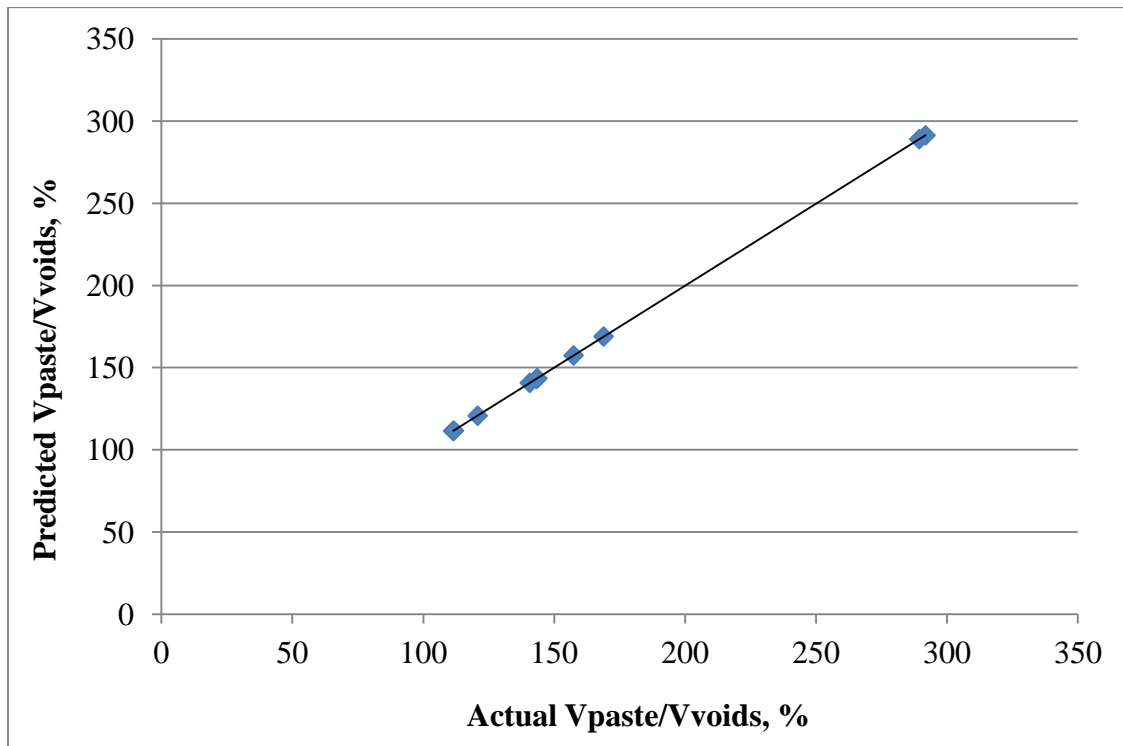


Fig. 5. Testing of the model based on 10 data points of Project A provided by Software A.

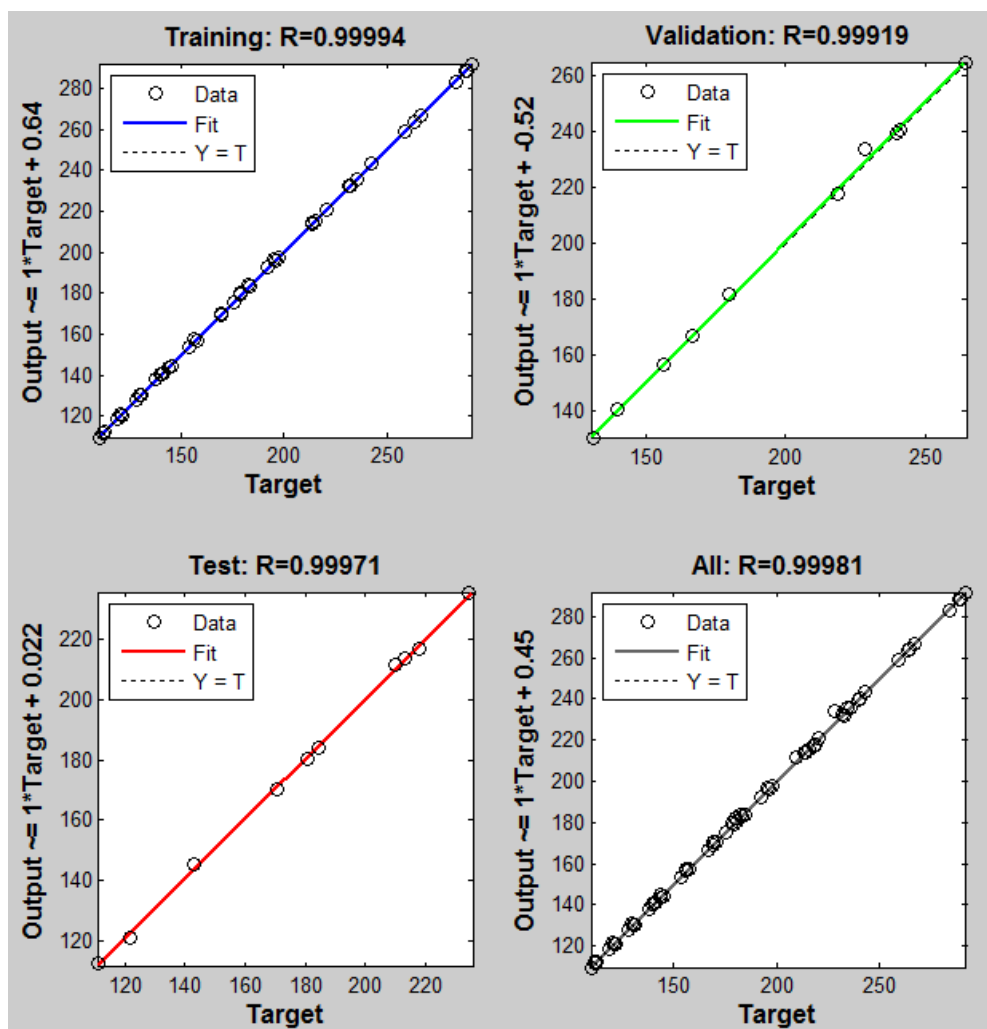


Fig. 6. Prediction of Vpaste/Vvoids based on 64 data points of Project A provided by Software B.

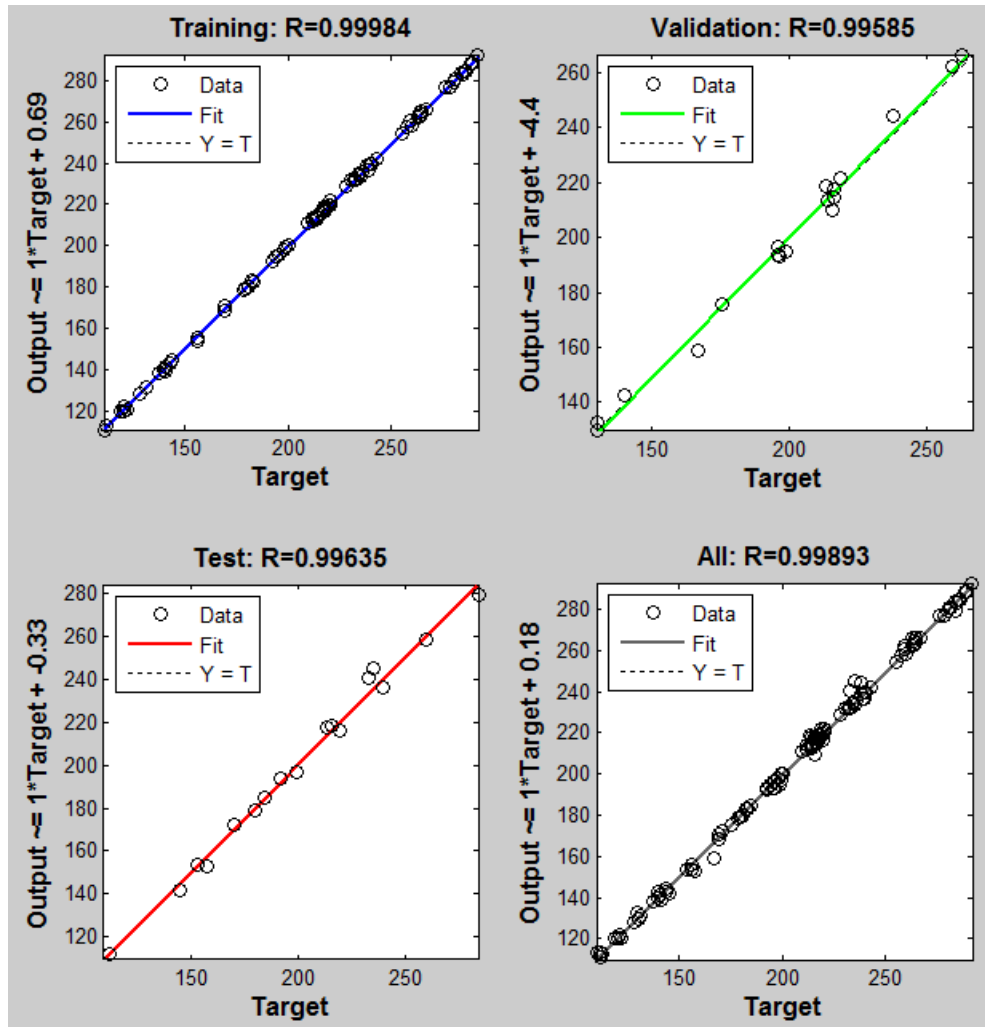


Fig. 7. Prediction of Vpaste/Vvoids based on the combined data set (118 data points) of Projects A and B provided by Software B.

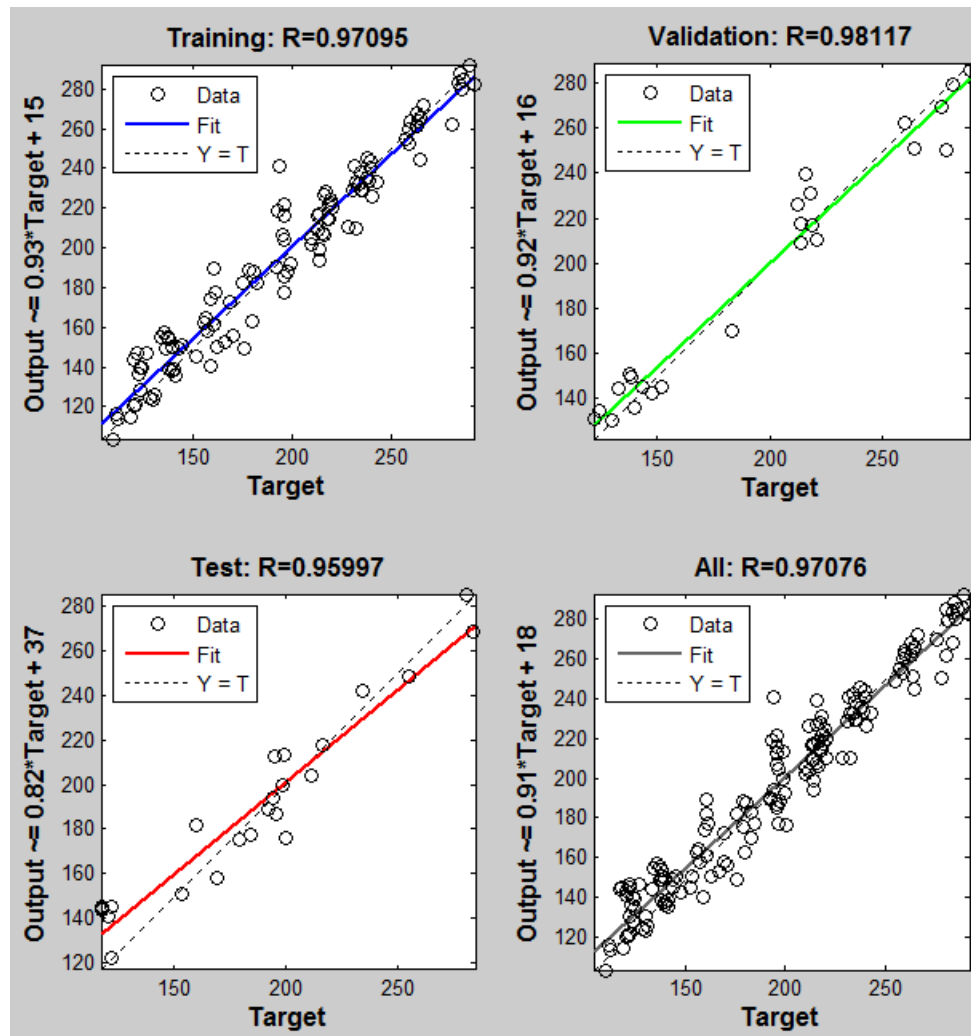


Fig. 8. Prediction of Vpaste/Vvoids based on the combined data set (178 points) of Projects A, B, C and D provided by Software B.

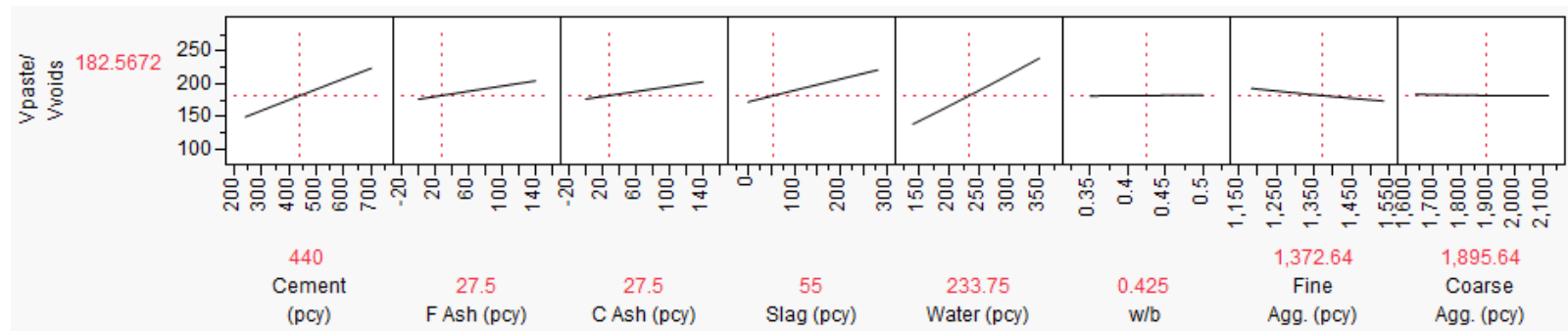


Fig. 9. Example of a prediction profiler provided by Software A.

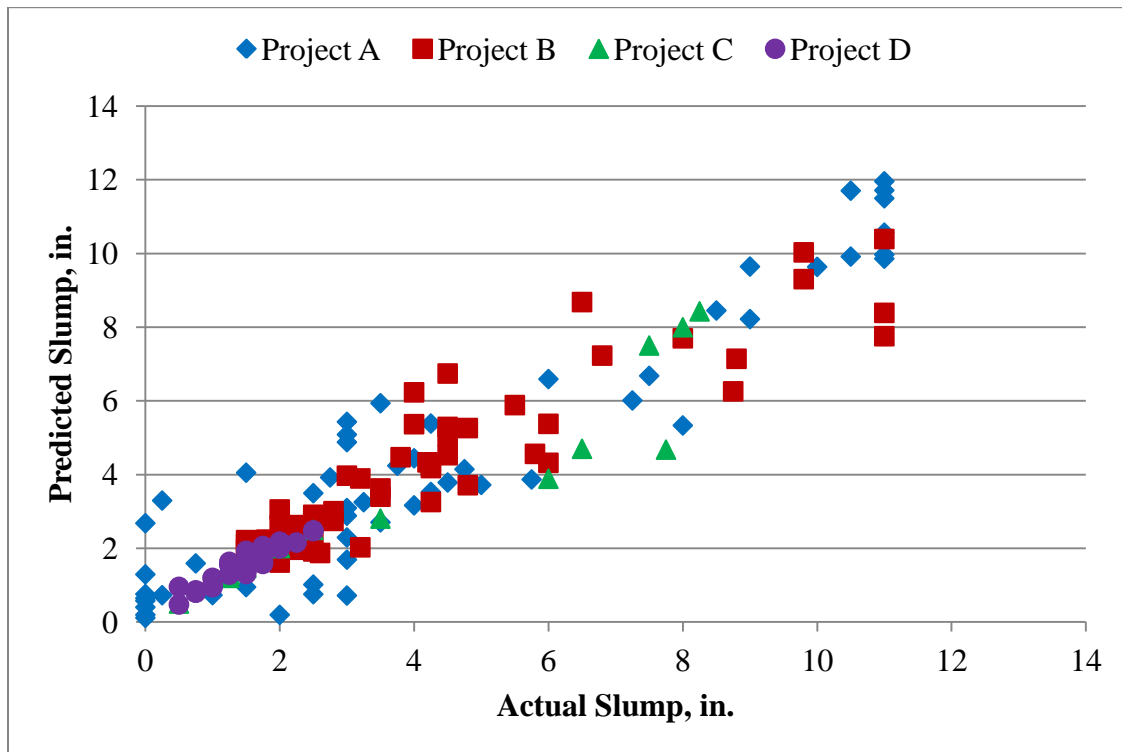


Fig. 10. Prediction of slump for each project provided by Software A.

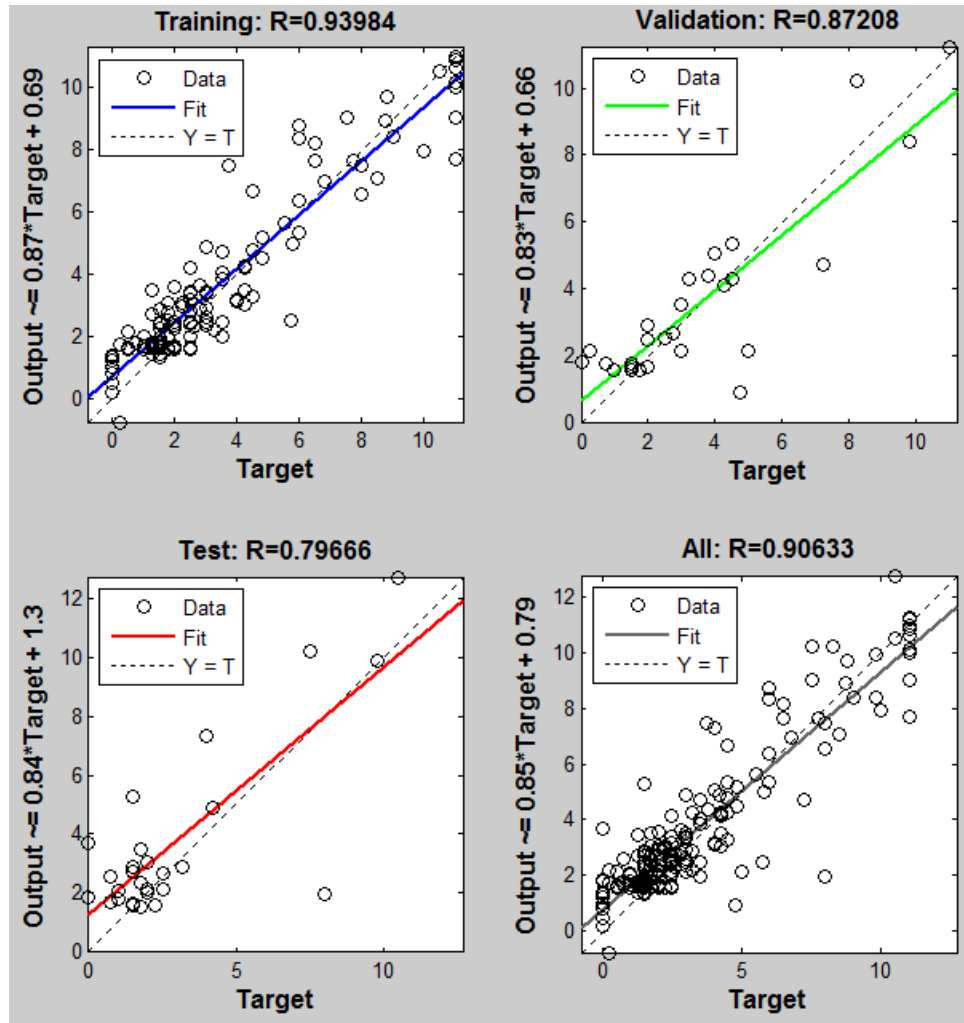


Fig. 11. Prediction of slump based on the combined data set (178 data points) of Projects A, B, C and D provided by Software B.

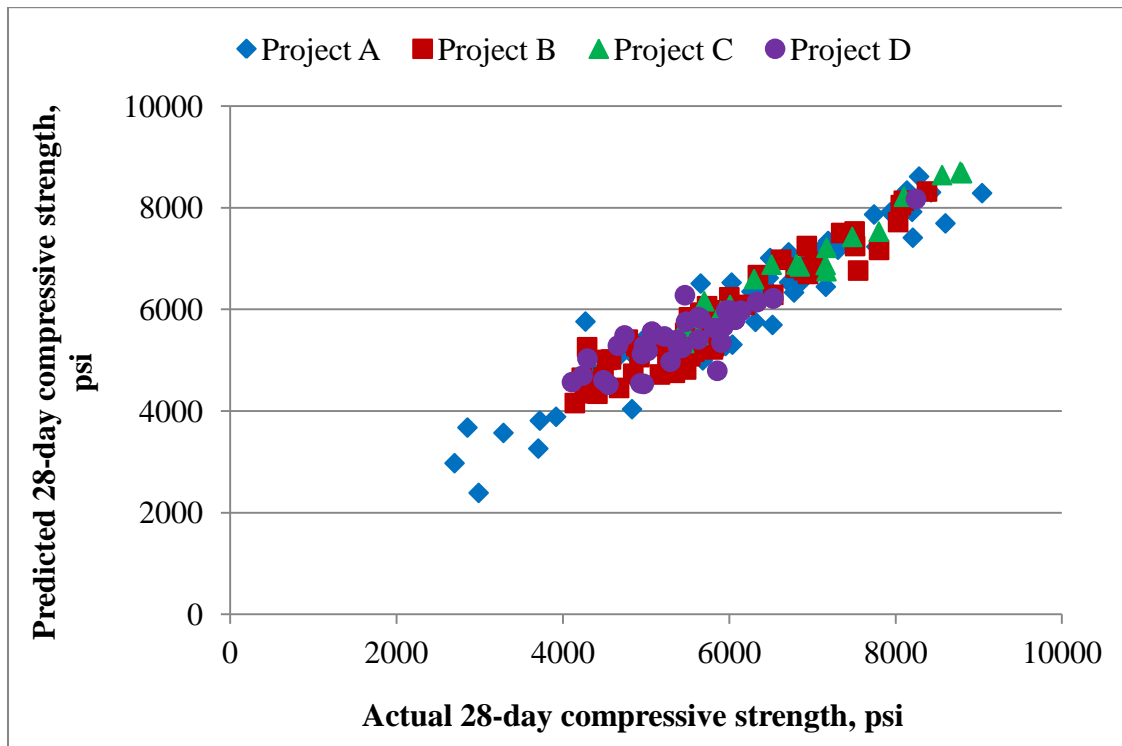


Fig. 12. Prediction of strength for each project provided by Software A.

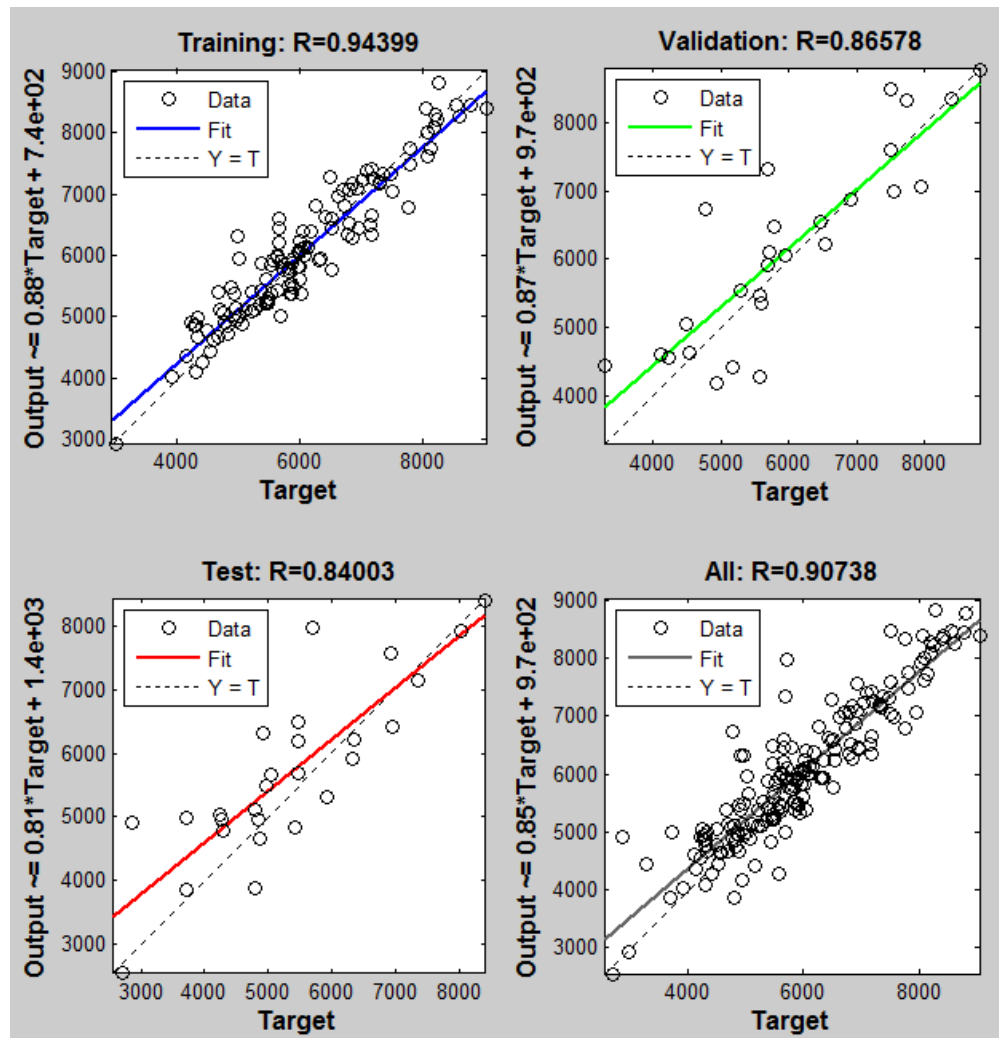


Fig. 13. Prediction of strength on the combined data set (178 data points) of Projects A, B, C and D provided by Software B.

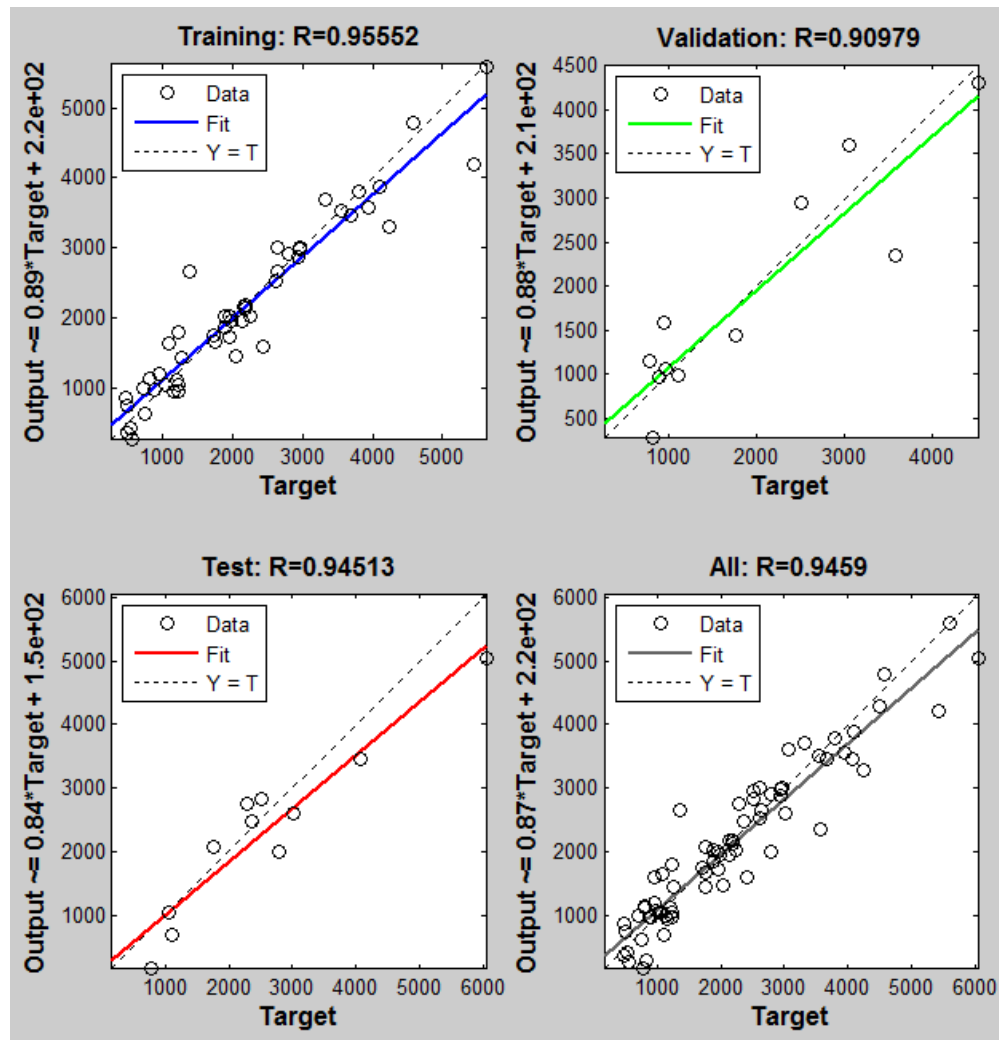


Fig. 14. Prediction of chloride penetration based on the combined data set (72 data points) of Projects A and C provided by Software B.

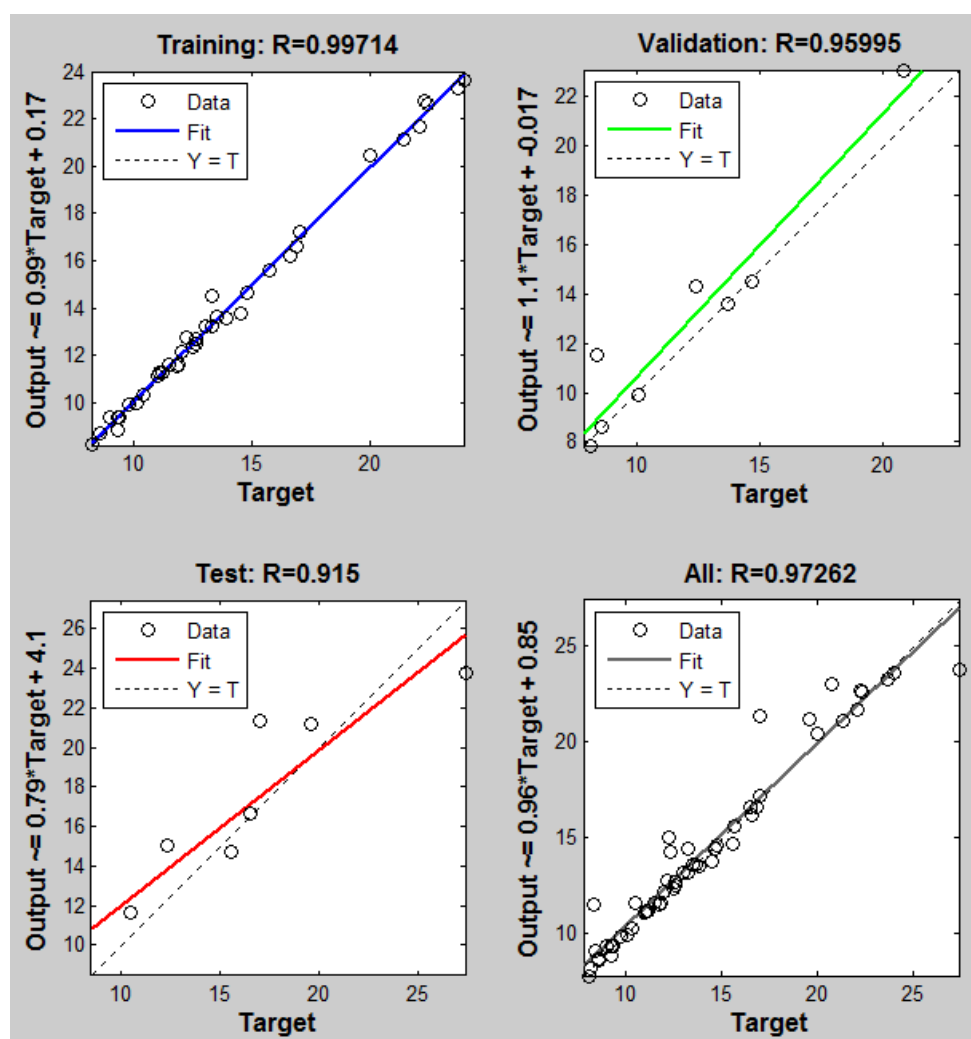


Fig. 15. Prediction of surface resistivity based on 54 data points of Project B provided by Software B.

CHAPTER 5. DISCUSSION AND CONCLUSIONS

As part of the transition between prescriptive-to-performance based specifications, this study aimed to develop a mix proportioning tool that can be used to determine the required concrete components' amount based on the desired fresh and hardened concrete properties.

The work was analyzed under 3 different studies to understand the effect of paste quantity, paste quality and aggregate systems on overall concrete performance. Therefore, in this chapter, the conclusions from each study will be individually presented because it is essential to understand the results in the development of the mix proportioning tool.

The Effect of Paste-to-Voids Volume Ratio on the Performance of Concrete Mixtures (Paste Quantity)

The purpose of this study was to investigate the minimum paste volume required with an appropriate water-to-binder ratio (w/b) to achieve required workability, durability, and strength requirements of concrete mixtures for pavements. Based on the findings of this study, the following conclusions can be drawn:

- Approximately 1.5 times more paste by volume is required than voids between the aggregates to obtain a minimum workability. Below this threshold value, the use of water-reducing admixtures provides little benefit.

- For a given w/b, increasing cementitious content does not significantly improve compressive strength once the critical minimum is reached. The critical value is about twice the voids content of the aggregate system.
- For a given w/b, increasing paste content increases chloride penetrability and air permeability.
- The results suggest the addition of supplementary cementitious materials (SCMs) would help improve workability, long-term strength, and durability in portland cement concrete (PCC) pavements.

These results suggest that the strength and durability of concrete could be improved by optimizing the mixture proportions.

Effect of Water-to-Binder Ratio, Air Content, and Type of Cementitious Materials on Fresh and Hardened Properties of Binary and Ternary Blended Concrete (Paste Quality)

The purpose of this study was to investigate the effect of water-to-binder ratio (w/b), air content, and type of cementitious materials on fresh and hardened properties of binary and ternary blended concrete mixtures in pavements. The following conclusions are made based on the results:

- When w/b was kept constant, increasing air content slightly increased workability.
- The ternary mixtures slightly retarded the setting time consistent with the effects of their ingredients.

- As expected, the compressive strength is strongly influenced by the w/b and air content.
- Binary and ternary mixtures containing Class C fly ash and slag cement exhibited higher compressive strength than the control mixture.
- Increasing w/b slightly decreased the surface resistivity.
- The surface resistivity results of binary and ternary mixtures were equal or higher than the control mixture.
- Surface resistivity is improved by the inclusion of SCMs, especially with Class F fly ash and slag cement.
- The shrinkage of binary and ternary mixture were comparable or less than the control mixture. Changing the SCM type and/or dosage did not significantly affect the shrinkage of binary and ternary mixtures.

Development of a Performance-Based Mix Proportioning Tool Using the Artificial Neural Network Modeling

The objective of this study was to develop a performance-based mix proportioning tool. A statistical artificial neural network (ANN) modeling software was used to account for the effect of all variables on each tested concrete property. The following conclusions are made based on the results:

- Portland cement concrete (PCC) is a complex and heterogeneous material. It is composed of aggregates, cement, and water. Supplementary cementitious materials (SCMs) and chemical admixtures may also be included. Each component has a complex physical and chemical variability. Changing the

characteristics of these materials such as the amount, chemistry, and fineness of cementitious materials; and shape, texture, and gradation of aggregate systems has a high impact on concrete properties. Given the fact that cement hydration is a chemical reaction, these complex variables also have interactions with each other which make concrete behavior even more complex to analyze. Therefore, to understand the overall concrete behavior in-depth, the effect of every component and their interactions with each other should be taken into consideration.

- Based on the performed analysis, predicted slump, strength, and durability were close to the actual tested property. The obtained coefficient of determination (R^2) ranged between 0.750 to 1.0, and the correlation coefficient (R) ranged between 0.906 to 1.0, depending on the complexity of the selected inputs.
- The addition of the aggregates generally tended to decrease the precision of the predictions due to the increased variability of the mix design parameters such as size, shape, texture, and gradation of aggregates.

Based on these findings, it can be concluded that the model provides predictions that are close to the actual numbers only when the selected aggregate systems is used, therefore the concept followed in this study is applicable, only if a similar aggregate system (similar shape, size, texture, and gradation) is chosen.

State of the Art Contributions to Engineering Practice

- This study applied an innovative concept to develop a nationally or universally applicable mix proportioning development tool. Although the current findings are not sufficient enough to develop such a tool, it provides predictions that are close to the actual values when the aggregate systems are similar to the one used in this study. The findings also suggest that this approach is promising and achievable upon calibration of the model.
- As distinct from the other studies, this study applies a new concept by using the parameter of “Paste-to-voids volume ratio ($V_{\text{paste}}/V_{\text{voids}}$) for the first time in concrete pavements.
- There is a perception in the concrete industry that the higher the cement content used; the higher the strength will be achieved. However, the findings of this study showed that this perception is invalid by demonstrating that for a given w/b, increasing cementitious content does not significantly improve compressive strength once a critical minimum is reached. The critical value is about twice the voids content of the aggregate system.
- There is a perception that the use of water-reducing admixtures can help improving the workability at any level. However, the findings of this study showed that this perception is invalid by demonstrating that approximately 1.5 times more paste by volume is required than voids between the aggregates to obtain a minimum workability. Below this threshold value, the use of water-reducing admixtures provides little benefit.

- Findings of this study showed that ternary blended mixtures have the ability to improve concrete performance by improving workability, durability and strength.
- Findings of this study clarified that once adequate paste is obtained to fill the voids and separate the aggregate particles, then the quality of paste, not the quantity, dominates the trends.

Limitations of the Proposed Mix Proportioning Tool

The proposed mix proportioning tool aims to predict the required quantities of locally available materials to achieve the desired fresh and hardened concrete properties.

While it may be desirable to develop so-called “optimum” quantities of ingredients, this concept has to be approached with care. Reports in the literature and the findings of this study showed that there may be no such thing as an “optimum”. This is firstly because the parameters used to define the optimum are not always clearly defined. The second reason is that the lowest cost or lowest binder content mixture to achieve a given potential durability may not be the same as that required to achieve required workability. In such cases the greater amount will be required to avoid compromising overall system performance. Therefore when determining the required amount of materials, it is wise to ensure that workability, durability, and strength are all achieved. Failure to achieve or disregarding one or more of these properties would result in poor pavement performance.

Because of the observed strong influence of the aggregate systems (and likely cementitious chemistry), the mix proportioning tool reported here is limited in its ability to predict actual values at any location with a high level of confidence when a different

aggregate system is used. The approach of using $V_{\text{paste}}/V_{\text{voids}}$ in an ANN system appears to have global application, but the direct model will have to be locally calibrated for materials and required performance parameters.

Summary

The findings of the three studies were combined to analyze the effect of binder systems with different types and content; the paste quality; and size, shape, texture and gradation of different aggregate systems primarily on workability, durability, and strength. A total of 178 mixtures were prepared with had 7 different gradation systems, 12 binder systems, 25 binder contents, 6 different w/b, and 3 different nominal air content. As part of this study, ANN modeling software was used to account for the effect of all variables on each tested concrete property. The following conclusions are made based on the results:

- Approximately 1.5 times more paste by volume is required than voids between the aggregates to obtain a minimum workability.
- For a given w/b, increasing cementitious content does not significantly improve compressive strength once the critical minimum is reached. The critical value is about twice the voids content of the aggregate system.
- For a given w/b, increasing paste content decreases durability by increasing chloride penetrability and air permeability.
- The addition of SCMs helps improve workability, long-term strength, and durability.

- Ternary mixtures overall performed in accordance with their ingredients; however the degree of improvement that they contribute varies based on the selected dosage and type of SCMs.
- Based on the performed ANN analysis, predicted slump, strength, and durability were close to the actual tested property. The obtained coefficient of determination (R^2) ranged between 0.750 to 1.0, and the correlation coefficient (R) ranged between 0.906 to 1.0, depending on the complexity of the selected inputs. However, the addition of the aggregates generally tended to decrease the precision of the predictions due to the increased variability of the mix design parameters such as size, shape, texture, and gradation of aggregates.
- The proposed approach is promising in terms of predicting the values of the tested properties based on the mix design variables. However, considering that the cementitious materials and aggregate characteristics vary depending on the location along with the environmental conditions that the concrete may be exposed to (such as presence of sulfate and deicing chemicals), it is challenging to propose a universally or nationally applicable statistical model that could be used on different projects in various locations.

CHAPTER 6. RECOMMENDATIONS

The proposed mix proportioning tool is promising and achievable in terms of predicting the values of the tested properties based on the mix design variables, or predicting the amount and type of materials needed to achieve the desired concrete properties. Therefore, the findings of this study can be implemented in real time although; the proposed mix proportioning tool is not completely ready for prime time. Considering the complexity of concrete and its components, to increase the applicability of the proposed tool universally with a larger data set, it is recommended that further research will be beneficial that investigates the effect of size, gradation, shape, and texture of aggregate systems as the available data used in this study is not adequately wide enough to establish a reliable relationship using a statistical approach. Upon using this approach with a larger data set, the proposed model will be beneficial for performance-based specifications. If a model is to be useful, more detailed information of materials such as cement chemistry and fineness should be included in it to fully evaluate the effect of cementitious materials on tested properties. Additional tests such as early-age strength, air-void systems, and freeze-thaw should be conducted to determine the effect of concrete components on these properties. Different statistical models may be used to investigate the precision of the applied method.

APPENDIX A. THE APPLICATION OF X-RAY FLUORESCENCE TO ASSESS PROPORTIONS OF FRESH CONCRETE

A paper published in the proceedings of the 10th International Conference on Concrete

Pavements (ISCP), Quebec, Canada

July 8-12, 2012

Ezgi Yurdakul¹; Peter C. Taylor²; and Halil Ceylan³

Abstract

Any transportation infrastructure system is concerned with durability and performance issues. The proportioning and uniformity control of concrete mixtures are critical factors that directly affect the longevity and performance of concrete pavements. Currently, the only means available to monitor mix proportions of any batch are to track batch tickets created at the batch plant. This does not take into account potential errors in loading materials into storage silos, calibration errors, and addition of water after dispatch. Therefore, there is a need for a rapid, cost-effective, and reliable field test that estimates the proportions of as delivered concrete mixtures.

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In addition, performance based specifications will be more easily implemented if there were a way to readily demonstrate whether any given batch is similar to the proportions already accepted based on laboratory performance testing. This paper describes a preliminary investigation into the potential use of a portable x-ray fluorescence (XRF) technique to assess the proportions of concrete mixtures as they are delivered. Tests were conducted on the raw materials, paste and mortar samples using a portable XRF device. There is a reasonable correlation between the actual and calculated mix proportions of the paste samples, but data on mortar samples was less reliable.

Introduction

Performance based specifications need test methods that can prove that a mixture will perform as required. One approach would be to validate and characterize the mixtures before construction starts. All that is needed during construction then is to prove that the mixture delivered is similar to that tested and accepted in the lab.

X-ray fluorescence (XRF) is a method that is used to determine the element concentration of samples (EPA, 2007). This study used an XRF device that is a portable battery powered scanner to analyze elements of cementitious materials, and paste and mortar samples made with those materials. Ideally, using such a device will be cost-effective, especially given the fact that the cost of well-conducted testing and quality control is small when compared to the cost of removing and replacing failed concrete (Broton and Bhatti, 2004).

Proportioning and uniformity of concrete mixtures are critical factors that can directly affect the longevity and performance of concrete pavements (Wang and Hu,

2005; Kropp and Hinsdorf, 1995). At present the only means available to monitor mix proportions of any given batch are to track batch tickets created at the batch plant or to submit hardened samples to a central laboratory for XRF analysis. However, batch tickets do not take into account potential errors in loading materials into storage silos, calibration errors, and addition of water after dispatch. Laboratory XRF analysis is expensive and time consuming. Therefore, there is a need for a rapid, cost-effective, and reliable field test that estimates the proportions of as delivered concrete mixtures.

The literature on applying the XRF technique to concrete samples is limited, particularly with the use of portable devices. Speed, accuracy, and precision are among the advantages of using the XRF technique in analyzing the chemical composition of samples; however the specimen preparation is challenging (Broton and Bhatta, 2004). Accurate quantitative XRF analysis requires a homogeneous and flat surface (Broton and Bhatta, 2004). Field studies have shown that the comparability of the obtained test results with confirmatory samples is mostly affected by the heterogeneity of the sample (EPA, 2007). This is achieved in a central laboratory by grinding and mixing the sample and embedding it in a glass matrix. However, this is not possible when using a portable device. In a field device, the aperture is considerably larger, leading to a reduction in precision but removing the need to prepare a special sample for analysis. It should be noted that concrete is heterogeneous at almost all scales from mm down to nm, including within individual aggregate particles. Obtaining a representative sample for micro analysis is therefore always a challenge.

This paper describes a preliminary investigation into the potential use of a portable XRF technique to assess the proportions of concrete mixtures as they are delivered. Tests were conducted on the raw materials, paste and mortar samples using a portable XRF device. There is a reasonable correlation between the actual and calculated mix proportions of the paste samples, but data on mortar samples was less reliable.

Methodology

Materials

The following materials were obtained:

- ASTM C150 Type I ordinary portland cement
- ASTM C618 Class F fly ash
- ASTM C618 Class C fly ash
- ASTM C989 ground granulated iron blast-furnace slag
- ASTM C1240 silica fume
- No. 4 nominal maximum size concrete sand

Samples Tested

In order to determine the accuracy of the portable XRF device and establish whether there were reasonable correlations between the designed and tested values, the following samples were prepared:

Powder

5 different types of cementitious materials (Type I portland cement, Class C fly ash, Class F fly ash, silica fume, slag cement) were tested to analyze the chemical

compositions of the powder materials. 6-micron polypropylene sheets were used to cover the surface of the materials to minimize contamination (Figure 1).

Fine and Coarse Aggregates

In order to eliminate the effect of moisture, oven-dried fine and coarse aggregates were tested.

Paste

15 paste mixes at w/b ratio of 0.45 were prepared in accordance with ASTM C305. Samples were molded in accordance with ASTM C109. Three cubes (2*2*2-in.) were prepared per mixture. 5 different cementitious materials (Type I portland cement, Class C fly ash, Class F fly ash, silica fume, slag cement) were tested. The supplementary cementitious materials (SCM) replacement levels were fixed at 0, 20, and 40% by mass.

Mortar

15 different mortar mixes at w/b ratio of 0.45 were prepared in accordance with ASTM C305 and tested. Samples were also molded into three cubes (2*2*2-in.) per mixture. The cementitious blends used were the same as those in the paste mixtures. The cementitious to sand ratio was fixed at 1:3 by mass.

Test Procedure

A handheld XRF device (Niton XL3t900GOLDD+ analyzer) was obtained from Thermo Scientific to test and analyze elements. The weight of this portable device is less than 3 lbs. The dimensions are 9.60*9.05*3.75-in.

The device was equipped with a 50 kV x-ray tube with an 8-mm aperture. The measurement time is user-selectable. Up to 30 seconds of testing time is known to be adequate for initial screening whereas longer measurement times (up to 300 seconds) are needed to meet higher precision and accuracy requirements (EPA, 2007). Since the testing period affects the limit of detection, the testing was conducted for 15 minutes per sample to provide sufficient time for a reasonably repeatable analysis.

The electromagnetic radiation of wavelengths of x-rays ranges between 0.1 Å and 20 Å (Broton and Bhatti, 2004). The necessary wavelengths are produced by an x-ray tube in which the electrons are accelerated from an emitting source toward the target material (Broton and Bhatti, 2004). Under radiation from an x-ray source, a sample will emit characteristic X-ray intensities depending on characteristics of the beam, sample elemental concentration, powder particle size distribution, degree of compaction and the compounds in the matrix (Proverbio and Carassiti, 1997). A detector that collects and reports the intensities of the emitted x-rays, that in turn can be used in a calibrated system to determine the relative proportions of elements in the sample.

The tests were conducted on three samples per each mixture and the results were averaged. The prepared paste and mortar samples were tested 1 day after mixing in a hardened condition. The samples were not crushed or powdered prior to analyzing because grounding the samples would not reflect the field conditions.

Sample Placement

X-ray signal decreases as the distance from the source is increased. Therefore, in order to minimize the variations and maintain the same distance between sample and

detector for each sample, the XRF device was attached to its portable test stand for all tests (Figure 2). A cover was used to protect operators during use.

Detecting Elements

The lighter the element, the more difficult it is to detect emitted x- rays. Table 1 presents the elemental limits of detection for a SiO₂ matrix of the portable XRF device. Those elements are the most common elements in cementitious materials that the device could detect.

According to the product specification sheet, the presented limits of detection (LOD) are dependent on the testing time, interferences and level of statistical confidence; and are calculated as three standard deviations (99.7% confidence interval) for each element, using 60-second analysis times per filter. No value was for silicon was provided by the manufacturer because the calibration was based on silicon being one of the major elements under test.

Among the elements listed in Table 1, the percentage of calcium oxide, silica and alumina describe the primary compounds in the cement and significantly affect the hydrated cement properties (Kosmatka et al. 2002).

It was reported that the device does not detect elements lighter than Mg therefore interpretation of the data will have to compensate for the fact that the device is not able to detect hydrogen and oxygen (and so water).

Results and Discussion

Analysis of Raw Materials

The portable XRF device reported the test results as elemental mass percentage. Data was converted into oxides using their atomic weights. Some of the total values add up to more than 100%, and the variance from 100% is an indication of the error of the device. This is normal practice even though compounds in the cement are rarely in oxide form (Kosmatka et al. 2002). The summarized data are shown in Table 2. Only the elements showing non-zero results were utilized in the analyses.

The tested cementitious materials were in the form of powder. The aggregates were oven-dried, but not ground, prior to testing.

The analytical results of the portland cement obtained from the portable XRF were compared with the requirements of ASTM C 150 for Type I portland cement as shown in Table 3.

Comparison of the results between the portable device and the standard shows that the obtained test results are mostly within the expected range. However, the observed SO₃ content reported by the portable device at 8.55% is well above expected levels. In addition, the observed Al₂O₃ and Fe₂O₃ content are slightly higher than the expected levels. The observed SiO₂ is slightly lower than the expected level. This difference may be due to the uniformity of the sample as tested.

Analysis of Fine Aggregate

The oven-dried fine aggregate was tested in a plastic sample cup covered with a 6-micron polypropylene sheet. For comparison, a sample of the fine aggregate sample

was obtained using a riffle splitter and ground to less than 50 micron. This sample was then tested using a laboratory XRF. The results are presented in Table 4.

There was a large difference between the Balance values (the percentage of undetected elements) from the handheld device and laboratory instrument. This difference is unlikely to be a result of the moisture content as both samples were oven-dried.

It should be noted that the fine aggregate sample was crushed to 50 micron before lab testing while the sample tested using the portable device was not ground. Sampling error may therefore account for differences between the two sets of data.

Analysis of Paste

The test results from paste mixtures are presented in Table 5. The percentage of detected elements was decreased in paste mixtures compared to the cementitious materials. The magnitude of the Balance is roughly equivalent to the percentage of water in the mixture.

The solver function in Excel program was used to calculate the proportions of the cementitious materials based on a least differences approach. The solver varied the amount of SCM in each set, compared the calculated oxides with the measured. The total of the cementitious materials, aggregates and water (if considered in the calculation) was fixed as 1 and the SCM dosage was selected as variables. The solver function reported the SCM dosage that yielded the lowest error. The analysis of paste containing 20% F fly ash by using the solver function is presented in Table 6.

Figure 3 presents the relationship between the cumulative error and the calculated SCM content and is a visual representation of the sensitivity of the approach. It is promising that for each of the mixtures there was a clear minimum error. The data sets shown in Figure 3 are for the 40% SCM mixtures.

Figure 4 presents the relationship between the tested and batched SCM contents. The calculated SCM content was based on analysis using only the reported oxides. This figure shows that the portable device provides an adequate correlation between the real mix proportions for both binary and ternary paste mixtures as there is less than 10% of variation between the predicted values and the actual SCM content.

Figure 5 presents the relationship between the tested and actual SCM contents when the presence of the water is included in the calculation. Water in the mixture is dealt with by assuming that the “Balance” in the reported results is a measure of water content. In this case, the results are less promising. When Figure 4 and Figure 5 are compared, it can be observed that the consideration of water increases the prediction error of the mix proportions.

Analysis of Mortar

The test results of mortar mixtures are presented in Table 7. The undetected element percentage is significantly increased, likely due to the presence of moisture, small detection area (8 mm in diameter) and heterogeneity of the tested samples. The XRF device provided sufficient accuracy for finely grounded homogenous powder samples. However, once the heterogeneity of the increased, the percentage of the total detected elements decreased.

Similar to the analysis on paste mixes, the calculated SCM content was predicted by using the solver function, this time including the data from the sand analysis. The solver reported the percentage of sand content to be around 30% by mass which is close to the actual mix. Figure 6 demonstrates the relationship between the tested and actual SCM content. The inclusion of sand increased the error between the predicted and the actual percentages of SCMs.

To assess the effect of potential errors in detecting sand in the mortar sample because of the relatively small field of influence of the x-ray beam, the calculations were repeated with a range of fixed sand contents forced into the model for the 40% SCM mixtures. This relationship is presented in Figure 7. Based on this figure, when assumed sand content is between 12% and 18%, the predicted SCM content is the closest to the actual SCM content. This clearly illustrates a non-representative amount of sand was being detected by the beam.

Assuming a fixed 15% of sand the relationship between the actual and calculated SCM content was calculated as presented in Figure 8. The error between actual and predicted SCM content is lower than the variation when the sand content was not fixed, but is still unacceptably high.

Conclusions and Recommendations

When the paste was tested, the predicted SCM contents were close to the actual batched values. However, once the system was complicated with the addition of fine aggregates, Balance was significantly increased and the predictions did not match the

batched values. The following conclusions, which are limited to the materials used in this study, can be drawn:

1. When water is not included in the model, there is a reasonable correlation between the actual and calculated mix proportions of paste.
2. The percentage of the detected elements was decreased in mortar mixtures compared to the paste mixtures.
3. The accuracy of the approach decreased when the heterogeneity of the system increased.

This approach is not ready for prime time but shows promise if the technology and the models can be refined. More accurate results may be obtained if the model were to be calibrated for a given mix.

Acknowledgment

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The opinions, findings, and conclusions presented here are those of the authors and do not necessarily reflect those of the research sponsors.

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**Table 1. Elemental limits of detection of the Niton XL3t900GOLDD+ analyzer for a
SiO₂ matrix**

Element	Limit of Detection (ppm)
Ba	35
Sr	3
Fe	35
Mn	60
Ti	10
Ca	50
K	40
S	70
P	250
Al	500
Mg	3500

Table 2. Test results of raw materials, %

Oxides	Cement	Std. dev.*	C Ash	Std. dev.*	F Ash	Std. dev.*	Slag	Std. dev.*	Fine Agg.	Std. dev.*	Coarse Agg.	Std. dev.*
CaO	62.95	0.118	26.35	0.059	15.03	0.039	40.86	0.078	10.96	0.014	60.67	0.109
SiO ₂	18.21	0.107	30.88	0.113	49.51	0.128	34.37	0.122	56.75	0.135	4.51	0.062
Al ₂ O ₃	3.67	0.191	15.23	0.234	12.08	0.185	9.93	0.225	3	0.064	1.76	0.136
Fe ₂ O ₃	4.63	0.016	9.4	0.023	11.17	0.024	0.79	0.006	1.42	0.009	0.74	0.006
MgO	3.12	1.169	2.64	1.383	1.29	0.595	8.47	0.909	3.34	0.595	0	1.902
K ₂ O	0.77	0.011	0.42	0.007	2.11	0.014	0.41	0.007	0.61	0.006	0.47	0.008
SO ₃	8.55	0.042	5.14	0.030	2.52	0.020	4.69	0.032	0.35	0.010	0.94	0.020
TiO ₂	0.16	0.003	1.57	0.008	0.8	0.005	0.41	0.003	0.06	0.002	0.07	0.002
BaO	0.03	0.002	0.57	0.003	0.47	0.003	0.05	0.002	0.04	0.001	0.02	0.002
SrO	0.03	0.032	0.28	0.001	0.21	0.001	0.03	0.001	0.02	0.001	0.02	0.001
Mn ₂ O ₃	0.48	0.009	0.06	0.004	0.11	0.006	0.38	0.007	0.16	0.006	0.02	0.004
Total	102.61		92.53		95.3		100.4		76.7		69.23	

Table 3. The comparison of the test results obtained from the portable XRF and the ASTM C150 recommended chemical composition range

Chemical Composition, %	ASTM C150 min- max	XRF Portable Device
SiO₂	18.7-22.0	18.21
Al₂O₃	4.7-6.3	3.67
Fe₂O₃	1.6-4.4	4.63
CaO	60.6-66.3	62.95
MgO	0.7-4.2	3.12
SO₃	1.8-4.6	8.55

**Table 4. Fine aggregate test result comparison between portable device and XRF
core scanner, %**

Oxides	Desktop	Portable
CaO	8.91	10.96
SiO₂	65.24	56.75
Al₂O₃	7.26	3.00
Fe₂O₃	1.77	1.42
MgO	3.04	3.34
K₂O	1.47	0.61
Na₂O	1.95	
SO₃	0.21	0.35
TiO₂	0.11	0.06
BaO	0.02	0.04
SrO	0.03	0.02
Mn₂O₃	0.06	0.16
P₂O₅	0.07	0.00
LOI	9.30	
Balance	0.55	23.30
Total	99.45	76.70

Table 5. Test results of paste, %

Oxides	OPC	20 F	40 F	20 C	40 C	20 SL	40 SL	20F 20SL	10F 20SL
CaO	48.45	40.44	35.03	44.42	36.45	44.73	41.21	37.83	41.90
SiO₂	14.68	19.99	23.32	19.41	18.45	18.92	20.75	22.82	22.03
Al₂O₃	2.32	4.04	4.77	5.21	6.09	3.70	3.96	4.64	4.76
Fe₂O₃	3.29	4.19	5.21	4.14	4.60	2.73	2.17	3.69	3.26
MgO	0.00	0.00	0.00	2.71	1.71	2.46	2.21	1.79	2.46
K₂O	0.93	1.35	1.21	0.48	0.62	1.03	0.90	1.12	1.01
SO₃	6.96	5.37	5.00	4.69	6.00	4.81	4.96	4.65	4.50
TiO₂	0.12	0.21	0.30	0.36	0.55	0.16	0.19	0.25	0.21
BaO	0.03	0.09	0.15	0.11	0.17	0.03	0.03	0.09	0.06
SrO	0.02	0.05	0.07	0.06	0.09	0.02	0.02	0.05	0.03
Mn₂O₃	0.33	0.28	0.23	0.28	0.21	0.32	0.30	0.27	0.31
P₂O₅	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Balance	22.87	24.00	24.69	18.14	25.06	21.09	23.30	22.78	19.48
Total	77.13	76.00	75.31	81.86	74.94	78.91	76.70	77.22	80.52

Table 6. Example of the solver function, %

	Cement	F ash	C ash	Slag cement	Total
Measured	0.8	0.2	0	0	1
Theory (solver)	0.81	0.19			1
	Oxides		Theory	Measured	Delta
	CaO		47.32	44.42	2.90
	SiO ₂		13.69	19.41	5.72
	Al ₂ O ₃		2.76	5.21	2.45
	Fe ₂ O ₃		3.48	4.14	0.66
	MgO		2.34	2.71	0.37
	K ₂ O		0.58	0.48	0.10
	SO ₃		6.42	4.69	1.74
	TiO ₂		0.12	0.36	0.23
	BaO		0.02	0.11	0.08
	SrO		0.02	0.06	0.03
	Mn ₂ O ₃		0.36	0.28	0.08
	P ₂ O ₅		0.00	0.00	0.00
	Difference				14.36

Table 7. Test results of mortar mixtures, %

Oxides	OPC	20 F	40 F	20 C	40 C	20 SL	40 SL	20F20SL	10F20SL
CaO	35.57	31.40	27.75	32.94	29.33	33.68	32.55	29.72	32.25
SiO₂	13.44	17.07	20.56	15.79	17.87	16.16	17.41	19.69	17.86
Al₂O₃	1.66	2.54	3.25	3.18	4.63	2.44	2.87	3.18	2.90
Fe₂O₃	2.48	2.97	3.65	2.90	3.38	2.03	1.95	2.66	2.36
MgO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
K₂O	0.34	0.51	0.82	0.60	0.38	1.00	0.44	0.99	0.52
SO₃	4.96	4.59	3.80	4.75	4.62	4.22	3.98	3.22	3.89
TiO₂	0.09	0.16	0.22	0.24	0.41	0.11	0.14	0.18	0.15
BaO	0.04	0.07	0.08	0.07	0.09	0.04	0.04	0.06	0.05
SrO	0.02	0.03	0.04	0.03	0.05	0.02	0.02	0.03	0.02
Mn₂O₃	0.21	0.18	0.16	0.18	0.15	0.22	0.20	0.18	0.20
P₂O₅	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Balance	41.17	40.49	39.67	39.33	39.09	40.08	40.39	40.08	39.80
Total	58.83	59.51	60.33	60.67	60.91	59.92	59.61	59.92	60.20



Figure 1. Sampling of powder materials.

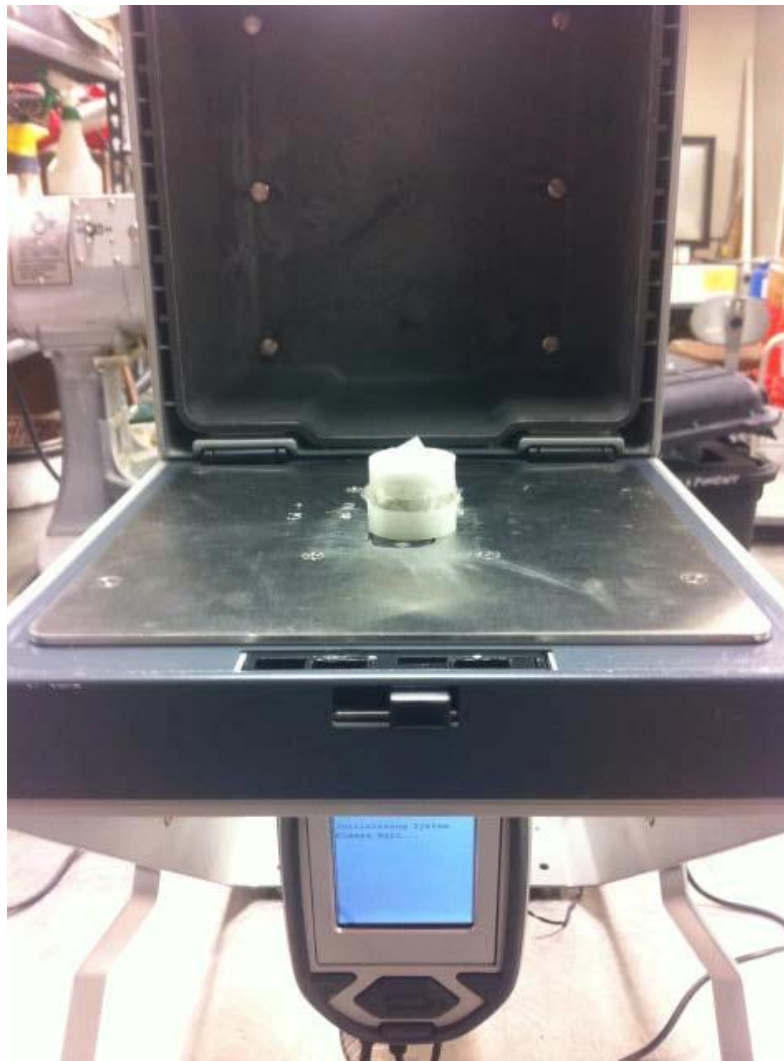


Figure 2. Portable test stand.

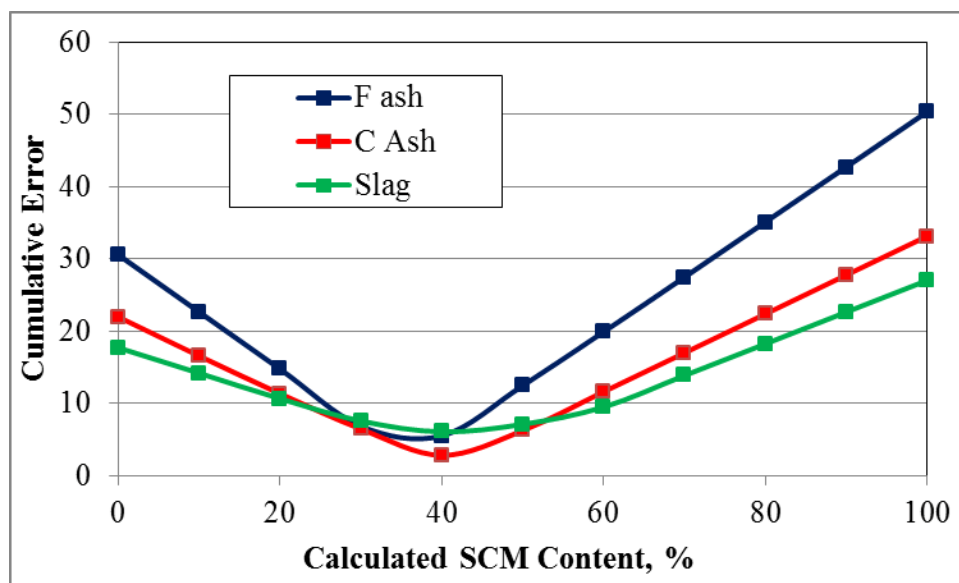


Figure 3. The relationship between cumulative error and calculated SCM content.

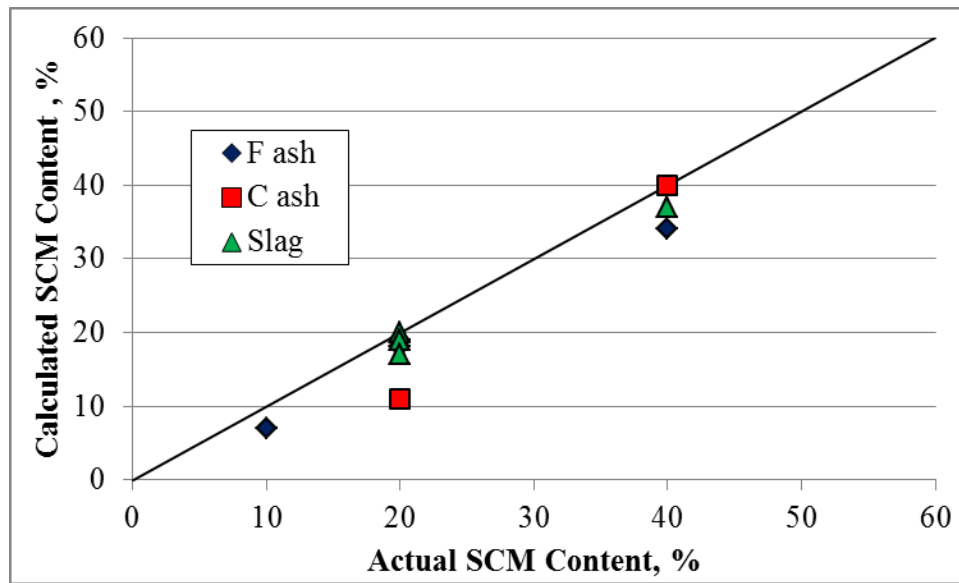


Figure 4. The relationship between tested and designed SCM content.

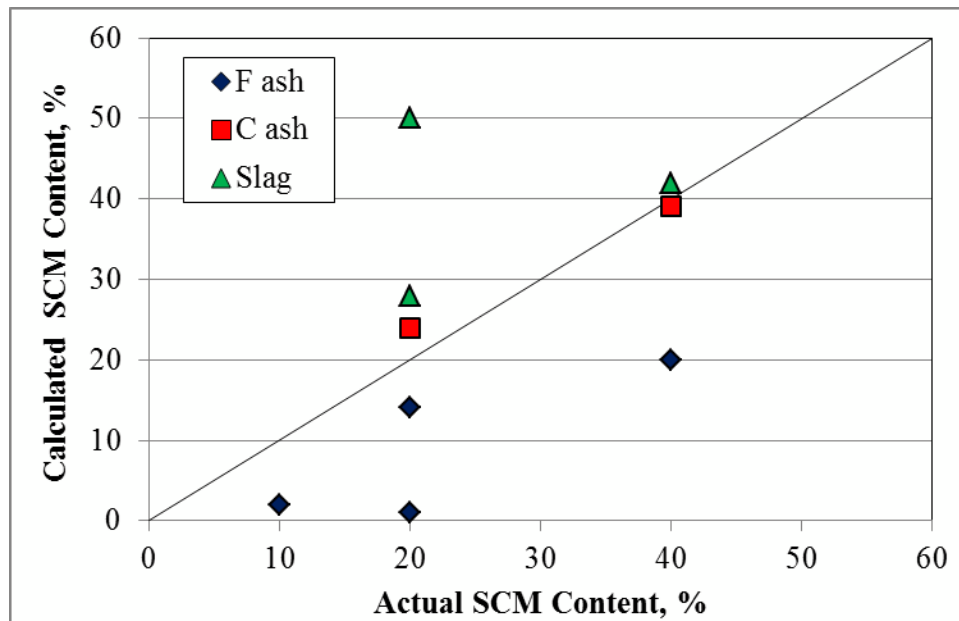


Figure 5. The relationship between tested and actual SCM content when the water presence is included.

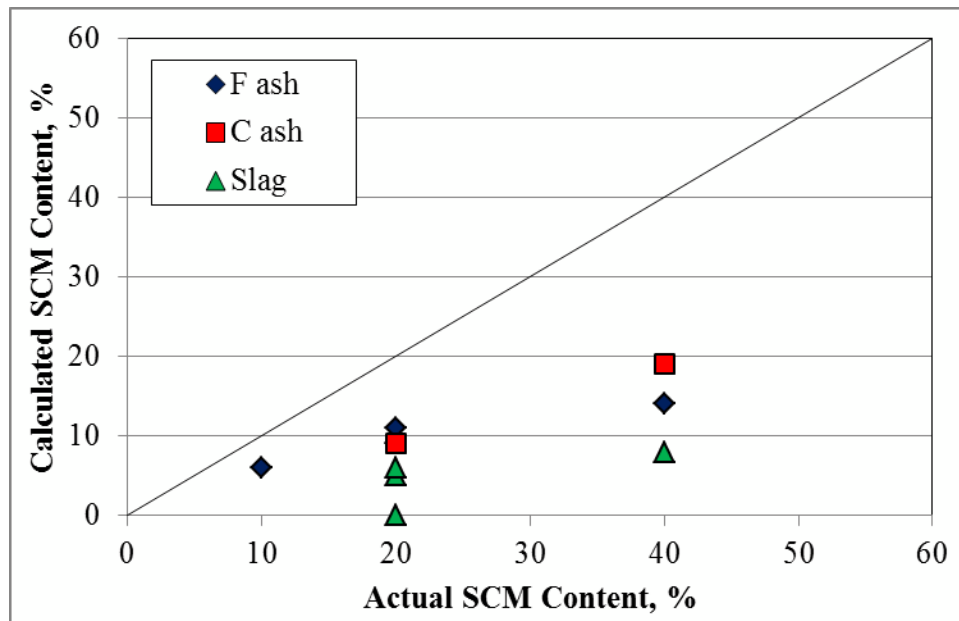


Figure 6. The relationship between tested and designed SCM content.

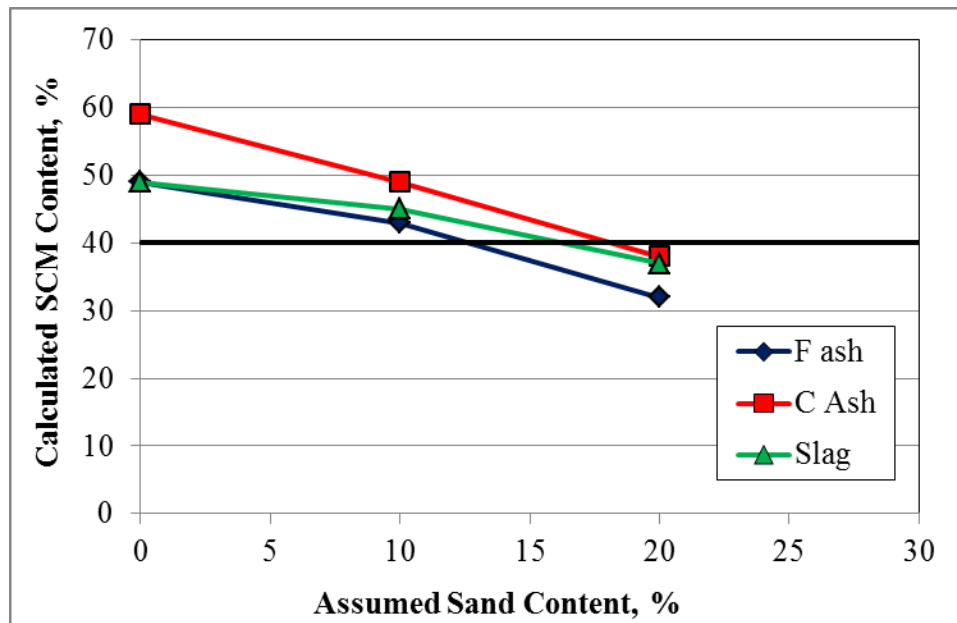


Figure 7. The calculated SCM content for varying sand contents forced into the model.

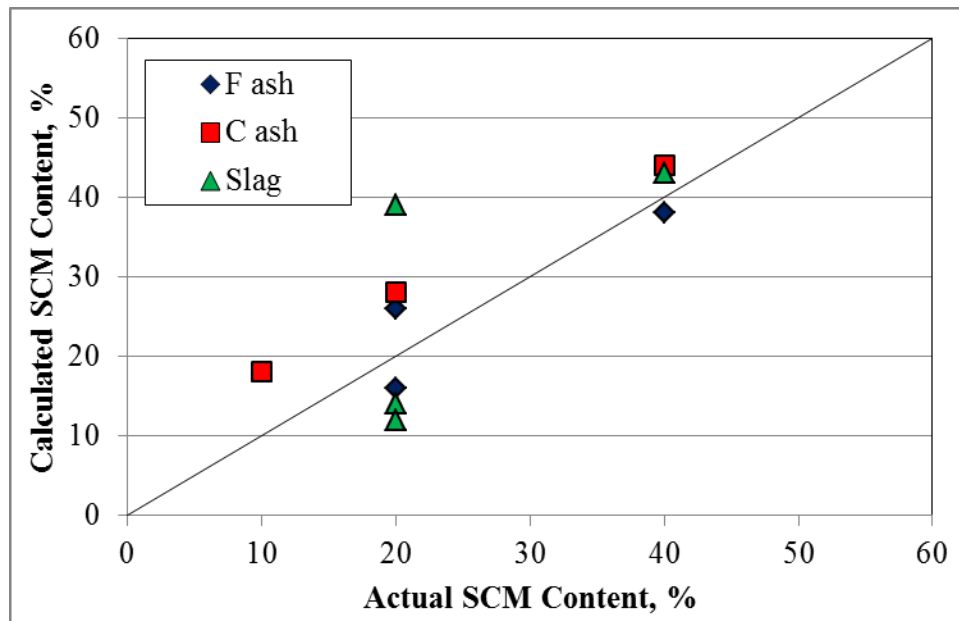


Figure 8. The relationship between tested and designed SCM content.

APPENDIX B. MINIMIZING CEMENTITIOUS CONTENT FOR PERFORMANCE AND SUSTAINABILITY IN RIGID PAVEMENTS

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Abstract

The production of each ton of portland cement clinker emits approximately 1 ton of carbon dioxide. Therefore, there is a demand on the industry to reduce its carbon footprint by environmental agencies. A solution for the concrete construction industry is to use cement more efficiently. However, many concrete specifications impose minimum cementitious contents that may be in excess of those required to achieve desired durability and strength, leading to increased costs and increased carbon loading on the environment. In addition, in some cases, excessive cementitious content adversely affects concrete performance and durability by causing shrinkage related cracking. Therefore, minimizing the cementitious amount will not only reduce the cost but also lead to a more sustainable method of constructing performance-based rigid pavements.

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The main purpose of this research is to investigate the minimum cementitious content required with an appropriate water to cementitious ratio (w/cm) to meet given workability, strength, and durability requirements in a rigid pavement; and so to reduce carbon dioxide emissions, energy consumption, and cost.

This paper presents an experimental program that was conducted on 32 concrete mixtures with w/cm ranging between 0.35 and 0.50; and cementitious contents ranging from 400 to 700 pcy. 16 mixtures were prepared using ASTM Type I ordinary portland cement and 16 contained ASTM C618 class C fly ash at 20% of portland cement replacement level. Hardened concrete properties such as compressive strength, chloride penetration and air permeability were determined up to 90 days. The test results showed that strength is a function of w/cm and independent of the cementitious content after a certain cementitious content is reached, for a given w/cm . Chloride penetration increases as w/cm or cementitious content increases. At constant w/cm , air permeability also increases as cementitious content increases. For the aggregate system used in this work, 500 pcy is found to be the most appropriate cementitious content that provides a workable mixture whilst meeting the desired strength and durability performance of mixtures. Based on these findings, it is possible to reduce the use of cementitious content without sacrificing the desired strength and durability.

Introduction

Cement, the main component of concrete, is a common material used in all kinds of construction. Cement content is perceived to control concrete strength. Based on this perception, a minimum cement content is often specified that may exceed the amount needed to achieve the desired strength and durability. However, once the cement content reaches an optimum value, using more cement does not achieve higher strength (Nawy, 2008). In some cases, excessive cement content adversely affect concrete performance and durability by causing shrinkage and cracking. Therefore, this excessive amount should be minimized not only for improving the performance of rigid pavements but also to prevent its negative impact on costs and environment because:

- cement is the most expensive component in concrete.
- each ton of portland cement clinker production emits approximately 1 ton of carbon dioxide (Mehta, 1999).
- cement production emits approximately 5% of global carbon dioxide (CO₂) and 5% of global energy consumption (Hendriks et al., 2004)..

The findings of this research will indicate ways to use cement more efficiently by showing that strength is largely independent of cementitious content after a certain cementitious content is reached, for a given w/cm. These findings will have an impact on the concrete pavement industry because minimizing the cementitious content in concrete will not only reduce costs but also may lead to more sustainable methods for rigid pavements.

Methodology

Materials

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

- ASTM C150 Type I ordinary portland cement
- ASTM C618 Class C fly ash
- 1-inch nominal maximum size crushed limestone
- No. 4 nominal maximum size concrete sand
- ASTM C494 Type F polycarboxylate based high range water reducer (HRWR)

Test Variables

This test program includes 32 concrete mixtures with w/cm ranging between 0.35, 0.40, 0.45 and 0.50; and cementitious content ranging from 400, 500, 600 and 700 pcy. 16 mixtures were prepared using ASTM Type I ordinary portland cement and 16 contained ASTM C618 class C fly ash at 20% of portland cement replacement level. Fine and coarse aggregates were combined at a ratio of 1:1.38 to maintain the same void content for all the mixtures. If necessary, slump measured by ASTM C143, was adjusted to a minimum of 2-in. using a high range water reducer. No air entraining admixture was used.

Test Procedures

4×8-in concrete cylinders were cast in accordance with ASTM C31. Compressive strength was determined at 1, 3, 28 and 90-days according to ASTM C39. 2- and 1-in. thick disks were cut from cylinders for chloride penetration resistance tests (ASTM

C1202), and air permeability tests, based on the University of Cape Town Method (Alexander et al., 2007) respectively. Due to the high permeability of concrete samples at early ages, the rapid chloride penetration test could not be conducted on samples at 1 and 3-days. Therefore, only 28 and 90-day test results are presented. Air permeability was determined at the age of 1, 3, 28 and 90-days.

Results and Discussion

Table 1 presents the test results of 32 concrete mixtures.

Compressive Strength

Compressive strength test was performed on all 32 mixtures at 1, 3, 28 and 90-days (Table 1).

Plain Concrete

As shown in Figure 1, for a given w/cm of 0.40, after strength reaches its maximum value at 500 pcy of cementitious content, further increasing cementitious amount does not increase the compressive strength at any age. This statement also applies to the mixtures with w/cm of 0.35 and 0.45 as similar results and trends were obtained.

Strength increased when cementitious content was increased from 400 pcy to 500 pcy, because in order to obtain a certain degree of strength, an adequate amount of paste is required. Especially at mixtures having very low w/cm, this trend is more obvious.

The compressive strength values in Figure 2 are within the 9% error band (ASTM C39) as presented therefore when the overall values and trends are evaluated, these trends may be considered as roughly straight lines. This indicates that for concretes

having high w/cm, strength is not affected by increasing the cementitious content for both portland cement concrete and concrete with Class C fly ash.

Concrete with Class C Fly Ash

Figures 1 and 2 also show the effect of adding Class C fly ash on compressive strength. At later ages, mixtures containing Class C fly ash show an overall higher compressive strength than mixtures without fly ash. However, at early ages (1 and 3-day), mixtures with Class C fly ash show lower compressive strength than mixtures with only portland cement. As expected, the addition of Class C fly ash contributes to the strength gain, because of its slow pozzolanic reactivity, the strength development at early ages is less than normal portland cement concrete.

Figure 3 demonstrates the effect of w/cm on 1-day compressive strength. Overall, for a given cementitious content, increasing w/cm decreases compressive strength. This result is consistent with the literature that strength is a function of w/cm, when cementitious content is kept constant.

Mixtures with 400 pcy of cementitious content do not follow the decreasing trend when w/cm was increased from 0.35 to 0.40 due to the workability, consolidation and compaction problems associated with inadequate paste content in mixtures at low w/cm.

Based on these findings, when w/cm is constant, for the aggregate system used in this study, 500 pcy is found to be the most appropriate cementitious content when desired workability and strength are considered for rigid pavements.

Chloride Penetration

Rapid chloride penetration test was performed on all 32 mixtures at 28 and 90-days (Table 1). Testing for the cementitious content at 400 pcy could not be conducted on plain concrete mixtures due to their low paste content causing severe consolidation and honeycombing problems at all w/cm levels.

In this research study, the observed test results were compared with the values given by the ASTM C1202 standard (Table 2).

According to Figure 4, as the curing time is extended from 28 days to 90 days, the chloride penetration resistance of all mixtures are increased thus less chloride ions penetrate through the concrete samples. This finding is in accordance with the literature (Naik et al., 1994; Naik et al., 1996; Thomas and Bamforth, 1999; Hassan et al., 2000; Khatib and Mangat, 2002). Overall, for all mixtures, increasing cementitious content increases the chloride penetration. It is likely that chlorides penetrate the paste faster than the aggregate. Therefore, the increased chloride penetration may be due to the increased paste content which will increase the volume of material able to transmit chlorides. The coefficient of variation of this test method is stated as 12.3% for single operator (ASTM C1202 standard). Given this high variability and obtaining similar results between the plain concrete and mixtures with Class C fly ash, for given age and cementitious content; according to the presented data, a certain statement cannot be provided regarding the effect of using Class C fly ash on chloride penetration. However, there is information stated in the literature (Thomas and Bamforth, 1998; Khatib and

Mangat, 2002) that the reduction rate of chloride ion penetration of concrete containing Class C fly ash over time is significantly higher than the portland cement concrete.

The concrete mixtures with w/cm of 0.35, 0.40 and 0.45 show similar results as the mixtures with w/cm of 0.50 that chloride penetration decreases when concrete age increases; and increasing cementitious content increases the chloride penetration.

According to Figure 5, when the cementitious content is kept constant, increasing w/cm also increases the chloride penetration because the capillary porosity increases.

Given the fact that increasing cementitious content further increases the chloride penetration, minimizing cementitious content will be efficient for both improving the performance and, reducing the cost (i.e., replacing more expensive paste with cheaper aggregate). However, inadequate amounts of cementitious materials at 400 pcy caused consolidation problems. Therefore, when w/cm is constant, for the aggregate system used in this study, 500 pcy is found to be the most appropriate cementitious content (provides less than 4000 coulombs of chloride penetration for all tested w/cm levels) regarding the desired workability and chloride penetration resistance for rigid pavements.

Air Permeability

Air permeability index is the negative log of the Darcy coefficient of permeability (m/s) and uses a log scale (Buenfeld and Okundi, 1998). Thus, lower air permeability index indicates higher permeability. The air permeability coefficients are affected by the changes in curing duration, test age and concrete composition (Dinku and Reinhardt, 1997).

Plain Concrete

Test results are presented in Table 1. Air permeability test could not be performed on plain concrete mixtures with 400 pcy of cementitious content because their low paste content caused high porosity.

According to Figure 6, for a w/cm of 0.35, increasing cementitious content from 400 pcy to 500 pcy decreases air permeability because mixtures having low cementitious content at low w/cm have inadequate paste content, thus high porosity. However, the rest of the mixtures exhibit such a trend that when w/cm is constant, increasing cementitious content increases permeability because similarly to the chloride penetration, air tends to penetrate through the less dense system. In addition, permeability decreases as concrete age increases. This result is expected because cement hydration continues over time and as hydration continues the pore sizes get smaller and concrete becomes more impermeable.

Concrete with Class C Fly Ash

According to the overall trends presented in Figure 7, for a given w/cm of 0.40, increasing cementitious content increases air permeability. In addition, permeability decreases as concrete age increases. This result confirms the previous findings in the literature (Naik et al., 1994). At early ages, plain concrete mostly exhibited higher resistance against air permeability than mixtures with Class C fly ash. However, at later ages, concretes with Class C fly ash became less permeable than plain concrete because the permeability resistance gain rate of Class C fly ash mixes is higher than plain concrete, within time. This information is also consistent with the literature (Naik et al.,

1994; Hassan et al., 2000). Mixtures with w/cm of 0.45 and 0.50 also showed similar results as the mixtures with w/cm of 0.40.

Therefore, when w/cm is constant, for the aggregate system used in this study, 500 pcy is found to be the most appropriate cementitious content that is able to be consolidated and provides the desired air permeability resistance for rigid pavements.

Conclusions

Based on the obtained test results, following conclusions can be drawn:

1. Strength is a function of w/cm and independent of the total cementitious content: for a given aggregate system, there is a certain paste content after which strength does not change significantly.
2. Chloride penetration increases as w/cm or cementitious content increases, when one parameter is fixed.
3. For a given w/cm, air permeability increases as cementitious content increases.
4. When w/cm is constant, for the aggregate system used in this study: to design for sustainable, workable, and performance-based rigid pavements, 500 pcy is found to be the most appropriate cementitious content beyond which strength does not improve significantly and permeability (i.e., chloride ion and air) is adversely affected.
5. The results suggest that the 20% Class C fly ash replacement would help improve workability, long-term strength, and durability in rigid pavements.

These results suggest that the strength and durability of concrete could be improved by optimizing the mixture proportions. However, further research is needed to investigate the different aggregate gradation systems on concrete performance.

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Table 1. Hardened concrete properties

No	Cement (pcy)	C Ash (pcy)	Water (pcy)	w/cm	Strength (psi)				RCP (coulombs)		API (log value)			
					1- day	3- day	28- day	90- day	28- day	90- day	1- day	3- day	28- day	90- day
1	400		140	0.35	1,120	2,467	3,919	4,573	N/A	N/A	N/A	N/A	N/A	N/A
2	320	80	140	0.35	956	2,342	4,950	6,091	N/A	1,256	9.27	9.32	10.09	10.15
3	500		175	0.35	3,909	6,403	8,208	9,520	1,199	1,208	10.61	10.81	10.61	11.34
4	400	100	175	0.35	2,078	4,369	7,938	9,251	1,748	1,019	10.21	10.57	10.93	11.17
5	600		210	0.35	3,930	5,640	8,427	9,532	1,770	1,392	10.45	10.81	11.06	11.29
6	480	120	210	0.35	2,295	4,482	6,782	7,983	2,034	1,080	9.98	10.42	10.69	10.98
7	700		245	0.35	3,907	5,337	8,137	8,986	1,980	1,533	10.22	10.66	10.92	11.03
8	560	140	245	0.35	2,978	5,289	7,742	9,265	2,131	1,102	10.08	10.48	10.53	10.78
9	400		160	0.40	1,584	2,886	4,314	5,284	N/A	N/A	N/A	N/A	N/A	N/A
10	320	80	160	0.40	1,762	3,740	7,162	7,463	1,710	955	10.25	10.50	10.91	11.16
11	500		200	0.40	2,410	3,714	7,947	9,185	1,547	1,307	10.34	10.46	11.00	11.48
12	400	100	200	0.40	2,096	3,690	7,308	9,325	2,185	824	9.96	10.26	10.76	10.79
13	600		240	0.40	2,744	4,099	6,492	7,840	2,505	2,206	10.12	10.51	10.70	10.70
14	480	120	240	0.40	2,218	4,049	7,317	8,655	2,635	1,650	10.00	10.16	10.51	10.88
15	700		280	0.40	2,901	4,327	6,715	7,977	2,511	1,938	9.93	10.45	10.56	10.31
16	560	140	280	0.40	2,359	4,014	7,364	8,673	3,023	2,328	9.96	10.27	10.56	10.55

Table 1. Hardened concrete properties (cont.)

No	Cement (pcy)	C Ash (pcy)	Water (pcy)	w/cm	Strength (psi)				RCP (coulombs)		API (log value)			
					1-day	3-day	28- day	90- day	28- day	90- day	1-day	3- day	28- day	90- day
17	400		180	0.45	1,962	3,362	4,793	4,690	N/A	N/A	N/A	N/A	N/A	N/A
18	320	80	180	0.45	1,530	3,141	4,272	5,348	N/A	N/A	N/A	N/A	N/A	N/A
19	500		225	0.45	1,649	3,729	6,521	7,541	2,626	N/A	10.01	10.40	10.65	10.67
20	400	100	225	0.45	1,283	2,826	6,086	6,922	2,951	1,488	9.56	10.14	10.43	10.65
21	600		270	0.45	2,311	3,868	5,960	7,171	3,677	2,063	9.88	10.44	10.70	10.56
22	480	120	270	0.45	1,943	3,378	6,780	8,108	N/A	2,553	9.69	10.28	10.68	10.69
23	700		315	0.45	2,197	3,519	5,693	6,775	3,540	2,854	9.85	10.30	10.66	10.44
24	560	140	315	0.45	2,080	3,381	6,855	7,919	N/A	3,576	9.64	10.15	10.58	10.46
25	400		200	0.50	1,947	3,370	4,876	5,262	N/A	N/A	N/A	N/A	N/A	N/A
26	320	80	200	0.50	1,222	2,709	5,570	7,094	2,790	1,083	9.38	9.98	10.73	10.99
27	500		250	0.50	1,950	3,225	5,849	6,934	3,062	2,561	10.33	10.21	10.62	10.66
28	400	100	250	0.50	1,335	2,651	5,957	6,852	2,937	1,355	9.25	9.93	10.44	10.66
29	600		300	0.50	1,897	2,978	5,475	5,912	4,104	2,566	9.62	10.15	10.52	10.24
30	480	120	300	0.50	1,546	2,874	6,039	7,590	4,077	2,757	9.53	9.98	10.44	10.47
31	700		350	0.50	1,708	2,747	4,915	6,589	6,050	4,259	8.33	8.95	9.93	10.42
32	560	140	350	0.50	1,206	2,712	5,560	7,127	4,510	4,610	9.38	8.41	10.28	10.39

Table 2. Chloride penetration based on charge passed (ASTM C1202)

Charge Passed (coulombs)	Chloride Penetration
> 4,000	High
2,000 – 4,000	Moderate
1,000 – 2,000	Low
100 – 1,000	Very Low
<100	Negligible

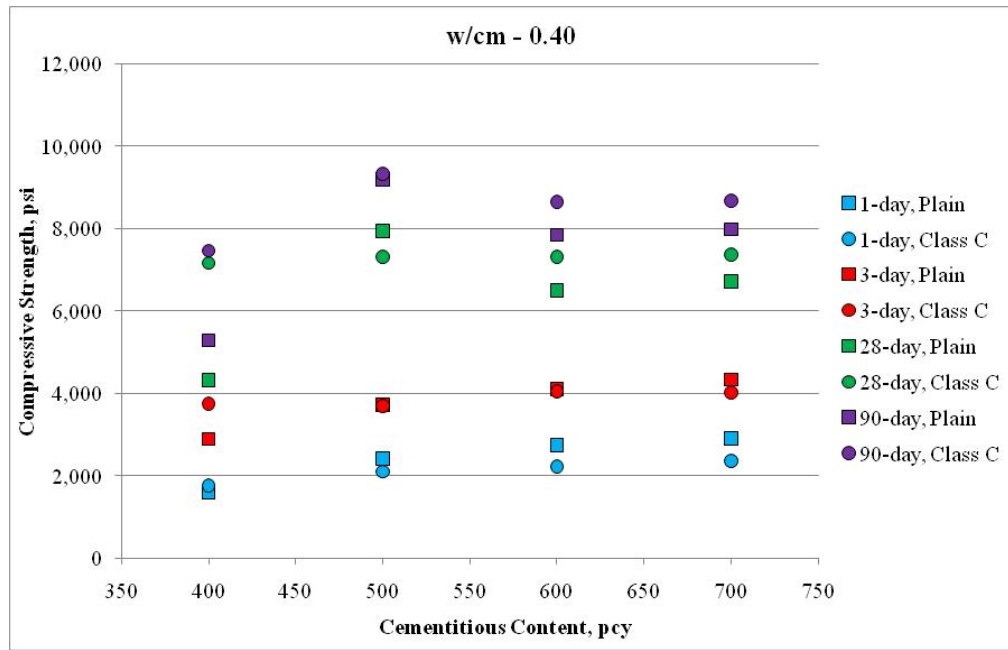


Figure 1. Effect of cementitious content on compressive strength for w/cm of 0.40.

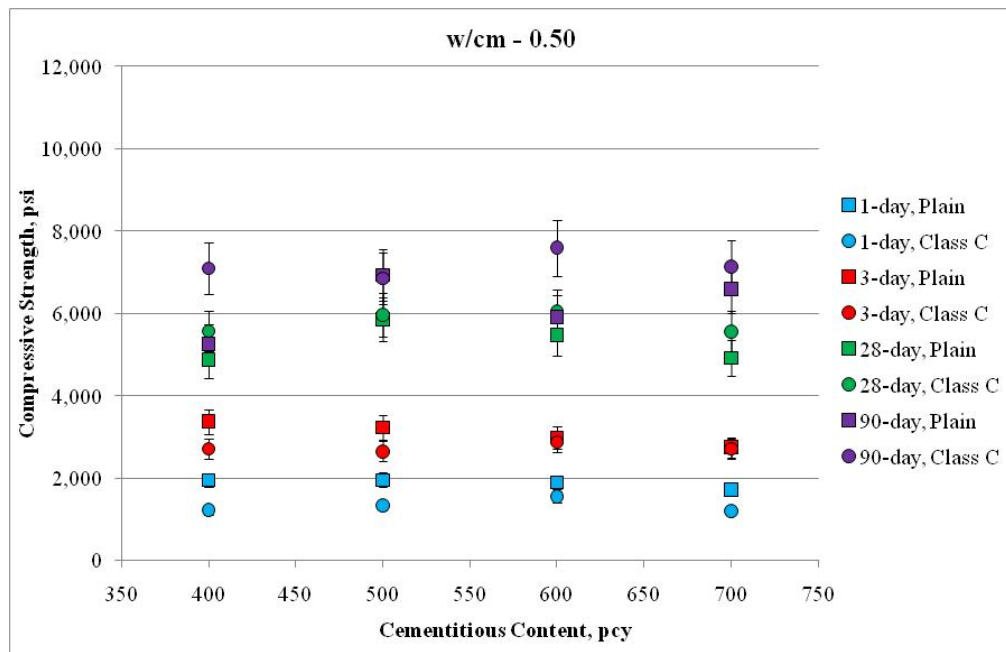


Figure 2. Effect of cementitious content on compressive strength for w/cm of 0.50.

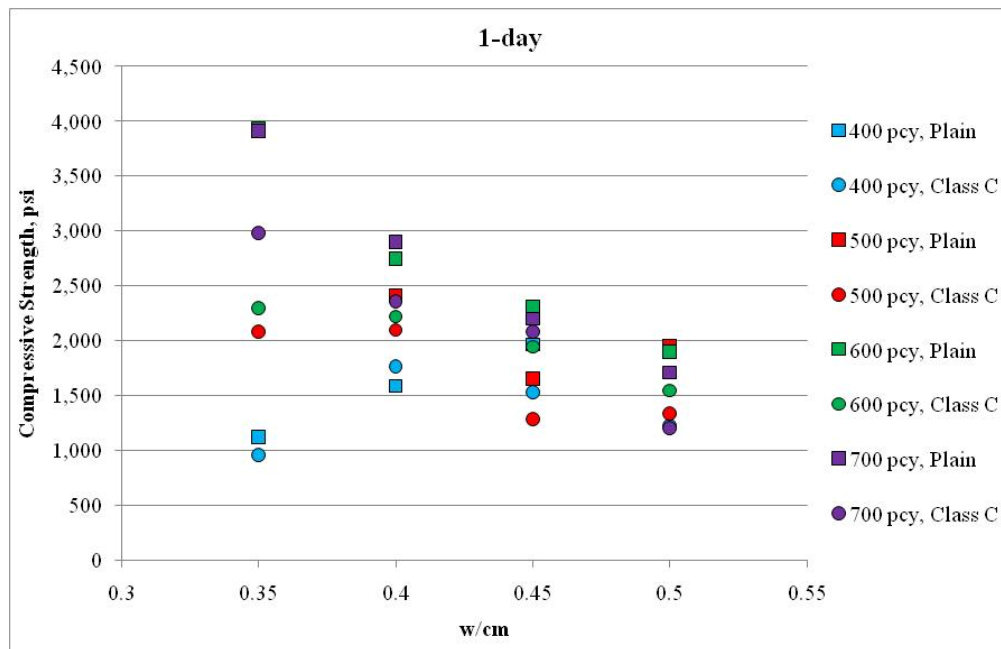


Figure 3. Effect of w/cm on 1-day compressive strength.

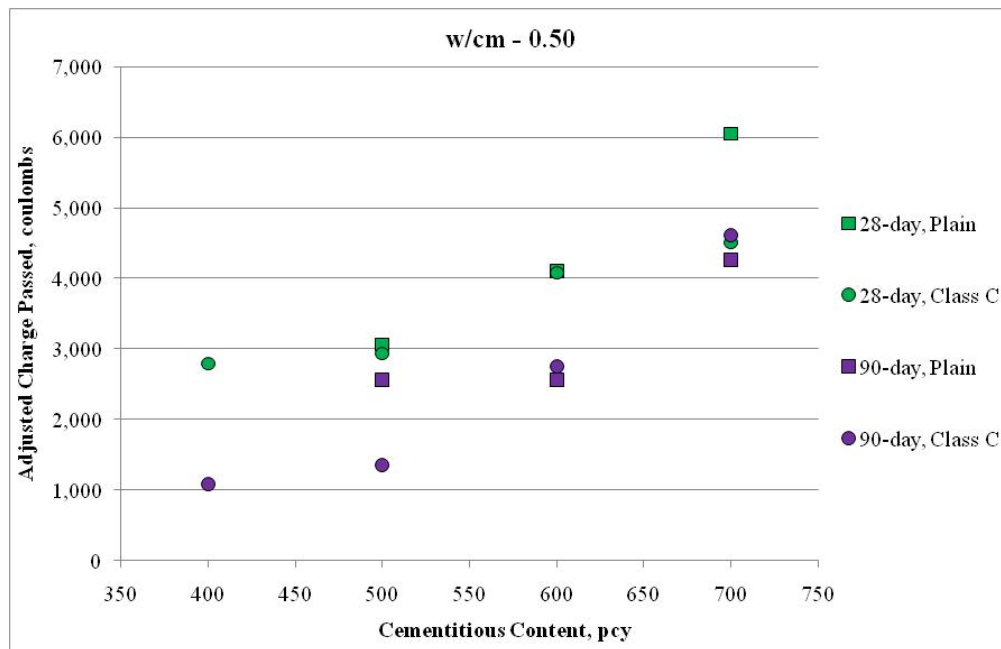


Figure 4. Effect of cementitious content on chloride penetration for w/cm of 0.50.

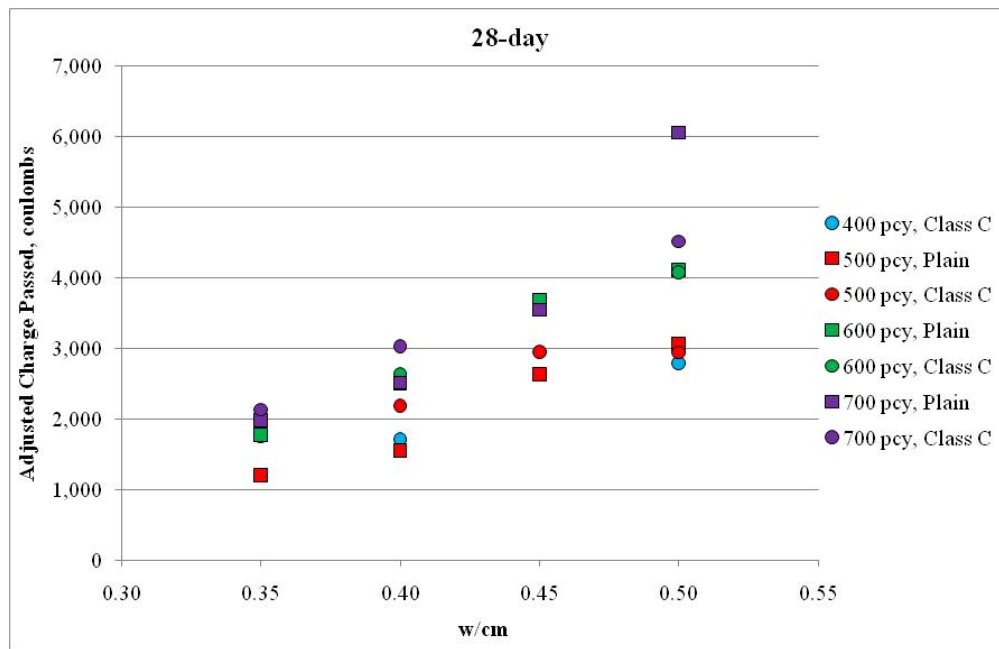


Figure 5. Effect of w/cm on 28-day chloride penetration.

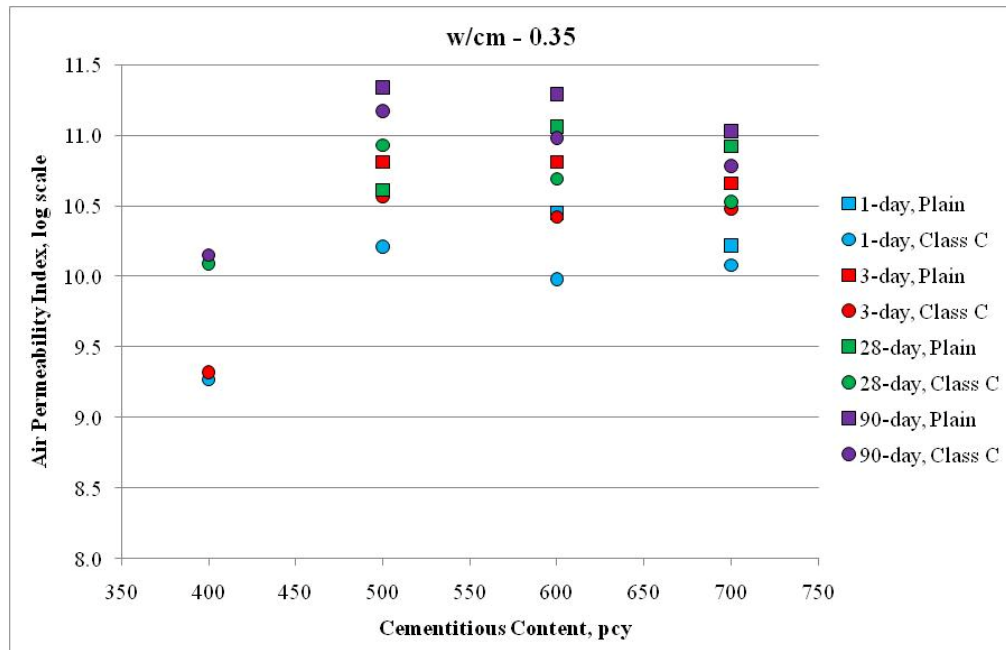


Figure 6. Effect of cementitious content on air permeability for w/cm of 0.35.

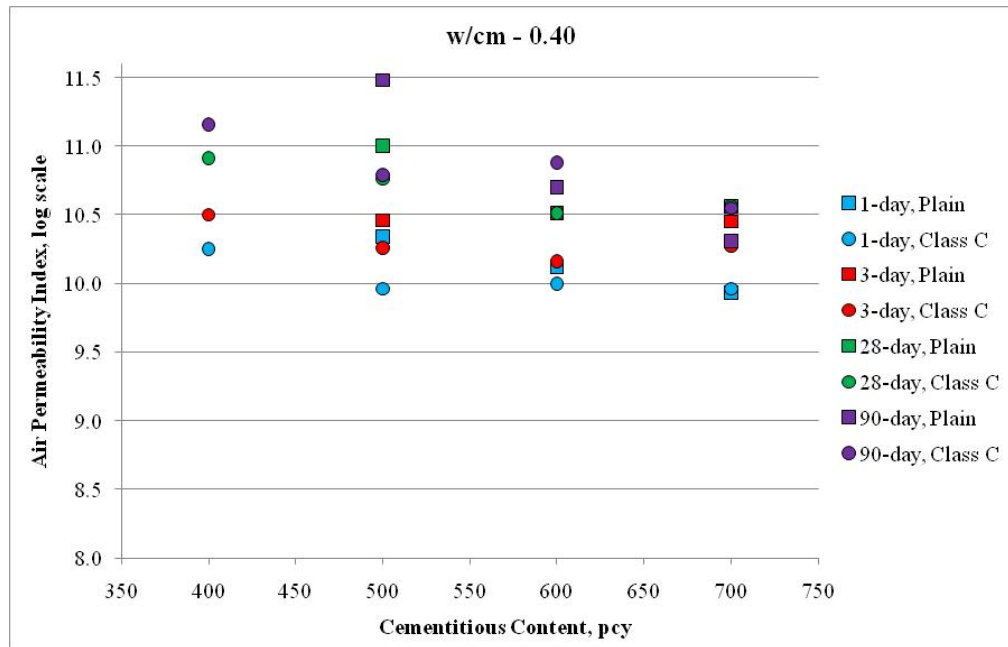


Figure 7. Effect of cementitious content on air permeability for w/cm of 0.40.

APPENDIX C. DEVELOPMENT OF A PROTOCOL TO ASSESS INTEGRAL WATERPROOFING ADMIXTURES

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Abstract

Concrete durability is a direct function of permeability, therefore reducing permeability will improve the potential durability of a given mixture. One approach to improving permeability of a mixture is to add chemical compounds, known as integral waterproofing admixtures, which help to fill and block capillary pores.

Currently there are no standard approaches to evaluate the effectiveness of integral waterproofing admixtures or to compare them in the United States. 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.

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This study investigated the fresh and hardened properties of mixtures containing commercially available integral waterproofing admixtures. The aim was to develop a protocol that would help owners and specifiers compare different products and to evaluate their effects on concrete mixtures.

In this experimental program, concrete mixtures were prepared with a fixed water-to-cement ratio and cement content. One mixture was prepared as a control mixture, five mixtures were prepared using the recommended dosage of different waterproofing admixtures, and five mixtures were prepared using double the recommended dosages. Fresh and hardened properties were assessed at various ages. The data are presented and recommendations for a testing protocol provided.

Introduction

- Concrete contains various types of pores (1, 2): Pores in the hydrated cement paste (gel pores, capillary pores, hollow-shell pores, air voids).
- Pores in the aggregates.
- Pores associated with the interfacial transition zone (ITZ).
- Voids due to construction deficiencies, e.g. honeycombing due to poor compaction.

Among these pores, those in the aggregate generally play only a minor role in deleterious moisture transportation, except for those prone to D-cracking (3). The space not filled by the cement or the hydration products consists largely of capillary pores. (See references 1 through 5.) The relative amounts and percolation of the different types and sizes of pores affect concrete properties such as strength, shrinkage, creep,

permeability, and ionic diffusion (5). From a strength and elasticity point of view, the important factor is the total pore volume; however, permeability is primarily controlled by the amount and continuity of the capillary pores in the paste system that are larger than 50 nm. (See references 1 through 8.)

Long-term concrete performance is strongly influenced by concrete durability (8). The durability of concrete is a direct function of the permeability because it controls the rate of penetration of fluids, gas and ions that may contain aggressive chemicals and the movement of water during heating or freezing. (See references 1 through 9.) Therefore, reducing permeability will improve the potential durability of a given mixture. (See references 4 through 11.)

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 (7, 8, 9). The inclusion of supplementary cementitious materials, such as fly ash, slag cement and silica fume, can decrease permeability (1, 2, 4, 7). However, the scope of this study is integral waterproofing admixtures.

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” (See references 3, 9, 12, 13, and 14.) The effectiveness of the admixtures depends on their dosage and chemical interactions with cement paste matrix (14, 15, 16). An integral

waterproofing admixture can be in powder, liquid, or suspension form. ACI 212.3R-10 (16) classifies these products in three categories:

- Hydrophobic chemicals: These materials are based on soaps and long-chain fatty acid derivatives, vegetable oils, and petroleum. These materials provide a water-repellent lining in the pores; however the pores remain physically open.
- Finely divided solids: These materials include inert and chemically active fillers (such as talc, bentonite, clay etc.). These materials reduce permeability by filling up voids and physically restrict the water penetration through the pores.
- 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.

Although some researchers argue the effectiveness of these admixtures in the long term (4, 9), these admixtures claim to provide concrete with lower permeability, lower water absorption, slower ingress of aggressive elements, and even to self-heal minor cracks (11, 14). Like all additives, these materials may have side effects on other properties of the mixture such as setting time and strength.

Although, integral waterproofing admixtures have been commonly used on bridge decks, foundation walls, sewage, tunnels, and pavements where deicing salt usage is extensive (11, 13), at present there are no standard approaches to evaluate the efficiency of these products or to compare them in the United States.

The European Standard EN 934-2 (18) only evaluates the effectiveness of waterproofing admixtures on permeability by testing the capillary absorption. However,

depending on the formulation, different waterproofing 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., the U.S. Army Corps of Engineers CRC C48-92, DIN 1048, BS EN 12390, ASTM C1202, ASTM C1556, ASTM C1585) have been used to evaluate the effectiveness of admixtures (16), and rarely are the same tests used on more than one product. These test methods are selected based on the waterproofing admixture types (i.e., hydrophobic, crystalline, solids). For instance, crystalline admixtures are commonly tested using the German test method DIN 1048, which is a penetration method that reports the water penetration depth as a simplified indicator of water permeability. On the other hand, pore blocking or water repellent types of admixtures are commonly tested in accordance with the BS 1881-122, which is an absorption method. Therefore, a standard protocol needs to be developed that could recommend various permeability measuring test methods, as well as other fresh and hardened properties that are of interest for a particular application.

This study investigated the fresh and hardened properties of mixtures containing a selection of commercially available integral waterproofing admixtures. The aim was to develop a protocol that would help owners and specifiers compare different products and to evaluate their effects on concrete mixtures.

In this experimental program, 11 concrete mixtures were prepared with a fixed water-to-cementitious materials ratio (w/cm) and cement content: 1 mixture was prepared as a control mixture, 5 mixtures were prepared using the recommended dosage

of different waterproofing admixtures, and 5 mixes were prepared using double the recommended dosage for each product. Slump, air content, setting time, compressive and flexural strength, Wenner Probe surface resistivity, rapid chloride penetration (RCP), air permeability, shrinkage, water absorption, and sorptivity tests were conducted at various ages.

Methodology

Materials

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

- ASTM C150 Type I, low-alkali, portland cement.
- 1-inch (25.4 mm) nominal maximum-size crushed limestone coarse aggregate.
- No. 4 nominal maximum-size concrete sand.

After reviewing 13 commercially available admixtures produced by different manufacturers, 5 integral waterproofing admixtures were selected. In the interest of confidentiality, the products are referred to as A, B, C, D, and E in the remainder of this paper.

Mix Design

The integral waterproofing admixtures are intended to be used in well-proportioned concrete mixtures with a w/cm of 0.45 or lower (16). In this study, a fixed cement content, aggregate system, and w/cm were selected for consistency.

The full test program included 11 mixes with a constant w/cm of 0.45 and a fixed cement content of 564 lb/yd³ (335 kg/m³). One mixture was prepared as control mixture (designated as “0”), five mixtures were prepared using the manufacturers’ recommended dosage (labeled “R”) of the different products, and five mixes were prepared using double the recommended dosage (“2R”).

The proportioning recommendations and mixing specifications of each product were reviewed. The selected cement content and w/cm were within the acceptable range for four products. Admixture “E” recommended using higher cementitious content and lower w/cm than the selected values due to its water-reducing effect. Since the main focus of this study was to evaluate the effectiveness of these products on reducing permeability, the selected cement content and w/cm were also applied deliberately on concretes containing admixture “E”.

Based on an assessment of the combined aggregate system using the tools developed by Shilstone (19), the ratio of fine aggregate to total aggregate selected was 0.42.

To minimize the effect of variables in the test matrix, water-reducing admixtures, air- entraining admixtures, and supplementary cementitious materials were not used in this study.

Specimens and Testing

For each mixture, sixteen 4- x 8-inch (100 x 200 mm) concrete cylinders were prepared in accordance with ASTM C31 and stored in the fog room in accordance with ASTM C192 until testing. The tests conducted are listed in Table 1. Although the

hardened concrete properties were tested at a range of ages, this paper presents mostly the 28-day data and 56-day flexural strength results.

Results and Discussion

Workability

A slump test was conducted to assess the effect of the addition of integral waterproofing admixtures on workability. The test results show that products generally tend to increase the workability at the recommended dosage (Figure 1). However, admixture “E” increased the workability by about 50 percent compared to the control mix.

It is recommended that the slump of mixtures containing integral waterproofing admixtures should not be less than 80 percent of the control mixture.

Air Content

The test results show that the air content of the mixtures containing waterproofing admixtures is mostly within 2 percent of the control mix, other than the mixture containing product “B” (Figure 2). The air content of the mix with “B” at the recommended dosage was 4 percent more than the control mixture.

It is recommended that the air contents of mixtures containing integral waterproofing admixtures should not differ more than 2 percent from the control mixture.

Setting Time

The effect of the integral waterproofing admixtures on final setting time is plotted in Figure 3 along with 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. The addition of waterproofing admixtures tested does not significantly affect the setting time at the recommended dosages. However, when the selected dosage is higher than the recommended dosage, the setting time may be significantly affected, depending on the admixture type.

It is recommended that setting time of the mixes containing the integral waterproofing admixtures should not be less than 60 min and more than 90 min of the control mixture.

Strength

The effects of the admixtures on 28-day compressive strength and 56-day flexural strength are illustrated in Figure 4 and Figure 5, respectively.

ASTM C494 requires that mixtures with special admixtures should not have less than 90 percent of the strength of the control mix. According to this specification, admixtures “B” and “C” did not meet this criterion at the recommended dosages.

The strength of the mixes containing the integral waterproofing admixtures should not be less than 90 percent of the control mixture.

Surface Resistivity

The effect of integral waterproofing admixtures on 28-day surface resistivity was tested using the Wenner Probe and is illustrated in Figure 6. As expected, the addition of integral waterproofing admixtures increased the surface resistivity by approximately 7 to 30 percent compared to the control mix at the recommended dosages.

Given the objective of using the waterproofing admixtures mixtures, incorporating integral waterproofing admixtures should provide a minimum of 10 percent higher surface resistivity compared to the control mix.

Rapid Chloride Penetration

The effect of integral waterproofing admixtures on 28-day RCP is illustrated in Figure 7.

The addition of admixture “A” and “B” at the recommended dosage increased the chloride penetration by 27 percent and 41 percent, respectively. However, considering that the precision of the test means that two samples may differ by 42 percent according to ASTM C1202, the test results may be considered to be acceptable. Overall, considering the high variability of this test method, the chloride penetration of the mixtures containing the waterproofing admixtures is recommended to be equal or less than the control mixture.

Air Permeability

Air permeability testing was conducted in accordance with the University of Cape Town Method (20) except that dry air was used instead of oxygen as the test gas. The test results are presented in Figure 8.

Air permeability index (API) is the negative log of the D'Arcy coefficient of permeability (m/s) and uses a log scale (21). Higher API values indicate higher impermeability (22). As reported by Alexander and Beushausen (23), the following interpretation can be applied to the results:

- $API > 10.0$ – Excellent.
- $9.5 < API < 10.0$ – Good.
- $9.0 < API < 9.5$ – Poor.
- $API < 9.0$ – Very poor.

Based on the provided classification, all the mixtures (including the control) may be considered “excellent” with an API value higher than 10. The differences between the test mixtures and the control are relatively small, with the greatest benefit exhibited by product “C.” The API of mixtures containing the waterproofing admixtures should have an index value that is a minimum of 0.05 greater than the control mixture.

Water Absorption

Absorption is another parameter that indicates concrete durability (24). The effect of integral waterproofing admixtures on 28-day water absorption is illustrated in Figure 9.

The 28-day water absorption test results showed that the addition of the waterproofing admixtures provided ~40 percent less permeable voids volume compared to the control mixture, at the recommended dosage. However, admixtures “A” and “B” did not improve the water absorption. Admixture E provided ~50 percent less absorption compared to the control mixture and thus improved the impermeability. The permeable

pore space voids volume of the mixes containing integral waterproofing admixtures should be a minimum of 10 percent lower than the control mixture. Shilstone (25) was of the opinion that a value of less than 12 percent indicated a “good” mixture.

Sorptivity

Sorptivity is also an indicator of the concrete durability as it measures the rate of absorption. This makes the sorptivity test suitable for evaluation of the effects of curing on a surface in addition to assessing the quality of the concrete mixture.

The effect of integral waterproofing admixtures on 28-day secondary sorptivity is illustrated in Figure 10.

The 28-day secondary sorptivity test results showed an overall trend that the addition of the waterproofing admixtures provided lower sorptivity than the control mixture, as desired. Especially, admixtures “C” and “D” significantly decreased (up to 50 percent) the rate of absorption.

Overall, the sorptivity of the mixes containing integral waterproofing admixtures should be minimum of 10 percent lower than the control mixture.

Shrinkage

The 28-day shrinkage test results are presented in Figure 11. According to the ASTM C494 limit, when the length change of control is less than 0.03 percent (which it is less than 0.03 percent in this study), the addition of waterproofing admixtures should not shrink more than 0.01 percent of the control mixture. Therefore, all the products shrank within the acceptable limit.

Overall, as stated in the ASTM C494 Type S Special Admixtures, the increase of the length change of the mixtures containing integral waterproofing admixtures over control mixture should not be more than 0.01 percent.

Recommended Limits

The recommended limits of the tested properties are summarized and presented in Table 2.

The requirements for water-resisting admixtures specified by the European Standard EN 934-2 (18) are presented in Table 3. Although these requirements do not cover as wide a range of properties as in this study, the proposed recommended limits and the EN 934-2 specified requirements for a given property are similar for compressive strength and air content. However, the capillary absorption test in EN 934 was for mortars, whereas in this work, tests are conducted on concrete. The capillary absorption is related to the 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 ITZ surrounding each (26, 27). It is therefore not surprising that the requirement provided for the capillary absorption in EN 934-2 is different than the proposed requirement in our study.

Conclusions and Recommendations

The following conclusions are drawn from this study:

- Recommended limits were developed, based on the data collected and some of the recommendations of ASTM C 494.

- Four of the five products tested performed satisfactorily in the fresh state.
- Three of the products performed satisfactorily in the mechanical tests.
- All of the products performed satisfactorily in at least half of the potential durability tests.

Consideration may be given to conducting an additional test mixture with proportions adjusted to accommodate significant effects on properties such as strength or air entrainment.

Acknowledgment

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Table 1. Test matrix

Fresh Properties	Method	No. of Specimens	Age (days)
Slump/Slump flow	ASTM C143/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	28
Flexural strength	ASTM C78	4 per age	56
Surface resistivity	Wenner Probe	3 per age	28
Rapid chloride penetration	ASTM C1202	3 per age	28
Air permeability	Uni. of Cape Town	3 per age	28
Water absorption	ASTM C642	3 per age	28
Sorptivity	ASTM C1585	3 per age	28
Shrinkage	ASTM C157	4 per age	28

Table 2. Recommended limits for concretes containing integral waterproofing admixtures

	Property	Test method	Recommended limit
<i>"Do not harm"</i>	Workability	ASTM C143	Slump of mixes containing integral waterproofing admixtures should not be less than 80% of the control mixture.
	Air content	ASTM C231	Air contents of mixes containing integral waterproofing admixtures should not differ more than 2% from the control mixture.
	Setting time	ASTM C403	Setting time of the mixes with the integral waterproofing admixtures should not be less than 60 min and more than 90 min of the control mixture.
	Strength	ASTM C39/C78	Mixes with the integral waterproofing admixtures should not have less than 90% of the compressive/flexural strength of the control mix.
	Shrinkage	ASTM C157	The 28-day shrinkage of the mixes with the integral waterproofing admixtures should not be more than 0.01% of the control mixture.
	Property	Test method	Recommended limit
<i>Permeability enhancements</i>	Surface resistivity	Wenner Probe	Mixtures with the integral waterproofing admixtures should provide a minimum of 10% higher resistivity at 28-day compared to control mix.
	Chloride penetration	ASTM C1202	The 28-day chloride penetration of the mixes with the integral waterproofing admixtures should be equal or less than the control mix.
	Air permeability	University of Cape Town	The 28-day air permeability index of mixes with the integral waterproofing admixtures should be a minimum of 0.05 greater than control mix.
	Water absorption	ASTM C642	The 28-day permeable pore space voids volume of the mixes with the integral waterproofing admixtures should be a minimum of 10% lower than the control mixture.
	Sorptivity	ASTM C1585	The 28-day sorptivity of the mixes with the integral waterproofing admixtures should be a minimum of 10% lower than the control mixture.

Table 3. Specific Requirements for Water-Resisting Admixtures (at equal consistence or equal w/cm ratio*) (adapted by EN934-2:2000 (18))

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 $\leq 60\%$ 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 $\leq 2\%$ V/V above control mix unless otherwise stated by the manufacturer.

*All tests shall be performed either at equal consistence or equal w/cm ratio.

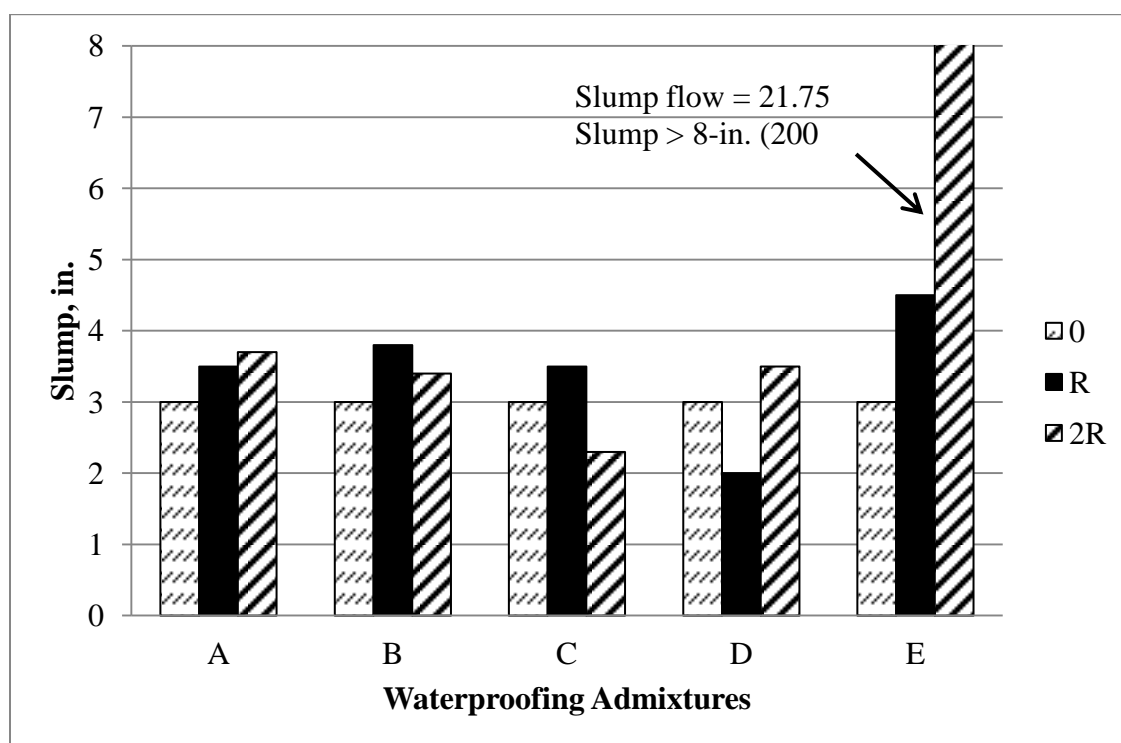


Figure 1. Effect of integral waterproofing admixtures on concrete workability.

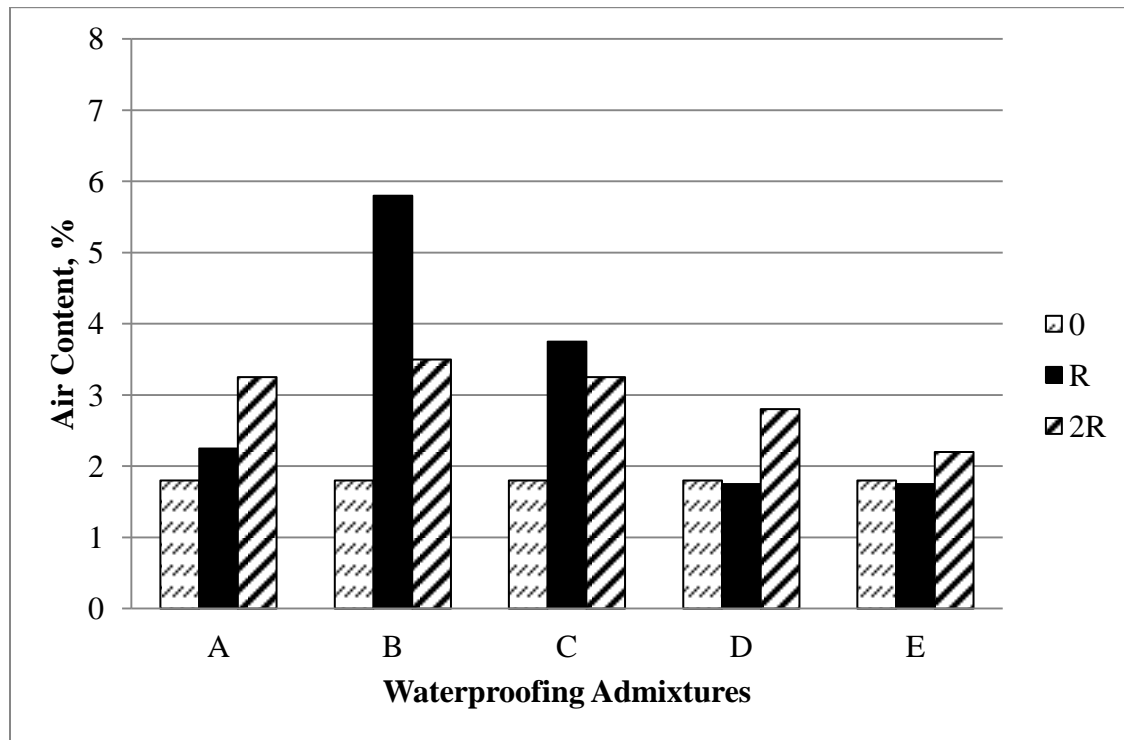


Figure 2. Effect of integral waterproofing admixtures on air content.

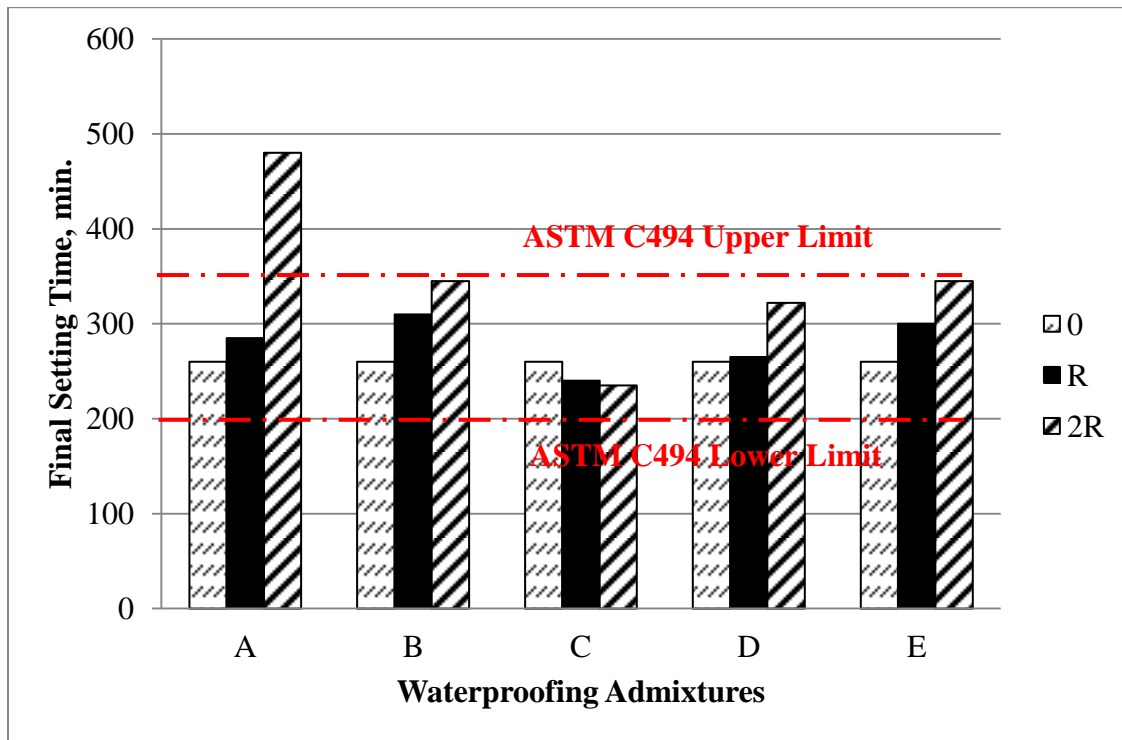


Figure 3. Effect of integral waterproofing admixtures on final setting time.

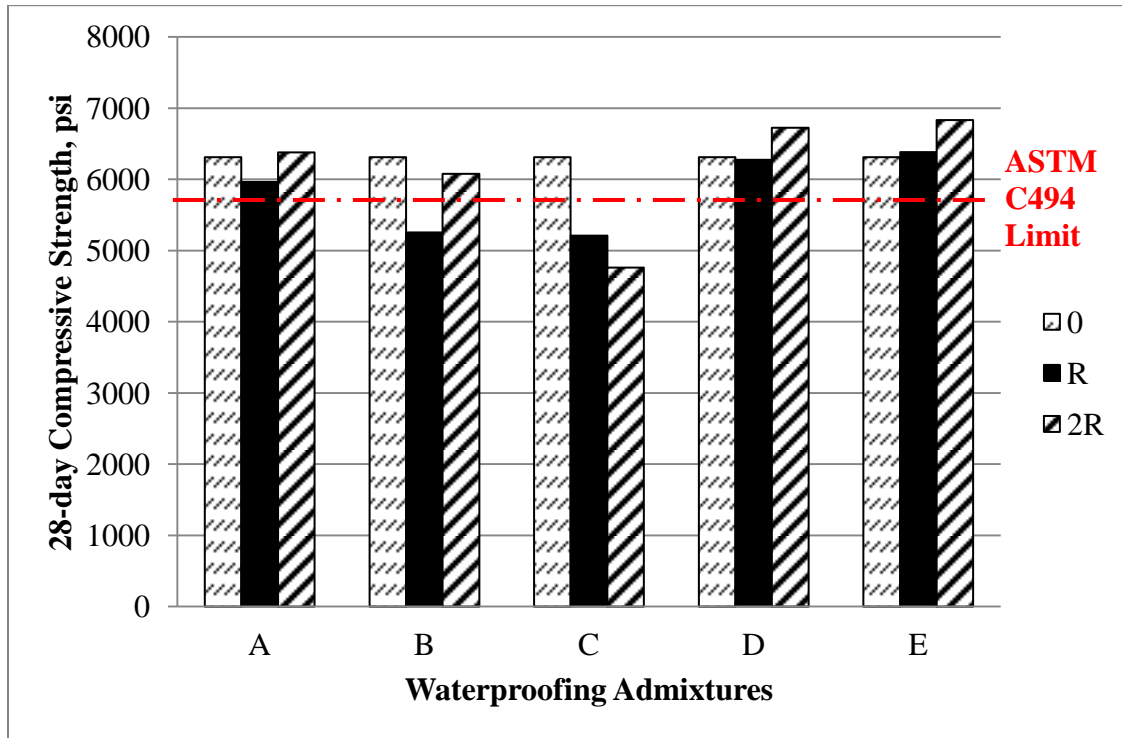


Figure 4. Effect of integral waterproofing admixtures on 28-day compressive strength.

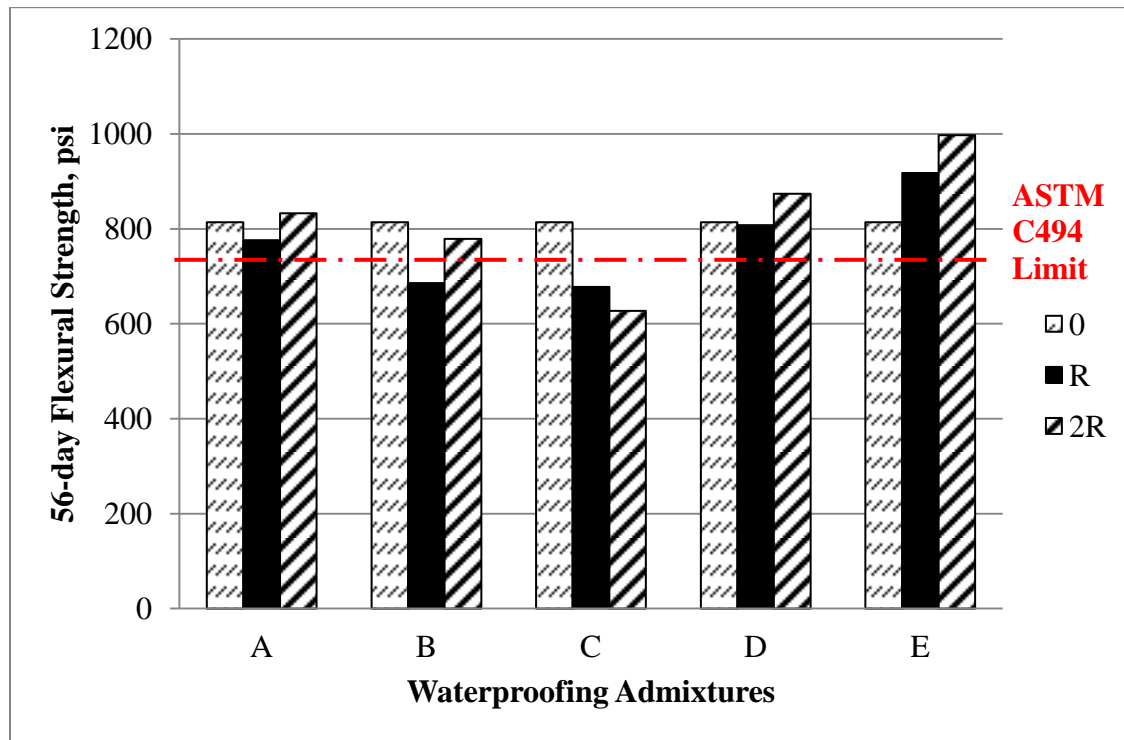


Figure 5. Effect of integral waterproofing admixtures on 56-day flexural strength.

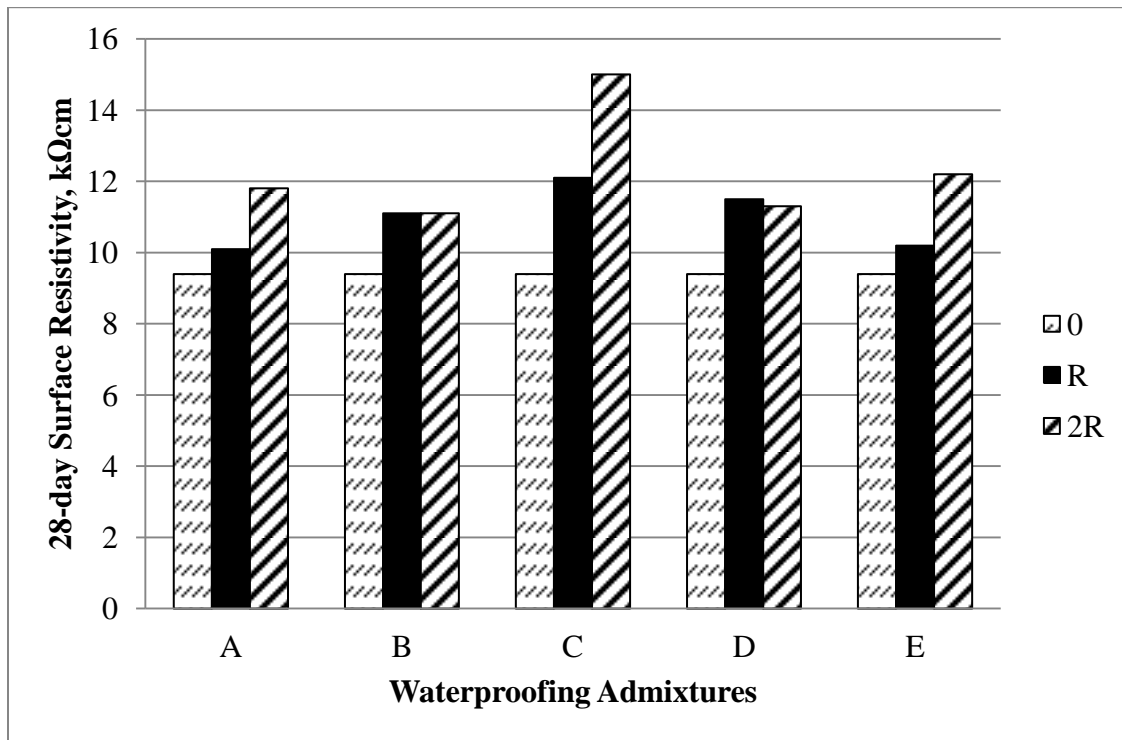


Figure 6. Effect of integral waterproofing admixtures on 28-day surface resistivity.

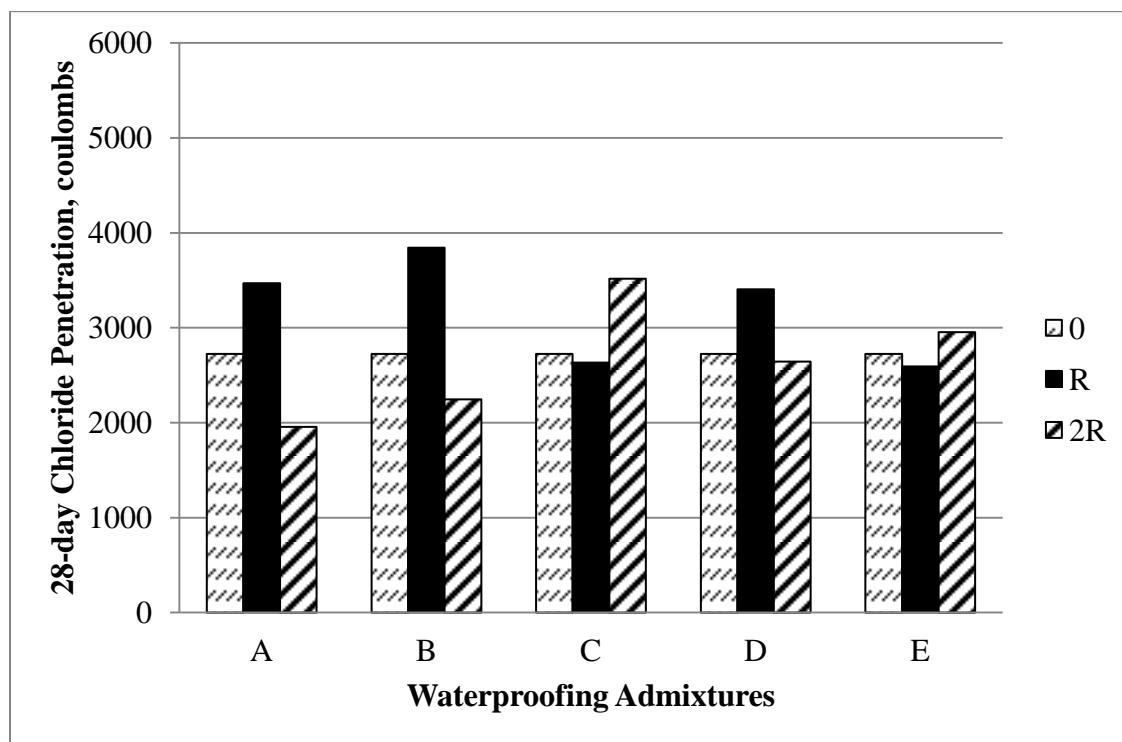


Figure 7. Effect of integral waterproofing admixtures on 28-day rapid chloride penetration.

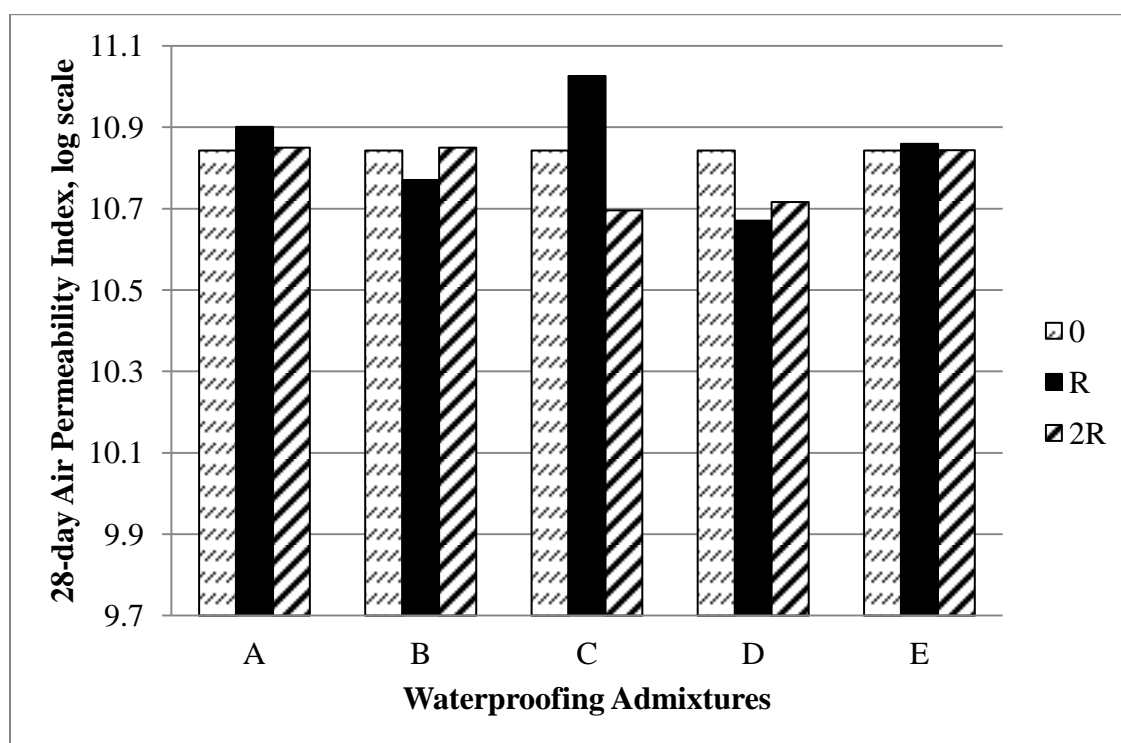


Figure 8. Effect of integral waterproofing admixtures on 28-day air permeability.

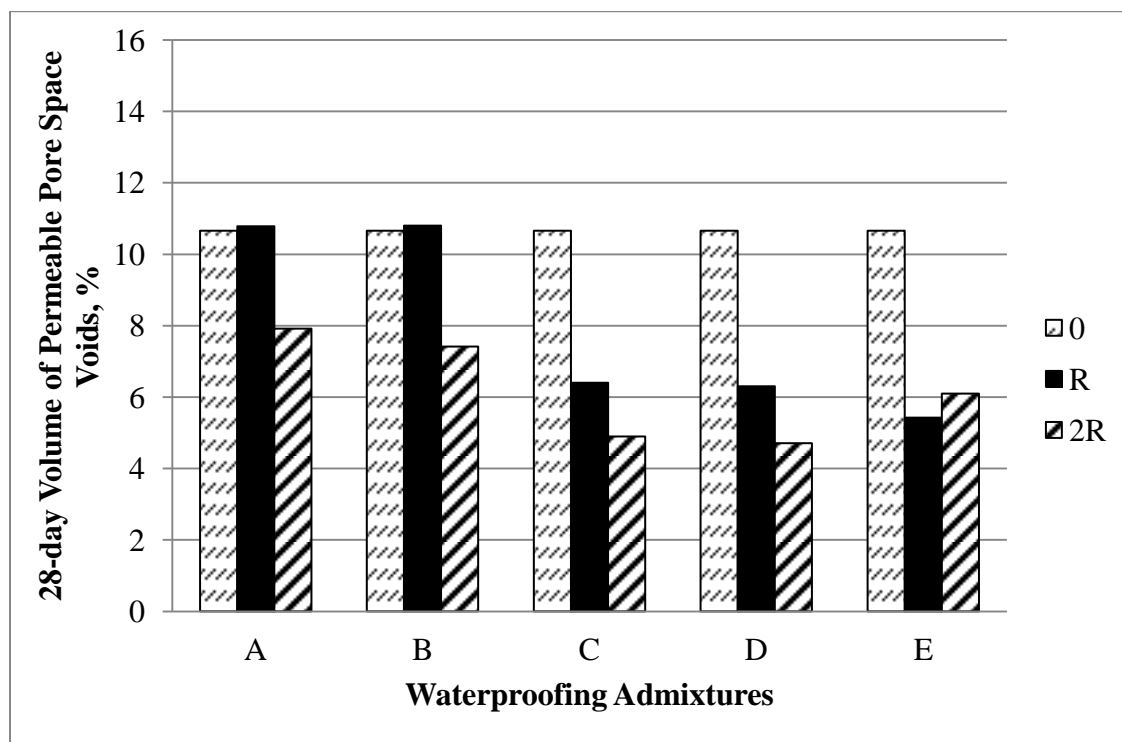


Figure 9. Effect of integral waterproofing admixtures on 28-day water absorption.

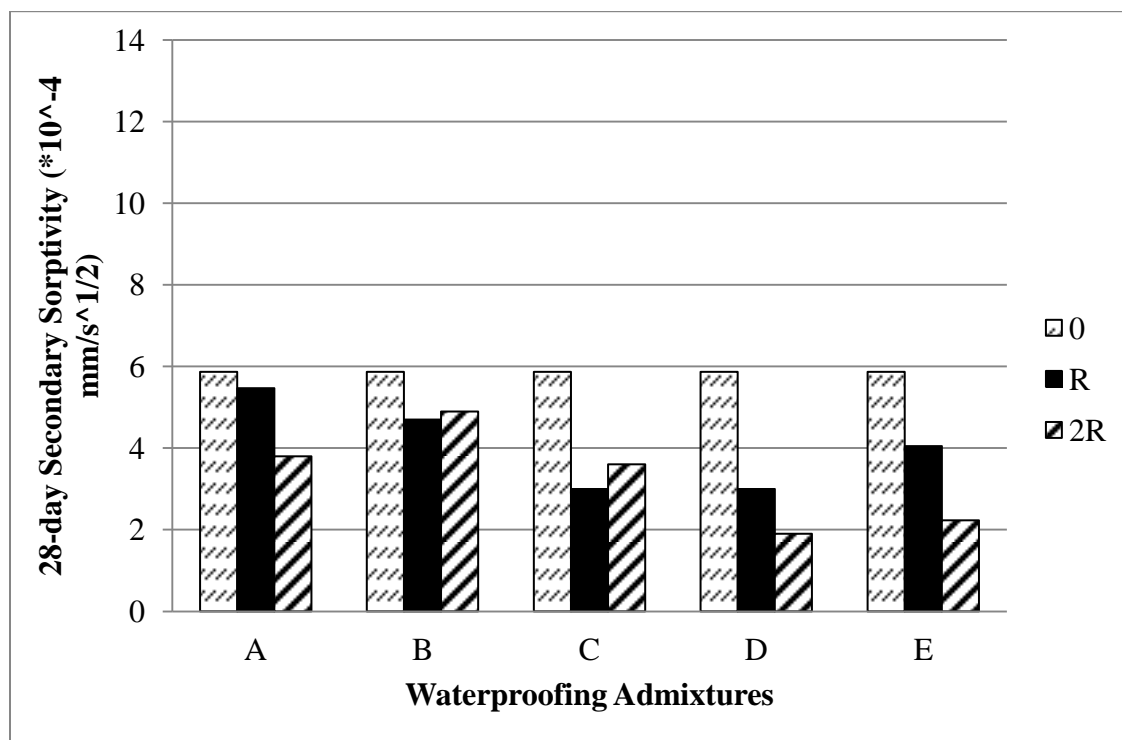


Figure 10. Effect of integral waterproofing admixtures on 28-day secondary sorptivity.

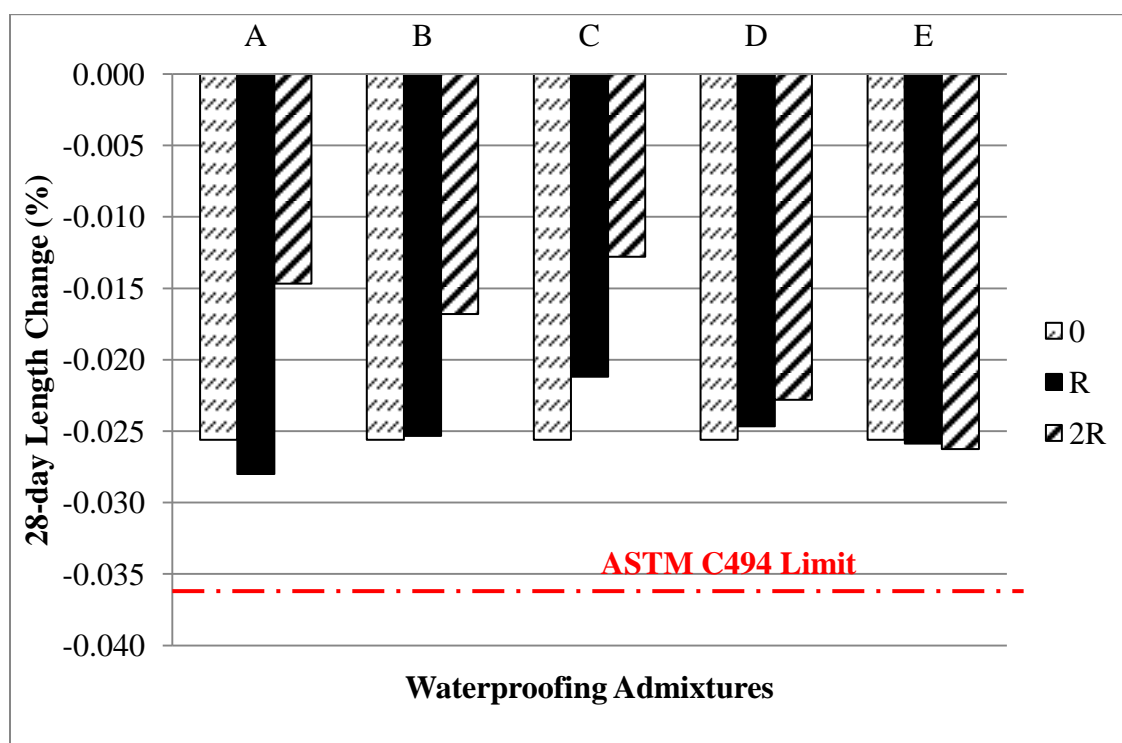


Figure 11. Effect of integral waterproofing admixtures on 28-day shrinkage.

APPENDIX D. PRELIMINARY INVESTIGATION ON DETERMINING THE MINIMUM CEMENT CONTENT IN RIGID PAVEMENTS

A paper presented at the 90th Annual Transportation Research Board (TRB) Meeting,

Washington DC, USA

January 23-27, 2011

Ezgi Yurdakul^{1,*}; Peter C. Taylor²; Halil Ceylan¹; and Fatih Bektas²

Abstract

This paper presents the preliminary results of an experimental program that consists of testing of concrete mixtures with varying water-to-binder ratios (w/b) and cementitious contents. The purpose of this laboratory study is to investigate the minimum cement content that can be used in rigid pavements without sacrificing the performance (i.e., strength and durability). Initially, 16 mixes using only portland cement and 48 mixes incorporating supplementary cementitious materials, namely class F fly ash, class C fly ash and slag as portland cement replacement at levels of 20%, 20% and 40%, respectively, were planned. This paper reports the results of a subset of this study. Concrete mixtures with w/b ranging from 0.43 to 0.65 and cementitious content ranging from 400 lb/yd³ to 700 lb/yd³ were designed. Compressive strength, chloride penetration and air permeability were determined.

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The findings of the study with the available data are as follows: strength is a function of w/b and independent of the binder content; air permeability increases as w/b and binder content increase; among all the mixtures containing different type and amount of cementitious materials, slag cement provides the lowest 28 day chloride penetration whereas class C fly ash results in the highest chloride penetration.

APPENDIX E. INVESTIGATING THE MINIMUM BINDER CONTENT REQUIREMENT BY USING CLASS F FLY ASH IN CONCRETE PAVEMENTS

A paper presented at the International Conference on Construction, Architecture and Engineering, Athens Greece

June 20-23, 2011

Ezgi Yurdakul^{1,*}; Peter C. Taylor²; Halil Ceylan¹; and Fatih Bektas²

Abstract

Many of the state Department of Transportation agencies in the USA specify a minimum cement content range in between 500 and 600 lb/yd³ (pcy). However, minimum cement content is often specified conservatively and may exceed the amount needed to achieve the desired strength and durability. This research study was conducted to investigate the minimum binder content by comparing the obtained test results with the current binder content specifications for portland cement concrete pavements. Considering the high usage of supplementary cementitious materials in concrete to improve its long-term serviceability, reduce cost and be sustainable; Class F fly ash was included in the experimental matrix.

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In this experimental program, 32 concrete mixtures with water-to-binder ratio (w/b) ranging between 0.35 and 0.50; and binder content ranging from 400 to 700 pcy were used. 16 mixtures were prepared using ASTM Type I ordinary portland cement and 16 contained Class F fly ash at 20% portland cement replacement level. Compressive strength, chloride penetration and air permeability were determined at various ages.

The test results showed that strength is a function of w/b and, for a given w/b; it is independent of binder content after certain content is reached. Chloride penetration increases as w/b or binder content increases. Air permeability increases as w/b or binder content increases. For the materials used in this study, 500 pcy cementitious is found to be the most appropriate amount that provides a workable mixture with typical normal strength (28-day compressive strength ranging between 3000 and 6000 psi) and durability in concrete pavements.