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Superabsorbent Polymers for Internally Cured Concrete

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16. Abstract

Two commercial superabsorbent polymer (SAP) formulations were used to internally cure cement pastes, mortars, and concretes with a range of water-to-cement ratios (w/c 0.35–0.52). The following properties were determined as a function of cement chemistry and type, use of chemical admixtures, use of slag, and batching parameters: SAP absorption capacity, fresh mixture workability and consistency, degree of hydration, volumetric stability, cracking tendency, compressive and flexural strength, and pumpability. SAP internal curing agents resulted in cementitious mixtures with improved hydration, accelerated strength gain, greater volumetric stability, and improved cracking resistance while maintaining sufficient workability to be pumped and placed without sacrificing compressive or flexural strength. When using SAP, batching adjustments prioritized the use of water reducing admixture instead of extra water to tune workability. While the benefits of SAP internal curing agents for low w/c mixtures were expected, SAP-containing mixtures with w/c ≥ 0.42 displayed accelerated strength development and decreased cracking tendency.

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EXECUTIVE SUMMARY

Introduction

This project investigates the use of superabsorbent polymer (SAP) particles as internal curing agents in cementitious mixtures that are currently specified for INDOT applications such as bridge decks and full-depth patching of concrete pavements. A materials-centric research approach was employed to identify suitable commercial SAP formulations for use in internally cured concrete mixtures and to develop successful methods of implementation and evaluation of fresh and hardened SAP-cured mixtures.

Methods

A series of laboratory-based experiments were designed to determine the following: (1) the effects of such variables as cement chemistry and type, use of chemical admixtures, and use of slag cement on the absorption of commercial SAP; (2) successful batching sequences and adjustments for SAP-cured mixes; (3) the impact of SAP on early-age properties of cementitious mixtures; and (4) whether SAP-mortar mixtures had sufficient workability to be pumped.

SAP absorption was measured gravimetrically during immersion in simulated pore fluids prepared using three cements of different chemical compositions and two different types: (1) systems containing slag cement and (2) systems with and without (high range) water reducing admixture ((HR)WRA) and air entraining (AE) admixture. The following properties (measurements) were determined as a function of SAP composition and batching parameters for mortars and concretes: workability (slump, flow retention); consistency (air content); volumetric stability (warping, autogenous shrinkage, drying shrinkage); cracking tendency (restrained ring test); strength (compression and flexural tests); and pumpability (time trials with recirculating pump). Cement pastes were used for measurements of degree of hydration (isothermal calorimetry, non-evaporable water). The microstructure of internally cured paste and mortar samples was analyzed, and thermogravimetric analysis was performed on pastes with different cement types and sources cured with and without SAP and with and without external curing compound.

Findings

Different commercial SAP formulations composed of acrylate- and acrylamide-based crosslinked polymers with dry particle sizes less than 0.3 mm displayed different absorption capacities and swelling kinetics when immersed in simulated pore solutions than in pure water. While SAP absorption was slightly to moderately sensitive to cement chemistry and type, the presence of water reducing and air entraining admixtures did not affect SAP absorption behavior. SAP equilibrium absorption in Type I cement pore fluid ranged from 30 to 60 g fluid per g dry SAP.

Concrete properties were found to be insensitive to whether SAP was added with the cement or with the aggregate in batching sequence. All results were presented for SAP dosage of 0.2% by weight of binder with SAP added as dry particles. Results indicated that no extra water needs to be added to mixtures containing SAP to account for water absorption by SAP particles. In such cases, workability should be adjusted using (HR)WRA to ensure the target slump value.

SAP-containing mortars with higher w/c values (≥0.49) displayed accelerated compressive and flexural strength gains compared to SAP-free mortars, consistent with the accelerated hydration observed via calorimetry for SAP-containing pastes and signifying that SAP internal curing could be employed in situations when rapid strength development is desired. Additionally, SAP and external curing compound displayed similar increases in CH formation when used together or separately across cement sources and types.

Warping and drying shrinkage of SAP-containing pastes were greater when extra water was added to the mixtures. Both SAP formulations were effective in reducing autogenous shrinkage. Additionally, SAP can be used to decrease cracking tendency of both high and low w/c mixes. Mortars containing SAP were pumpable, indicating that SAP internal curing is feasible for field application.

Implementation

While field trials are still needed, the laboratory outcomes of this project suggest that the use of SAP internal curing agents would lead to the formation of concrete mixtures with improved hydration, accelerated strength gain, greater volumetric stability, and improved cracking resistance while maintaining sufficient workability to be pumped and placed without sacrificing compressive or flexural strength. Laboratory results also indicate that incorporation of SAP in fresh concrete mixes would have similar hydration benefits as the application of curing compound after concrete has set (this aspect should be verified under field exposure conditions). Mixture composition adjustments should prioritize the use of (HR)WRA instead of extra water to attain a target slump value, as addition of extra water will almost always lead to reductions in strength, increased warping, and higher levels of drying shrinkage. At the recommended dosage of 0.2% dry SAP by weight of binder, approximately one pound of SAP would be needed per cubic yard of concrete, potentially delivered in dissolvable bags similar to those used by industry to introduce other types of admixtures.

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1. INTRODUCTION

1.1 Motivation

An internal curing strategy being investigated by construction groups across the world is to utilize superabsorbent polymer (SAP) particles within cementitious mixtures. As initially conceived, SAP has been added to low water-to-cement (w/c) ratio mixtures—including high and ultra-high performance concretes—to control hydration and limit self-desiccation and autogenous shrinkage (Mechtcherine et al., 2014). However, researchers have discovered additional benefits of SAP, including improvement in the freeze-thaw resistance of mixtures with w/c ~ 0.45–0.50 (Mechtcherine et al., 2017) and reduction of thermal expansion at early ages (up to 7 days) for mixtures with w/c ~ 0.25–0.35 (Wyrzykowski & Lura, 2013).

Compared to internal curing with lightweight aggregate, SAP particles do not require pre-wetting and are thus expected to be easier to incorporate at ready mixed concrete plants to achieve a targeted w/c) (Barrett et al., 2015) Since SAP performance can be determined from testing performed on trial batches, small adjustments to the mixture compositions can be effectively implemented to create SAP-concrete mixtures with good workability. One technical challenge with deploying this internal curing strategy is to identify suitable SAP formulations and dosages for a given construction application, as there are a number of commercially available SAP products with different chemical compositions and particle sizes and shapes, attributes which affect their absorption performance and thus their effectiveness as internal curing agents.

1.2 Project Goal and Objectives

The overall goal of this project was to utilize SAP particles to create internally cured cementitious mixtures that display increased hydration and improved mechanical properties and durability compared with conventional mixtures currently specified for INDOT bridge decks (Class C) as well as full-depth patching of pavements. To achieve this goal, a materials-centric research approach was employed to identify suitable commercial SAP formulations and dosages for use in internally cured mixtures as well as develop successful methods of implementation and evaluation of fresh and hardened SAP-cured mixtures. This research project was designed to accomplish the following key objectives.

1. Determine the effects of such variables as cement chemistry and type, use of chemical admixtures, and use of slag cement on the absorption of commercial SAP.
2. Provide guidance on batching sequence and adjustments for SAP-cured mixes, including admixture dosage and the amount of additional water (if any) required to facilitate internal curing without compromising mixture workability and mechanical properties.
3. Measure the impact of SAP on early-age properties of cementitious mixtures, including degree of hydration, warping, shrinkage, and cracking tendency.
4. Demonstrate that SAP-mortar mixtures have sufficient workability to be pumped.

1.3 Organization of Report

Chapter 2 provides a review of relevant literature about the structure and properties of SAP and the use of SAP particles in concrete applications. Chapter 3 summarizes the key findings on SAP absorption capacity in simulated pore fluid with and without the use of additional admixtures. Chapters 4 and 5 summarize the best strategies for the batching sequence and mixture adjustments that should be made when incorporating SAP into cementitious materials. Chapter 6 presents an in-depth study on the impact of SAP on the hydration of cement paste and mortar, including a comparison with use of curing compounds. Chapter 7 discusses the impact of SAP on warping of cement pastes, and Chapter 8 discusses the impact of SAP on the autogenous and drying shrinkage of mortar. Chapter 9 summarizes the impact of SAP on cracking tendency of mortars. Chapter 10 discusses some preliminary experiments to quantify the pumpability of SAP-mortar mixtures. Chapters 11 and 12 provide some brief concluding remarks and implementation guidance based on the research outcomes of the project.

2. LITERATURE REVIEW

2.1 Absorption and Characterization of SAP Particles

The key performance metric for SAP particles is absorption capacity, which depends on the particle's composition and size as well as the composition of the absorbed fluid and, in particular, the concentration of cations in solution (Erk & Bose, 2018; Krafcik et al., 2017). This has clear implications for the use of SAP particles in real cementitious mixtures in which the exact ion composition and concentration of pore fluid changes as a function of time, cement type, and chemical composition of cement and cementitious binders (Montanari et al., 2018).

Gravimetric "tea bag" absorption measurement using simulated or extracted pore solution is a well-established method for characterizing SAP absorption behavior over time in cementitious environments (Krafcik & Erk, 2016; Schröfl et al., 2017). These measurements are described in Section 3.2. Such simple absorption measurements typically overestimate the amount of fluid absorbed by SAP particles in real construction applications. Nonetheless, this method provides a fast and reliable way of estimating absorption capacity of SAP particles (Schröfl et al., 2022).

2.2 Performance of SAP Internal Curing Agents in Concrete Applications

One of the original applications of SAPs in cementitious materials was as "water-entraining admixtures" to prevent self-desiccation, which is of critical importance

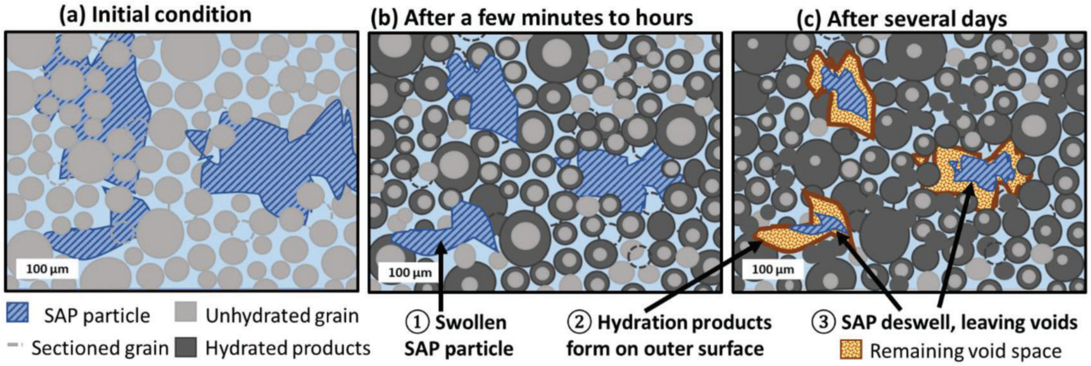


Figure 2.1 Cross-section of a cement mixture containing SAP particles and cement grains: (a) immediately after mixing and placement when SAP absorbs fluid and increases in size; (b) after a few minutes to multiple hours (before final setting); and (c) after a few days when a substantial amount of cement has reacted and the SAP particles have released a majority of their fluid (adapted with permission from Erk and Bose, 2018).

in mixtures with relatively low water to cement ratios (w/c) such as high and ultra-high performance concrete (Jensen & Hansen, 2001a, 2001b; Jensen & Hansen, 2002). If self-desiccation—i.e., drop in the internal relative humidity (RH) of the mixture—is unmitigated, the mixture will undergo shrinkage which can lead to the development of cracks and, ultimately, reduced strength and durability (Schröfl et al., 2022).

A 2014 international round-robin study demonstrated impressive reductions in autogenous shrinkage of SAP-cured high-performance concrete compared with SAP-free mixtures (Type I ordinary Portland cement, w/c = 0.3; 10% silica fume and 0.3% dry SAP by weight of cement) (Mechtcherine et al., 2014). For example, one SAP composition resulted in average reductions of autogenous strain of 109%, 84%, and 73% over the first 3 days (respectively) of aging while at longer times, reductions of 58%, 52%, and 42% were observed at ages of 7, 14, and 28 days (respectively).

As shown in Figure 2.1, SAP particles first absorb then desorb fluid over time within the cement paste matrix (Erk & Bose, 2018). Initially, dry SAP particles absorb a portion of the mixing water which reduces the effective w/c and decreases mixture workability (Mechtcherine et al., 2015). SAP presoaking prior to mixing with other raw materials can mitigate this workability loss (Kong & Zhang, 2014); however, the addition of SAP in its dry state ensures better particle dispersion in the mixture (Mönnig, 2009). When dry SAP is utilized, mixture workability can be increased through the addition of extra water, use of high-range water reducing admixture (HRWRA), or both (Justs et al., 2015; Kang et al., 2018; Snoeck et al., 2015). The amount of extra water is typically determined by one of two approaches: (1) 5% extra water by weight of binder is added when using SAP of any type (Mechtcherine et al., 2015), or (2) extra water is added according to the equilibrium (long-time) absorption capacity of the SAP being used (Laustsen et al., 2015).

A few recent studies have demonstrated the success of SAP internal curing agents at industrial scales. De Meyst et al. (2020) recently completed a large-scale demonstration of SAP particle's ability to reduce autogenous shrinkage and mitigate cracking of a high performance concrete restrained wall (2 m × 0.05 m × 1.5 m) placed on a slab (2 m × 0.95 m × 0.2 m) containing embedded fiber optic sensors and mechanical strain gauges. Tenório Filho et al. (2020) found that the method of incorporating SAP into real world mixtures at the concrete production plant will influence autogenous shrinkage, with reduced shrinkage observed when SAP particles were added to the materials belt with other dry components compared to when SAP was added directly to the truck after the (wet) mixing procedure was completed. When SAP was added to the truck, it resulted in agglomeration of SAP particles and reduced internal curing performance. In a follow-up demonstration, the same team of researchers (Tenório Filho et al., 2021) erected concrete walls that remained crack-free after 5 months while the SAP-free reference walls cracked after 5 days (14 m × 2.75 m × 0.80 m; 0.44 w/c; SAP-dosage was 0.38% by weight of cement).

3. OBJECTIVE 1: SAP CHARACTERIZATION

3.1 Overview

This section describes the research results obtained during the investigation of industrially sourced SAPs for internal curing of cementitious mixtures. The objective of this study was to characterize the selected SAPs and their behavior in cementitious systems. Characterization of the SAPs themselves, in addition to the evaluation of their effects on cementitious mixtures, provides valuable insight into the structure-property-performance relationship between SAP and internally cured concrete (using the same SAP particles). As such, characterization of SAP can be used to aid with SAP selection and processing design. Characterization of the selected SAPs and their behavior in cementitious systems was accomplished through scanning electron microscopy analysis and gravimetric absorption capacity tests conducted in reverse osmosis (RO) water and in cement pore fluids derived from cementitious systems of varying chemistry. The primary finding from these evaluations was that different SAPs have different sensitivities to the chemistry of cementitious systems with respect to absorption behaviors. A complete, detailed description of the experimental methods, quantitative results and supporting discussion was published in *Construction and Building Materials* in 2022 (refer to Appendix A).

3.2 Methods

Six different, industrially produced SAPs were sourced from BASF and SNF Floerger companies. For each SAP, gravimetric absorption capacity in both RO and simulated pore solution created by hand mixing a cement solution with water to cement ratio (w/c) of 10 for 30 seconds, letting the mixture settle for 1 minute, and decanting the fluid. Buzzi Type I cement was used except where noted otherwise. SAP absorption capacity through equilibrium (2 hours) was quantified using the "tea bag" gravimetric absorption test (Mechtcherine et al., 2018; Montanari et al., 2017).

The absorption capacity, Q (grams of absorbed fluid per grams of dry SAP particles), was calculated using the following formula:

$$Q = \frac{m_{wet} - m_{bag} - m_{dry}}{m_{dry}} \qquad \text{(Equation 3.1)}$$

While gravimetric absorption capacity, Q, as measured in a lab, is a useful characterization technique for SAP, it should be noted that it does not accurately quantify the absorption capacity or kinetics of SAP for *in situ* concrete applications. This is due to system differences, including the effects of SAP particles' confinement by cement paste and aggregate present in the actual concrete (Schröfl et al., 2022).

All six of the previously mentioned, industrially sourced acrylic acid-based SAPs that were screened fell into high and low absorption capacity groupings based on these gravimetric swelling tests. One representative formulation from each absorption capacity grouping was selected for use in all subsequent studies. These two formulations were termed, respectively, "low absorption" (LA) and "high absorption" (HA) SAP, based on their relative equilibrium absorption capacities (34.43 g/g and 49.05 g/g for LA and HA, respectively). The LA- and HA-SAP were imaged with backscattered scanning electron microscopy (SEM) to facilitate size and morphology analysis.

To investigate whether the addition of chemical admixtures, such as (high range) water reducing admixture ((HR)WRA) and air entraining admixture (AEA) had an effect on the absorption capacities of SAP and on the kinetics of absorption by SAP, another set of gravimetric absorption tests was carried out in simulated Buzzi Type I pore fluid that contained varying dosages of a Type A/F admixture (Master Glenium 7511, BASF, ASTM, 2019) by itself or in combination with AEA (MasterAir AE 200, BASF, ASTM, 2016) (ASTM, 2016; ASTM, 2019). Gravimetric absorption tests were also conducted for both SAP formulations in simulated pore fluid from two additional Type I cements (sourced from Lehigh and St. Marys), Buzzi Type III cement, and Buzzi Type I cement with 30% slag replacement (mill certificates can be found in Appendix B. For brevity, throughout this report the cements used are referred to according to their first initial and type where relevant (ex: "B Type I" is Buzzi Type I, while "B Type III" is Buzzi Type III).

3.3 Results

3.3.1 SAP Formulations

Both LA- and HA-SAPs were composed of particles with irregular shapes. The dry particle size of the LA- and HA-SAP were calculated to be 116.0 ± 70.0 μm and 48.9 ± 21.9 μm, respectively. The scanning electron microscopy (SEM) images of dry SAP particles are shown in Figure 3.1.

3.3.2 SAP Absorption

3.3.2.1 Absorption capacity in B Type I simulated pore fluid. After the first 30 minutes of exposure to the simulated pore solution (Figure 3.2b), the LA-SAP particles had a significantly (~30%) lower absorption capacity compared to HA-SAP particles. The crossover in relative absorption capacity between the two SAPs that occurred around 30 minutes is an important reminder that absorption kinetics may be more important than equilibrium (long-time) absorption capacity in determining the effects of a specific SAP on the flow behavior of a fresh cementitious mixture. Typically, smaller SAP particles (like HA-SAP used in this study) absorb fluid more quickly than larger SAP particles (Zhu et al., 2015). However, the opposite is seen here, indicating that the chemical composition of the two SAP formulations were different and that perhaps the fast-absorbing LA-SAP particles contain a greater fraction of ionizable functional groups (e.g., acrylic

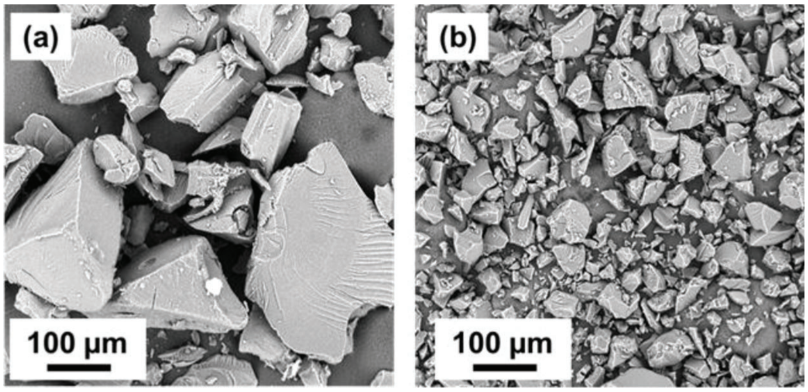


Figure 3.1 SEM images of the dry (a) LA-SAP and (b) HA-SAP particles used in the experiments.

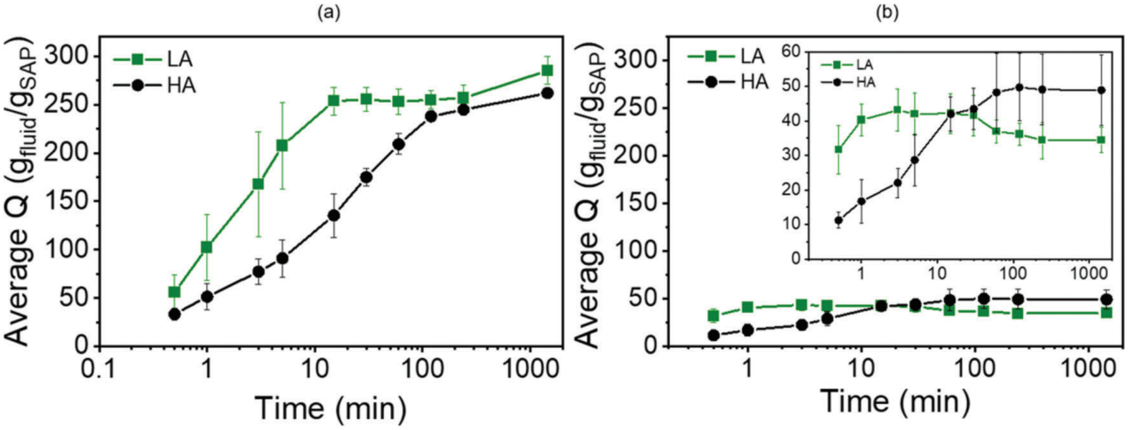


Figure 3.2 Absorption capacity of SAP as a function of immersion time in (a) RO water and (b) simulated pore solution from B Type I cement (inset displays the same data using a smaller range of values on a vertical axis to show greater detail).

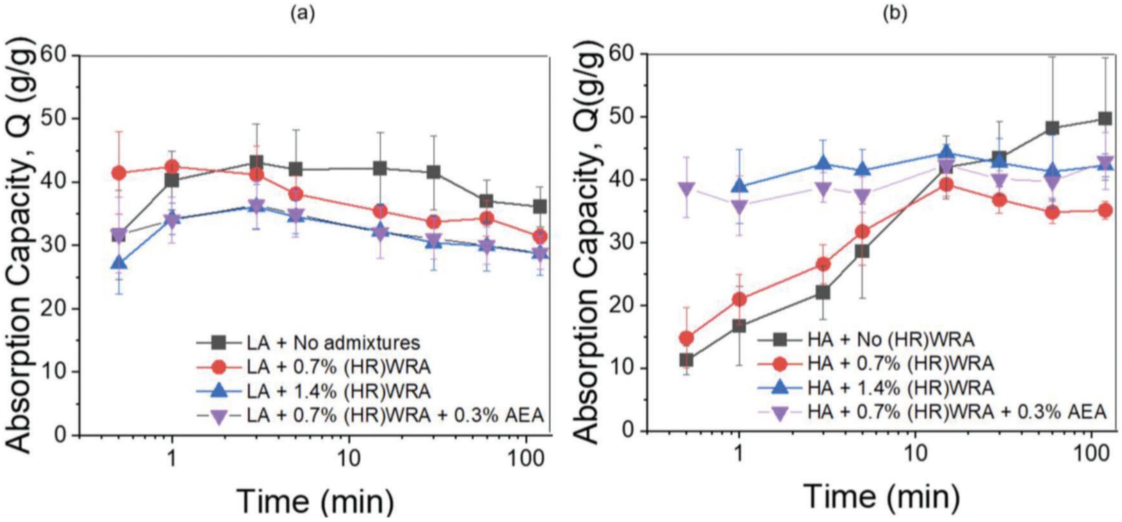


Figure 3.3 Absorption capacity as a function of immersion time in B Type I simulated pore solution with varying (HR)WRA and AEA dosages for (a) LA-SAP and (b) HA-SAP.

acid) which are known to facilitate relatively rapid absorption and desorption in pore fluids (Krafcik & Erk, 2016; Zhu et al., 2015).

There was no significant difference in pore fluid absorption capacity of LA-SAP in the presence of (HR)WRA at a dosage of 0%, 0.7%, or 1.4% by weight of cement (Figure 3.3a). Nor was there any significant difference in pore fluid absorption capacity when (HR)WRA was added to the simulated pore fluid in combination with 0.3% AEA. This suggests that the use

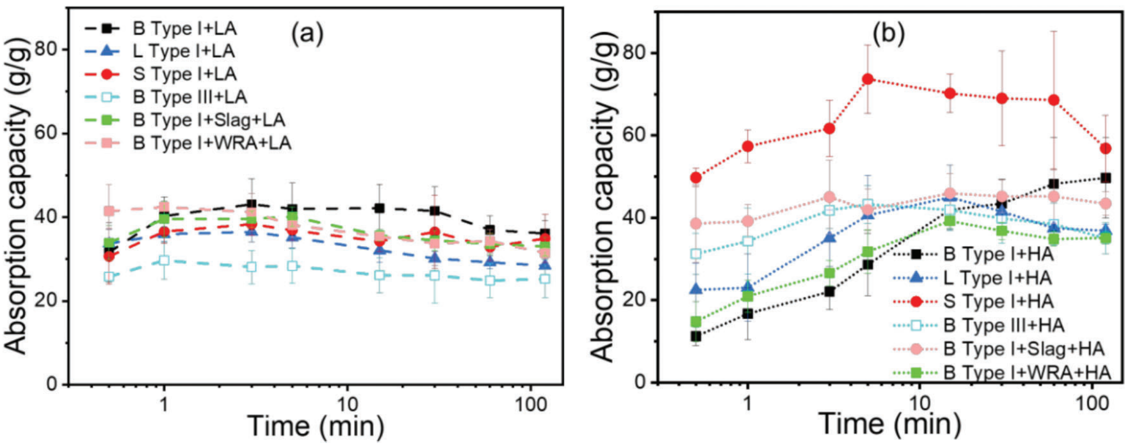


Figure 3.4 Absorption capacity of (a) LA-SAP particles and (b) HA-SAP particles as a function of immersion time in pore solution. Absorption capacity of the LA- and HA-SAP particles are plotted using dashed and dotted lines, respectively.

of common concrete admixtures does not affect the LA-SAP's function as an internal curing agent.

As shown in Figure 3.3b, the absorption capacity of HA-SAP was elevated through the first 5 minutes in the presence of larger dosages of chemical admixtures (1.4% (HR)WRA and 0.7% (HR)WRA + 0.3% AEA). Starting at 15 minutes and continuing through 2 hours, the absorption capacity of HA-SAP was comparable at all admixture dosages. As such, any effect of chemical admixtures on HA-SAP is likely limited to the first minutes of mixing.

3.3.2.2 Absorption capacity in simulated pore fluid made from different cement sources and types. The absorption capacity of the LA- and HA-SAP particles in pore solutions of different compositions are shown in Figure 3.4. The tests were denoted as X+Y where X and Y represents the binder used to create the pore solution and type of SAP particles, respectively. For instance, B Type I+Slag+LA represents absorption capacity tests conducted using LA-SAP particles in pore solution comprised of Buzzi Type I cement and slag cement. The absorption capacity tests indicate that pore solution composition had less influence on the absorption capacity LA-SAP particles, when compared to the HA-SAP particles.

After the first 30 minutes of exposure to the B-Type I pore solution, the LA-SAP particles had a significantly (~30%) lower equilibrium absorption capacity compared to HA-SAP particles. While SAP compositions are unknown, it has been demonstrated previously that either an increase in anionicity (Krafcik & Erk, 2016) or increase in crosslinking density will decrease the equilibrium absorption capacity of SAP (Davis et al., 2019; Zhu et al., 2015). In addition, the initial absorption kinetics for the first few minutes indicate that the LA-SAP particles absorbed fluid more rapidly than the HA-SAP particles even though the HA-SAP particles were of smaller average size. While insights into this phenomenon are not possible without information regarding SAP composition, it has been seen in the past that smaller particles generally absorb more quickly (Zhu et al., 2015). However, it is also possible that the smaller average size of the HA-SAP particles gave rise to a phenomenon known as "gel blocking" where the flow of water into the teabag is physically blocked as the small particles aggregate and swell (Kelly, 2017).

The varying composition of the pore solution had less impact on the absorption capacities of LA-SAP particles, when compared to the HA-SAP particles. In fact, a closer look at the equilibrium absorption capacities (at 2 hour) indicates that the upper and lower equilibrium absorption capacities obtained for LA-SAP particles only differ by 30%. It has been shown by previous research in Erk's group that the increase in acrylamide content in the SAP composition would result in lesser vulnerability to pore solution composition (Bose et al., 2021; Erk & Bose, 2018; Krafcik & Erk, 2016). The lowest absorption capacities of LA-SAP particles obtained for the B Type III pore solution can easily be explained by the fineness of Type III cement, which, at 593 m^2/kg, was highest of the four different cement compositions used in this study (B Type I: 412 m^2/kg, L Type I: 418 m^2/kg, S Type I: 376 m^2/kg). The higher fineness of the cement grains would allow dissolution more quickly, and thus the pore solution would contain greater concentrations of alkali ions. Greater amount of alkali ions present in the pore solution of the gravimetric test would result in greater complexation with the polymer backbone, thus resulting in overall lower absorption capacities.

The difference in pore solution composition had a greater effect on HA-SAP particles, when compared to the LA-SAP particles, as evidenced by the much wider range in both initial (40 g/g for HA vs. 20 g/g for LA) equilibrium (30 g/g for HA vs. < 20 g/g for LA) absorption capacities across pore solution concentrations.

3.4 Conclusions and Implications

The following conclusions resulted from this SAP characterization study.

- Short-term SAP absorption behavior did not predict the long-term (i.e., equilibrium) absorption capacity of SAP in simulated pore fluid.
- Absorption capacities of SAP immersed in cementitious pore fluid were reduced compared to SAP immersed in pure water.
- The equilibrium absorption capacities of both LA- and HA-SAP were insensitive to (HR)WRA and AEA dosage in simulated pore fluids.
- HA-SAP displayed a greater absorption capacity sensitivity to changes in cement chemistry than LA-SAP.

A number of implications resulted from this study. First, relative absorption kinetics of SAPs may display opposite behavior to relative SAP equilibrium absorption capacities. Thus, when selecting SAP for internal curing, short-term absorption behavior (<30 minutes) could serve as a selection criterion in addition to, or instead of, the long-term equilibrium absorption capacity (>2 hrs), depending on design needs. Second, common concrete admixtures did not affect the equilibrium absorption behavior of either SAP studied. This suggests that typical concrete admixtures can be used in combination with SAP without any significant change in the action of SAP. The effect of changes in cement source and chemistry on SAP internal curing behavior could be minimized by selecting nonionic SAPs.

4. OBJECTIVE 2A: EVALUATION OF BATCHING SEQUENCE AND VERIFICATION OF SAP IN CONCRETE

4.1 Overview

This section describes the research results obtained during the investigation of batching sequence and verification of SAP's presence when using SAP. The objective of this study was to determine the effects of the order of addition of SAP during the concrete batching sequence on the resultant concrete properties as well as to determine methods for verification of the presence of SAP particles in fresh concrete. This objective was accomplished first by evaluating two batching sequences with respect to the effect on slump, air content, compressive strength, and microstructure of concrete, and second by participation in a RILEM TC 260-RSC interlaboratory study on SAP verification in as-delivered concrete. The primary finding from the batching sequence study was that previously mentioned concrete properties were insensitive to the point of addition of SAP (i.e., it did not matter whether SAP was added to the cement or to the aggregate in batching sequence). The primary finding from the SAP presence verification study was that qualitative assessment could be performed on-site in minutes via sieving, while quantitative assessment required lab-scale testing.

4.2 Methods

Two approaches to adding dry SAP particles during the concrete mixture batching sequence were evaluated: (1) SAP-to-cement and (2) SAP-to-aggregate. Both sequences were prepared in a Lancaster low shear mixer (Lebanon, PA) using oven-dried aggregates with additional water added to bring the aggregates to saturated surface dry conditions (SSD). In both sequences, fine and coarse aggregate were mixed first for 2 minutes. This was followed by addition of cement and then followed by addition of water, (HR)WRA, and AEA. B Type I cement (13.6 kg), #23 fine aggregate (9.6 kg), #8 coarse aggregate (13.6 kg), tap water (2.5 kg) MasterGlenium 7511 (1.0% bwoc), MasterAir AE 200 (0.3% bwoc), and LA-SAP were used. All components were mixed for another 2 minutes before testing. Typical lab procedure for batching sequence is SAP-to-cement where dry SAP particles are stirred into cement to promote distribution of the SAP particles throughout the mix before the addition of water. To evaluate sensitivity of properties to batching, a SAP-to-aggregate method was tested, concrete was batched starting by mixing dry fine and coarse aggregate with SAP before the addition of cement, water, (HR)WRA, and AEA.

Air content of fresh concrete was measured using ASTM C231 (ASTM, 2017). Slump (ASTM, 2015) was measured immediately and at 30 minutes after the addition of water to the mix. Concrete was cast into 4 × 8″ cylinders for compression testing at 7 and 28 days (ASTM, 2018a). Three specimens were cast for each mix order for testing at each age. Microstructure samples were cut from purpose-cast cylinders at 7 days and hydration halted with isopropanol for backscattered SEM analysis.

Qualitative and quantitative verification of SAP in fresh mortar were tested using two acrylate SAPs of unknown composition with different maximum particle sizes, referred to here as "large" and "small." High aggregate load mortar (cement:fine aggregate of 1:5.5) at 0.40 w/c with 0.15% SAP bwoc and 0.8% (HR)WRA was used for testing.

4.3 Results

4.3.1 Fresh Concrete Properties

The SAP-to-cement sequence resulted in concrete with air content of 5.8%, an initial slump of 6.6 inches, which increased to 6.8 inches at 30 minutes. The SAP-to-aggregate sequence produced concrete with a lower air content of 4.4% and lower slump of 4.5 inches, which fell to 3.7 inches at 30 minutes. The targets established with the input of the study advisory committee for air content and slump were 5%–8% and 5–8 inches, respectively. The SAP-to-cement concrete fell within these ranges, while the SAP-to-aggregate was slightly under in both air content and slump.

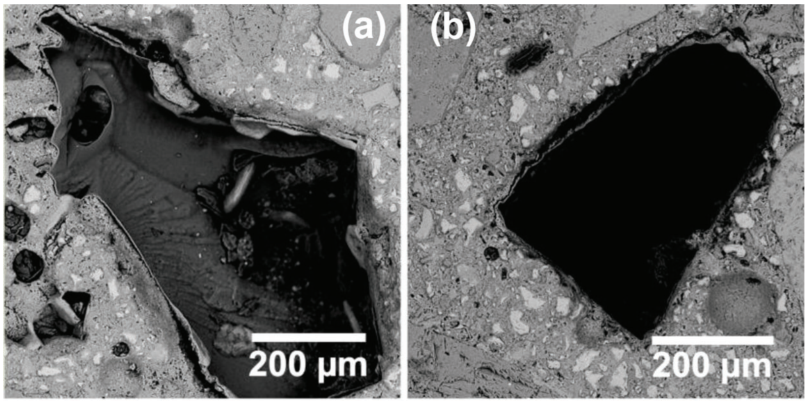


Figure 4.1 SEM micrographs of 7-day 0.42 w/c concrete samples with (a) LA-SAP added to cement and (b) LA-SAP added to aggregate. The remnants of the dehydrated LA-SAP particle were clearly visible as the grey film in the void shown in (a). The HA-SAP particle remnants were less visible but still present in the void shown in (b).

4.3.2 Hardened Concrete Properties

SAP-to-cement and SAP-to-aggregate orders of addition displayed comparable compressive strength at 7 days (6,440 psi and 6,270 psi, respectively; p-value = 0.22, all t-tests were two-tailed hypothesis tests conducted at the 5% significance level). At 28 days, SAP-to-cement (7,212 psi) displayed increased strength over SAP-to-aggregate (6,945 psi). While this difference in compressive strength is statistically significant (p-value = 0.04), it only amounts to a 4% improvement in compressive strength.

SEM analysis showed similar microstructure and SAP distribution between the two orders of addition (Figure 4.1). Prior to analysis, SAP distribution in the SAP-to-aggregate sample was of particular interest—would the SAP particles stick or otherwise concentrate around the aggregates, potentially disrupting the interfacial transition zone? No evidence of this behavior was observed.

4.3.3 Verification of SAP

Qualitative verification of SAP was performed via sequential sieving of approximately 0.2 kg of the prepared mortar. First, mortar was rinsed through a 2 mm (#10) sieve with tap water to remove aggregates. The remaining slurry was then rinsed through a 0.063 mm (#230) sieve with tap water to collect SAP particles. At this time, positive or negative verification of the presence of SAP in the original mortar was possible via visual observation and tactile inspection of the soft wet SAP particles (Figure 4.2). The entire procedure was completed in less than 10 minutes. Any sieves of comparable magnitude mesh sizes could be used to similar effect.

Quantitative verification of SAP required careful collection of all SAP from a known quantity of mortar. To promote complete separation of SAP from higher density aggregates and cement, approximately 2 L of mortar was massed and placed in a five-gallon bucket along with an excess of tap water (approximately 10 L). The contents of the bucket were swirled into a whirlpool for 1 minute and then allowed to settle. SAP is less dense than water and did not settle with the aggregates and the majority of the cement. All liquid and floating solids (including SAP) were poured off through a 0.125 mm sieve. Contents of the sieve were examined for qualitative verification of SAP and collected. The collected sample was dried to constant mass, then oven-heated to 500°C for 3 hours, to burn off the SAP fraction of the sample without degrading any mineral content. The sample was then massed again to find the mass of SAP burned off, which was compared to the expected mass of SAP in the starting mortar sample. The percent error in experimental measurement of SAP was higher (14%) for the small SAP particles than for the large SAP particles (9%) due to difficulty in reclaiming the sample from the 0.125 mm sieve. Sample collection was performed in approximately 10 minutes, with a total experimentation time of 3 days. A similar magnitude sieve of a different mesh size could be used.

4.4 Conclusions and Implications

The following conclusions resulted from this batching sequence study.

- The slump and air content of SAP-to-cement fell within the target ranges for concrete in this study, while the slump and air content of SAP-to-aggregate fell slightly below the target ranges for concrete in this study.
- The batching sequence of SAP-to-cement concrete yielded comparable or minorly improved strength at both 7 and 28 days compared to SAP-to-aggregate concrete.
- SAP showed no signs of interference with the interfacial transition zone, "sticking" or concentrating around the aggregates, or agglomeration within the cement matrix in the SEM analysis of either order of addition.
- Qualitative verification of the presence of SAP in fresh mortar was performed in minutes using field-ready equipment.
- Quantitative verification of the amount of SAP in fresh mortar was performed at lab-scale with 9%–14% error.

SAP A: large particles

SAP B: small particles

Figure 4.2 SAP collected on a 0.063 mm sieve from fresh mortar samples for visual and tactile verification.

A number of implications resulted from this study. First, for the same AEA and (HR)WRA dosage and conventional SAP dosages, addition of SAP-to-cement yielded higher air content and slump values, suggesting that this order of addition would require lower admixture dosages than that of cement-to-aggregate to achieve the same target air content and slump values. Second, SAP distribution within the mixture was insensitive to the order of addition. Seven-day compressive strength of concrete was also insensitive to the order of addition of SAP and the increased strength gain at 28 days displayed in SAP-to-cement concrete was minimal. Combined, these results indicate that in terms of hardened properties, concrete internally cured with SAP yields similar properties regardless of order of addition of SAP, meaning that the order of addition could be selected to optimize processing workflow without sacrificing material properties. Furthermore, verification of SAP in as-delivered concrete could be performed in minutes on-site, with follow-up quantitative lab-scale testing of delivered SAP dosage possible within days.

5. OBJECTIVE 2B: EVALUATION OF THE INFLUENCE OF MIXTURE COMPOSITION ADJUSTMENTS

5.1 Overview

This section describes the research results obtained during the investigation of the influence of changes in the quantities water and (HR)WRA in SAP mortars on their fresh and hardened properties. This objective was accomplished by changing the composition of mortars using three different approaches and evaluating the effects of these changes with respect to flow, compressive and flexural strengths, and microstructure. The primary finding was that mortar mixes with w/c ≥ 0.42 and enough (HR)WRA to produce a flow of 110% ± 10% could be modified by addition of a typical SAP dosage (0.2% by weight of cement) and still develop a flow of at least 45% at the time of casting, which would be sufficient for casting of adequate quality specimens. A complete, detailed description of the experimental methods, quantitative results and supporting discussion was published in *Construction and Building Materials* in 2022 (refer to Appendix A).

5.2 Methods

Three approaches were used for mixture compositions: *Approach I*, where only extra water was added to compensate for SAP absorption and no (HR)WRA was used; *Approach II*, where only (HR)WRA was used and the water amount was kept constant; and *Approach III*, where both extra water and (HR)WRA were varied. The amount of extra water added was determined using two common methods: (1) addition of a fixed (i.e., 5%) amount and (2) addition of an amount determined by measuring the pore solution equilibrium absorption capacity of the specific SAP (Mechtcherine et al., 2014, 2017; Wyrzykowski et al., 2020). While the *ex situ* equilibrium absorption capacity of SAP would not equal its *in situ* absorption capacity, this experimental measure provided a consistent means by which to customize the extra water dosage to the specific SAP.

The compositions of the mortar mixtures used for each of the three approaches are reported in Table 5.1. The samples are identified as X_A%_B% where X represents the type of SAP used (if any), A is the % of extra water added to compensate for SAP absorption and B is the % of (HR)WRA used to maintain flow. In this report, the dosages of extra water and (HR)WRA are always given as percent by weight of cement (% bwoc) or percent by weight of binder (% bwob). For example, No SAP_7%_0% did not include any SAP, had 7% extra water, and no (HR)WRA, while LA_5%_0.3% used LA-SAP, had 5% extra water, and 0.3% (HR)WRA.

Approach III was also applied to design mixes with alternate cement sources, types, and with partial slag replacement as shown in Table 5.2. These mixes were evaluated via the mortar flow table test from 10 minutes to 120 minutes after the addition of water to cement (ASTM, 2020c). Compressive and flexural strength were tested at 3, 7, and 28 days with three specimens for each mix at each age (ASTM, 2020a, 2020b).

TABLE 5.1
Composition of mortar mixtures

Mix Design	Approach Number	Sample	Water (g)	SAP (g)/ (% bwoc)	(HR)WRA (mL)/ (% bwoc)	$(w/c)_{total}$	Extra Water (% bwoc)	Initial % Flow (at 10 min)
1	I	No SAP_0%_0%	1,610	0/0	0/0	0.42	0	66
2[1]	I	LA_0%_0%	1,610	7/0.2	0/0	0.42	0	21
3[1]	I	HA_0%_0%	1,610	7/0.2	0/0	0.42	0	31
4	I	No SAP_5%_0%	1,786	0/0	0/0	0.47	5	100
5	I	LA_5%_0%	1,786	7/0.2	0/0	0.47	5	49
6	I	HA_5%_0%	1,786	7/0.2	0/0	0.47	5	64
7	I	No SAP_7%_0%	1,852	0/0	0/0	0.49	7[2]	102
8	I	LA_7%_0%	1,852	7/0.2	0/0	0.49	7[2]	62
9	I	No SAP_10%_0%	1,954	0/0	0/0	0.52	10[2]	124
10	I	HA_10%_0%	1,954	7/0.2	0/0	0.52	10[2]	100
11	II, III	No SAP_0%_0.5%	1,610	0/0	17.5/0.5	0.42	0	113
12	II	LA_0%_1.45%	1,610	7/0.2	50.8/1.45	0.42	0	104
13	II	HA_0%_1.15%	1,610	7/0.2	40.3/1.15	0.42	0	120
14	III	LA_0%_0.5%	1,610	7/0.2	17.5/0.5	0.42	0	57
15	III	HA_0%_0.5%	1,610	7/0.2	17.5/0.5	0.42	0	93
16	III	No SAP_5%_0.3%	1,786	0/0	10.5/0.3	0.47	5	120
17	III	LA_5%_0.3%	1,786	7/0.2	10.5/0.3	0.47	5	63
18	III	HA_5%_0.3%	1,786	7/0.2	10.5/0.3	0.47	5	91
19	III	No SAP_7%_0.27%	1,852	0/0	9.5/0.27	0.49	7[2]	124
20	III	LA_7%_0.27%	1,852	7/0.2	9.5/0.27	0.49	7[2]	83
21	III	No SAP_10%_0.13%	1,954	0/0	4.4/0.13	0.52	10[2]	135
22	III	HA_10%_0.13%	1,954	7/0.2	4.4/0.13	0.52	10[2]	98

Note: All mixes were prepared using B Type I cement. Cement and sand amounts were fixed at 3,500 g and 9,625 g, respectively, for all mixes.

[1] Mix was not cast due to insufficient workability (flow at 10 minutes was less than 45%).

[2] Extra water percent was determined by the equilibrium absorption capacity in pore solution of the relevant SAP (7% for LA-SAP and 10% for HA-SAP.) Full equilibrium absorption capacity results are reported in Section 3.3.

TABLE 5.2
Composition of 0.42 w/c mortar mixtures with varying binder compositions

Mix Design	Sample	Cement (g)	Slag (g)	Water (g)	SAP (g)/(% bwoc)	(HR)WRA (mL)/ (% bwoc)	Initial % Flow (at 10 min)
23[1]	B Type I_No SAP_0.5%	3,500	0	1,610	0/0	17.5/0.5	113
24[2]	B Type I_HA_0.5%	3,500	0	1,610	7/0.2	17.5/0.5	93
25	L Type I_No SAP_0.4%	3,500	0	1,610	0/0	14/0.4	107
26	L Type I_HA_0.4%	3,500	0	1,610	7/0.2	14/0.4	66
27	S Type I_No SAP_0.5%	3,500	0	1,610	0/0	17.5/0.5	120
28	S Type I_HA_0.5%	3,500	0	1,610	7/0.2	17.5/0.5	88
29	B Type III_ No SAP_0.9%	3,500	0	1,610	0/0	31.5/0.9	110
30	B Type III_ HA_0.9%	3,500	0	1,610	7/0.2	31.5/0.9	56
31	B Type I + Slag_No SAP_0.3%	2,450	1,050	1,610	0/0	10.5/0.3	104
32	B Type I + Slag_HA_0.3%	2,450	1,050	1,610	7/0.2	10.5/0.3	32

Note: All mixes designed following Approach III. Total binder, sand, and water amounts were fixed at 3,500 g, 9,625 g, and 1,610 g, respectively, for all mixes

[1] Same as Mix Design 11 in Table 5.1.

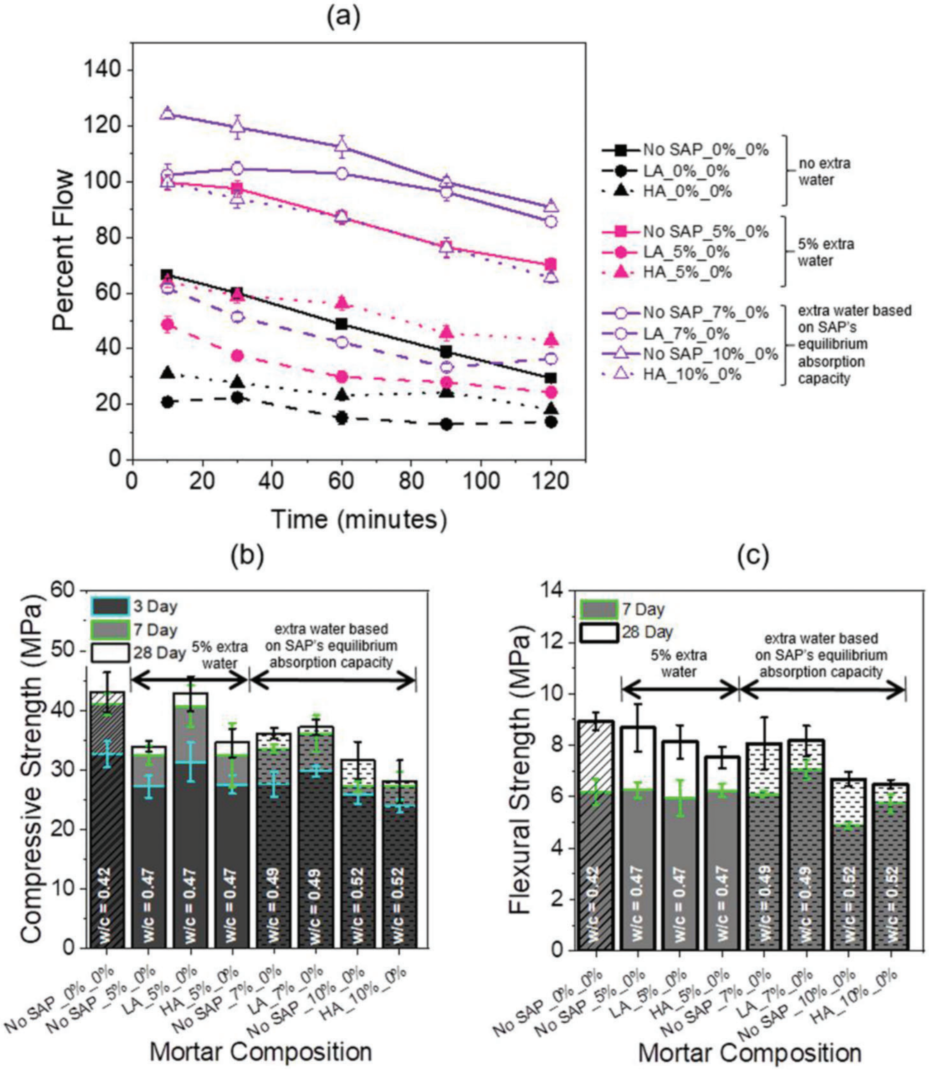
[2] Same as Mix Design 15 in Table 5.1.

5.3 Results

5.3.1 Results from Approach I (addition of 5% of extra water)

For each w/c, samples containing SAP had a lower percent flow than the reference sample without SAP (Figure 5.1a). For example, the No SAP_0%_0% mixture had an initial (i.e., 10 minutes after mixing) percent flow of 66%, while LA_0%_0% mixture had an initial percent flow of 21% and HA_0%_0% had an initial percent flow of 31%. When present, SAP absorbed mixing water and as expected, the percent flow of the mortar decreased (Mechtcherine et al., 2015).

Interestingly, the addition of LA-SAP resulted in a 10–16 percentage points greater drop in percent flow of mortar than the addition of HA-SAP, despite the fact



Note: No (HR)WRA was added to these set of mixes. In Figures (b) and (c), bars with diagonal hatches represent mortars with w/c 0.42 prepared without extra water, bars with no hatches represent mortar mixes with 5% extra water, and bars with horizontal dashes represent mortar mixes with extra water amount based on SAP's equilibrium absorption capacity in synthetic pore solution.

Figure 5.1 (a) Percent flow over time of fresh mortar mixes, (b) compressive strength, and (c) flexural strength of mortar containing extra water to compensate for SAP absorption.

that at the equilibrium time ($t_{abs} > 2$ hrs), HA-SAP had a higher absorption capacity in simulated pore solution than LA-SAP (refer back to Figure 3.2b). As previously noted in Section 3.3.2.1, LA-SAP displayed a higher absorption capacity than HA-SAP during the first 30 minutes of gravimetric absorption tests in simulated pore solution. Thus, while equilibrium absorption capacity was useful for characterizing SAPs of different compositions, it did not logically predict the relative effect of SAP composition on percent flow of mortar.

Based on laboratory observations, mortars with an initial flow of less than 45% had insufficient workability to be cast into viable specimens. As such, mortars with initial flow of less than 45% were not used to cast specimens for mechanical testing. The flow of mortars containing SAP without extra water (Table 5.1, Mix Designs 2 and 3) was significantly less than 45%, confirming the need for attention to mix design when using SAP.

While the addition of 5% extra water to cementitious mixtures containing SAP is a common approach (Mechtcherine et al., 2017; Wyrzykowski et al., 2020), it did not result in mortars with sufficient workability for the LA-SAP. The addition of 5% extra water produced an initial percent flow of 49% which had fallen to 38% by 30 minutes and was thus insufficient for casting. As such, the addition of 5% extra water to SAP-containing mixtures does not universally guarantee sufficient workability for all mixing and placement timescales.

At a given w/c, the addition of SAP resulted in comparable or improved values of compressive and

TABLE 5.3
Percent of 28-day compressive and flexural strength of mortars attained by 7 days

Mix Design	Sample	% of 28-Day Compressive Strength Attained by 7 Days	% of 28-Day Flexural Strength Attained by 7 Days
1	No SAP_0%_0%	95	69
4	No SAP_5%_0%	96	72
5	LA_5%_0%	95	73
6	HA_5%_0%	94	83
7	No SAP_7%_0%	93	76
8	LA_7%_0%	97	87
9	No SAP_10%_0%	86	73
10	HA_10%_0%	97	89

flexural strengths. At w/c $\geq$ 0.49 the strength development was accelerated when using SAP, suggesting that the addition of SAP could benefit even high w/c mixes, which are not typically considered as candidates for internal curing.

SAP-containing 28-day mortar samples with 5% extra water (LA_5%_0% and HA_5%_0%) showed comparable (HA_5%_0%, p-value = 0.71) or improved (LA_5%_0%, p-value = 0.007) compressive strength and comparable (LA_5%_0%, p-value = 0.453; HA_5%_0%, p-value = 0.122) flexural strength when compared to the reference of the same w/c (No SAP_5%_0%) (Figure 5.1b and c). In fact, LA_5%_0% not only showed significantly increased compressive strength compared to the same (i.e., 0.47) w/c reference (p-value = 0.007) but displayed compressive strength comparable to the 0.42 w/c reference (p-value = 0.925), despite the fact that it had a w/c value ~12% higher.

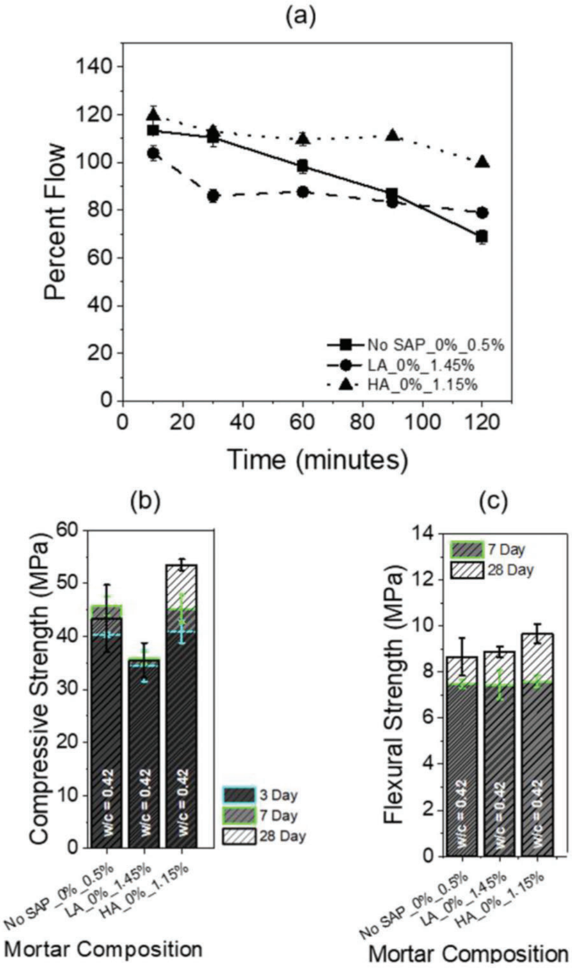
As SAP is an internal curing agent, it is useful to consider the indicators of hydration kinetics in evaluating its effects. In Figure 5.1b and c, it is visible that the relative strength gains from 7 to 28 days are not always the same between a reference sample (in this case sample with 5% extra water but no SAP) at a certain w/c and a SAP-containing sample at the same w/c. To quantify that difference, Table 5.3 shows the percent of 28-day compressive and flexural strengths attained by 7 days for each sample prepared using *Approach I* (just addition of extra water). This metric serves as an indicator of rate of strength development. By 7 days, both of the SAP + 5% extra water samples (mix designs 5 and 6) attained, respectively, 95% and 94% of their 28-day compressive strength. These values were very similar to the 96% of the 28-day compressive strength attained by the reference sample (mixture 4, No-SAP + 5% of extra water). Interestingly, at 7 days samples from mixture 6 (HA_5%_0%) attained a higher percent of their 28-day flexural strength (83%) than those observed for either mixture 5 (LA_5%_0%) or the reference mix design 4 (No SAP_5%_0%), 73% and 72%, respectively.

Mix designs 7, 8, 9, and 10 contained extra water in the amounts determined from the equilibrium absorption capacity data for LA-SAP (mix designs 7 and 8) and HA-SAP (mix designs 9 and 10). Both the LA- and HA-samples attained compressive and flexural strength values comparable to those obtained from reference mortar with the same w/c value. The results of the t-tests (comparing each sample to its associated reference) resulted in p = 0.33 and p = 0.85 for 7% extra water compressive and flexural strength values, respectively; and p = 0.25 and p = 0.38 for 10% extra water compressive and flexural strength values, respectively. These similar values of strengths suggest that the addition of SAP may not decrease compressive or flexural strength at a given total w/c.

For both LA_7%_0% and HA_10%_0%, there was an accelerated strength gain through 7 days for the SAP-containing samples compared to their No-SAP references (see Table 5.3). The difference in percentage strength gained between the two samples can be interpreted as a difference in the rate of strength development. LA_7%_0% showed 4 percentage points accelerated compressive strength gain and 11 percentage points accelerated flexural strength gain over No-SAP_7%_0%. HA_10%_0% displayed more aggressive accelerated strength gain of 11 percentage points for compressive strength gain and 16 percentage points accelerated flexural strength gain over No SAP_10%_0%. This increase in percentage of 28-day compressive strength attained by 7 days for higher w/c mortars suggests that high-w/c mortars internally cured with SAP may hydrate more rapidly (and develop higher early-age strength) than similar mortars without SAP, while attaining comparable 28-day strengths.

5.3.2 Results from Approach II (addition of (HR)WRA while keeping the amount of water constant)

In Approach II, the (HR)WRA dosage was varied for each mix to achieve a set target flow of 110% ± 10% at 10 minutes after the addition of water. As seen in Figure 5.2a, the target flow was achieved for all three sample mortars containing no SAP, LA-SAP, and HA-SAP (mix designs 11, 12, and 13, respectively, in Table 5.1). The (HR)WRA dosage required did not correspond to the relative equilibrium absorption capacities of the low- and high-absorption SAPs. Specifically, the mortar containing LA-SAP required a higher (HR)WRA dosage (1.45%) than the mortar



Note: The required dosage of (HR)WRA was defined as the amount needed to achieve the mortar flow of 110% ± 10% 10 minutes after the addition of water to the mixture. In Figures (b) and (c), diagonal hatches indicate mortar with w/c 0.42 without extra water, consistent with representations of the types of mortar mixtures given in Figure 5.1.

Figure 5.2 (a) Percent flow over time of fresh mortar mixes, (b) compressive strength, and (c) flexural strength of mortar samples with the addition of (HR)WRA and no extra water.

containing HA-SAP (1.15%) to achieve the target flow, indicating that absorption capacity is not the only property of SAP that affects the flow of mortar.

The 28-day values of compressive and flexural strengths of both SAP-containing mortars were maintained compared to the no SAP reference mortar (Figure 5.2b,c). However, at 3 and 7 days, the compressive strength of LA-SAP mortar was lower relative to the no SAP reference (3-day p-value = 0.03, 7-day p-value = 0.001, 28-day p-value = 0.13, at the 5% significance level), likely due to the high (HR)WRA dosage of 1.45% retarding early compressive strength development (Esen & Orhan, 2016; Han et al., 2013). This suggests that early and long-term strength can be maintained for SAP-cured mortars if a (HR)WRA dosage needed to achieve target flow is below the onset of segregation.

5.3.3 Results from Approach III (addition of (HR) WRA and extra amount of water)

Approach III, where (HR)WRA dosage was adjusted such that a mortar without SAP at the given w/c flowed at least 110% ± 10%, produced mortar with sufficient flow for casting for all mixes tested (Figure 5.3), suggesting that this method may represent a viable general approach when employing SAP for internal curing purposes.

The general trends in compressive and flexural strength previously observed in *Approach I* were also observed in *Approach III* (Figure 5.4). Specifically, at a given w/c, compressive and flexural strengths were either preserved or improved when using SAP at all ages. As such, in order to maximize compressive and flexural strength values, no extra water should be added

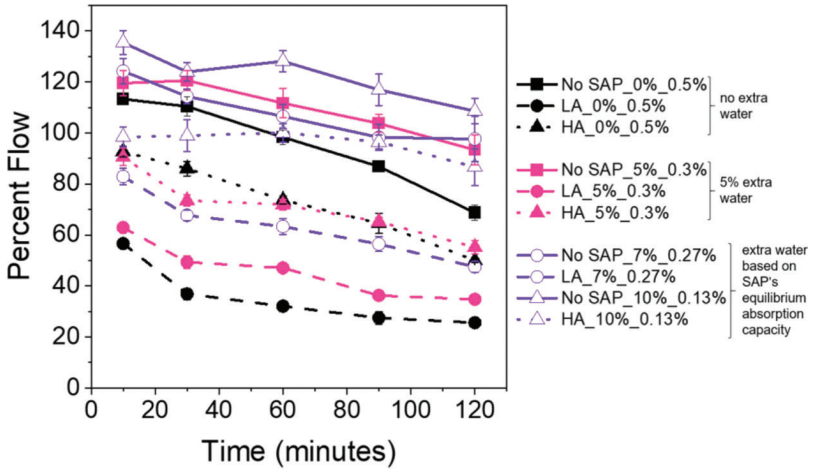


Figure 5.3 Percent flow of mortars over time with and without extra water and with the (HR)WRA dosage necessary to achieve at least 110% ± 10% flow in No SAP mortars at the given total w/c 10 minutes after the addition of water to the mix.

when using SAP if sufficient flow can be achieved with the use of the (HR)WRA.

For the HA_10%_1.3% SAP-containing specimens with w/c of 0.52 there was a 13 percentage point accelerated compressive strength gain through 7 days compared to No SAP_10%_1.3% specimens. There was no similar accelerated strength gain for the 0.49 w/c SAP-containing specimen, i.e., LA_7%_2.7%. While the average 7-day compressive strength of LA_0%_0.5% was higher than its average 28-day compressive strength, the two values were comparable (p-value = 0.126). In *Approach I*, the SAP-containing samples at both 0.49 and 0.52 w/c displayed accelerated strength gain with greater acceleration associated with the higher w/c values (Section 3.2.4). In *Approach III*, the accelerated strength gain due to the addition of SAP to 0.52 and 0.49 w/c mixtures may have been partially or completely counteracted by the addition of the polycarboxylate-based (HR)WRA as polycarboxylate-based admixtures have been shown to retard hydration and strength development (Li et al., 2017). When using (HR)WRA, a higher w/c (0.52) was necessary before any accelerated strength gain due to the addition of SAP was observed.

5.3.4 Results from Approach III with Varying Binders

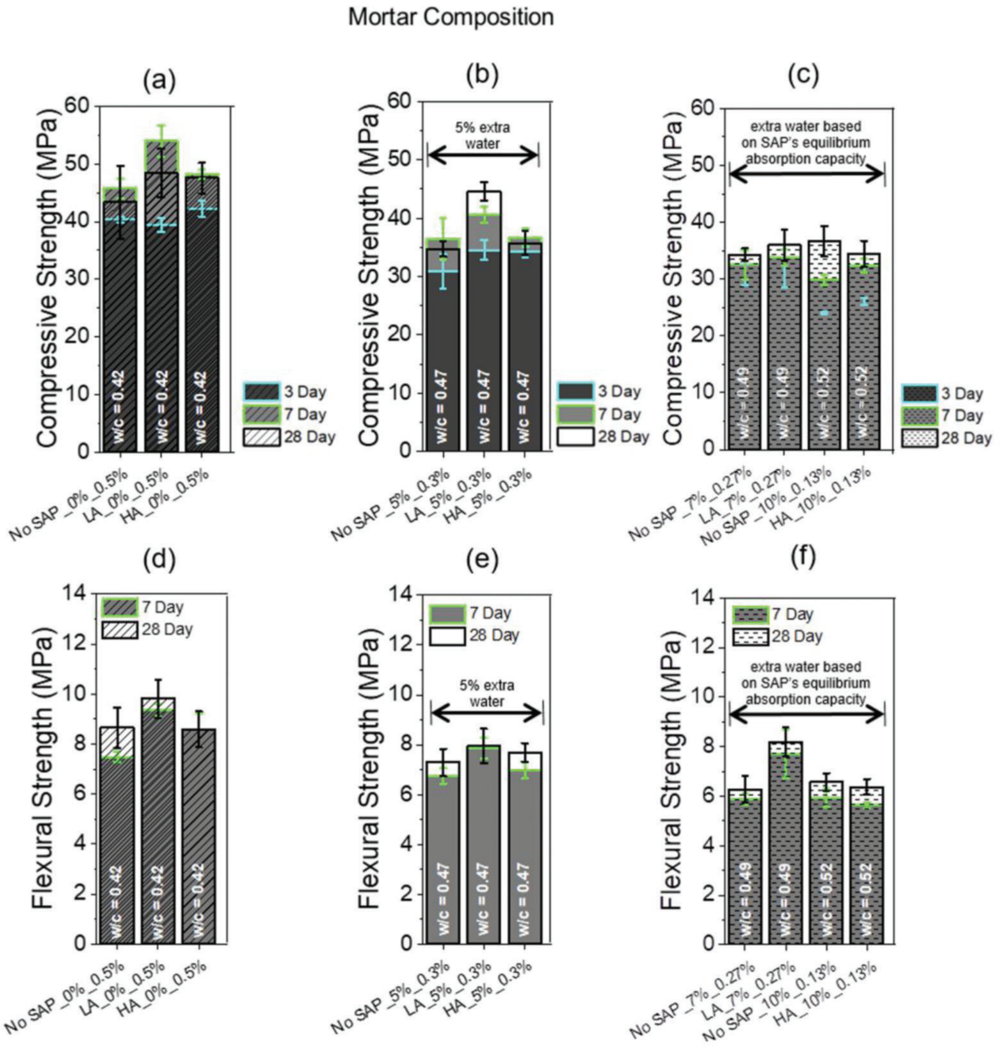
The percent mortar flow of mortars prepared with cements of different chemical compositions and different types is shown in Figure 5.5. B Type I was the cement used in all mixes previously detailed in Table 5.1. As expected, the addition of HA-SAP decreased the percent flow for all mixes. As shown in Figure 5.5a, the reduction in flow with the addition of HA-SAP to Type I mortars ranged from 20 to 42 percentage points. Figure 5.5b shows that when varying binder type among Type I cement, Type III cement, and Type I cement with 30% slag replacement, the addition of HA-SAP resulted in a wider range of reduction in percent flow. B Type I displayed the least change with a 21 point reduction in flow, while B Type III and B Type I + Slag displayed flow reductions of 54 and 72 points, respectively. As noted in Section 3.3.2.2, the absorption capacity of HA-SAP was dependent on pore solution composition and higher at short times for simulated pore solution containing B Type III cement and B Type I cement with 30% slag replacement. This increased absorption capacity at short times would result in a greater reduction in percent mortar flow as observed.

All Type I mortars flowed at least 45% at 10 minutes, sufficient to be cast into well-consolidated samples. However, B Type I + Slag_HA_0.3% had a percent mortar flow at 10 minutes of 32%. While Type I mortars with such low percent flow were unable to be cast into well-consolidated samples, compression and flexure samples of B Type I + Slag_HA_0.3% were able to be cast with some care. Additional (HR)WRA could be added to reach a target flow.

The compressive and flexural strength of mortars prepared with cements of different chemical compositions and different types are shown in Figure 5.6. All three Type I cement mortars followed the same compressive and flexural strength trends observed previously for Approach III (Section 5.3.3). Type I mortars displayed comparable compressive and flexural strength at all ages both with and without HA-SAP.

For B Type III mortars, the addition of HA-SAP resulted in a 22% increase in 3-day compressive strength over that of its No SAP reference, while yielding comparable 7- and 28-day compressive and flexural strengths. This suggests that the use of HA-SAP with Type III cement enhances the acceleration of strength development.

The addition of HA-SAP to B Type I mortar with 30% slag replacement resulted in an increase in 3- and 28-day compressive strength ($p = 0.007$ and $p = 0.015$, respectively). The increase in 3-day compressive strength is of particular interest given the significant



Note: Figures (a) and (d) show samples with no extra water. In Figures (b) and (e), samples with 5% of extra water added. In Figures (c) and (f), the amount of extra water was determined based on the equilibrium absorption capacity of the SAP used.

Figure 5.4 (a–c) Compressive strength and (d–f) flexural strength of mortar samples with the (HR)WRA dosage necessary to achieve at least 110% ± 10% flow in No SAP mortar at the given total w/c 10 minutes after the addition of water to the mix.

reduction in early age compressive strength when using slag replacement, compared to straight Type I cement. Flexural strength was comparable in slag replacement mortars with and without SAP at all ages.

These findings build on those in Section 5.3.3, showing that the addition of SAP to 0.42 w/c mortars without the addition of extra water resulted in comparable or improved compressive and flexural strength values for all binder systems tested.

5.4 Conclusions and Implications

The following conclusions resulted from this mixture composition adjustment study.

- Mix design adjustments were required when SAP was added to a 0.42 w/c Type I OPC mortar with a cement:sand ratio of 1:2.75 in order to cast well-consolidated specimens.
- In all mix designs tested across binder systems, when w/c was held constant, the addition of SAP preserved or increased compressive and flexural strength through 28 days. In other words, the addition of SAP alone did not decrease compressive or flexural strengths through 28 days.
- Adjustment of (HR)WRA alone could be used to achieve an initial target mortar flow of 110% ± 10 when using SAP.
- In high w/c (≥0.49) mixtures, the addition of SAP accelerated compressive and flexural strength development in samples without (HR)WRA (*Approach I*) and accelerated compressive strength development in samples with w/c = 0.52 and (HR)WRA (*Approach III*). It should be noted however, that increased w/c may negatively alter transport and durability properties, which were not evaluated in the course of this study.

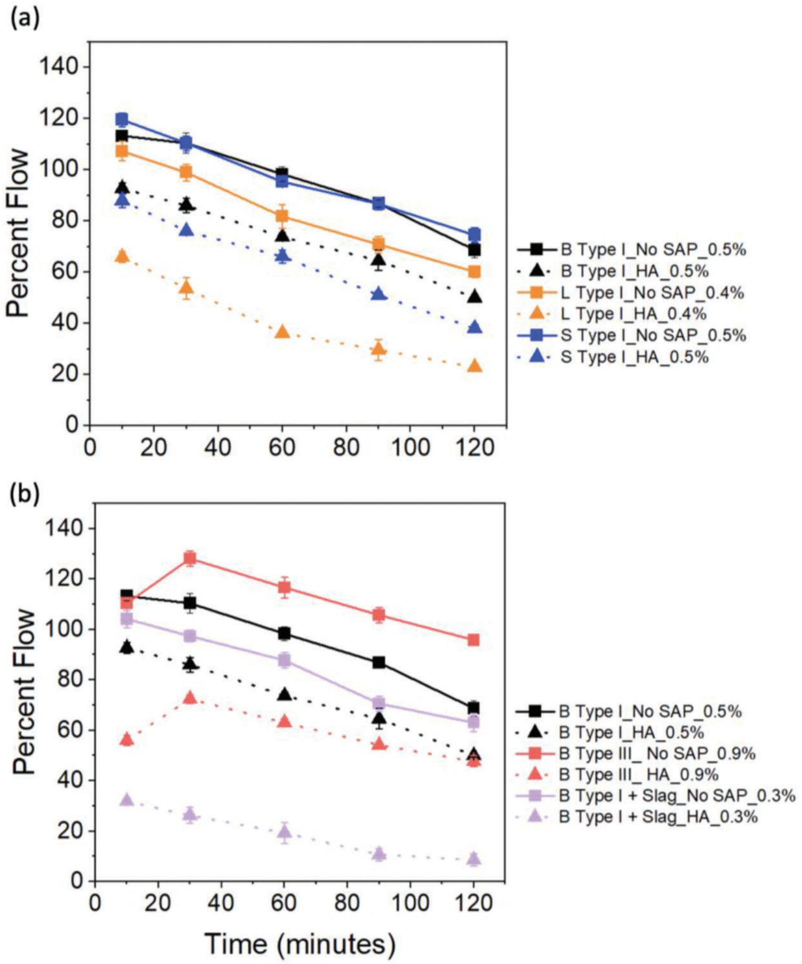


Figure 5.5 Percent flow of 0.42 w/c mortars over time with the (HR)WRA dosage necessary to achieve at least 110% ± 10% flow in No SAP mortars at the given total w/c 10 minutes after the addition of water to the mix. In (a) the source of Type I cement was varied. In (b) the binder type was varied.

- Type I mortar mixtures with w/c ≥ 0.42 and enough (HR) WRA to produce an initial flow of 110% ± 10% could be modified by addition of a typical SAP dosage (0.2% by weight of cement) and still develop a flow of at least 45% at 10 minutes after the addition of water, which was sufficient for casting of adequate quality specimens.

A number of implications resulted from this study. First, extra (HR)WRA could be used to increase flow when using SAP for all binder systems tested. Second, *Approach III* demonstrated a potential "rule of thumb" for mixture design adjustments when using SAP. SAP-free mixtures that have sufficiently high initial flow values (110% ± 10 for mortar) can be modified to include a typical 0.2% dosage of SAP to maintain or improve compressive and flexural strength without sacrificing overall workability. Second, the accelerated compressive and flexural strength gain displayed by SAP-containing mortars at higher w/c values (≥0.49) suggests that SAP may benefit mixtures at w/c above those usually considered for internal curing.

Workability of SAP-containing mixtures can be increased with (HR)WRA alone in order not to compromise the values of the compressive and flexural strengths. However, in instances where increased (HR)WRA dosage is cost-prohibitive or ultra-high workability is required, extra water could be added in a fixed amount of 5% (by weight of binder) as a reasonable offset to the water absorbed by SAP (Assmann & Reinhardt, 2014; Justs et al., 2015; Krafcik & Erk, 2016; Mechtcherine et al., 2017; Oh & Choi, 2018; Snoeck et al., 2017; Wyrzykowski et al., 2020) with minimal compromise of compressive and flexural strength.

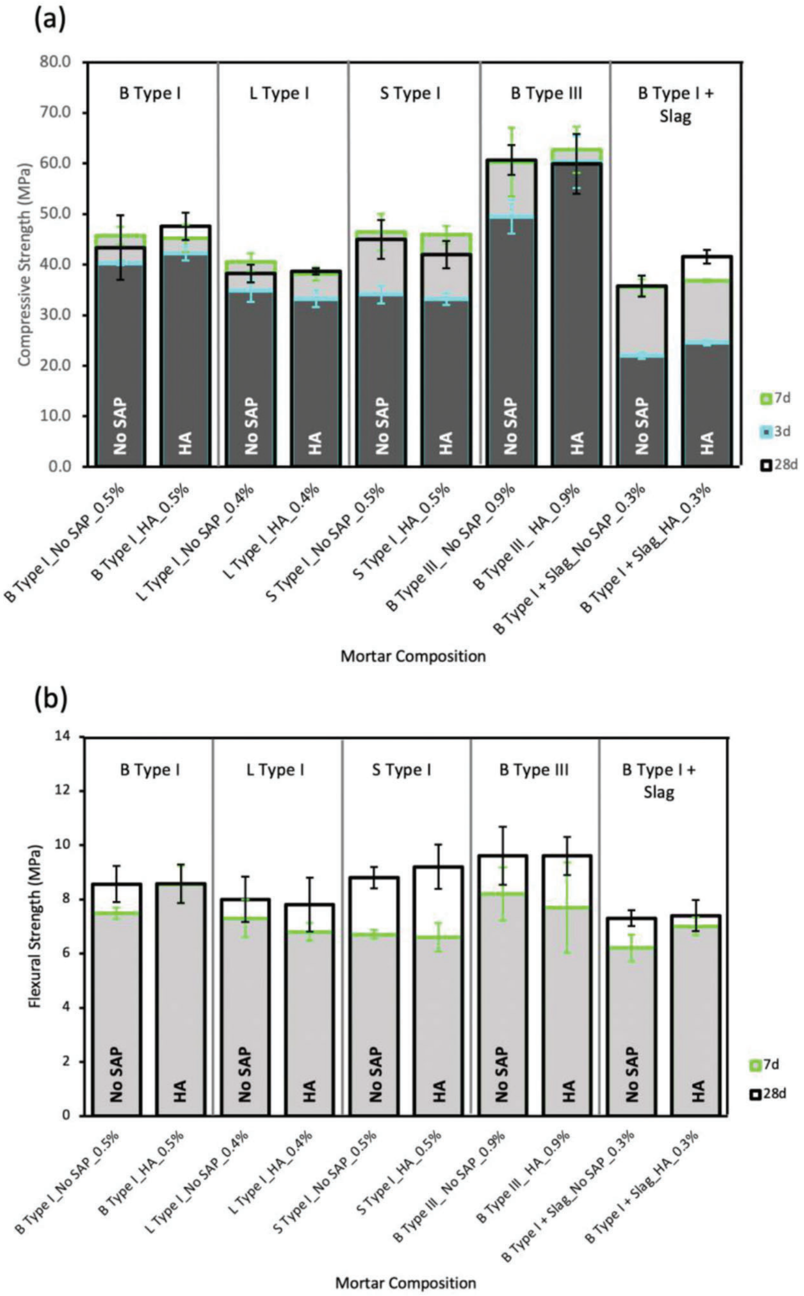


Figure 5.6 (a) Compressive strength and (b) flexural strength of 0.42 w/c mortar samples with varying binder sources and types with the (HR)WRA dosage necessary to achieve at least 110% ± 10% flow of No SAP mortar 10 minutes after the addition of water to the mix.

6. OBJECTIVE 3A: IMPACT OF SAP ON CEMENT HYDRATION

6.1 Overview

This section describes the research results obtained during the investigation of cement hydration when using SAP. The objective of this study was to evaluate the effect of internal curing with SAP on cement hydration. This objective was accomplished by conducting isothermal calorimetry and non-evaporable water content testing on cement pastes with and without SAP. Microstructural analysis of hydration products was performed on both mortar and paste samples. In addition, to determine the combined effects on hydration of internal and external curing, thermogravimetric analysis was conducted on cement paste samples internally cured with SAP and externally treated with curing compound. The primary finding was that both LA- and HA-SAP accelerated and increased hydration in both Type I and Type III cement systems, with HA-SAP having a more outsized effect in some systems. Curing compound used in combination with SAP did not significantly increase hydration beyond what was seen with SAP or curing compound alone.

6.2 Methods

Cement hydration was investigated using 0.42 w/c cement paste samples made with deionized water. When used, SAP dosage was 0.2% by weight of binder (bwob) and (HR)WRA dosage was 0.7% bwob.

A TA Instruments TAM Air isothermal calorimeter (New Castle, DE) was used to measure hydration kinetics for freshly mixed pastes. Cement paste samples prepared for isothermal calorimetry analysis were mixed using a Renfert Twister vacuum mixer (Charles, IN). Since paste samples were mixed outside of the calorimeter, the initial reactions due to aluminate phases dissolution were not recorded. Once the mixing was completed (about 2 minutes after adding water to the cement), about 12 ± 3 mg of paste was added to glass ampoules, which were immediately sealed, weighed, and placed into the calorimeter for 48 hours. The bath temperature of the calorimeter was maintained at (23 ± 0.02)°C. For each paste composition, three ampoules were tested, and the reported results represent the average of these three readings.

To determine the non-evaporable water content of 7-day cement paste, ground cement paste was first dried in an oven at 105°C for 24 hours, then ignited in a muffle furnace at 1,050°C for 3 hours. The mass of the samples before and after ignition was used to calculate the non-evaporable water content using the following equation:

$$W_n = \frac{m_{105} - m_{1050}}{m_{1050}} - LOI \qquad \text{(Equation 6.1)}$$

where W_n is the non-evaporable water content (g/g cement paste), m_{105} is the dry mass (g), m_{1050} is the ignited mass (g), and LOI is the loss on ignition of cement.

Selected 28-day mortar samples described in Section 5.3.3 were prepared for backscattered SEM analysis. For the mortar samples containing SAP particles, at least 30 random SAP voids were imaged and further analyzed using ImageJ software to quantify size of the voids following the methodology described in Bose et al., 2021.

The effect of external curing compound in combination with SAP internal curing on hydration was measured using thermogravimetric analysis to determine the percent of calcium hydroxide (CH % (g/g)) in cement paste samples with and without SAP, with and without external curing compound, and with multiple cement types and sources. A poly-alphamethylstyrene-based external curing compound, 2255-WHITE (W. R. Meadows, Hampshire, IL), was used. Cement paste samples were cast, then demolded after 24 hours, at which point a thin layer of curing compound was applied to a subset of the samples. All samples were stored at 23°C and 50% RH until tested at 7 days. A small sample of powder (15 to 25 mg in size) was used for TGA measurements using a 2050 Thermogravimetric Analyzer manufactured by TA Instruments (New Castle, DE). Care was taken to ensure that the sample used for analysis did not come from the surface of the sample and did not include any of the curing compound itself. Analysis was performed in a nitrogen gas atmosphere, with a temperature range of 23°C to 1,000°C at a heating rate of 10°C/min.

6.3 Results

6.3.1 Isothermal Calorimetry

As shown in Figure 6.1a, the slope of the heat flow curves during the acceleration period (up to 8 hrs) is higher for the pastes containing Type III cement, when compared to Type I cement pastes. Additionally, higher total heat at the end of 3 days was observed for Type III pastes, when compared to Type I pastes (Figure 6.1b). This is unsurprising as the Type III cement is much finer (a 44% increase in Blaine fineness) when compared to Type I cement, and thus it is expected to hydrate at the higher rate.

The presence of HA-SAP particles significantly increased both, the height of main hydration peak (between 4 and 16 hours) and total heat generated in B Type I cement paste, when compared to B Type I+No SAP. The increase in height of main peak is an indication of the increase in the effective w/c (Justs et al., 2013). This implies that majority of the HA-SAP particles released their stored water by the end of the acceleration period (i.e., by 16 hours), thus facilitating hydration reactions of C_3S. When compared to B Type III+No SAP, the height of the heat flow peak for B Type III+HA paste was only slightly higher. However, when compared to the B Type III+No-SAP paste, the main hydration peaks for the SAP pastes were

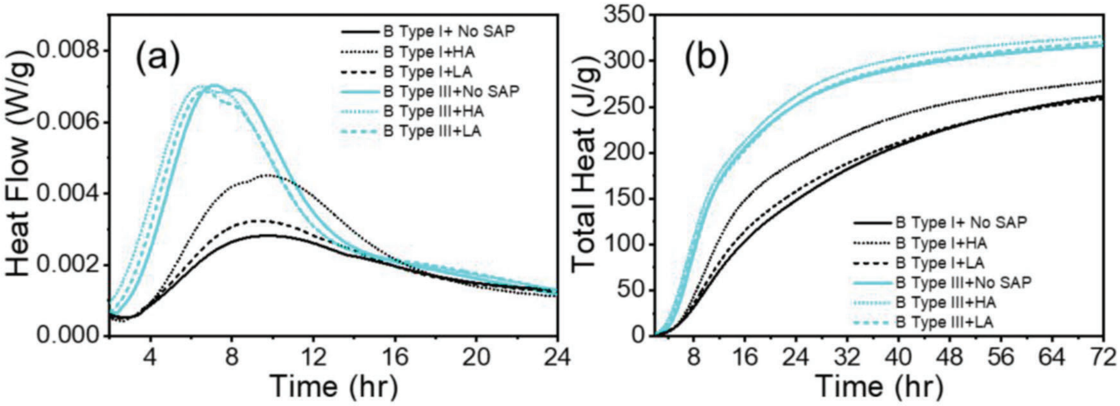


Figure 6.1 Isothermal calorimetry analysis of 0.42 w/c cement paste showing the effect of Type I and Type III cement from the same source on (a) heat flow (24 hours) and (b) total heat (72 hours).

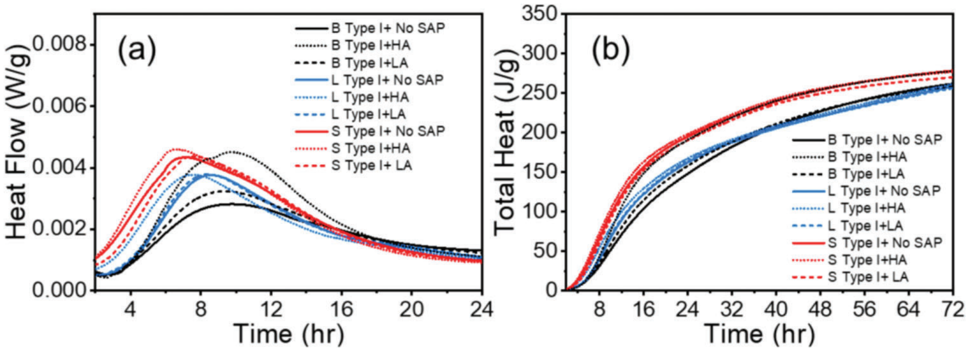


Figure 6.2 Isothermal calorimetry analysis of 0.42 w/c cement paste showing the effect of different sources (and thus different chemistries) of Type I cement on (a) heat flow (24 hours) and (b) total heat (72 hours).

noticeably shifted to the left, indicating the acceleration of hydration.

The presence of LA-SAP particles only slightly increased the height of the main hydration peak for B Type I+LA, when compared to B Type I+No SAP. This might indicate that the SAP particles released the water during the acceleration period. Since LA-SAP particles' absorption capacity was lower than that of HA-SAP particles, the lower amount of water stored and released by the LA-SAP could result in smaller increase in the effective w/c values when compared to HA-SAP-containing pastes. No major difference in total heat attained was observed for the LA-SAP-containing pastes, when compared to the reference pastes.

Figure 6.2 shows the effect of different sources (and thus different chemistries) of Type I cement on the isothermal calorimetry analysis of internally cured cement pastes. S Type I pastes displayed earlier setting and higher total hydration (total heat) when compared to the B Type I or L Type I pastes. These outcomes are consistent with the initial setting time reported in the cements' mill certificates. S Type I had the highest total amount of alkali oxide content. This would result in dissolution of the greater amount of alkali ions in the pore solution and contribute towards earlier attainment of higher rate of hydration. The presence of HA-SAP particles increased the rate of hydration and resulted in comparable or higher total hydration in pastes from all three cement sources. The presence of LA-SAP particles had no impact on the hydration of S or L Type I when compared to the corresponding reference paste without SAP.

As shown in Figure 6.3, reduced total hydration was observed for all three pastes containing 30% slag, despite the fact that they had a higher hydration rate during the first 4–8 hrs. This has been reported by previous researchers (Gruyaert et al., 2010). Additionally, no significant impact of internal curing with HA- or LA-SAP particles was observed in the pastes containing slag. Previous researchers have reported a reduction in main hydration peak and a delay in hydration kinetics in slag containing pastes without any SAP (Moon et al., 2018). It is possible that this effect negated the greater hydration due to the presence of HA-SAP particles.

6.3.2 Non-Evaporable Water Content

The non-evaporable water content (W_n) at 7 days is shown in Figure 6.4. Irrespective of the chemical composition of Type I cements, the W_n values at 7 days were comparable for samples with and without SAP. Despite the accelerated hydration observed through 72 hours via isothermal calorimetry (Section 6.3.1) in

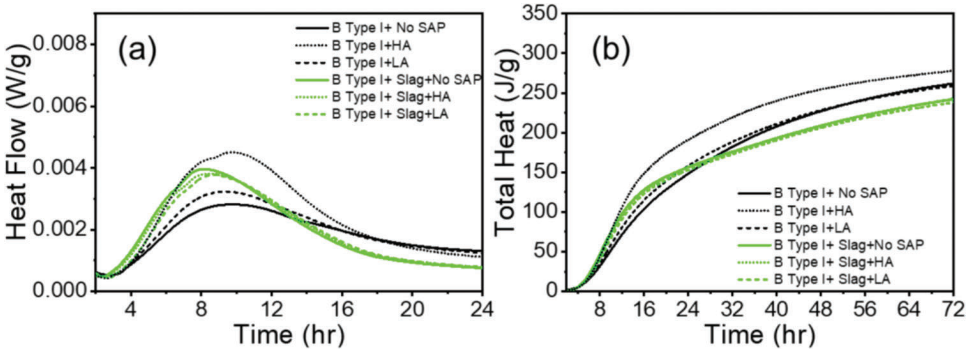


Figure 6.3 Isothermal calorimetry analysis of 0.42 w/c cement paste showing the effect of partial replacement of cement by slag on (a) heat flow (24 hours) and (b) total heat (72 hours).

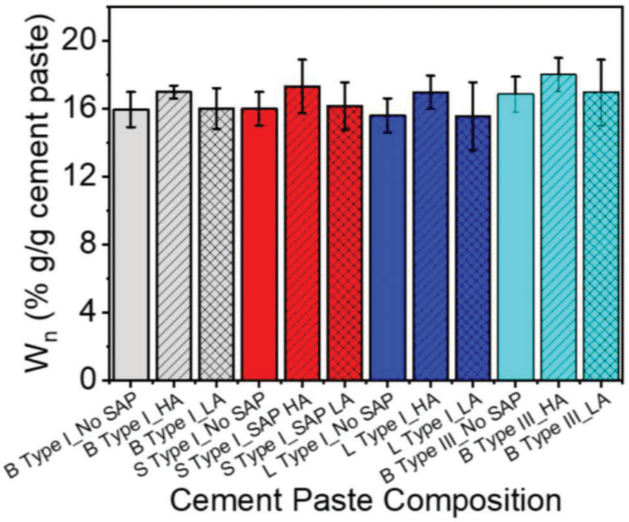


Figure 6.4 Non-evaporable water content (W_n) of 0.42 w/c cement pastes at 7 days. Hatched bars represent samples containing HA-SAP particles and cross-hatched bars represent samples containing LA-SAP particles.

Type III cement samples, no corresponding increase in W_n at 7 days was observed.

6.3.3 Microstructure of Mortar Samples

SEM micrographs of select w/c 0.42 mortars (Mix Designs ID 1, 2, and 3, see Table 5.1) are shown in Figure 6.5. In the B Type I_No SAP_0.5_0.42 sample (Figure 6.5a), microcracks were clearly visible throughout the cement paste matrix of the mortar. Microcracks were notably absent in the matrices of the corresponding SAP-containing samples (Figure 6.5b and Figure 6.5c). The lack of microcracks in these SAP-containing samples may have potentially helped them to achieve the later-age levels of compressive and flexural strengths which were either comparable to or improved compared to the levels of strengths in the reference specimens.

Analysis of the average diameter of the SAP-induced voids in mortars with SAP particles revealed that their average sizes were 302 μm and 127 μm, in samples containing LA- and HA-SAP particles, respectively. This relative size relationship was expected, as the average dry particle size of the LA-SAP particles was larger than that of the HA-SAP particles (see Section 3.3.1). However, the presence of larger SAP-induced voids does not necessarily mean that mechanical strength will be compromised. Other factors, such as SAP absorption kinetics and the subsequent release of water during hydration of cement might also play a role in determining the values of mechanical strength.

Some of the SAP-induced voids in the sample with HA-SAP-particles contained growths of identifiable hydration products. Figure 6.6a shows a SAP-induced void in a 0.42 w/c, B Type I, HA-SAP, 0.5% (HR)WRA (Mix Design 15) specimen with two spots (1 and 2) where energy-dispersive x-ray (EDX) analysis was conducted. In instances where there was substantial growth of hydration products around the vicinity of the SAP-induced voids, the perimeter of the SAP particle in the hardened matrix was difficult to distinguish as the significant growth of hydration product partially obscured the original dimensions of the swollen SAP particles. Figure 6.6b illustrates the relevant features of the SAP void: original perimeter of the SAP particle at the time of final set is outlined in red and hydration product growth is highlighted in cyan. Remnants of the dehydrated SAP, along with other intermixed hydration products were also present (region inside the SAP void that is not highlighted in Figure 6.6b). The EDX analysis of the locations shown in Figure 6.6a yielded the following atomic percentages (Ca: 27.4 ± 0.1%, O: 66 ± 1%, others (Na, Si, K, Al, Mg, S, Na): 6 ± 1%). Based on the EDX analysis and the morphology of the growth inside the SAP-induced voids it is postulated that it is most likely a calcium hydroxide (portlandite). Previous studies have also found portlandite in SAP-induced voids (Bose et al., 2021; Zhong et al., 2021). The growth of hydration products inside the SAP-induced voids was not observed in the LA-SAP-cured samples.

6.3.4 Microstructure of Paste Samples

Scanning electron micrographs of reference and internally cured cement pastes are shown in Figure 6.7. For each internally cured paste sample, the amounts of

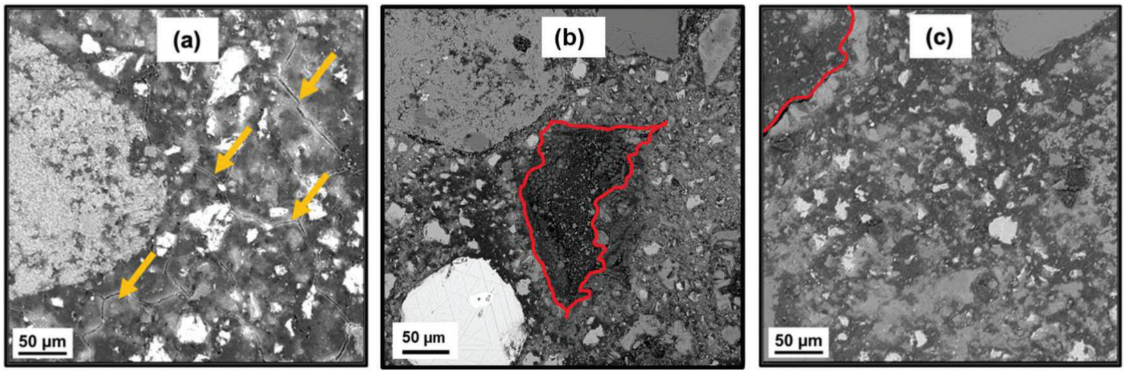


Figure 6.5 Representative SEM images of 28-day mortar samples (w/c 0.42): (a) B Type I_No SAP_0.5_0.42, (b) B Type I_LA_0.5_0.42, (c) B Type I_HA_0.5_0.42. The arrows in (a) indicate microcracks formed in the matrix of the mortar. The red outlines in (b) and (c) indicate the outer edges of deswelled SAP particles.

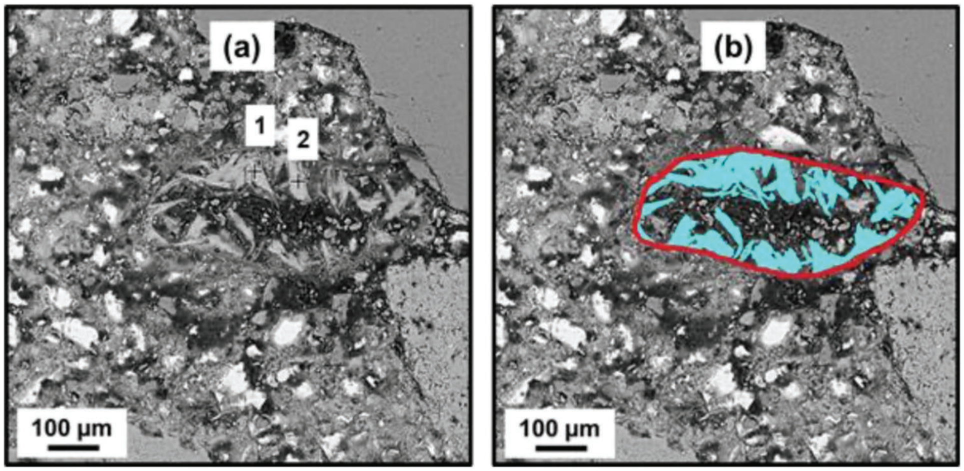


Figure 6.6 SEM image of a SAP-induced void in a 28-day mortar sample (0.42 w/c, B Type I, HA-SAP, 0.5% (HR)WRA, Mix Design 15): (a) with two locations used for EDX analysis (1 and 2) and (b) the SAP void outlined in red and the portlandite growth inside the void (highlighted in cyan).

hydration product observed within the SAP voids were measured (Figure 6.8) and are described below to ultimately determine the effects of the two compositions of SAP particles and cement source on the microstructure of hardened cement paste.

As seen in the micrographs in Figure 6.7(d–f), one of the features of the hydrated paste microstructures was the “in-filling” of the SAP voids with hydration product. Based on elemental analysis conducted on mortar specimens (Figure 6.6), the hydration products were likely primarily CH with some intermixed C-S-H.

To determine how the SAP composition affected the observed void-filling, the amounts (by area fraction) of hydration product observed within the SAP voids were quantified for each internally cured paste. Of the 30 or more SAP voids each that were analyzed for paste containing LA- or HA-SAP particles (Figure 6.8), more voids resulting from HA-SAP particles were filled with hydration products compared to voids resulting from LA-SAP particles for both B and S Type I cements. This increased in-filling of voids in specimens with HA-SAP might be, at least partially, the result of the higher total hydration observed in these specimens by isothermal calorimetry method (see Figure 6.2). While details regarding composition of the HA-SAP are unknown, some conclusions can be drawn from the free absorption capacity experiments conducted (Figure 3.2). HA-SAP displayed greater absorption capacity in the gravimetric absorption experiments, thus a larger amount of water is expected to be absorbed by the SAP particles, which would fuel hydration reactions in the vicinity of the void. Additionally, the crosslinking amount and the desorption kinetics of the SAP particles are also expected to play a role in influencing the growth of hydration products both inside and in the vicinity of the SAP voids.

In addition to SAP type, the cement source also played a role in impacting the hydrated paste microstructure. Although only minimal growth of hydration products was observed in the voids of LA-SAP particles in B Type I cement paste, more extensive deposits of hydration products were observed in some of the SAP voids present in S Type I cement. The higher alkali content of S Type I cement, when compared to B Type I cement, might have resulted in overall increase in alkali ion concentrations even within the LA-SAP voids. This

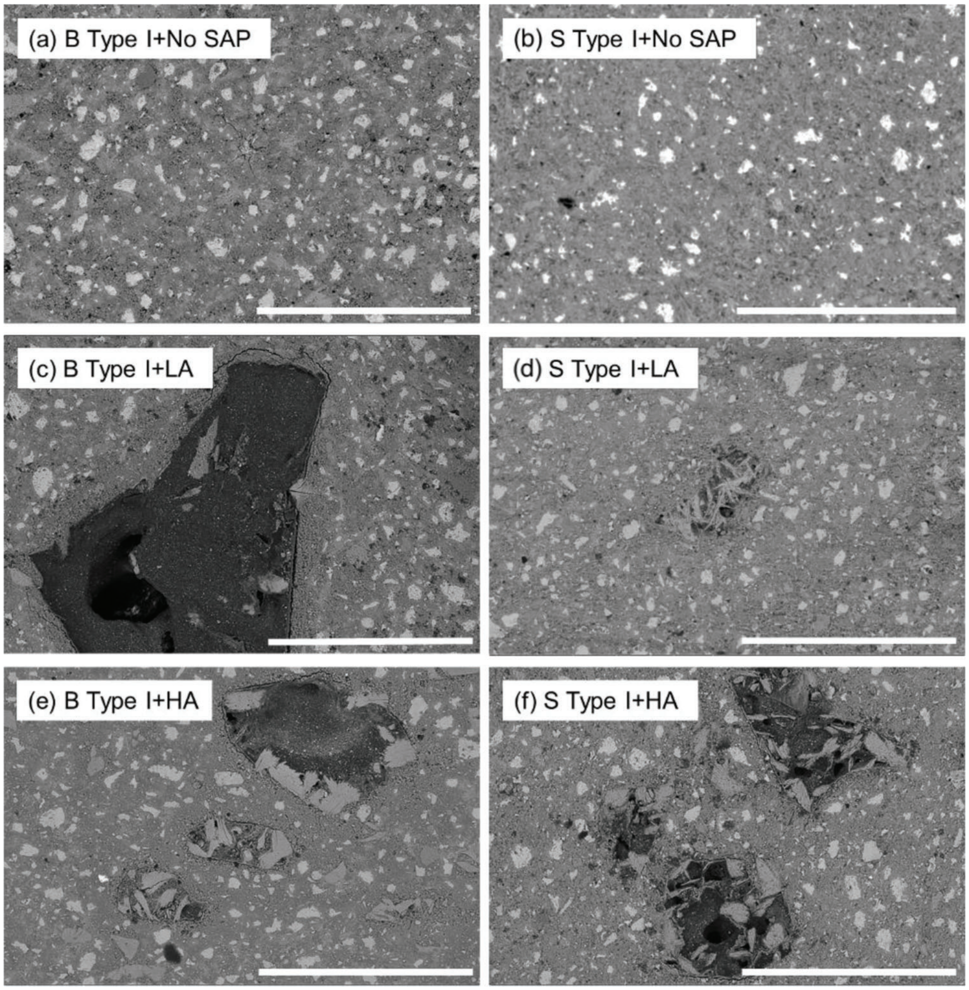


Figure 6.7 Microstructure of 7-days old 0.42 w/c pastes with LA- and HA-SAP particles. B Type I cement was used for (a), (c), and (e), while S Type I cement was used for (b), (d), and (f). Scale bar in each micrograph represents 400 μm. In (c) and (d), respectively, 4% and 83% of the SAP voids are filled with hydration products, while 14% and 18% of the SAP void are filled with hydration products in the upper-right SAP voids in (e) and (f), respectively.

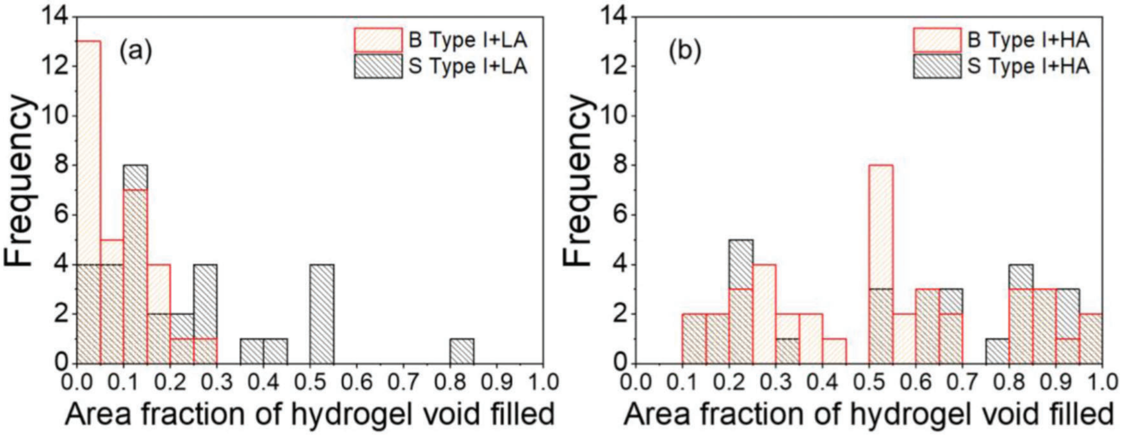


Figure 6.8 Results of microstructural analysis of 0.42 w/c pastes with (a) LA-SAP particles and (b) HA-SAP particles. Area of SAP particle voids filled with hydration products as a function of cement paste composition.

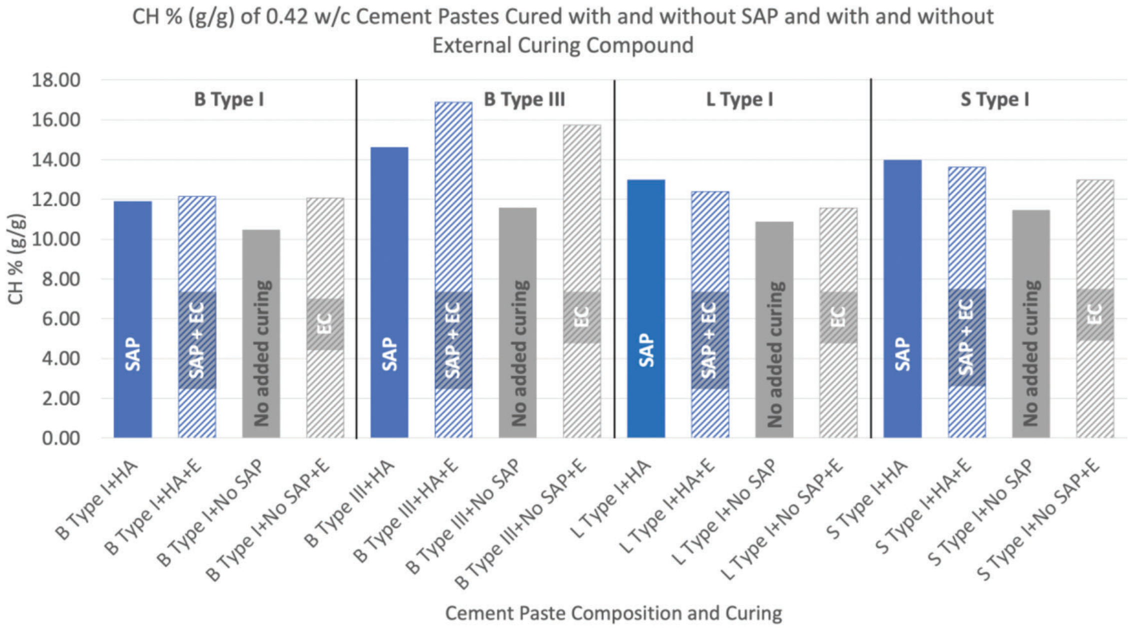


Figure 6.9 CH% (g/g) of 0.42 w/c cement pastes prepared using different cement types and sources, with and without SAP and with and without external curing compound. CH% determined via TGA.

would result in accelerated hydration even within the SAP voids and thus formation of hydration products both inside and in the vicinity of the voids.

6.3.5 Impact of Curing Compound

The amounts of calcium hydroxide (CH%) present in hydrated cement pastes determined using the TGA method are shown in Figure 6.9. These results were all obtained from 0.42 w/c pastes with and without SAPs, with and without application of external curing compound, using cements of multiple types and sources. Since CH is a product of the cement hydration reaction, increased CH% serves as an indicator of increased degree of hydration. For all Type I cement sources, pastes with some form of additional curing, whether that be internal curing with SAP, external curing compound, or a combination of the two showed comparably increased CH% over the reference without any additional curing method. For the Type III cement, the highest CH% came from the use of internal curing with SAP and external curing compound together.

6.4 Conclusions and Implications

The following conclusions resulted from this cement hydration study.

- Heat flow plots and total heat plots from isothermal calorimetry analysis of Type I and Type III cement pastes indicated that the presence of either LA- or HA-SAP particles contributed to accelerated and increased hydration of the cement paste regardless of cement source.
- Heat flow from isothermal calorimetry analysis of B Type I cement with 30% slag replacement showed accelerated hydration with both LA- and HA-SAP but did not display an increase in total heat.
- Microstructural refinement typically associated with SAP internal curing of lower w/c mixtures, namely the absence of microcracks compared to SAP-free samples, was observed in mortars at a w/c of 0.42.
- Microstructural analysis of both B and S Type I cement pastes with HA-SAP particles revealed significant presence of hydration products in the SAP voids.
- For 0.42 w/c Type I cement pastes, internal curing with SAP, external application of curing compound, and a combination of the two all yielded similarly increased CH% over the reference without any additional curing method for a given cement source.

A number of implications resulted from this study. First, the accelerated hydration observed in SAP-containing pastes supports the accelerated strength development previously observed in mortar samples (Sections 5.3.1 and 5.3.4), signifying that SAP internal curing could be employed in situations when rapid strength development is desired. Second, the absence of microcracks in SAP-containing mortars supports previous findings of increased durability with the use of SAP (Beushausen et al., 2014; Mechtcherine et al., 2021) and suggests SAP delayed time to cracking (which is further investigated in Section 9). Third, SAP and external curing compound displayed similar increases in CH formation when used together or separately across cement sources and types, indicating that incorporation of SAP in fresh concrete mixes could have similar hydration benefits as the application of curing compound after concrete has set.

7. OBJECTIVE 3B: IMPACT OF SAP ON WARPING OF CEMENT PASTES

7.1 Overview

This section describes the research results obtained during the investigation of warping of cement pastes internally cured with SAP. The objective of this study was to determine the impact of SAP on warping of cement pastes with and without extra water and prepared using varying SAP dosages. This was accomplished by casting 1.3-cm thick cement paste beams with and without SAP and measuring the degree of warping over time when stored at constant temperature and relative humidity. The primary finding was that SAP increased warping and that the addition of extra water exaggerated that effect.

7.2 Methods

B Type I cement paste beams with the dimensions 100.0 cm × 6.1 cm × 1.3 cm were cast at different w/c values with and without LA- and HA-SAP. The beams were stored in a moist room for 24 hours to ensure no cracking occurred before or during demolding. After demolding, beams were sealed with aluminum tape on all but the finished top surface and stored in an environmental chamber with a temperature of 25°C and a relative humidity of 50%. The gap between the midpoint of the beam and a bar flushed with edges of the beam was measured and reported as the warping deformation. Figure 7.1a shows the beam specimens stored in the environmental chamber and Figure 7.1b shows example of the warping deformation for paste with w/c of 0.52 and no SAP).

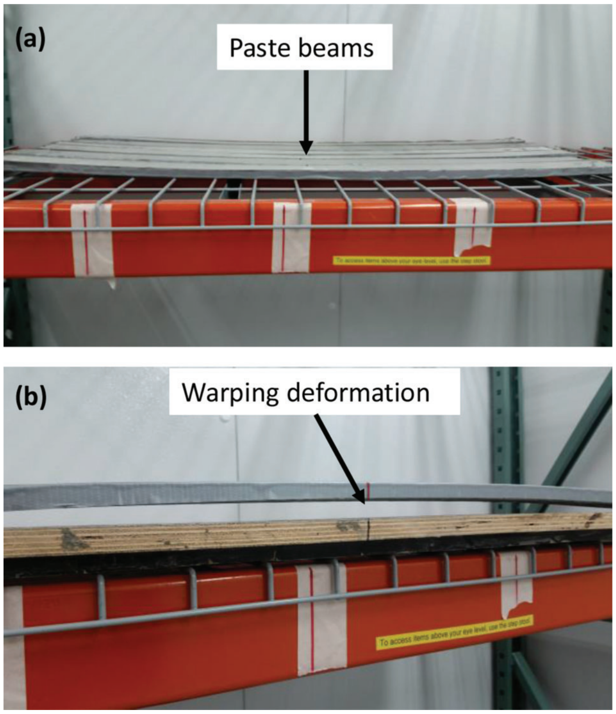


Figure 7.1 Beam specimens for warping tests: (a) specimens sealed and stored in an environmental chamber and (b) w/c of 0.52 No-SAP specimen positioned on the top of the board for measurement of warping deformations.

7.3 Results

The warping deformation of paste beams with varying w/c values made with and without LA- and HA-SAP are shown in Figure 7.2. The addition of either SAP at a w/c of 0.42 increased the warping deformation with the HA-SAP resulting in significantly more warping than the LA-SAP. As expected from previous studies (Amirkhanian & Roesler, 2017; Wei et al., 2017), an increase in w/c resulted in an increase in warping, as can be seen when comparing the 0.42, 0.49, and 0.52 w/c reference samples without SAP. For both SAPs, the specimens prepared at higher w/c values (0.49 w/c and 0.52 w/c) and containing SAP showed higher warping deformation compared to their corresponding control specimens (i.e., with higher w/c values but no SAP).

7.4 Conclusions and Implications

The following conclusions resulted from this warping study.

- The addition of SAP increased warping, with HA-SAP showing a greater increase than LA-SAP.
- Warping was greater in cement pastes with higher w/c.

One major implication resulted from this study. The addition of extra water to SAP-containing pastes to increase workability resulted in increased warping. As such, extra water usage should be avoided with SAP internal curing.

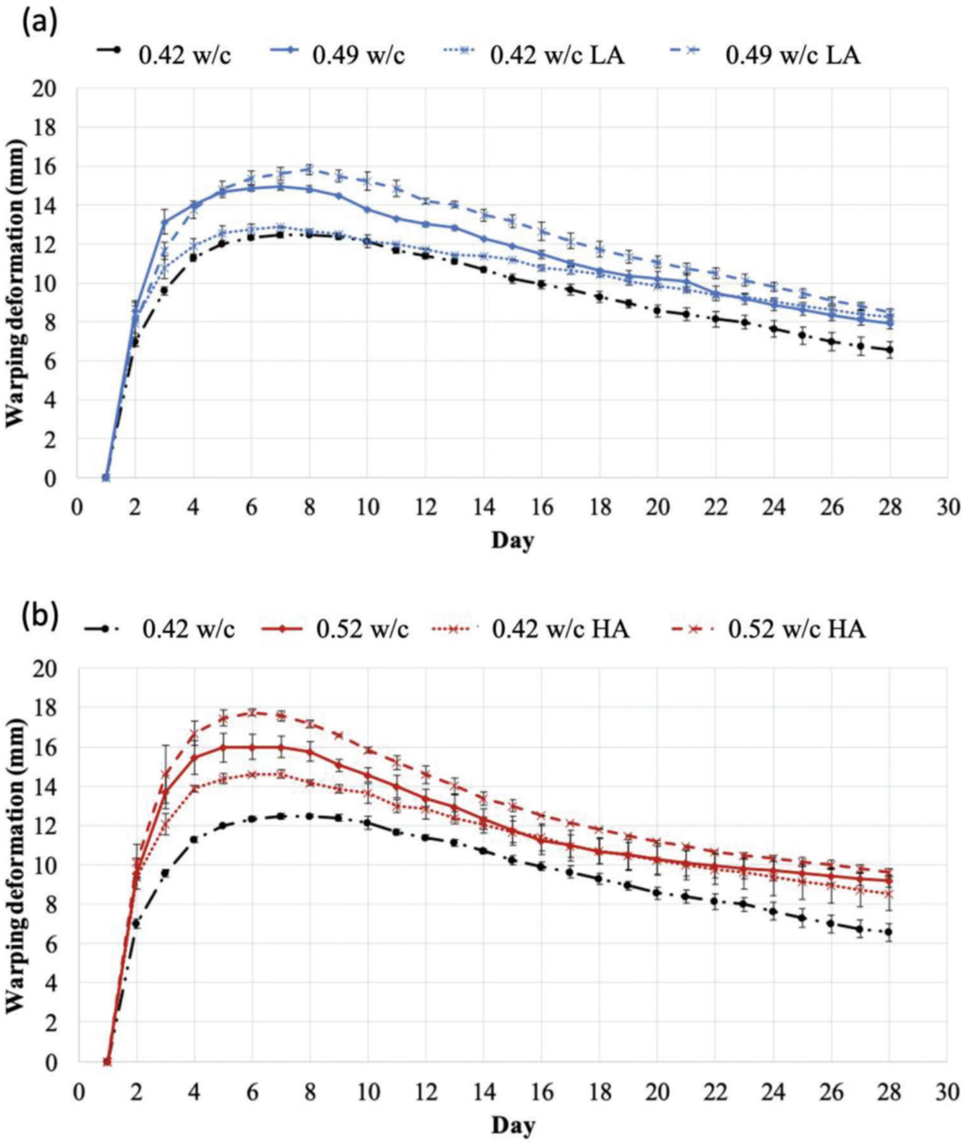


Figure 7.2 Warping deformation (mm) of cement paste beams with and without extra water and with and without (a) LA-SAP or (b) HA-SAP.

8. OBJECTIVE 3C: IMPACT OF SAP ON SHRINKAGE OF MORTAR

8.1 Overview

This section describes the research results obtained during the investigation of autogenous and drying shrinkage of mortar internally cured with SAP. The objective of this study was to quantify the impact of SAP on shrinkage of mortars. This objective was accomplished by measuring autogenous shrinkage and change in length of mortars over time. The primary finding was that in mixtures that displayed significant autogenous shrinkage (e.g., those containing Type III cement or those prepared at a low w/c of 0.35), the addition of both LA- and HA- SAP reduced autogenous shrinkage, while in 0.42 w/c mortars, SAP did not reduce drying shrinkage.

8.2 Methods

Autogenous shrinkage was measured following ASTM-C1698, which was modified to begin recording data immediately after casting. Linear autogenous strain, ε, was calculated with the following formula:

$$\varepsilon = \frac{\Delta L(t) - \Delta L(0)}{0.382 + \Delta L(0)} \times 10^6 \qquad \text{(Equation 8.2)}$$

$\Delta L(t)$ is the length of the tube at time t and $\Delta L(0)$ is the length of the tube at the sample's time of final set. The final setting time of cement paste was estimated from isothermal calorimetry experiments.

Drying shrinkage of mortars was determined following ASTM C157/C157M.

8.3 Results

8.3.1 Autogenous Shrinkage

Autogenous shrinkage of mortars with 0.42 w/c is shown in Figure 8.1. The presence of SAP particles reduced autogenous shrinkage of SAP-cured mortars, when compared to the reference mortar (B Type III No SAP_0.5_0.42). Type I mortars did not experience any significant autogenous shrinkage strains. Also, it is interesting to note that HA-SAP was more successful in mitigating autogenous shrinkage of mortars, than the LA-SAP and that this effect was particularly pronounced in Type III cement mortars.

The addition of HA-SAP particles induced an early age expansion in the mortars. This effect was particularly prominent in the Type III mortar mixture. Bentz et al. (2015) reported similar expansive strains due to addition of SAP to mortars and indicated that this might be due to the formation of ettringite in early stages of hydration. The needle-like morphology of ettringite would result in development of expansive strains in the microstructure. Although this phenomenon would be prevalent in the reference mortar too, it was likely suppressed by the autogenous shrinkage and chemical shrinkage of the reference mortars. The measured autogenous shrinkage is a reflection of the self-desiccation of the mortar and any expansion produced due to formation of hydration products (such as ettringite). Since most of the self-desiccation was mitigated due to the presence of HA-SAP particles, the expansive strains became the dominant factor in determining autogenous shrinkage strains of B Type III_HA_1.15_0.42_mortar. The slightly higher amount of sulfate ions presents in the Type III cement used, when compared to Type I cement, might have also contributed to higher rate of formation of ettringite in the Type III mortars. Other researchers have reported this expansive strain to be the result of thermal expansion and abrupt desorption of the retained water in the SAP after final set (Montanari et al., 2017).

To further investigate the effect of internal curing on autogenous shrinkage of Type I mortars, a set of experiments were carried out at a lower w/c (w/c = 0.35) for 5 days. Figure 8.2 shows the autogenous shrinkage of mortars with w/c of 0.35. As seen in the figure, the presence of SAP particles reduced the shrinkage strains, and this effect was particularly significant in the mortars containing HA-SAP.

8.3.2 Drying Shrinkage

Drying shrinkage data for mortars with and without SAP and with varying values of w/c are shown in Figure 8.3. HA-SAP-containing mortars showed increased drying shrinkage compared to their No SAP counterparts at the same w/c (0.42 and 0.52), Furthermore, an increase in w/c resulted in an increase in drying

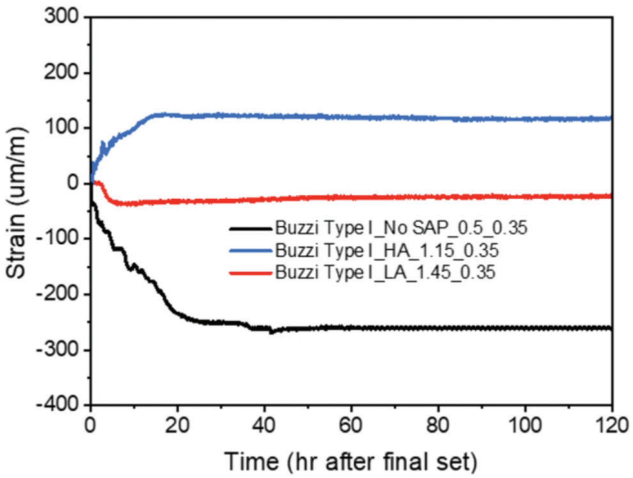


Figure 8.2 Autogenous shrinkage of B Type I mortars with a w/c of 0.35.

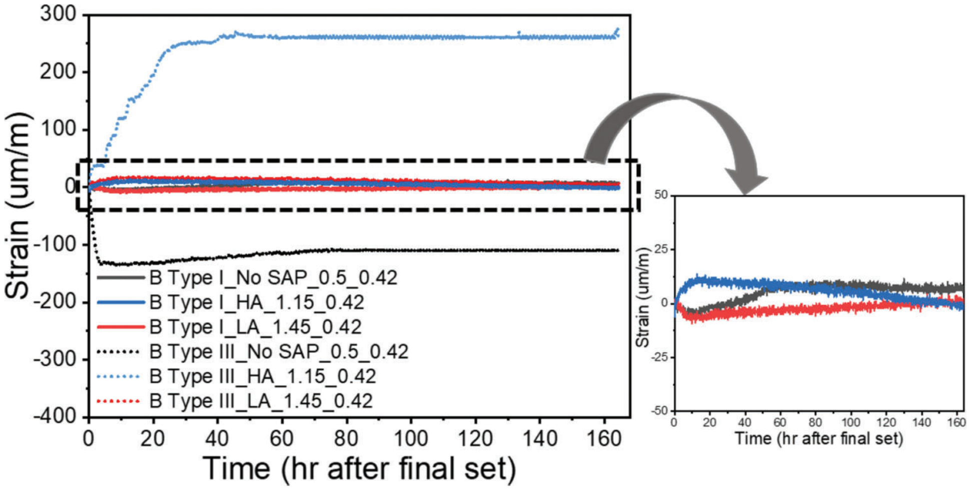


Figure 8.1 Autogenous shrinkage of B Type I and B Type III mortars with a w/c of 0.42. For easier visualization of the Type I mortars, the right-side plot shows the expanded segment of the plot to the left.

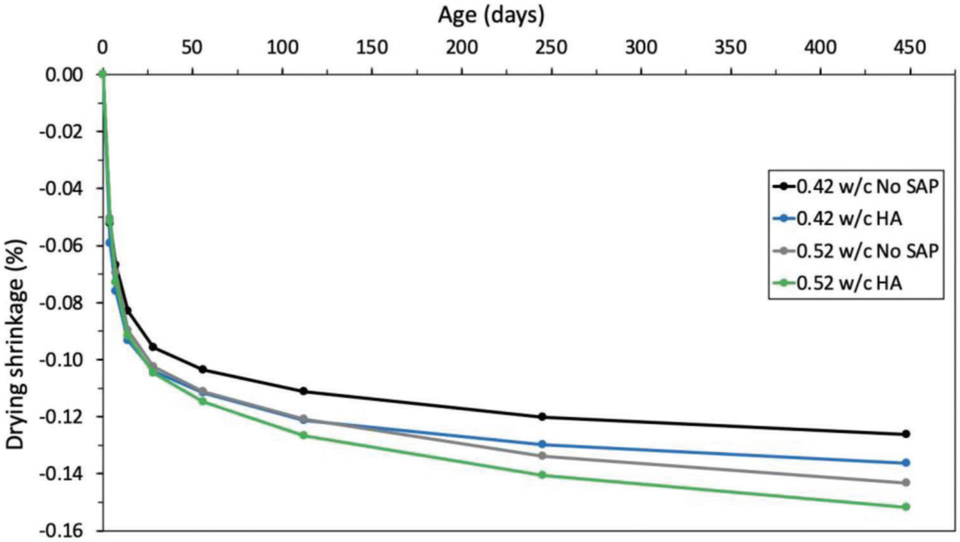


Figure 8.3 Drying shrinkage (%) over time of mortars with varying w/c values and with and without HA-SAP.

shrinkage. Additionally, drying shrinkage increases with increasing w/c without SAP, as expected (Yurtdas et al., 2006). The mechanism that led to increased warping with the addition of SAP (Chapter 7) likely also contributed to the increase in drying shrinkage with the addition of HA-SAP. Water sequestered within the SAP is unavailable for evaporation until later ages, which maintains the total water in the specimen at a higher level for longer period of time than that associated with mortars without SAP.

8.4 Conclusions and Implications

The following conclusions resulted from this shrinkage study.

- In mortars that displayed autogenous shrinkage (0.35 w/c B Type I and 0.42 B Type III), the addition of LA-SAP mitigated autogenous shrinkage, while HA-SAP led to early-age expansion rather than autogenous shrinkage.
- HA-SAP increased drying shrinkage at all w/c.
- Increased w/c values increased drying shrinkage.

A number of implications resulted from this study. First, when autogenous shrinkage is experienced, the addition of SAP could mitigate or even reverse autogenous shrinkage. Second, addition of extra water should be avoided to minimize drying shrinkage.

9. OBJECTIVE 3D: IMPACT OF SAP ON CRACKING TENDENCY

9.1 Overview

This section describes the research results obtained during the investigation of cracking tendency of SAP-containing mortar under conditions of restrained shrinkage. The objective of this study was to determine the impact of SAP on the cracking tendency of mortar at 0.35 and 0.42 w/c. This objective was accomplished by conducting the ring test on mortars with and without SAP and both values of w/c. The primary finding was that the addition of SAP increased time to cracking and reduced average stress rate in Type I mortars at both w/c values.

9.2 Methods

Mortars were prepared as described in Section 5.2. The B Type I mortars with w/c of 0.42 had the same relative composition as Mix Designs 11, X, and Y from Table 5.1. The B Type I mortars with w/c of 0.35 were batched using Mix Designs 11, 12, and 13 with the amount of water lowered to yield w/c of 0.35. After batching, mortars were cast into a ring mold according to ASTM C1581/C1581M with a properly sized cardboard tube serving as the outer ring and four strain gauges distributed evenly inside the inner steel ring (ASTM, 2018b). One day after casting, the outer cardboard ring was removed and the top surface of the specimen was sealed with self-adhesive aluminum tape to restrict drying. Strain gauge measurements were collected until the sample cracked. The ring had four strain gauges placed at regular intervals. The diametrically opposed strain gauge measurements were averaged for data analysis.

9.3 Results

9.3.1 0.35 w/c Mortars Results

Strains generated in the steel ring supporting the w/c = 0.35 mortars are shown in Figure 9.1. The No-SAP mortar served as a reference. This mortar cracked at an age of 4.25 days. The time-to-cracking values increased for both mortars containing SAPs and were, respectively, 5.25 days for LA-SAP and 5.0 days for HA-SAP.

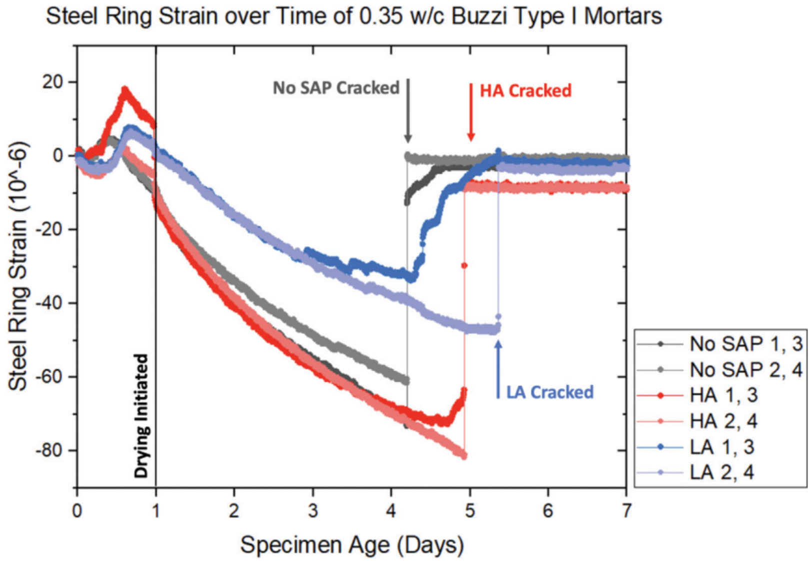


Figure 9.1 Steel ring strains over time for 0.35 w/c B Type I mortars with and without HA- and LA-SAPs.

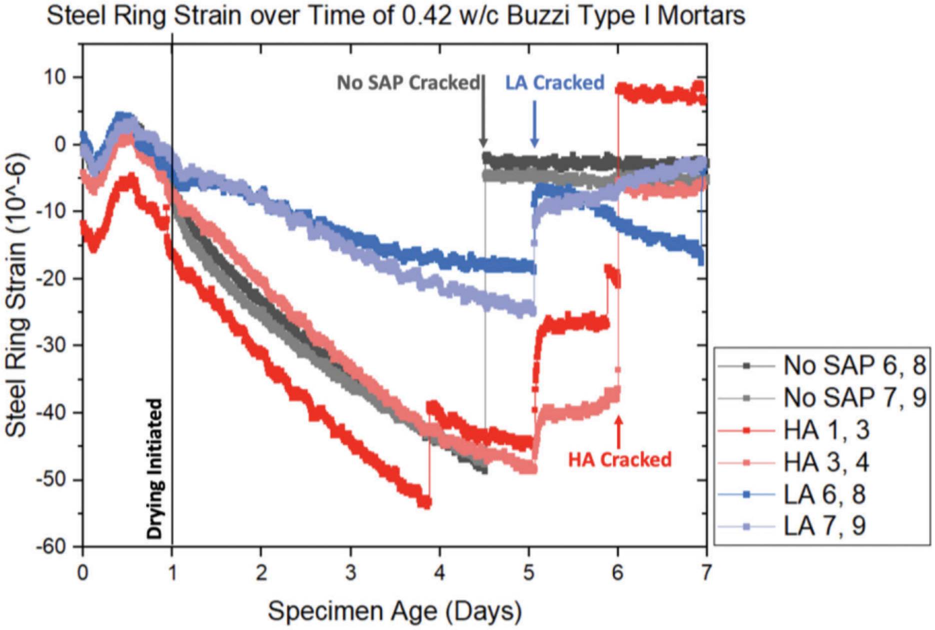


Figure 9.2 Steel ring strain over time of 0.42 w/c B Type I mortars with and without HA- and LA-SAP.

These higher time-to-cracking values represented 24% and an 18% increase for LA-SAP and HA-SAP mortars, respectively, over the values observed for No SAP reference mortar.

9.3.2 0.42 w/c Mortars Results

Strains generated in the steel ring supporting the 0.42 w/c mortars are shown in Figure 9.2. The No SAP mortar served as a reference. This mortar cracked at an age of 4.5 days. The time-to-cracking values increased for both mortars containing SAPs and they were, respectively, 5.0 days for LA-SAP and 6.0 days for HA-SAP. These higher time-to-cracking values represent 11% and 33% increases for 0.42 w/c LA-SAP and HA-SAP mortars, respectively, over the 0.42 w/c No SAP reference mortar. Of additional note, the time-to-cracking of 4.5 days for the 0.42 w/c No SAP mortar was exceeded not only by the 0.42 w/c mortars containing SAP, but also by both 0.35 w/c mortars containing SAP, despite their reduced values of the water-to-cement ratio.

9.4 Conclusions and Implications

The following conclusions resulted from this cracking tendency study.

- SAP-containing mortars at both 0.35 and 0.42 w/c cracked at later ages than reference mortars without SAP.
- SAP-containing mortars at 0.35 w/c cracked at later ages than 0.42 w/c mortars without SAP, despite having a lower w/c.

A number of implications resulted from this study. First, SAP can be used to decrease cracking tendency of both high and low w/c mixes. Second, 0.35 w/c mortars with SAP have longer time- to-cracking than 0.42 w/c mortars without SAP, meaning SAP can be used assure the high performance of lower w/c mixes as it decreases their cracking tendency.

10 OBJECTIVE 4: SAP-MORTAR PUMPABILITY

10.1 Overview

This section describes the research results obtained during the investigation of the pumpability of SAP-containing mortar. The objective of this study was to determine whether SAP-containing mortar was pumpable for placement. This objective was accomplished by batching and then continuously pumping SAP-containing mortars in recycling mode. The primary finding was that 0.42 w/c mortars containing SAP were pumpable for at least half an hour.

10.2 Methods

SAP-containing mortars were prepared as described in Section 5.2 (Table 5.1, Mix Designs 12, 13, 15). Mortars were then transferred into a progressive cavity pump (IMER Small 50) with a capacity of 50 liters and pumped continuously at a flow rate of 2 to 4 liters per minute through a 3-meter long, 1-inch diameter hose in recycling mode. Trials to demonstrate pumpability lasted for at least 30 minutes or until the pumping pressure exceeded 200 psi, indicating formation of some sort of blockage in the pump.

10.3 Results

Mortars with (HR)WRA dosed to achieve an initial percent flow of 110% ± 10% (Mix Designs 12 and 13: B Type I mortar with LA- and HA-SAP, respectively) were successfully pumped in recycling mode for half an hour. Mix Design 15 (B Type I mortar with HA-SAP, 0.5% (HR)WRA, and an initial percent flow of 93%) was pumpable for 25 minutes before the pumping pressure exceeded 200 psi (mixture became unpumpable).

10.4 Conclusions and Implications

The following conclusions resulted from this pumpability study.

- SAP-containing mortars with an initial percent flow of 110% ± 10% were pumpable for half an hour.
- The addition of the (HR)WRA alone produced pumpable mortar containing SAP.
- The lower initial percent flow mortar resulted in a reduced duration of pumpability.

The primary implication resulting from this study was that mortars containing SAP, with sufficient percent flow, were pumpable, indicating that SAP internal curing is feasible for field application.

11. SUMMARY

The experimental results obtained over the course of this project suggest that the use of SAP internal curing agents would lead to the formation of concrete mixtures with improved hydration (Chapter 6), accelerated strength gain (Chapter 5), greater volumetric stability (Chapter 8) and improved cracking resistance (Chapter 9) while maintaining sufficient workability to be pumped and placed in the field without sacrificing compressive or flexural strength (Chapter 5). Results also indicated that incorporation of SAP in fresh concrete mixes could have similar hydration benefits as the application of curing compound after concrete has set (Chapter 6). Mixture composition adjustments should prioritize the use of (HR)WRA instead of extra water to tune workability, as extra water will almost always lead to reductions in strength (Chapter 5), increased warping (Chapter 7), and increased drying shrinkage (Chapter 8). While the benefits of SAP internal curing agents for low w/c ratio mixtures were expected, a number of benefits were observed for SAP-containing mixtures with w/c ≥ 0.42, including decreased cracking tendency (Chapter 9) and accelerated strength development (Chapter 5), signifying that SAP internal curing could be employed in situations when rapid strength development is desired.

Overall, the two commercial SAP formulations resulted in some notable differences in performance of internally cured mixtures. The two SAP formulations were expected to differ with respect to their responses in cementitious systems due to differences in both their physical particle size and polymer chemistry. This was confirmed by differences in their absorption capacities and kinetics of absorption observed during immersion in the simulated pore fluids (Chapter 3). In particular, as shown in Figure 3.4, one SAP formulation (HA-SAP) was much more sensitive to cement chemistry and type compared with the other SAP formulation (LA-SAP). This larger observed variation in absorption for HA-SAP with variation in cement chemistry and type could explain why mixtures containing HA-SAP displayed greater variation in degree of hydration (Figures 6.1 and 6.2) and shrinkage (Figure 8.1) in comparison to mixtures containing LA-SAP. If so, LA-SAP could potentially be utilized across many different cement chemical compositions and types without causing significant variation in internal curing performance. However, more experiments are needed to more completely define the structure-property-performance relationships of SAP internal curing agents in concrete.

12. IMPLEMENTATION

SAPs used in this study were acrylate and acrylamide based crosslinked polymers with dry particle sizes less than 0.3 mm. SAP equilibrium absorption in Type I cement pore fluid ranged from 30 to 60 g fluid per g dry SAP.

All results were presented for SAP dosage of 0.2% by weight of binder with SAP added as dry particles. The research results indicate that no extra water needs to be added to mixtures containing SAP to account for water absorption by SAP particles. In such cases, workability should be adjusted using (HR)WRA to ensure the target slump value.

As dissolvable bags are used for delivering other types of additives and admixtures, there is potential for SAP to be added to concrete mixtures in a similar way. At the recommended SAP dosage of 0.2% by weight of binder, approximately one one-pound dissolvable bag of SAP would be needed per cubic yard of concrete.

Based on the research results, which considered SAPs of varying chemistries, particle sizes, and sensitivity to the cementitious environment, it appears that various types of SAP could be successfully used to produce workable mixtures with increased hydration, reduced cracking tendency, reduced autogenous shrinkage, and comparable or improved compressive and flexural strength. Additionally, test results indicate that the extent of hydration was comparable for SAP-containing mixtures with and without curing compound and SAP-free mixtures with curing compound in a lab environment. Therefore, under the lab conditions no additional hydration benefits of using curing compound were realized in mixtures with added SAP. However, it should be recognized that field concrete may benefit from external curing. In such cases, curing compound could be used without negative effects hydration mechanisms facilitated by addition of SAP.

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Zhu, Q., Barney, C. W., & Erk, K. A. (2015). Effect of ionic crosslinking on the swelling and mechanical response of model superabsorbent polymer hydrogels for internally cured concrete. *Materials and Structures*, *48*(7), 2261–2276. https://doi.org/10.1617/s11527-014-0308-5

APPENDICES

Appendix A. Evaluation of Mix Design Strategies to Potimize Flow and Strength of Mortar Internally Cured with Superabsorbent Polymers

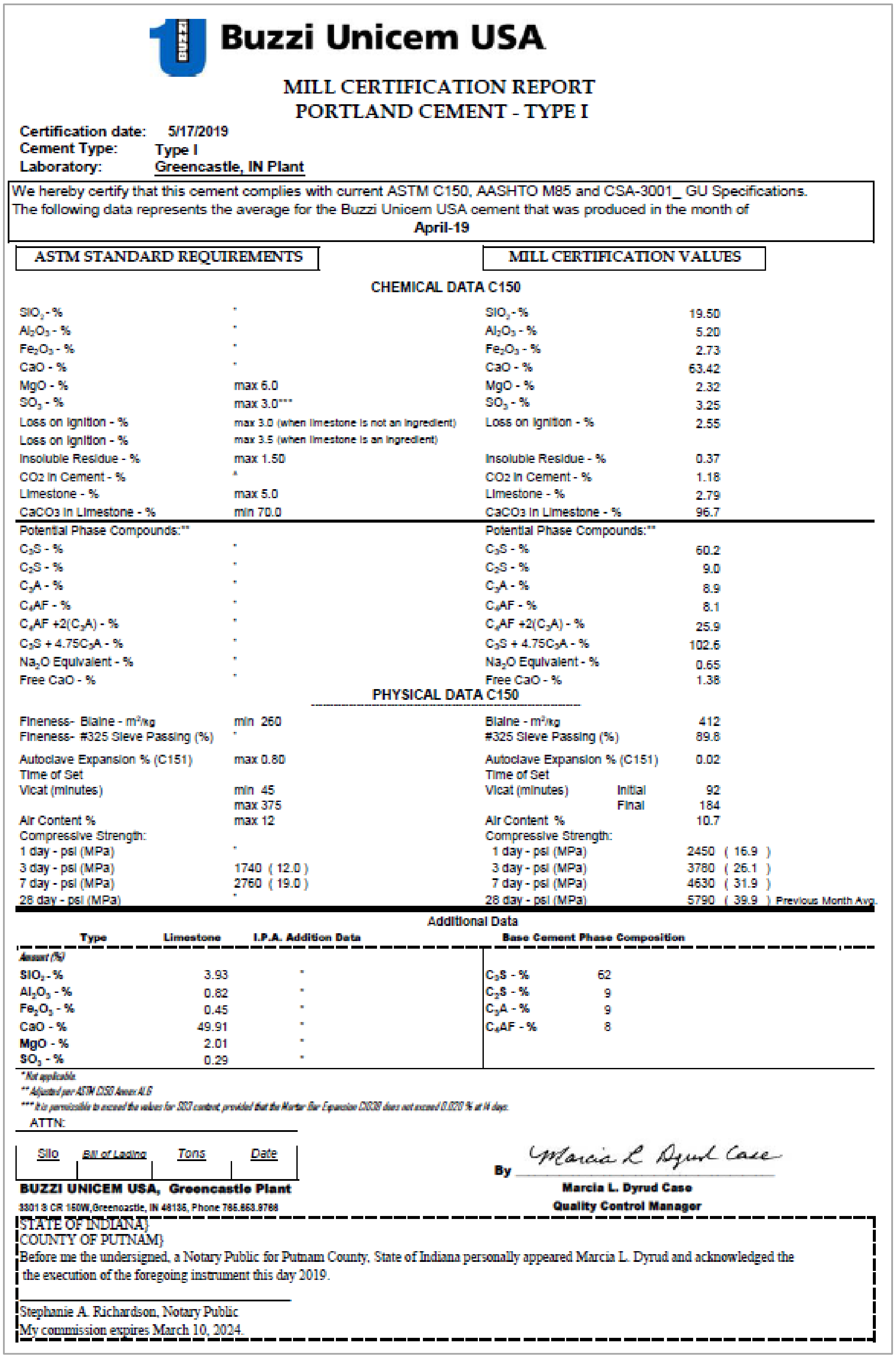
Appendix B. Mill Certificates

APPENDIX A. EVALUATION OF MIX DESIGN STRATEGIES TO POTIMIZE FLOW AND STRENGTH OF MORTAR INTERNALLY CURED WITH SUPERABSORBENT POLYMERS

Adams, C. J., Bose, B., Olek, J., Erk, K. A. (2022). Evaluation of mix design strategies to optimize flow and strength of mortar internally cured with superabsorbent polymers. *Construction and Building Material*, *324*, 126664. https://doi.org/10.1016/j.conbuildmat.2022.126664

Publication abstract: A straightforward mix design method was developed for proportioning mortars containing superabsorbent polymers (SAPs). When modified by introduction of a typical amount of SAP (i.e., 0.2% by weight of cement), the 0.42 w/c ordinary Portland cement (OPC) mortars required addition of extra water and/or high-range water reducing admixture (HRWRA) to achieve a minimum target percent flow in mortar flow table tests. At high w/c (≥0.49), SAP accelerated compressive and flexural strength development. In all mortars tested, the addition of SAP either preserved or increased compressive and flexural strength values relative to SAP-free mortar with the same w/c.

APPENDIX B. MILL CERTIFICATES



Buzzi Unicem USA

MILL CERTIFICATION REPORT
PORTLAND CEMENT - TYPE I

Certification date: 5/17/2019
Cement Type: Type I
Laboratory: Greencastle, IN Plant

We hereby certify that this cement complies with current ASTM C150, AASHTO M85 and CSA-3001_ GU Specifications.
The following data represents the average for the Buzzi Unicem USA cement that was produced in the month of
April-19

CHEMICAL DATA C150

ASTM STANDARD REQUIREMENTS		MILL CERTIFICATION VALUES		
SiO_2 - %	*	SiO_2 - %		19.50
Al_2O_3 - %	*	Al_2O_3 - %		5.20
Fe_2O_3 - %	*	Fe_2O_3 - %		2.73
CaO - %	*	CaO - %		63.42
MgO - %	max 6.0	MgO - %		2.32
SO_3 - %	max 3.0***	SO_3 - %		3.25
Loss on ignition - %	max 3.0 (when limestone is not an ingredient)	Loss on ignition - %		2.55
Loss on ignition - %	max 3.5 (when limestone is an ingredient)			
Insoluble Residue - %	max 1.50	Insoluble Residue - %		0.37
CO2 in Cement - %	*	CO2 in Cement - %		1.18
Limestone - %	max 5.0	Limestone - %		2.79
CaCO3 in Limestone - %	min 70.0	CaCO3 in Limestone - %		96.7
Potential Phase Compounds:**		Potential Phase Compounds:**		
C_3S - %	*	C_3S - %		60.2
C_2S - %	*	C_2S - %		9.0
C_3A - %	*	C_3A - %		8.9
C_4AF - %	*	C_4AF - %		8.1
$C_4AF+2(C_3A)$ - %	*	$C_4AF+2(C_3A)$ - %		25.9
$C_3S + 4.75C_3A$ - %	*	$C_3S + 4.75C_3A$ - %		102.6
Na_2O Equivalent - %	*	Na_2O Equivalent - %		0.65
Free CaO - %	*	Free CaO - %		1.38

PHYSICAL DATA C150

Fineness- Blaine - m^2/kg	min 260	Blaine - m^2/kg		412
Fineness- #325 Sieve Passing (%)	*	#325 Sieve Passing (%)		89.8
Autoclave Expansion % (C151)	max 0.80	Autoclave Expansion % (C151)		0.02
Time of Set		Time of Set		
Vicat (minutes)	min 45	Vicat (minutes)	Initial	92
	max 375		Final	184
Air Content %	max 12	Air Content %		10.7
Compressive Strength:		Compressive Strength:		
1 day - psi (MPa)	*	1 day - psi (MPa)		2450 (16.9)
3 day - psi (MPa)	1740 (12.0)	3 day - psi (MPa)		3780 (26.1)
7 day - psi (MPa)	2760 (19.0)	7 day - psi (MPa)		4630 (31.9)
28 day - psi (MPa)	*	28 day - psi (MPa)		5790 (39.9) Previous Month Avg.

Additional Data

Type	Limestone	I.P.A. Addition Data	Base Cement Phase Composition	
Amount (%)				
SiO_2 - %	3.93	*	C_3S - %	62
Al_2O_3 - %	0.82	*	C_2S - %	9
Fe_2O_3 - %	0.45	*	C_3A - %	9
CaO - %	49.91	*	C_4AF - %	8
MgO - %	2.01	*		
SO_3 - %	0.29	*		

** Not applicable*
*** Adjusted per ASTM C150 Annex A1.6*
**** It is permissible to exceed the values for SO3 content, provided that the Mortar Bar Expansion C1038 does not exceed 0.020 % at 14 days.*

ATTN:

Silo	Bill of Lading	Tons	Date

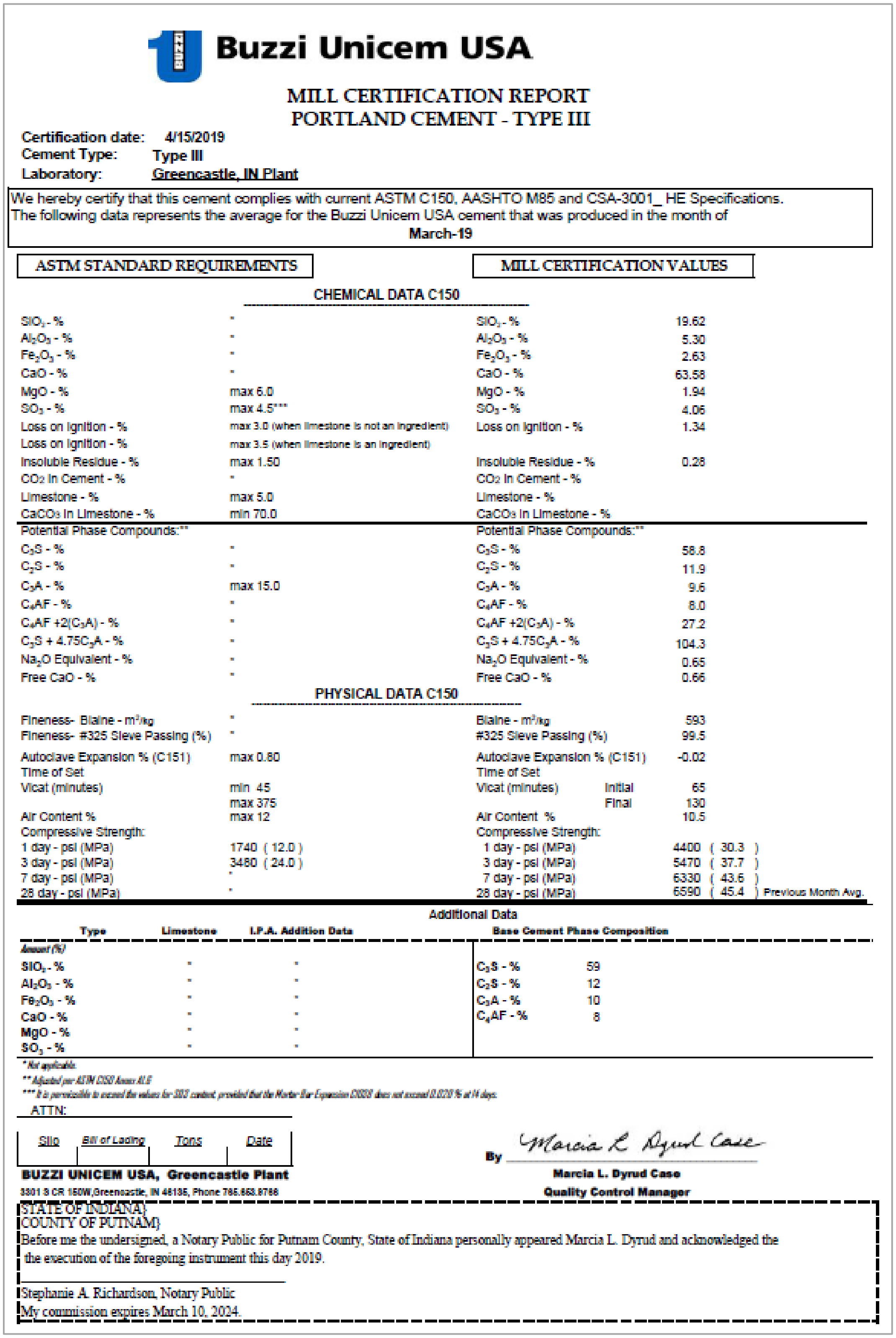
By Marcia L Dyrud Case

Marcia L. Dyrud Case
Quality Control Manager

BUZZI UNICEM USA, Greencastle Plant
3301 S CR 150W,Greencastle, IN 46135, Phone 765.653.9766

STATE OF INDIANA}
COUNTY OF PUTNAM}
Before me the undersigned, a Notary Public for Putnam County, State of Indiana personally appeared Marcia L. Dyrud and acknowledged the the execution of the foregoing instrument this day 2019.

Stephanie A. Richardson, Notary Public
My commission expires March 10, 2024.



Buzzi Unicem USA

MILL CERTIFICATION REPORT
PORTLAND CEMENT - TYPE III

Certification date:	4/15/2019
Cement Type:	Type III
Laboratory:	Greencastle, IN Plant

We hereby certify that this cement complies with current ASTM C150, AASHTO M85 and CSA-3001_ HE Specifications.
The following data represents the average for the Buzzi Unicem USA cement that was produced in the month of
March-19

CHEMICAL DATA C150

ASTM STANDARD REQUIREMENTS		MILL CERTIFICATION VALUES	
SiO_2 - %	*	SiO_2 - %	19.62
Al_2O_3 - %	*	Al_2O_3 - %	5.30
Fe_2O_3 - %	*	Fe_2O_3 - %	2.63
CaO - %	*	CaO - %	63.58
MgO - %	max 6.0	MgO - %	1.94
SO_3 - %	max 4.5***	SO_3 - %	4.06
Loss on Ignition - %	max 3.0 (when limestone is not an ingredient)	Loss on Ignition - %	1.34
Loss on Ignition - %	max 3.5 (when limestone is an ingredient)		
Insoluble Residue - %	max 1.50	Insoluble Residue - %	0.28
CO_2 in Cement - %	*	CO_2 in Cement - %	
Limestone - %	max 5.0	Limestone - %	
$CaCO_3$ in Limestone - %	min 70.0	$CaCO_3$ in Limestone - %	
Potential Phase Compounds:**		Potential Phase Compounds:**	
C_3S - %	*	C_3S - %	58.8
C_2S - %	*	C_2S - %	11.9
C_3A - %	max 15.0	C_3A - %	9.6
C_4AF - %	*	C_4AF - %	8.0
C_4AF +2(C_3A) - %	*	C_4AF +2(C_3A) - %	27.2
C_3S + 4.75C_3A - %	*	C_3S + 4.75C_3A - %	104.3
Na_2O Equivalent - %	*	Na_2O Equivalent - %	0.65
Free CaO - %	*	Free CaO - %	0.66

PHYSICAL DATA C150

ASTM STANDARD REQUIREMENTS		MILL CERTIFICATION VALUES		
Fineness- Blaine - m^2/kg	*	Blaine - m^2/kg		593
Fineness- #325 Sieve Passing (%)	*	#325 Sieve Passing (%)		99.5
Autoclave Expansion % (C151)	max 0.80	Autoclave Expansion % (C151)		-0.02
Time of Set		Time of Set		
Vicat (minutes)	min 45	Vicat (minutes)	Initial	65
	max 375		Final	130
Air Content %	max 12	Air Content %		10.5
Compressive Strength:		Compressive Strength:		
1 day - psi (MPa)	1740 (12.0)	1 day - psi (MPa)		4400 (30.3)
3 day - psi (MPa)	3480 (24.0)	3 day - psi (MPa)		5470 (37.7)
7 day - psi (MPa)	*	7 day - psi (MPa)		6330 (43.6)
28 day - psi (MPa)	*	28 day - psi (MPa)		6590 (45.4) Previous Month Avg.

Additional Data

Type	Limestone	I.P.A. Addition Data	Base Cement Phase Composition	
Amount (%)				
SiO_2 - %	*	*	C_3S - %	59
Al_2O_3 - %	*	*	C_2S - %	12
Fe_2O_3 - %	*	*	C_3A - %	10
CaO - %	*	*	C_4AF - %	8
MgO - %	*	*		
SO_3 - %	*	*		

** Not applicable.*
*** Adjusted per ASTM C150 Annex A1.6*
**** It is permissible to exceed the values for SO3 content, provided that the Mortar Bar Expansion C1038 does not exceed 0.020 % at 14 days.*

ATTN:

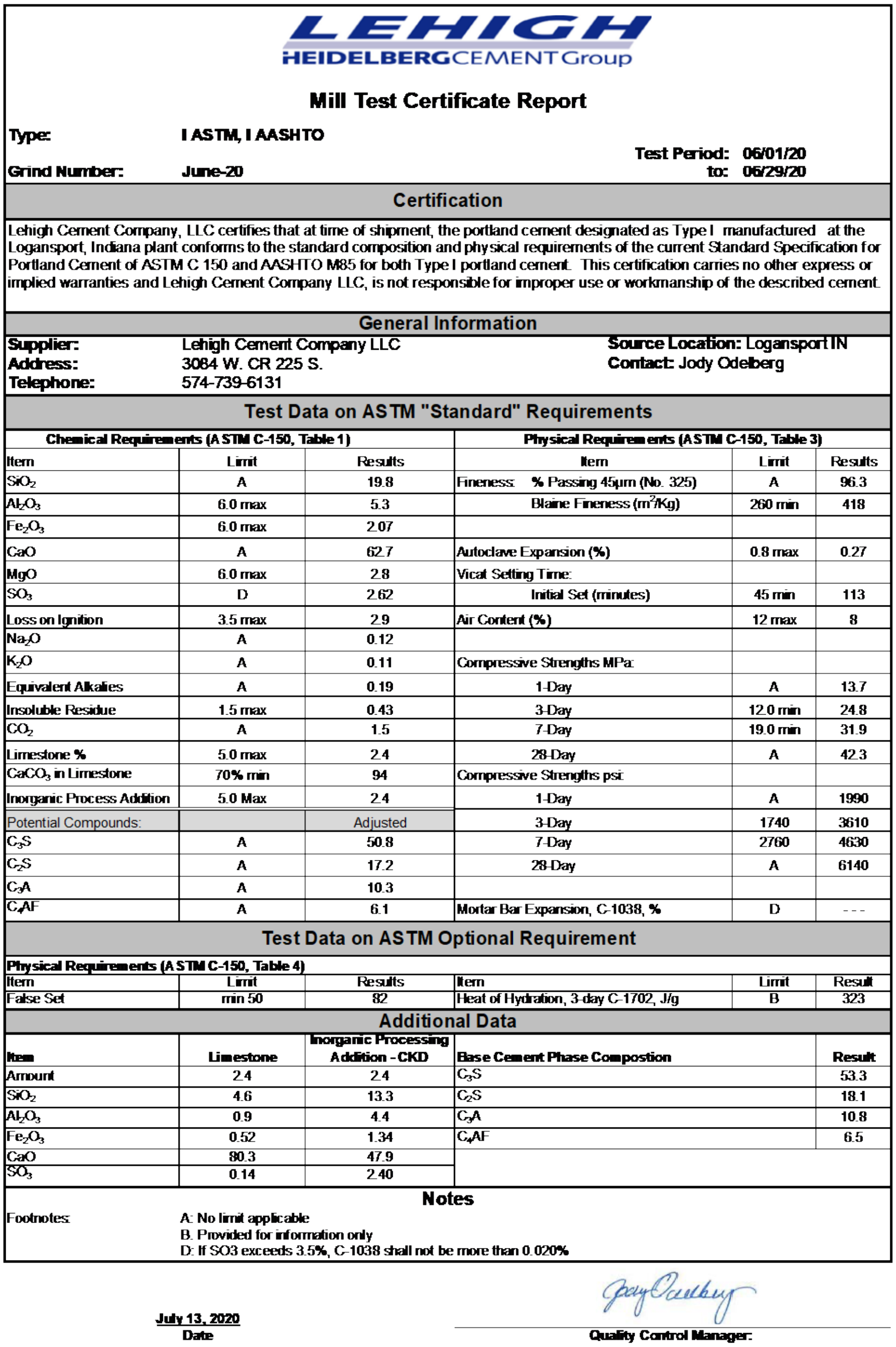
Silo	Bill of Lading	Tons	Date

BUZZI UNICEM USA, Greencastle Plant
3301 S CR 150W,Greencastle, IN 46135, Phone 765.653.8766

By Marcia L Dyrud Case
Marcia L. Dyrud Case
Quality Control Manager

STATE OF INDIANA}
COUNTY OF PUTNAM}
Before me the undersigned, a Notary Public for Putnam County, State of Indiana personally appeared Marcia L. Dyrud and acknowledged the the execution of the foregoing instrument this day 2019.

Stephanie A. Richardson, Notary Public
My commission expires March 10, 2024.



LEHIGH
HEIDELBERGCEMENT Group

Mill Test Certificate Report

Type: **I ASTM, I AASHTO**

Test Period: **06/01/20**
to: **06/29/20**

Grind Number: **June-20**

Certification

Lehigh Cement Company, LLC certifies that at time of shipment, the portland cement designated as Type I manufactured at the Logansport, Indiana plant conforms to the standard composition and physical requirements of the current Standard Specification for Portland Cement of ASTM C 150 and AASHTO M85 for both Type I portland cement. This certification carries no other express or implied warranties and Lehigh Cement Company LLC, is not responsible for improper use or workmanship of the described cement.

General Information

Supplier: Lehigh Cement Company LLC
Address: 3084 W. CR 225 S.
Telephone: 574-739-6131

Source Location: Logansport IN
Contact: Jody Odelberg

Test Data on ASTM "Standard" Requirements

Chemical Requirements (ASTM C-150, Table 1)			**Physical Requirements (ASTM C-150, Table 3)**		
Item	Limit	Results	Item	Limit	Results
SiO_2	A	19.8	Fineness: % Passing 45µm (No. 325)	A	96.3
Al_2O_3	6.0 max	5.3	Blaine Fineness (m^2/Kg)	260 min	418
Fe_2O_3	6.0 max	2.07			
CaO	A	62.7	Autoclave Expansion (%)	0.8 max	0.27
MgO	6.0 max	2.8	Vicat Setting Time:		
SO_3	D	2.62	Initial Set (minutes)	45 min	113
Loss on Ignition	3.5 max	2.9	Air Content (%)	12 max	8
Na_2O	A	0.12			
K_2O	A	0.11	Compressive Strengths MPa:		
Equivalent Alkalies	A	0.19	1-Day	A	13.7
Insoluble Residue	1.5 max	0.43	3-Day	12.0 min	24.8
CO_2	A	1.5	7-Day	19.0 min	31.9
Limestone %	5.0 max	2.4	28-Day	A	42.3
$CaCO_3$ in Limestone	70% min	94	Compressive Strengths psi:		
Inorganic Process Addition	5.0 Max	2.4	1-Day	A	1990
Potential Compounds:		Adjusted	3-Day	1740	3610
C_3S	A	50.8	7-Day	2760	4630
C_2S	A	17.2	28-Day	A	6140
C_3A	A	10.3			
C_4AF	A	6.1	Mortar Bar Expansion, C-1038, %	D	- - -

Test Data on ASTM Optional Requirement

Physical Requirements (ASTM C-150, Table 4)					
Item	Limit	Results	Item	Limit	Result
False Set	min 50	82	Heat of Hydration, 3-day C-1702, J/g	B	323

Additional Data

Item	**Limestone**	**Inorganic Processing Addition - CKD**	**Base Cement Phase Compostion**	**Result**
Amount	2.4	2.4	C_3S	53.3
SiO_2	4.6	13.3	C_2S	18.1
Al_2O_3	0.9	4.4	C_3A	10.8
Fe_2O_3	0.52	1.34	C_4AF	6.5
CaO	80.3	47.9		
SO_3	0.14	2.40		

Notes

Footnotes:
A: No limit applicable
B. Provided for information only
D: If SO3 exceeds 3.5%, C-1038 shall not be more than 0.020%

Jody Odelberg

July 13, 2020
Date

Quality Control Manager:

Votorantim Cimentos

St Marys Cement

Portland Cement Type I/II

Production Period: 5/01/2020 to 5/31/2020

STANDARD REQUIREMENTS

Chemical Data

Item	Spec. Limit	Results
SiO_2 (%)		19.7
Al_2O_3 (%)	*6.0 max*	4.9
Fe_2O_3 (%)		3.01
CaO (%)		62.3
MgO (%)	*6.0 max*	3.2
SO_3 (%)*	*3.0 max*	4.03
Loss of ignition (%)	*3.5 max*	1.1
Na_2O (%)		0.13
K_2O (%)		0.96
Insoluble residue (%)	*1.5 max*	0.20
CO_2 (%)		0.8
Limestone (%)	*5.0 max*	2.2
$CaCO_3$ in limestone (%)	*70.0 min*	88.8
Inorganic process addition(%)	*5.0 max*	0.0
Potential phase composition		
C_3S (%)		52
C_2S (%)		17
C_3A (%)	*8 max*	8
C_4AF (%)		9

Physical Data

Item	Spec. Limit	Results
Air Content of mortar (volume %)	*12.0 max*	9
Blaine fineness (m^2/kg)	*260.0 min*	376
Autoclave expansion (%)	*0.8 max*	0.12
Compressive strength (MPa/psi):		
1 day		20.7 [2997]
3 days	*12.0[1740] min*	29.7 [4302]
7 days	*19.0[2760] min*	35.5 [5148]
28 days *(previous month)*	*28.0[4060] min*	45.3 [6575]
Time of setting (minutes)		
(Vicat) Initial	*45 - 375*	87
(Vicat) Final		199
Mortar Bar Expansion (ASTM C1038) (%)*	*0.02 max*	0.009
Base Cement Phase Composition		
C_3S (%)		53
C_2S (%)		18
C_3A (%)		8
C_4AF (%)		9

OPTIONAL REQUIREMENTS

Item	Spec. Limit	Results	Item	Spec. Limit	Results
Equiv. Alkalies	*A*	0.76	False Set (%)	*50 min*	67
			Retained 325 (%)		2.51

Additional Data

Type	Limestone	Inorganic Processing Addition
Amount	2.2	0.0
SiO_2 (%)	4.7	
Al_2O_3 (%)	1.5	
Fe_2O_3 (%)	0.7	
CaO (%)	51.2	
SO_3 (%)	0.7	

A

June 22, 2020

St. Marys Cement
Charlevoix Cement Plant
16000 Bells Bay Road
Charlevoix, MI 49719
Tel: (231) 547-1362 - Fax: (231) 547-6202

Fred Hommel

Frederick Hommel
Lab Supervisor

Brand: NewCem®
Material: Slag Cement
Type: Grade 100

Material Certification Report

Test Period: 01-July-2020 to 31-July-2020
Lot Number: Multiple Lots

Certification

This cement meets the specifications of ASTM C989 and AASHTO M 302 for Grade 100 slag cement.

General Information

Supplier:	Holcim (US) Inc. d/b/a LafargeHolcim US	**Source Location:**	South Chicago Plant
Address:	8700 West Bryn Mawr Ave Chicago, IL 60631		2150 East 130th Street Chicago, IL 60633
Contact:		**Contact:**	Brian Borowski / (630) 561-1198

The following is based on average test data during the test period. The data is typical of product shipped from this source; individual shipments may vary.

Test Data on ASTM Standard Requirements

Chemical			Physical		
Item	**Limit [1]**	**Result**	**Item**	**Limit [1]**	**Result**
Sulfide Sulfur (S) (%)	2.5 max	0.9	+45 µm (No. 325) Sieve (%)	20 max	1.1
			Blaine Fineness (m^2/kg)	-	530.3
Sulfate Sulfur (as SO_3) [2] (%)	-	0.0	Air Content (%)	12 max	5.49
Aluminum Oxide (as Al_2O_3) (%)	-	10.2	Slag Activity Index (%)		
			Avg 7 Day Index	-	83
Chloride (Cl) (%)	-	0.054	Avg 28 Day Index (previous month's data)	95 min	106
Equivalent Alkalies (%)	-	0.56	Compressive Strength MPa (psi)		
			Slag + Reference Cement		
			7 Day	-	24.4 (3540)
			28 Day (previous month's data)	-	40.3 (5840)

Test Data on Reference Cement

Chemical			Physical		
Item	**Limit [1]**	**Result**	**Item**	**Limit [1]**	**Result**
Equivalent Alkalies (%)	0.60 - 0.90	0.78	7 Day	-	29.4 (4260)
			28 Day (previous month's data)	5000 min	38.2 (5540)

Notes (*1-5)

1 - Dashes in the limits columns means Not Applicable
2 - If calcium sulfate is added to slag cement, measure in accordance with Test Method C1038/C1038M. Slag cement with added calcium sulfate will not develop expansion exceeding 0.020% at 14 days.
3 - Information on Reference Cement test data available upon request.
4 - Specific Gravity: 2.97
5 - This data may have been reported on previous Material Certification Reports. It is typical of the cement being currently shipped.

Date Issued: 9/8/2020

Brian Borowski
Quality Manager, US MPC

About the Joint Transportation Research Program (JTRP)

On March 11, 1937, the Indiana Legislature passed an act which authorized the Indiana State Highway Commission to cooperate with and assist Purdue University in developing the best methods of improving and maintaining the highways of the state and the respective counties thereof. That collaborative effort was called the Joint Highway Research Project (JHRP). In 1997 the collaborative venture was renamed as the Joint Transportation Research Program (JTRP) to reflect the state and national efforts to integrate the management and operation of various transportation modes.

The first studies of JHRP were concerned with Test Road No. 1—evaluation of the weathering characteristics of stabilized materials. After World War II, the JHRP program grew substantially and was regularly producing technical reports. Over 1,600 technical reports are now available, published as part of the JHRP and subsequently JTRP collaborative venture between Purdue University and what is now the Indiana Department of Transportation.

Free online access to all reports is provided through a unique collaboration between JTRP and Purdue Libraries. These are available at http://docs.lib.purdue.edu/jtrp.

Further information about JTRP and its current research program is available at http://www.purdue.edu/jtrp.

About This Report

An open access version of this publication is available online. See the URL in the citation below.

Adams, C. J., Bose, B., Mann, E., Erk, K. A., Behnood, A., Castillo, A., Rodriguez, F. B., Wang, Y., & Olek, J. (2022). *Superabsorbent polymers for internally cured concrete* (Joint Transportation Research Program Publication No. FHWA/IN/JTRP-2022/04). West Lafayette, IN: Purdue University. https://doi.org/10.5703/1288284317366