Superabsorbent Polymer in Concrete to Improve Durability

Final Report
March 2025

National Concrete Pavement Technology Center

Tech Center

IOWA STATE UNIVERSITY
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16. Abstract The main objective of this research was to explore the optimized selection of superabsorbent polymer (SAP) products as internal curing materials in concrete and to examine the effects of the use of eight selected SAPs on properties that affect concrete performance, including shrinkage, hydration, permeability, and strength. Experimental results demonstrated that the use of SAPs improved the shrinkage and hydration of concrete while reducing strength and permeability, although the latter were still within acceptable bounds.			
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Final Report
March 2025

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EXECUTIVE SUMMARY

Internal curing involves adding small, uniformly distributed reservoirs of water within concrete. This method allows the concrete mixture to maintain a low water-to-cementitious materials (w/cm) ratio while gradually supplying the stored water to the concrete as the cement hydrates and the paste dries. Internal curing has been reported to be effective in reducing shrinkage cracking, improving the potential durability of concrete mixtures, and, most notably, reducing cracking caused by curling and warping in pavements and slabs on grade.

Superabsorbent polymers (SAPs) are synthetic materials that absorb significant amounts of water. When incorporated into concrete, they can act as tiny internal water reservoirs for internal curing. SAPs are commonly used in high-performance concrete, where the w/cm ratio is often relatively low, to improve cement hydration, densify the microstructure, and reduce concrete shrinkage.

In this project, eight SAP products with different properties were selected as internal curing materials. Criteria related to particle size and absorption/desorption rates were established to guide the selection of suitable products. The optimal contents of the SAPs were identified based on the absorption and desorption properties. Experimental results demonstrated that the incorporation of SAPs slightly reduced the strength and electrical resistivity of concrete, although these parameters remained within acceptable bounds, while the degree of hydration and shrinkage of the concrete were clearly improved.

INTRODUCTION

Problem Statement

Internal curing is the practice of providing small, well-distributed reservoirs of water throughout a concrete section so that the water-to-cementitious materials (w/cm) ratio of the mixture can be kept low but water can later be delivered to hydrate the cement as the system dries out. Internal curing has been reported to be effective in reducing shrinkage cracking, improving the potential durability of concrete mixtures, and, most notably, reducing warping and associated cracking in pavements and slabs on grade.

Currently, the use of lightweight fine aggregate (LWFA) is the most common practice in the United States to produce internally cured concrete. This method, however, necessitates the presaturation of aggregate at concrete batch plants in accordance with a set timeline. This may increase costs related to stockpile management and the costs and emissions associated with producing and hauling the LWFA. Furthermore, the use of LWFA can reduce the elastic modulus of concrete.

The use of superabsorbent polymers (SAPs) as a means of internal curing can address such problems while promoting hydration and reducing the risk of early-age cracking. However, there has been relatively little work conducted in the United States on these materials. Open questions include the following:

- How should SAP products be specified?
- How much of a given SAP is needed in a concrete mixture?
- Can SAPs be dry batched with additional water in a mixture without compromising performance?
- How are mixtures affected by the use of SAPs?

Objectives

This research aimed to assess the potential use of SAPs for internal curing in concrete mixtures, with specific objectives as follows:

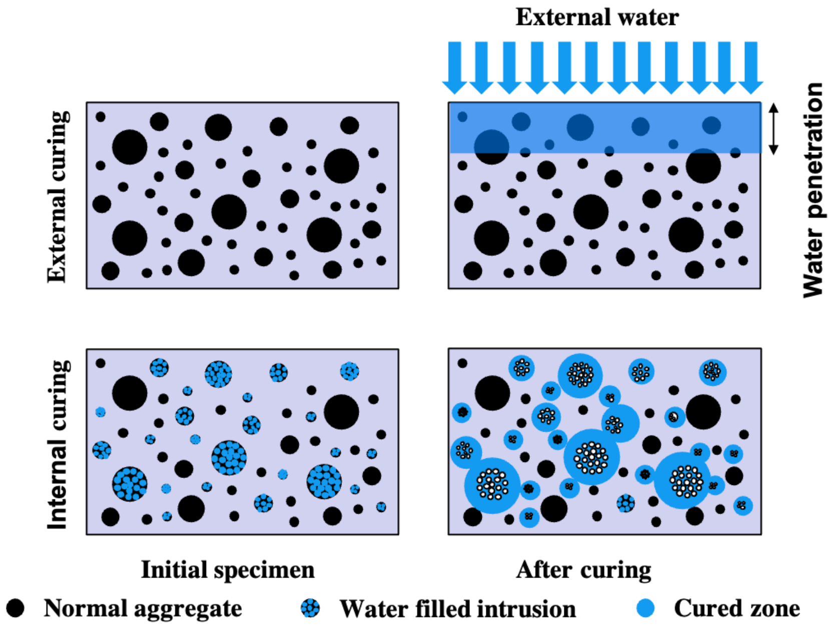
- Specify SAP products based on an investigation of their different types, absorption kinetics, and particle sizes
- Determine the quantity of SAP required for a concrete mixture
- Evaluate the feasibility of dry batching SAPs with additional water
- Assess the impact of SAPs that have been optimized for internal curing on properties that affect concrete performance, including strength and durability

INTERNAL CURING

Definition of Internal Curing

Concrete is a nonuniform composite material comprising combinations of aggregates and cementitious paste (Danish et al. 2020). When a cementitious material is mixed with water, the active components will hydrate to form hydration products, which interact physically causing the mixture to set and gain strength. If water is lost due to evaporation and/or desiccation at an early stage, cement hydration will slow or stop. This prevents the development of concrete strength and impermeability (Sanjuán and Muñoz-Martialay 1995, Liu et al. 2019) At the same time, concrete begins to shrink and generates internal stresses, increasing the risk of cracking (Jiang et al. 2005, Odler 2019).

Curing methods, including external curing and internal curing, are utilized to mitigate water loss, as shown in Figure 1.1. Conventional concrete surface curing methods include sprinkling extra water on the surface or covering the surface with burlap and plastic sheets to reduce water loss. However, these methods do not address water loss due to internal desiccation. In addition, externally supplied water will only penetrate a limited distance from the surface (see Figure 1.1). Internally stored water, on the other hand, can benefit the full depth of the concrete section.



Bentz and Weis 2011

Figure 1.1. External and internal curing

Internal curing can be achieved by the judicious use of absorbent materials, such as expanded shale (Bremmer and Ries 2009), clay (Bremmer and Ries 2009), fly ash (De La Varga et al. 2012), rice husk ash (Van et al. 2014, Henkensiefken et al. 2009), lightweight aggregate (LWA) (Henkensiefken et al. 2009), and SAP (Justs et al. 2015).

Benefits of internal curing include the following:

- Increased degree of hydration (and therefore better mechanical and durability properties)
- Reduced shrinkage (and therefore lower cracking risk)
- Reduced moisture differentials (and therefore reduced warping in thin slabs)

Required Properties of Internal Curing Agents in Concrete

Internal curing requires the use of materials able to freely release the stored water to the cement paste after setting, which implies that any potential internal curing medium must comply with two requirements. The first requirement is that water stored in the internal curing medium should have an equilibrium relative humidity (RH) of 99% (Jensen 1999).

Another requirement is that any potential internal curing medium should have kinetic availability to transfer the water to the cement paste as needed when the RH in the surrounding paste matrix drops. In addition, the stored water should be uniformly spatially distributed to reduce the internal stresses due to differential drying, which can be improved by decreasing the size and increasing the number of water-containing particles (Jensen and Lura 2006).

Basic Properties of SAP

SAP is a kind of functional macromolecule with a three-dimensional (3D) network structure that has significant water absorption and retention abilities. The use of SAP products in hygiene and agriculture has been extensively researched and implemented. SAPs are commonly synthesized from acrylic acid and derivatives, undergoing cross-linking reactions to create a highly absorbent structure. However, not all ready-made SAP is suitable and beneficial for cementitious building materials, as the majority of synthesized SAP products focus on performance in other industrial applications, such as the production of diapers or drug delivery systems in the hygiene industry (Elliot 2010, Sadeghi and Hossein 2008). Jensen and Hansen (2001) introduced these polymeric materials as a water-entraining additive in construction materials technology.

The properties desired for a SAP to be effective in concrete systems include the following:

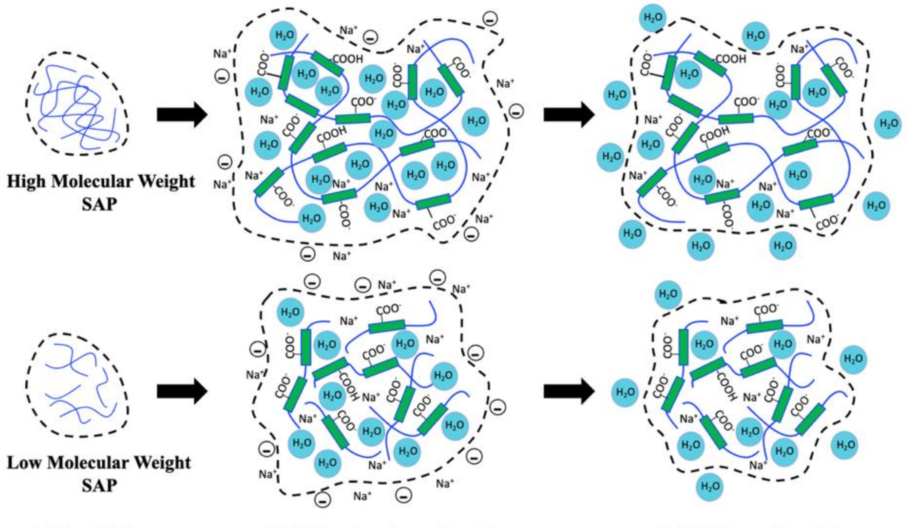
- Ability to rapidly absorb sufficient water during mixing
- Ability to retain integrity during mixing
- Ability to desorb when required (i.e., when RH drops below ~94%)
- Appropriate particle size distribution

Absorption and Desorption

The quantity of liquid absorbed, absorption kinetics and potential desorption, and stability of the product formed in solution depend on thermodynamics and the osmotic pressure differences between the gel network and the exterior environment (English et al. 1998).

Different from LWFA, dry SAP can be mixed with raw concrete materials before water is added during concrete mixing, thus facilitating the uniform dispersion of SAP. Therefore, SAP has to absorb the right amount of water from the mixture while it is still being mixed and transported. In addition, the desorption rate of SAP is a significant factor because it determines the timing and amount of water supplied to the paste (Schröfl 2012).

It is well known (Mignon et al. 2014, Chung 2004, Zhu et al. 2019, Mönnig 2015, Jensen 2010) that absorption and desorption kinetics are closely related to the cross-linking density, molecular structure (see Figure 1.2), and particle size of SAP. High molecular weight SAPs are capable of ionizing with hydrophilic groups such as carboxylic groups and hydroxyl groups when in contact with water. As shown in Figure 1.2a, dry SAP has a 3D network structure made up of tangled molecular chains. When the hydrophilic groups in the polymer chain are ionized, a difference in ionic concentration arises between the inside and outside of the molecule. This leads to the formation of gel caused by the successive ingress of water molecules into the internal structure. SAP containing different molecular weights exhibits quite different molecular bond expansion. Therefore, SAP with a higher molecular weight would have more internal network space, which corresponds to an enhanced retention capacity due to the migration of anions and cations. However, when the release of water from SAP occurs, the expansion of molecular bonds is also influenced by the molecular weight of SAP. Molecular bonds are restricted in their expansion as SAP's molecular weight increases. This is attributed to the weak bonding between the water molecules and hydrophilic groups in the molecular chain structure of SAP. For example, SAP with a smaller molecular weight has less 3D space formed to store water molecules after the expansion of the molecular chain. This in turn results in the water molecules absorbed by the hydrophilic groups being unable to escape easily. Therefore, the release of free water from the molecular chain structure is slower, as shown in Figures 1.2b and 1.2c.



(a) Dry SAP (b) SAP water absorption status (c) SAP desorption status

Zhu et al. 2019

Figure 1.2. Sorption kinetic behaviors of SAP with different molecular weights

In Figure 1.2, the kinetic behavior of SAP absorption and desorption in concrete provides insight into the physicochemical properties of the polymers added to the concrete. Time-resolved experiments on swelling must also consider the rate of absorption since it is considered one factor of assessment throughout the interval between mixing and placing concrete. After the final placement, no further water needs to be absorbed as the framework for the system is locked in. Hence, SAP's sorption properties determine how SAP will affect concrete's properties in both the short and long term.

Physical/Chemical Stability

SAP as an internal curing agent in concrete should have sufficient physical stability to remain whole during the rigors of mixing, transportation, and placement (Moini and Kabiri 2015, Ashkani et al. 2018). Generally, the mechanical strength of SAP is correlated with the cross-linking density inside its gel structure. When SAP has a higher cross-linking density, its gel strength becomes higher (Ashkani et al. 2018). However, to some extent, this will reduce the swelling capacity of the gel sample. Therefore, some researchers (Moini and Kabiri 2015, Li et al. 2018, Hu et al. 2012, Haraguchi 2011, Buchholz and Graham 1998) have proposed strategies to improve the swelling capacity by using double-networking and nanoparticles, which make the gel sample have both good water absorbency and mechanical properties. For example, Ma et al. (2004) studied ethylene glycol diglycidyl ether as a surface cross-linking agent to enhance the surface cross-linking of SAP particles, and the results showed that this method was effective at improving the gel strength of SAP.

In addition, SAPs need to retain their properties in concentrated solutions of cementitious pore solutions. When SAP particles are soaked in different ionic solutions (Na^+, Ca^{2+}, and Al^{3+}), the chemical bonds within the SAP microstructure should not decompose.

Particle Sizes

The mechanical performance of concrete paste is strongly influenced by the size and amount of voids in the hardened system. It is preferable to have a large number of small voids as opposed to a few large ones that will act as areas of stress concentration.

In addition, the maximum absorption capacity and absorption rate are influenced by the particle size of SAPs. The smaller the particle size of the SAP particles, the larger the relative exposure surface and the smaller the distance that water needs to travel to fully saturate the particle. Luís Pedro Esteves (2011) used Fick's second law of diffusion to show the nonlinear relationship between the absorption property of SAP and its particle size (diameter):

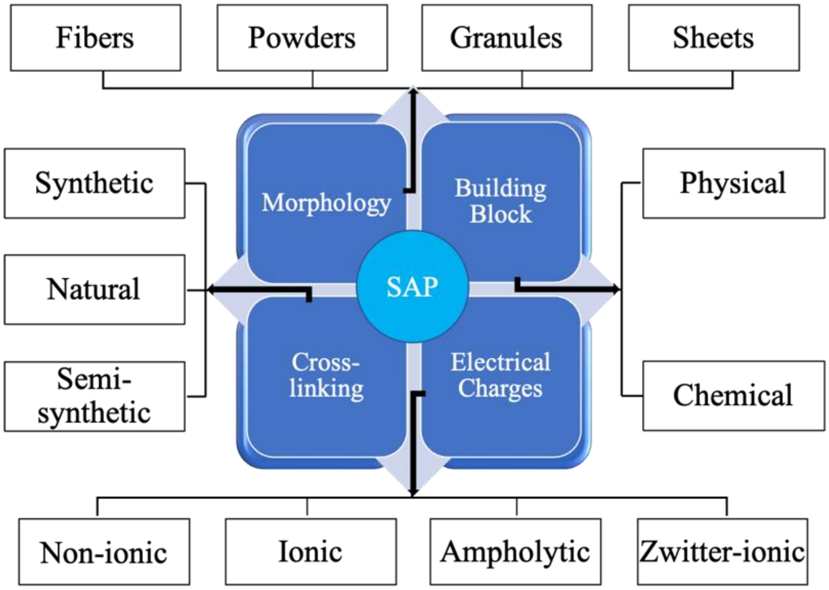
$$\frac{d_Q}{d_t} = k * (Q_{max} - Q) \tag{1}$$

where Q_{max} and Q are the swelling capacities at equilibrium and at any time t and k is the swelling constant rate, which depends on the particle size.

A solution/bulk polymerization technique is currently used to synthesize SAPs, in which a cross-linking bulk of randomly composed copolymers is broken into fine particles to meet a given particle size requirement. Consequently, SAP particles are irregularly shaped and their particle sizes are related to the selection mechanism (Buchholz and Graham 2007, Omidian et al. 1998). If inverse suspension polymerization is utilized, the SAP will be spherical, with a narrow size distribution (Daamen and Retorofu 1989, Dowding and Vincent 2000). Various dry SAP particle sizes, typically below 100 µm, have been used in cementitious materials (Esteves 2011, Snoeck et al. 2015, AzariJafari et al. 2016, Zhu et al. 2015, Snoeck 2015, Lee et al. 2013, Deng and Liao 2018, Ma et al. 2017, De Meyst et al. 2019, Ma et al. 2019, Jensen and Hansen 2002, Shen et al. 2016).

Chemical Characteristics of SAP

The potential efficiency of SAPs as internal curing agents in practical applications is affected by the swelling properties, sorption kinetics, type, dosage, and particle size of the SAPs. Additionally, the cross-linking density/degree is closely correlated with the swelling behaviors and sorption kinetics of SAP. The classification of SAP is based on morphology, chemical building blocks, cross-linking mechanisms, and the types of electrical charges present, as shown in Figure 1.3.



Mignon et al. 2019

Figure 1.3. Classification of SAP based on different characteristics

There are two main kinds of cross-links in the chains of most SAPs, namely, bulk or core cross-linking and surface cross-linking. The former occurs in the polymerization process of SAP production and is governed by the chains' reactivity ratios (Jensen 2011). Normally, the lower the cross-linking level, the higher the swelling capacity of the polymer. The latter can be modified in the post-process to improve the swelling rate and the resistance to pressure absorption of SAP, which contributes to maintaining the original shape of particles when immersed. Cross-linking of SAPs includes both physical cross-linking and chemical cross-linking. The weak physical bonds—like the hydrogen bonds in the physical cross-linking chain—are unstable, whereas the chemical covalent bonds are stable and strong.

Products are also classified based on the presence of electrical charges in the side chains of molecules as ionic (charged) and nonionic (neutral). Currently, commercially available SAPs are mostly ionic polymers.

Most SAPs used for internal curing are polyacrylate acids and polyacrylamides, both containing amide groups that can react with water to form carboxylate groups with hydrogen, which can be deprotonated, resulting in anionic charges left along the side chains of SAPs. The polyacrylic acids are the most used commercial polymers and are made by using the suspension polymerization technique. Moreover, these SAPs with high molecular weights (up to a few million g/mol) show a higher absorption capacity compared to other types of SAPs (Hewlett 2003). However, the absorption capacity of these commercial SAPs for application in concrete systems is reduced due to the presence of cations and anions in the cement paste.

It is necessary to understand the sorption kinetics between SAP and both water and pore fluid because the properties of SAP are affected by solution chemistry (Zohuriaan-Mehr 2006, Jabbar Braihi et al. 2014). Therefore, the reaction variables forming the SAP must be optimized to obtain a suitable balance among properties based on the target application. The SAP should achieve a stable thermodynamic equilibrium (swelling equilibrium at the balance of osmotic pressure), as shown in equation (2) (Richter 2008):

$$\Delta\pi = \Delta\pi_{elastic} + \Delta\pi_{mix} + \Delta\pi_{ion} + \Delta\pi_{bath} = 0 \tag{2}$$

where $\Delta\pi$ is the swelling equilibrium pressure of the hydrogel, $\Delta\pi_{elastic}$ is an elastic force exerted by the polymer network, $\Delta\pi_{mix}$ is the osmotic pressures generated by the polymer-solvent interactions during the mixing phase, $\Delta\pi_{ion}$ is the expansive pressure contributed by the electrostatic interactions of polyelectrolyte hydrogels, and $\Delta\pi_{bath}$ is the contribution of ionic strength from the surrounding bath solution.

In essence, most SAPs are a cross-linked structural polymer with a 3D network. The water molecules will first penetrate the cross-linked network, resulting in a difference in osmotic pressure $\Delta\pi_{mix}$ between the outside and inside of the polymer network. On the other hand, due to the interactions between polymers, the primary polymer network in the chain is limited to moving freely; therefore, an elastic force $\Delta\pi_{elastic}$ counteracts the expansive force.

Polyelectrolyte hydrogels consist of weak acidic and weak basic groups, respectively, which cause the hydrophilic group in the chain of SAP to ionize, yielding a difference in ion concentration between the internal solutions. When acrylic-based SAP is used in a cementitious environment, the hydrogel with acidic groups will be almost completely deprotonated; for example, almost all carboxylic groups have negative charges (English et al. 2000). Therefore, the neighboring chains with likewise charged groups will develop electrostatic repulsion $\Delta\pi_{ion}$

between chains, which contributes to the balance of the osmotic pressure (Richter 2008). The hydrogel system, however, must maintain electrical neutrality within itself; therefore, the counterions can’t be released from the system unless they’re replaced by another mobile ion from the surrounding environment. If the surrounding environment of SAP is the cement-based medium, the enriched calcium ions Ca^{2+} can exchange with the mobile ions of the gel in the SAP network, which will yield the osmotic pressure due to the different ion concentrations inside and outside of the polymer network structure.

Batching SAP

Different approaches to introducing particles into concrete mixtures have been studied. Each approach leads to different influences on the development of concrete properties. Table 1.1 lists the effects of different batching processes on concrete performance.

Table 1.1. SAP-modified concrete mixtures and properties

Concrete Types	Method of Adding SAP	Fineness	Content (%) by Mass of Cement or Binders	Mixing Procedure	Influences on Concrete Properties
SCLWC (AzariJafari et al. 2016)	Presoaked	1–4 mm and 0.3–1 mm	1.5	1. Mix aggregates and cementitious materials for 60 sec. 2. Add water and superplasticizer and mix for 5 min. 3. Add presaturated SAP and mix for 2 min.	1. Improves passing ability and decreases the static stability of fresh SCLWC mixtures. 2. Reduces the compressive strength of SCLWC mixtures at ages of 7 and 28 days. 3. Induces a negative effect on the electrical resistivity and water absorption of SCLWC. 4. Results in up to 50% reduction in total shrinkage of SCLWC compared to the reference mixture made without SAP.
HPC (Wang et al. 2009)	Prewetted	No greater than 500 µm	0.3, 0.5, and 0.7	1. Prewet the SAP. 2. Mix the prewetted SAP and other dry materials for 60 sec. 3. Add water and mix for 60 sec.	1. Obviously delays internal relative humidity (IRH) decline. 2. Mitigates autogenous shrinkage (AS) at an early age. 3. Decreases the compressive strength of concrete with a higher content of SAP or entrained water.
High-strength concrete (Kong et al. 2015)	Presoaked	180–420 µm	0.2 and 0.4	1. Mix cement, sand, and aggregate for 1 min. 2. Add water together with the presoaked SAP to the dry mixture and mix for 3 min.	1. Firmly alleviates the early-age shrinkage related to moisture loss (mainly consisting of AS and DS). 2. Postpones the drop of internal humidity. 3. Slightly reduces the compressive strength of HSCs.
HPC (Xiangmin 2013)	Presoaked	180–420 µm	0.2 and 0.4	1. Mix cement, sand, and aggregate for 1 min. 2. Add water together with the presoaked SAP to the dry mixture and mix for 3 min.	1. Increases the total porosity of the hardened cement paste. 2. Increases the gel pores and small capillary pores. 3. Increases the chloride diffusion coefficient.

Concrete Types	Method of Adding SAP	Fineness	Content (%) by Mass of Cement or Binders	Mixing Procedure	Influences on Concrete Properties
UHPC	Dry	<63 µm	0.206, 0.313, and 0.5	1. Mix SAP and all dry cementitious materials for 20 sec. 2. Add approximately 90% of water during a period of 30 sec. 3. Wet mix for 60 sec. 4. Add SP, the rest of water and mix for 90 sec. 5. Add fiber during a period of 45 sec. 6. Mix for 120 sec.	1. Reduces the internal relative humidity decrease and the autogenous shrinkage. 2. Decreases the compressive strength, flexural strength, and elastic modulus of the UHPC mixtures. 3. Delays the hydration time of the mixtures.
HPC (Assman and Reinhardt 2014)	Dry	60–125 µm	0.25	1. Mix cement powder, oven-dried aggregates, and dry SAP for 30 sec. 2. Add the first half of water and mix for 120 sec. 3. Add the second half of water together with the SP and mix for 5 min.	1. No change in compressive strength at 28 days. 2. Reduces autogenous shrinkage and drying shrinkage. 3. Significantly decreases the tensile creep of SAP modified concrete under the same total water content, the same strength, and the same paste volume.
Normal-strength concrete (Dang et al. 2017)	Non-absorbed/pre-absorbed	425–250, 180–150	0, 0.1, 0.2, and 0.3	1. Mix fine aggregate, coarse aggregate, cement, and fly ash for 30 sec. 2. Add water-reducing agent and concrete mixing water and mix for 100 sec. 3. Add non-absorbed or pre-absorbed SAP and mix for 60 sec.	1. Increases compressive strength of concrete. 2. Effectively reduces the shrinkage of concrete. 3. Improves the concrete's abilities of carbonation resistance and chloride penetration resistance. 4. Effectively improves the microstructure and makes the pore structure refined.
Normal-strength concrete (Liu et al. 2017)	Gel-type internal curing agent (ICA)	1–5 µm	3.65, 7.28, 10.93, and 14.56	1. Mix gravel, sand, cement, GGBS, and fly ash for 15 sec. 2. Add the water containing superplasticizer and the diluted ICA (with 50% of mixing water) and mix for 5 min.	1. Reduces the cracking area of concrete. 2. Delays the rates of decline of the IRH. 3. Mitigates the early-age cracking not only of hard-to-cure structures but also of concrete exposed to extreme weather conditions.

Concrete Types	Method of Adding SAP	Fineness	Content (%) by Mass of Cement or Binders	Mixing Procedure	Influences on Concrete Properties
Cellular concrete (Deng et al. 2016)	Millimeter-size spherical saturated SAP	0.5–2 mm	0.2	1. Mix cement, fine aggregate, superplasticizer, and saturated SAP for 1 min. 2. Add water and mix for 40 sec, then add fibers.	1. The compressive strength appears to decrease with the porosity under a given strain rate. 2. The value of average dynamic increase factor (DIF-fc) generally increases with rising porosity on a given stain rate level. 3. The dynamic increase factor for elastic modulus (DIF-E) decreases with porosity at the same level of strain rate.
Normal-strength concrete (Tenório Filho et al. 2020)	Dry	Mean particle size (d50) of 360 µm, d10 of 145 µm, and d90 of 460 µm	0.25	1. Mix dry materials for 1 min (including SAPs). 2. Add water and superplasticizer and mix for additional 2 min. 3. Add the additional entrained water and mix for additional 2 min.	1. No significant influence on the total shrinkage or the compressive strength. 2. Increases a number of bigger pores in the fresh concrete mixtures. 3. Decreases the shrinkage strain of concrete.

Dosage

Based on the principle of supplying a volume of water from SAP equal to the chemical shrinkage volume, the content of SAP addition in the concrete mixture design can be determined by equations (3) and (4) recommended by Bentz and Snyder (1999) and Montanari et al. (2017):

$$M_{SAP} = \frac{C_f CS \alpha_{max}}{\varphi_{SAP}} \tag{3}$$

$$\varphi_{SAP,30} = \frac{M_{SD,TB+SAP} - M_{SD,TB} - M_{D,SAP}}{M_{D,SAP}} \tag{4}$$

where M_{SAP} is the mass of the dry SAP (kg/m^3), C_f is the cement content of the mixture (kg/m^3), CS is the chemical shrinkage occurring during the hydration of the cement (this value is usually almost 0.06 kg water per kg of cement [Bentz 1997, Geiker 1983]), α_{max} is the expected maximum degree of hydration ranging from 0 to 1 (unitless), $\varphi_{SAP,30}$ is the absorption of the SAP in pore solution at 30 minutes (g/g), $M_{SD,TB+SAP}$ and $M_{SD,TB}$ are the mass of the tea bag with SAP and the tea bag in the soaked surface dry condition (g), respectively, and $M_{D,SAP}$ is the mass of the dry SAP (g).

In the literature (Savva and Petrou 2018), the absorption capacity of SAP per unit mass is 29 g/g using the tea bag method. The internal curing water requirement is 44 g/g calculated from the Bentz equation (Bentz and Snyder 1999). To hold the necessary quantity of internal curing water, the amount of SAP required is 0.18% of the cement mass, which equates to 1.5 g of SAP per 864 g of cement.

Presoaking

When LWFA is used for internal curing, it is presoaked before batching (Savva and Petrou 2018). This can lead to challenges in high-volume construction sites.

However, the absorption properties of SAP are quite different because the sorption of SAP is a physico-chemical process that is more efficient than the purely physical process in LWA. SAP particle sizes are often smaller with a higher absorption rate.

SAP particles are presoaked for different periods of time based on the corresponding requirement for the water content prior to mixing, and then the presoaked SAP particles are mixed with other concrete mixtures. However, there is no standard guidance for the sequence of presoaked SAP particles added to the concrete mixtures, as shown in Table 1.1.

The influence of presaturated SAP as an internal curing medium on the workability, early-age shrinkage, and mechanical properties of concrete was assessed by AzariJafari et al. (2016) and Kong et al. (2015). Their results revealed that the incorporation of presoaked SAP could not only improve the static stability and passing ability of fresh concrete but also mitigate the shrinkage at an early age, corresponding to an alleviation of the risk of shrinkage cracking, which was attributed to the compensation of water loss from the release of SAP. Additionally, a slight decrease in compressive strength was also reported due to the addition of presoaked SAP.

According to the data in Table 1.1, the additional presoaking of SAP is beneficial for performance improvement. However, it is still a challenge to precisely control the required water contents of the presoaked SAP at some specified period due to the release of water from the presoaked SAP during the mixing procedure.

Dry Batching

Much research has been conducted investigating dry SAP batching. It has been verified that this approach can distribute the SAP particles without agglomeration (Kang et al. 2017, Hasholt and Jensen 2015, Beushausen and Gillmer 2014). Attention has been paid to how the absorption rate of dry SAP affects the mixing and handling processes while achieving a suitable state of saturation. Lura et al. (2014) demonstrated that the absorption of 8 to 30 g/g of SAP was desirable for use in a highly alkaline solution, which is about 10 times less than in a tap water solution.

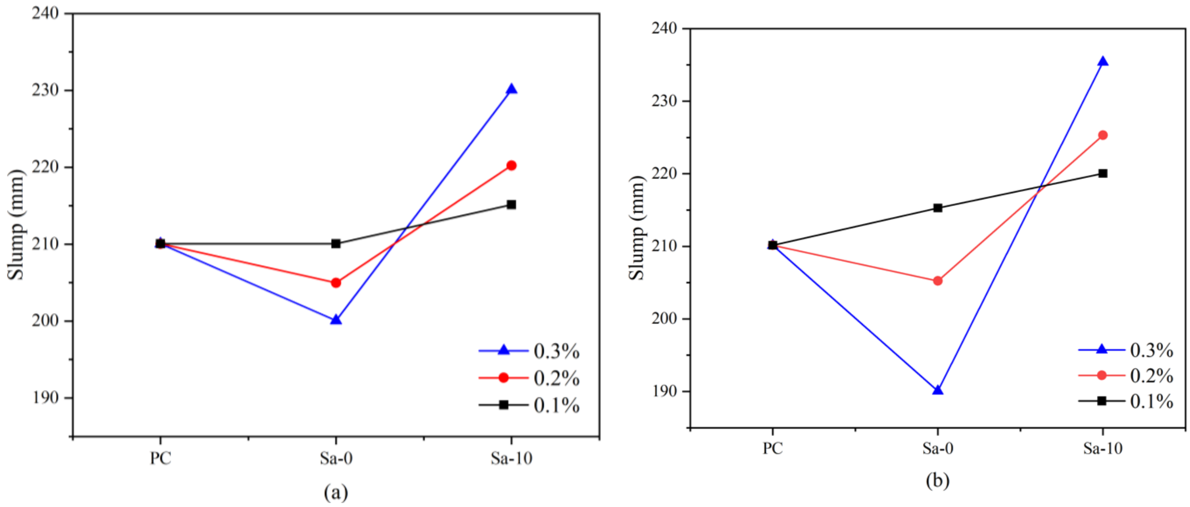
In summary, during the preparation of a concrete mixture, the addition of presoaked SAP is more efficient at enhancing workability compared to the use of dry SAP, whereas the second method can enhance uniform dispersion of the polymer (Mönnig 2015).

Effect of SAP Types and Dosages on Concrete Properties

Workability

When SAP is used in concrete, the effective water demand must be considered because of its effect on mixture workability. Jensen and Hansen (2002) studied a mixture with a water-cement (w/c) ratio of 0.5 and a SAP content of 0.4 wt.%. The results showed that adding SAP to the mixture would reduce the workability if additional water was not added (Schröfl 2012, Snoeck et al. 2014).

The effects of SAP on the workability of concrete correlate with factors such as particle sizes and water absorption rates (depending on whether the SAP is dry or presoaked). Dang et al. (2017) compared the influences of these factors on the workability of concrete, as shown in Figure 4.1. Results showed that, as expected, the use of prewetted SAP caused an increase in concrete slump, while the use of dry SAP resulted in the reduction of concrete workability and a delay of setting time (AzariJafari et al. 2016, Dang et al. 2017, Piérard et al. 2006). AzariJafari et al. (2016) showed that finer SAP could be more effective in improving workability than coarser SAP.



(a)

(b)

Dang et al. 2017

Figure 1.4. Effects of SAP on the slump of concrete for (a) SAP-a and (b) SAP-b, where Sa-0 is dry and Sa-10 is pre-absorbed (particle size: SAP-a > SAP-b)

Although some studies have shown that the cement hydration process influences the water release process of SAP, there is a need to correlate the rate of absorption of SAP with changes in the workability of fresh concrete. The reason for this is that the absorbency rate of SAP can impact fresh concrete's workability. As SAP absorbs water from a mixture, less water is available for workability. In addition, SAP produces gel-like structures that influence water distribution within mixtures, which further impacts workability. Considering specific SAP characteristics and concrete mix design, it is necessary to carry out experimental studies to correlate SAP absorption rate with changes in workability.

Strength

Several studies have investigated the impacts of SAP on concrete mechanical properties, including compressive strength, flexural strength, and splitting strength, but no consistent conclusions have been drawn.

Some researchers have observed that the use of SAP in concrete reduced the mechanical properties of concrete, which is attributed to the voids left by the drying SAP (Shen et al. 2016, Song et al. 2016, Woyciechowski and Kalinowski 2018, Liu et al. 2021, Shen et al. 2020, Lei et al. 2020, Mechtcherine et al. 2014). However, results from other researchers revealed that the mechanical properties of concrete were enhanced by the addition of SAP due to enhanced hydration (Jensen and Hansen 2001, Jensen and Hansen 2002, Piérard et al. 2006, Ghourchian et al. 2013, Liu et al. 2017, Memon et al. 2020, Al Saffar et al. 2019). A summary of the effects of SAP on the mechanical properties of concrete is shown in Table 1.2. It is notable that the mechanical properties of concrete mixtures modified with SAP are also influenced by the curing method (Piérard et al. 2006, Craeye and De Schutter 2009, Igarashi 2006, Lam 2005, Mechtcherine et al. 2009, Larianovsky 2007, Mechtcherine 2006).

Table 1.2. Effect of SAP on the mechanical properties of concrete at different ages

Concrete Type	w/c	Mechanical Property	Strength Values (without SAP) (MPa)			Strength Values (with SAP) (MPa)			Abs. (MPa)			Content of SAP (wt%)	References
			7d	28d	90d	7d	28d	90d	7d	28d	90d		
UHPC	0.15	Compressive strength	155	183	215 (365d)	134	166	186 (365d)	-21	-17	-29	0.206	Justs et al. 2015
			153	184	222 (365d)	122	149	174 (365d)	-31	-35	-48	0.313	
		Flexural strength	22	24	—	19	22	—	-31	-35	-35	0.206	
			21	26	—	16	19	—	-5	-7	—	0.313	
HPC	0.25	Compressive strength	79.14	80.9	75	71.1	80.2	92.9	-8.04	-0.7	17.9	0.18	Savva and Petrou 2018
			79.14	80.9	75	66.5	84.7	78.6	-12.64	3.8	3.6	0.2	
HPC	0.38	Compressive strength	48	59	61	47	56.5	58.5	-1	-2.5	-2.5	0.1	Dang et al. 2017
			48	59	61	46	56	59	-2	-3	-2	0.2	
			48	59	61	45	55.5	58	-3	-3.5	-3	0.3	
Normal concrete	0.45	Compressive strength	13.95	36.9	—	19.52	38.22	—	5.57	1.32	—	0.1	Karthikeyan et al. 2018
			13.95	36.9	—	20.35	38.81	—	6.4	1.91	—	0.2	
			13.95	36.9	—	20.88	40.3	—	6.93	3.4	—	0.3	
			13.95	36.9	—	19.2	39.11	—	5.25	2.21	—	0.4	
		Split tensile strength	19.2	3.75	—	2.63	3.8	—	-16.57	0.05	—	0.1	
			19.2	3.75	—	2.78	3.85	—	-16.42	0.1	—	0.2	
			19.2	3.75	—	2.85	4.2	—	-16.35	0.45	—	0.3	
			19.2	3.75	—	2.69	3.7	—	-16.51	-0.05	—	0.4	
		Flexural strength	2.56	4.25	—	2.93	4.29	—	0.37	0.04	—	0.1	
			2.56	4.25	—	3.15	4.32	—	0.59	0.07	—	0.2	
			2.56	4.25	—	3.19	4.9	—	0.63	0.65		0.3	
			2.56	4.25	—	3.06	4.3	—	0.5	0.05	—	0.4	
Normal concrete	0.5	Compressive strength	45	51	—	43	46	—	-2	-5	—	0.2	Piérard et al. 2006, Hasholt et al. 2010
	0.4		58	69	—	57	66	—	-1	-3	—	0.2	
	0.35		62.5	73	—	65	74.5	—	2.5	1.5	—	0.2	

Concrete Type	w/c	Mechanical Property	Strength Values (without SAP) (MPa)			Strength Values (with SAP) (MPa)			Abs. (MPa)			Content of SAP (wt%)	References
			7d	28d	90d	7d	28d	90d	7d	28d	90d		
Normal concrete	0.35	Compressive strength	13.95	36.9	—	19.52	38.22	—	5.57	1.32	—	0.1	Piérard et al. 2006
			13.95	36.9	—	20.35	38.81	—	6.4	1.91	—	0.2	
			13.95	36.9	—	20.88	40.3	—	6.93	3.4	—	0.3	
			13.95	36.9	—	19.2	39.11	—	5.25	2.21	—	0.4	
		Split tensile strength	2.56	3.75	—	2.63	3.8	—	0.07	0.05	—	0.1	
			2.56	3.75	—	2.78	3.85	—	0.22	0.1	—	0.2	
			2.56	3.75	—	2.85	4.2	—	0.29	0.45	—	0.3	
			2.56	3.75	—	2.69	3.7	—	0.13	-0.05	—	0.4	
		Flexural strength	2.56	4.25	—	2.93	4.29	—	0.37	0.04	—	0.1	
			2.56	4.25	—	3.15	4.32	—	0.59	0.07	—	0.2	
			2.56	4.25	—	3.19	4.9	—	0.63	0.65	—	0.3	
			2.56	4.25	—	3.06	4.3	—	0.5	0.05	—	0.4	
UHPC	0.3	Compressive strength	91 (3d)	120	122	82 (3d)	114	116	-9	-6	-6	0.2	Liu et al. 2021
			91 (3d)	120	122	76 (3d)	106	108	-15	-14	-14	0.4	
			91 (3d)	120	122	62 (3d)	101	105	-29	-19	-17	0.6	
HPC	0.3	Compressive strength	61.77	66.71	—	59.03	62.26	—	-2.74	-4.45	—	0.57	Shen et al. 2020
			61.77	66.71	—	56.06	58.16	—	-5.71	-8.55	—	0.86	
			61.77	66.71	—	42.44	49.28	—	-19.33	-17.43	—	1.14	
		Split tensile strength	4.36	4.74	—	3.95	4.23	—	-0.41	-0.51	—	0.57	
			4.36	4.74	—	3.44	3.78	—	-0.92	-0.96	—	0.86	
			4.36	4.74	—	3.33	3.56	—	-1.03	-1.18	—	1.14	
			97	107	—	76	93	—	-21	-14	—	0.6	

— No test results at corresponding ages were collected by the authors.

It should be noted that achieving a consistent w/cm ratio in a mixture may be challenging when SAP is withdrawing water from the paste while in a fresh state. This may be a cause for the lack of consistent findings in the literature.

The majority of research results show that the incorporation of SAP will decrease the mechanical properties of concrete, especially at early ages. However, these properties can be improved with the use of fibers or additives.

Shrinkage

Mitigating shrinkage is one of the primary benefits of internal curing in concrete mixtures. Volumetric changes in cement paste may be caused by different kinds of shrinkage: chemical/autogenous, plastic, and drying shrinkage.

Chemical/Autogenous Shrinkage

Definitions of autogenous and chemical shrinkage vary. For the purposes of this discussion, autogenous shrinkage is a result of water consumed in hydration leading to drying of the pores in low w/cm ratio (<0.4) mixtures where there is insufficient water to hydrate all of the cement. Internal curing is particularly effective at mitigating this mechanism.

Chemical shrinkage is considered to be the consequence of the volume of hydration products being smaller than the volume of the mixture of ingredients. There is no means of mitigating this.

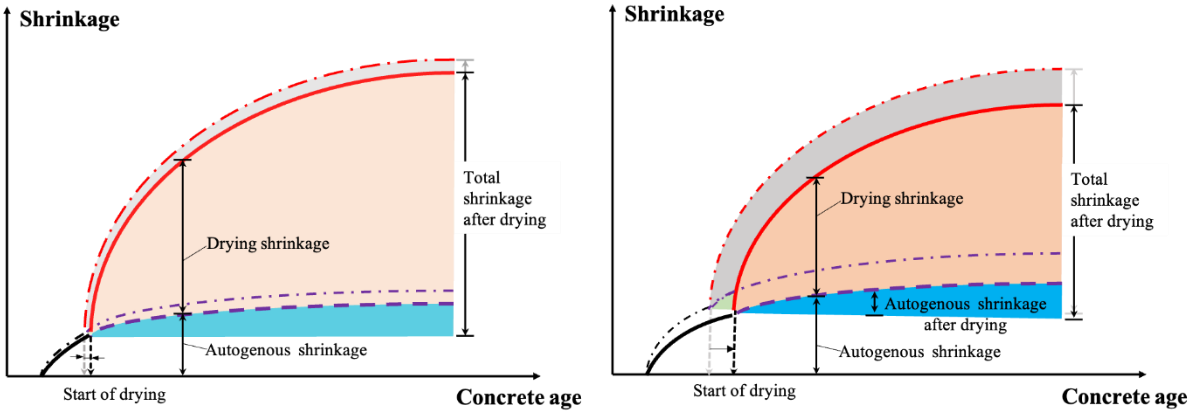
Plastic Shrinkage

Plastic shrinkage develops in the concrete plastic stage and is caused by capillary stresses due to water loss in fresh concrete. It occurs when the surface evaporation rate exceeds the rate at which the bleeding water reaches the surface of the concrete. The increase in capillary pressure and the plastic shrinkage rate leads to tensile stress in the surface layers with reduced moisture, while restraint is provided by the noncontracting inner layers (Sayahi et al. 2014, Saliba et al. 2011, Steyl 2016, Radocea 1992). This stress imbalance leads to the formation of short, shallow cracks that may facilitate the ingress of aggressive solutions (Mindess et al. 2003).

The risk of plastic shrinkage cracking is influenced by environmental humidity, temperature, wind, and surface area. Research has shown that the incorporation of SAP can reduce plastic shrinkage cracks by replacing water lost at the surface with evaporation (Friedrich 2012, Dudziak and Mechtcherine 2010, Rostami and Klemm 2019, Meyer et al. 2020, Cohen and Mobasher 1988).

Drying Shrinkage

Drying shrinkage is caused by water loss from hardened concrete. In terms of magnitude, it contributes the most to the total shrinkage of a system (Cohen and Mobasher 1988, Gribniak et al. 2013) (as shown in Figure 1.5).



Adapted from Gribniak et al. 2013

Figure 1.5. Drying shrinkage and autogenous versus concrete age in conventional concrete (left) and high-performance concrete (right) after internal curing

The drying shrinkage rate and the risk of cracking decrease with internal curing. However, the efficiency of SAP on the drying shrinkage rate depends on the water-to-binder ratio used, the content and type of SAP added, the external environment, and the size of the specimen (Jensen and Hansen 2002, Kong et al. 2015, Igarashi 2006).

Permeability

Porosity, pore size, and pore connectivity control the permeability of concrete (Liu et al. 2019, Liu et al. 2018). Permeability is responsible for the entry of harmful substances like chlorides and carbon dioxide. The modification of concrete with SAP should reduce permeability due to enhanced hydration. However, there are disconnected pores left by the SAP. It has been reported that SAP may swell and fill cracks if water penetrates the system (Hong and Choi 2017, Snoeck et al. 2018). For example, Snoeck et al. (2018) used neutron radiography to observe healed cracks to show that SAP decreased the permeability of healed cracks up to 100 µm wide.

Several papers have reported the influence of SAP on the permeability of concrete, as shown in Table 1.3. The effect of the incorporation of SAP on the resulting permeability of concrete has been attributed to SAP’s ability to improve the pore size distribution, facilitate the particle packing density, and enhance the interparticle bonding, leading to lower porosity and better strength and durability. In summary, the efficacy of SAP in decreasing the permeability of concrete is multifaceted. The outcome of experiments can vary due to different conditions, such as curing environments, concrete age, and SAP particle size. Furthermore, the level of permeability reduction depends on the extent to which the concrete matrix has internally cured, which in turn depends on the availability of water. Therefore, it is crucial for further research to customize SAP applications based on specific project requirements, such as water content, curing conditions, and SAP particle size, in order to achieve the desired reduction in concrete permeability. Additionally, combining SAP with other green cement-based materials like slag can further improve its effects on concrete properties.

Table 1.3. Effect of SAP on the permeability of concrete

Evaluation Indexes	Changes Compared to the Reference without SAP	SAP Contents	References
Water permeability	Same or decrease	0.33%, 0.58%, and 0.25%	Reinhardt and Assmann 2012, Reinhardt and Assman 2009
Oxygen permeability	Same or decrease		
Capillary suction	Decrease		
Chloride migration coefficient	Decrease	0.05%, 0.1%, 0.2%, and 0.3%	Hasholt and Jensen 2015
Chloride migration coefficient	Decrease	0.3%	Ouattara Coumoin et al. 2019
Carbonation depth Chloride migration coefficient	Decrease	0.1%, 0.2%, and 0.3%	Dang et al. 2017
Water absorption	Decrease	0.3%, 0.6%, and 0.9%	Ma et al. 2017
Chloride ion diffusion coefficient	Decrease		
Carbonation depth	Decrease		
Gaseous and fluid permeability	Decrease	—	Gräf and Düsseldorf 1986

Air Void Systems

The presence of a stable, adequate air void system is required for concrete exposed to freeze-thaw cycles. Chemical air-entraining agents (AEAs) are used to stabilize small air voids in a concrete mixture that act as pressure relief zones for water that moves and expands as it freezes (Dodson 1990). However, air content created by AEAs is variable with changes in the temperature, haul time, and vibration of the fresh mixture.

SAP has the potential to act as a physical air void after desorption (Jensen and Hansen 2001, Bentz and Jensen 2004). Caveats are that the spacing between voids may be higher than desired and that voids may become filled with hydration products (Justs et al. 2015, Xiangmin 2013, Justs et al. 2014, Sarbapalli et al. 2016, Esteves et al. 2014, Ma et al. 2015). SAP with an appropriate particle size has contributed to the air void system (Riyazi et al. 2017, Falikman 2020). Moreover, Mechtcherine et al. (2017) compared the effect of traditional AEA and SAP on the air void system of concrete. The results revealed that SAP created a more stable air void system in concrete compared to AEA, whereas it reduced the adverse effect of additional water on the mechanical performance of concrete compared to traditional AEA.

Specifications

Several methods have been recommended by the American Concrete Institute (ACI) to mitigate early-age shrinkage cracking. These can be divided into three categories (ACI 305R 2010). From the perspective of structures, the tensile strength of hardened concrete can be enhanced by adding reinforcing steel or fibers. From the perspective of materials, the consumption of water can be compensated by some internal curing using LWA or SAPs. From the point of view of construction, external curing methods can be adopted to minimize the moisture loss of concrete, such as water spraying, coverings, and membrane-forming curing compounds.

Internal curing in cement-based composites is regarded as an effective method of improving the performance of mixtures with a low w/cm ratio, which is particularly significant for the shrinkage properties of concrete (Bentz and Weiss 2011, ACI Committee 231 2010, ACI SP-256 2008, RILEM Technical Committee TC 196-ICC 2007). As an internal curing material, LWA is divided into LWFA and lightweight coarse aggregate (LWCA). However, LWFA is preferable to LWCA due to its more uniform distribution in the matrix and relatively lower cost and the availability of existing standards such as ASTM C1761 (2017). The dosage, preconditioning methods, mixing procedures, and other requirements are discussed in various specifications (Northeast Solite n.d., ESCSI n.d., NYSDOT n.d., INDOT 2015, Weiss and Montanari 2017). It is also necessary to measure the properties of LWFA, such as total, absorbed, and surface moisture, prior to use. Moreover, the amount of LWFA needed to develop an internally cured mixture is determined during mix design with various tools (Weiss and Montanari 2017, Mignon et al. 2017). In summary, research, design, guidelines, and implementation of LWFA for internal curing have proven to be feasible and effective.

SAP is promising for use as an internal curing material but has not found common use in the United States. SAP can be included in the group of concrete admixtures according to EN 206, since its dosage is 0.3% to 0.6% of the binder mass (Mignon et al. 2017). Introducing SAP into a concrete mixture affects the absorption of a portion of the mixing water in a way that dramatically exceeds the mass and volume of the added SAP. Therefore, SAP dosage should be based on the absorption rates of the product. A test method, referred to as the tea bag method, has been proposed that determines the free swell capacity of SAP powders in saline or other aqueous solutions (Weiss and Montanari 2017, Technical Committee ISO/TC 173 Subcommittee SC 3 2020). This method also notes that the paste volume, including SAP, shall be no more than 26.5% of the concrete design volume and specifies the maximum w/cm ratio (0.42) in concrete mixture design during construction.

A state-of-the-art report from RILEM Technical Committee 225-SAP published in 2012 summarized scientific knowledge and the potential application of SAP in concrete mixtures, with a focus on the improvement of the autogenous shrinkage and the freeze-thaw resistance of concrete (Mechtcherine and Reinhardt 2012). Subsequently, RILEM Technical Committee 225-

RSC, started in 2014, primarily facilitated the incorporation of SAP into construction practice by preparing technical recommendations for its use in concrete (Mechtcherine and Schroefl 2014).

Case Studies on the Use of SAP in Construction Projects

The following sections present case studies that describe some structures modified with SAP in order to achieve improved mixture performance.

Reduced Shrinkage Cracking

2006 FIFA World Cup Stadium in Kaiserslautern, Germany

The first construction project in the world that used SAP in a concrete structure was a pavilion built for the 2006 FIFA World Cup in Kaiserslautern, Germany (Figure 1.6). The pavilion was designed as a filigreed, thin-walled high-performance concrete structure with thin columns and no conventional reinforcement. Concrete performance parameters, including reduced autogenous shrinkage, high durability, enhanced ductility, and self-desiccation, were developed (Mechtcherine 2006).

Mechtcherine 2006

Figure 1.6. FIFA World Cup Pavilion in Kaiserslautern, Germany

China

Some large-scale structures using concrete modified with SAP as an internal curing agent have been constructed in China. A second double-lane of the Lanzhou-Urumgi railway was built using

1,050,000 yd^3 of bed slab concrete designed with a w/cm ratio of 0.37 in a windy and arid environment (Zhu et al. 2014). SAP was incorporated along with shrinkage-reducing admixtures to reduce the risk of moisture loss due to evaporation, thus reducing the risk of cracking due to plastic shrinkage (Liu et al. 2017, Mechtcherine 2016).

A 65 ft high by 235 ft long by 3 ft wide shear wall was built containing a novel kind of SAP (Liu et al. 2017). Field tests showed that no cracks formed in the SAP section for up to seven days, while cracks were observed in the concrete containing only an expansion agent.

SAP used as a type of chemical admixture was also applied in the construction of the China Zun tower (Liu et al. 2019, The Tower Info 2017). The results showed that when the SAP content was 0.56%, the shrinkage rate decreased by 46%, and the later compressive strength was not affected.

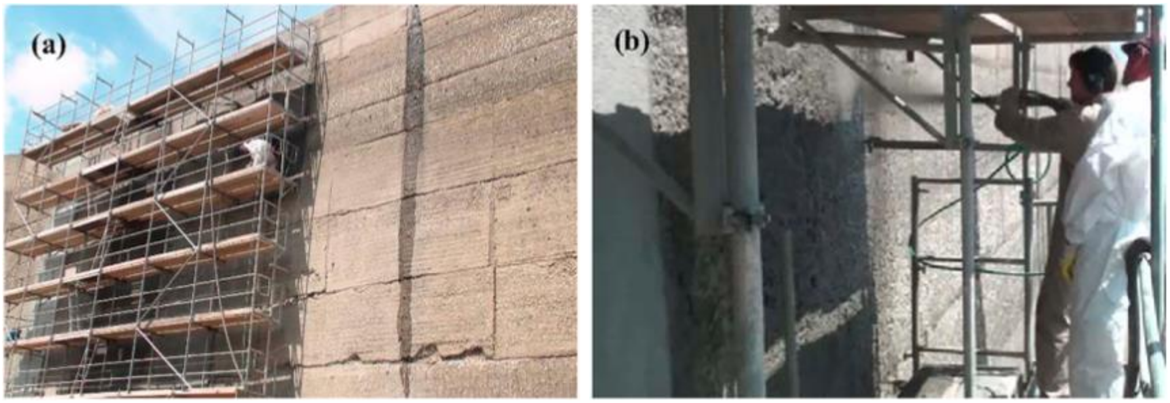
The Tower Info 2017

Figure 1.7. China Zun tower under construction

Freeze-Thaw Resistance of Concrete

The use of SAP can increase the freeze-thaw resistance of a mixture. This is attributed to the capabilities of SAP to absorb and retain water, which ensures that concrete does not dry out and crack during freeze-thaw cycles. Additionally, SAP can reduce concrete permeability significantly. Because SAP reduces the number of microcracks, water encounters more resistance when penetrating deeper into concrete during freeze-thaw cycles. In this way, repeated freezing and thawing cycles serve as a protective barrier against further damage. Furthermore, SAP enhances concrete's overall durability and resistance to freeze-thaw damage by refining its microstructure within cement paste.

The ability of SAP to improve the frost resistance of highly ductile short-fiber reinforced concrete (specifically, strain-hardening cement-based composite [SHCC]) was investigated by researchers at Technische Universität Dresden. In the summer of 2011, SAP was used as an additive to increase the freeze-thaw resistance of SHCC during repairs to the upper water reservoir of the Hohenwarte II pumping hydraulic power station in Thüringen, Germany (as shown in Figure 1.8). The risk of freezing damage to the reservoir walls was high due to their saturation and the variation in water levels. SAP was utilized as an additive to increase the frost resistance of the repair layer (Mechtcherine 2016, Brüdern and Mechtcherine 2010).



Mechtcherine 2016

Figure 1.8. (a) Condition of the upper water reservoir of the Hohenwarte II pumping hydraulic power station in Thüringen, Germany, before repair and (b) spray application of SHCC containing SAP as repair material

Improved Rheology

A 1991 patent application by Snashall (1991) explored the use of SAP to increase viscosity and decrease the rebound of shotcrete. The recommendation was to premix SAP with the aggregate and 10% to 15% of the aggregate's weight of water, let the mixture stand in the mixer for 10 minutes, and finally incorporate the cement and the rest of the mix water. According to a patent application by Jensen (2001), SAP can be added into a mixture in two ways: one is to add dry SAP to the nozzle to reduce the viscosity of a wet mixture, which requires SAP to absorb water quickly to achieve the desired viscosity before the shotcrete hits the wall; the other is to incorporate the presoaked or partially presoaked SAP into the concrete for internal curing purposes.

For example, SAP-modified concrete was applied to shotcrete wall panels for a construction project in in Lyngby, Denmark, as shown in Figure 1.9. In this case, the presence of 0.4% SAP in shotcrete with a w/c ratio of 0.4 acted as a rheology modifier because no other set-accelerating admixture was adopted in this construction project.

Jensen 2008

Figure 1.9. Shotcreting of wall panels with SAP-modified concrete in Lyngby, Denmark

Self-Sealing

When SAP comes into contact with water, it absorbs water and expands to form a flow barrier. Based on this mechanism, sealing composite materials were made by mixing modified SAP with rubber, as described by Tsubakimoto et al. (1987). These composite materials have been used to seal the joints of various building materials. They may be used like mortar to plug any gaps left by settlement during or after construction (Shimomura and Namba 1994). As reported by Buchholz and Graham (1998), this composite material was employed in the construction of the Channel Tunnel between France and England.

PHASE 1 – PASTE TESTING

An initial round of testing was conducted on pastes to assess which of eight SAP products showed the most promise for enhancing the properties of concrete.

Materials

Cement

Type II ordinary portland cement (OPC) was used as the cementitious material. The specific gravity of cement is 3.15 g/cm^3. The chemical composition, determined by X-ray fluorescence (XRF) spectrometry, and the physical properties are listed in Table 2.1.

Table 2.1. Chemical composition of cement from XRF test

Chemical Compositions	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O eq.	LOI	Specific Gravity	Blaine Fineness (m^2/kg)
OPC, %	23.16	4.52	4.65	63.17	2.31	0.59	0.23	0.46	0.89	3.15	385.2

Superabsorbent Polymers

Eight kinds of SAPs were initially investigated as internal curing materials in cementitious mixtures. The SAPs were as follows:

- Hydromax
- PX-1A-AW
- PX-1A-PS
- Waste Lock Type S
- Waste Lock 770
- BASF
- PX-1A-TA
- Pervious Enhancer Pro Zappatec

Their particle size distribution was measured by laser diffraction using the dry diffusion method, as shown in Figure 2.1.

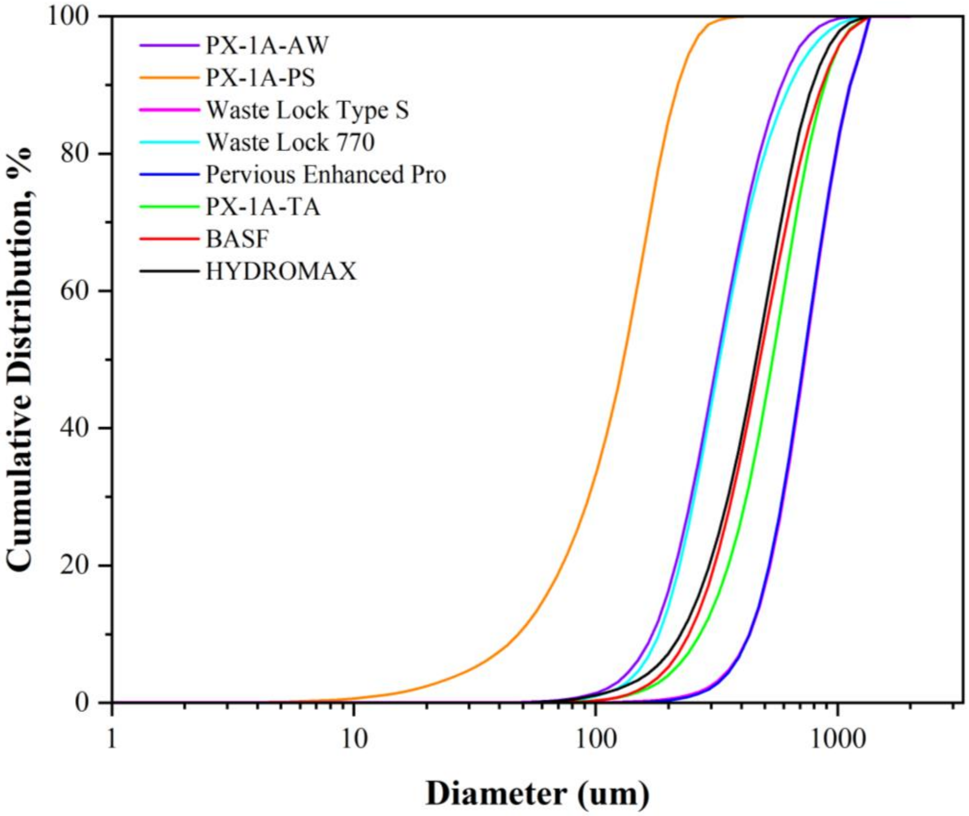


Figure 2.1. Particle size distribution of SAPs

Figure 2.2 illustrates one product in a dry state and after immersion in water. Dry SAPs were observed with a scanning electronic microscope (SEM), as shown in Figure 2.3. The images indicate that the majority of commercial SAPs used in this research have an irregular shape.

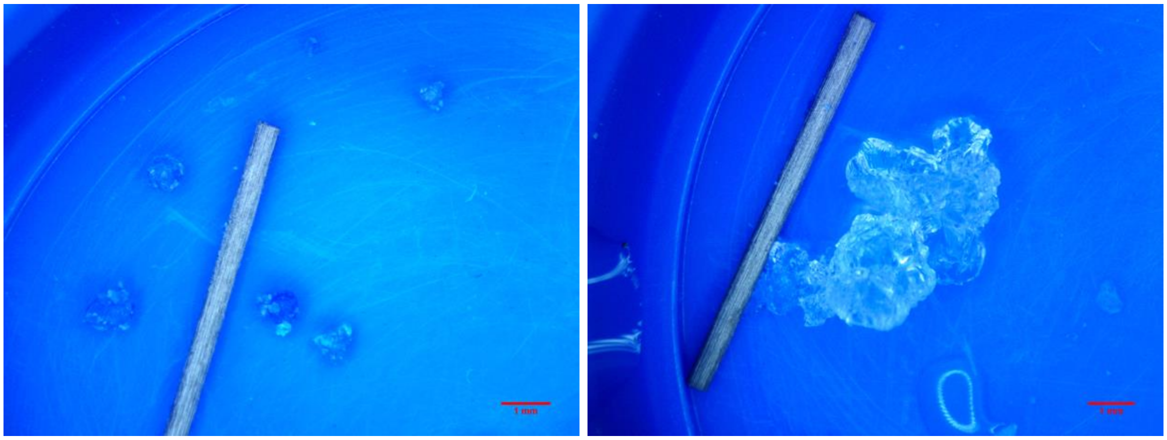


Figure 2.2. Micrographs of dry (left) and swollen (right) SAPs taken with an optical microscope (with 0.5 mm pencil lead as reference)

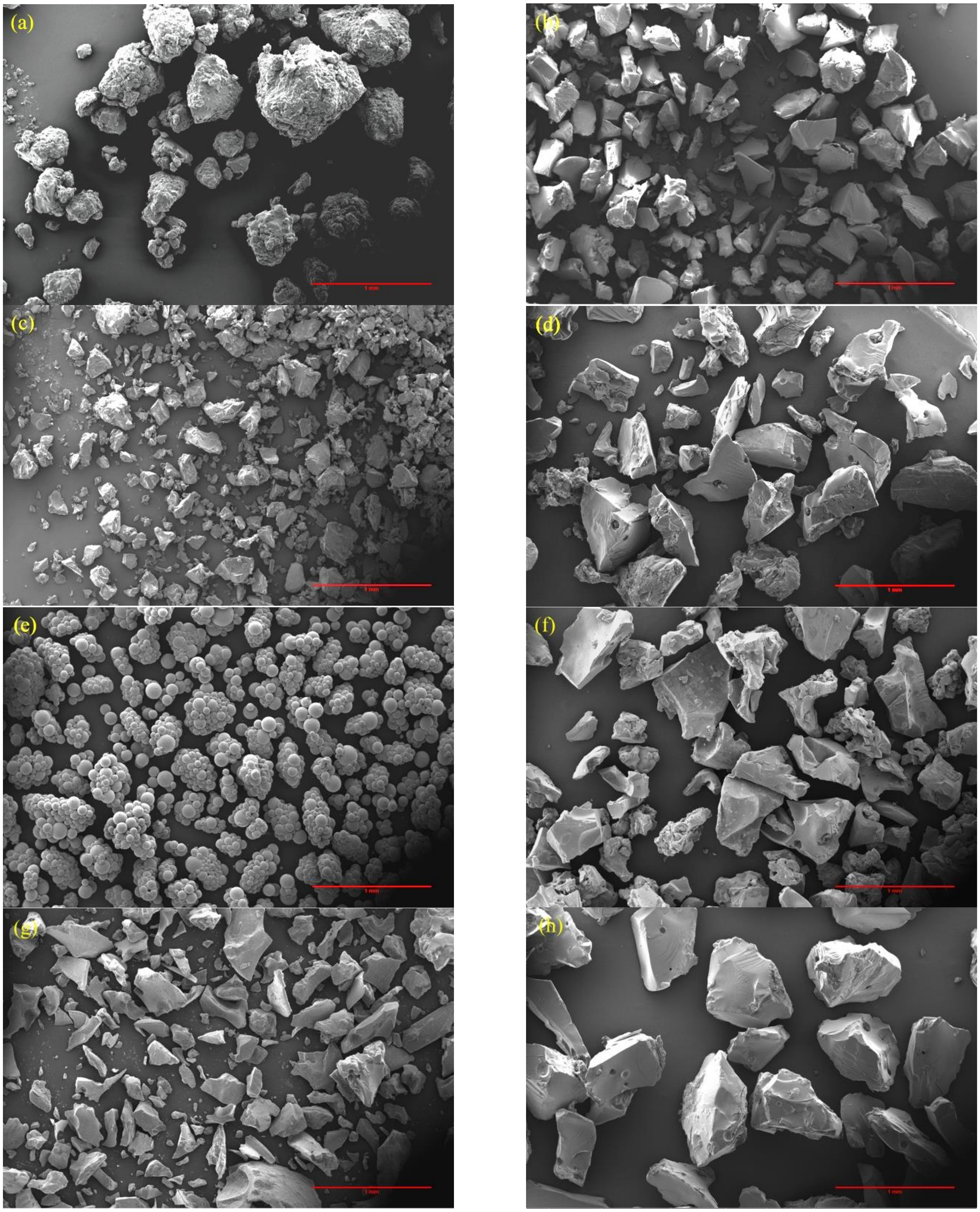


Figure 2.3. SEM images of SAPs: (a) Hydromax, (b) PX-1A-AW, (c) PX-1A-PS, (d) Waste Lock Type S, (e) Waste Lock 770, (f) BASF, (g) PX-1A-TA, and (h) Pervious Enhancer Pro Zappatec

Mixtures

All cement specimens for the paste experiments had the same w/c ratio as the corresponding concrete mixtures (0.42). The SAP content in the mixtures, except for the control group, was 0.4% by weight of cement. The proportions for the cement paste mixes used in the thermogravimetric analysis (TGA) test are shown in Table 2.2.

Table 2.2. Cement paste mixture design proportions for TGA test

Mixture	w/c	Cement (g)	Water (g)	SAP (g)
Control	0.42	900	378	-
Hydromax				3.6
BASF				3.6
PX-1A-TA				3.6

Experimental Methods

Cement Pore Solution Preparation

To conduct absorption and desorption tests of SAPs, tap water and a filtrate solution of paste were prepared as the test fluids. For the preparation of the filtered solution, a cement paste with a w/c ratio of 5 was mixed. After 3 hours, the initial fresh paste solution was filtered through a negative pressure filter setup. The resulting filtered solution was then transferred into a polypropylene bottle with a lid to prevent carbonation.

Absorption

The absorption capacities for the SAPs were determined using the tea bag method, and the liquids used were tap water and a cement paste with a w/c ratio of 5. The amount of fluid absorbed by the SAP was calculated based on equation (4):

$$m_{H_2O} = \frac{m_3 - m_2 - m_1}{m_1} \tag{4}$$

where m_{H2o} is the amount of water absorbed per gram of SAP at a certain moment (g), m_1 is the mass of dry SAP (g), m_2 is the mass of the tea bag saturated with a dry surface (g), and m_3 is the mass of the tea bag and SAP at a specific time (g).

Desorption

The desorption of SAPs with the filtrated solution was measured based on ASTM C1761/C1761M.

Results

Absorption

The results of SAP absorption (grams of fluid per gram of dry weight of SAP) over time in tap water and cement filtration solution (water:cement = 5:1) are shown in Figure 2.4 and Figure 2.5. As expected, the absorption rate of SAP in tap water was greater than in cement pore solution.

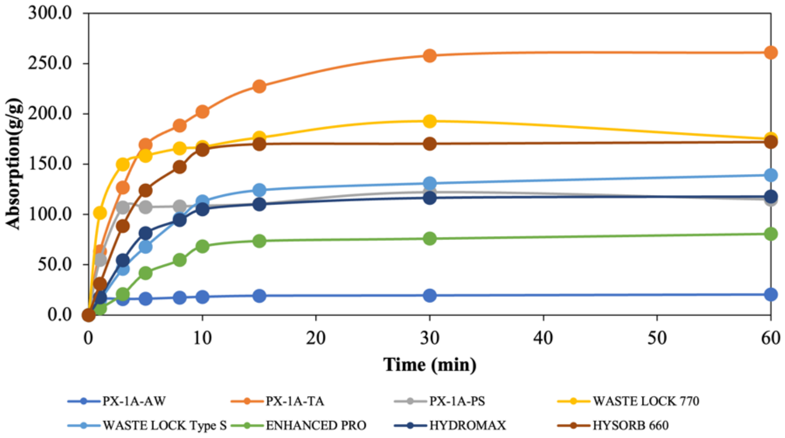


Figure 2.4. Absorption of various SAPs in tap water

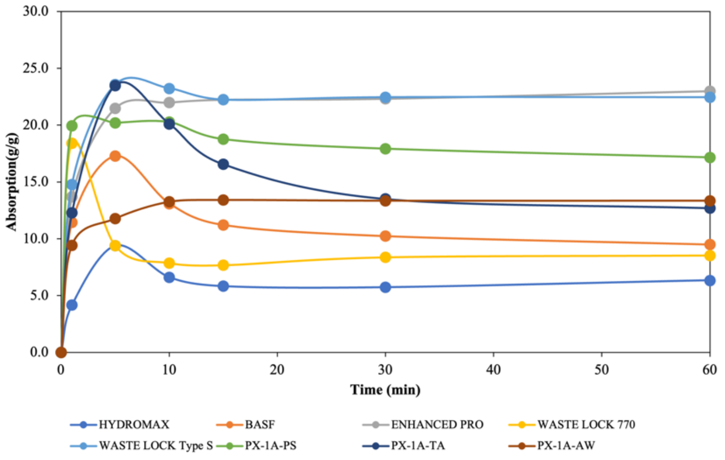


Figure 2.5. Absorption of various SAPs in cement pore solution

Desorption

The desorption of SAPs with the filtered pore solution is shown in Figure 2.6.

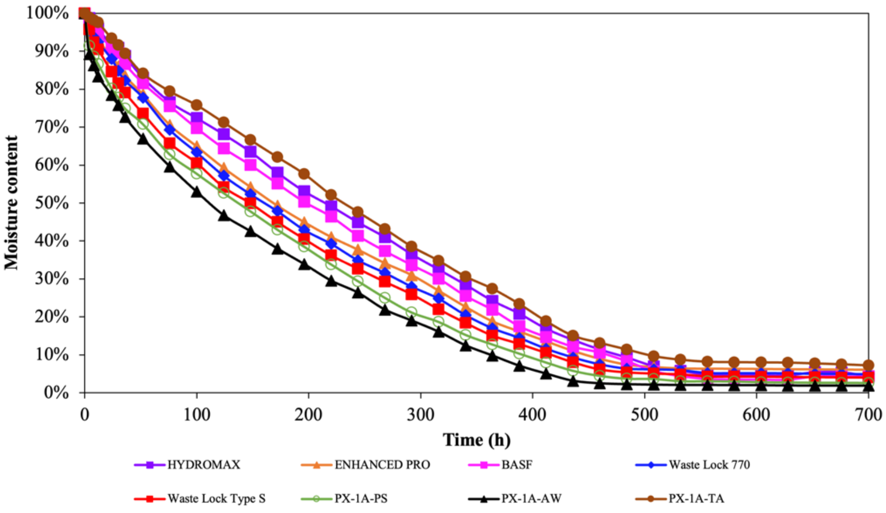


Figure 2.6. Desorption of different SAPs saturated with filtered pore solution

Discussion

The absorption behavior of the SAPs in tap water and cement filtrate solution is presented in Figure 2.4 and Figure 2.5. Firstly, the absorptivity determined in both liquids was different. The absorptivity measured in the filtered solution was less than that in tap water because the alkaline properties of the cement-filtered solution reduced the osmotic pressure responsible for the SAPs' fluid uptake under load-free conditions. Secondly, an increase in the absorptivity of the SAPs was observed within the first 15 minutes of immersion in tap water. After this time, the absorbance reached a plateau. However, the absorptivity in the cement filtrate solution initially increased and gradually decreased until it reached a constant value over time. The reduction in absorptivity is due to the interaction between the anionic carboxylate groups of the SAPs and the Ca^{2+} ions in the pore solution, which ultimately causes the polymer chains to lose their charge. Waste Lock 770 exhibited the highest initial absorption rate in both solutions, probably attributed to properties such as higher molecular weight and larger particle size.

The desorption behavior of SAPs over time was also assessed and is shown in Figure 2.6. The y-axis represents the normalized moisture content of the SAPs based on their initial and remaining water content during the drying process. In the first 100 minutes, the relationship between the amount of water lost and the elapsed time is almost linear. This stage represents water loss from the SAP surfaces due to weak van der Waals forces. The desorption curve flatlined later in the drying period because the bound water close to the cores of the SAP particles or the water trapped by the hydrogen was difficult to lose. The desorption rate for PX-1A-AW is the highest, while that for PX-1A-TA is the lowest.

Optimizing SAP type and dosage may be achieved by balancing water absorption and desorption behaviors with their effects on mechanical and durability properties. Therefore, the initial absorption rate, absorption volume, desorption mass, and particle size in this research were used as tools to select SAPs as internal curing materials for further testing in concrete.

For efficient internal curing, it is crucial to choose SAPs with a sufficient desorption capacity. At the same time, choosing SAPs with the appropriate particle size distribution is desired for effective internal curing because it improves the uniform dispersion of water throughout the concrete mix. The evaluation of the SAPs based on these factors is shown in Table 2.3.

Table 2.3. Evaluation of SAPs used as internal curing materials

	Initial Absorption Rate (Min)	**Absorption Volume (Min)**	**Desorption Mass (Min)**	**D50 (mm, Max)**
Preset Value	100	120	10	0.6
Hydromax	✕	✕	✓	✓
ENHANCED PRO	✕	✓	✓	✕
BASF	✓	✓	✓	✓
WASTE LOCK 770	✓	✕	✕	✓
WASTE LOCK TYPE S	✕	✕	✕	✕
PX-1A-PS	✕	✕	✓	✓
PX-1A-AW	✕	✕	✕	✓
PX-1A-TA	✓	✓	✓	✕

✕ and ✓ indicate whether the SAP sample fails to meet or meets the target requirement, respectively.

Based on the evaluation shown in Table 2.3, Hydromax, BASF, and PX-1A-TA were chosen as internal curing materials for further testing in Phase 2.

PHASE 2 – CONCRETE TESTING

Materials

The same cement used in the Phase I testing and the three SAP materials highlighted in Table 2.3 were used in Phase 2, along with the aggregate and chemical admixture products described in the following sections.

Aggregates

The coarse aggregate used was crushed limestone from Martin Marietta in Ames, Iowa, with a nominal maximum size of 1 in. The river sand used was fine aggregate provided by Hallett Materials in Ames, Iowa. The basic physical properties of the coarse aggregate and sand, such as gradation, absorption, specific gravity, moisture content, dry-rodded unit weight (DRUW), and fineness modulus, were determined according to the relevant ASTM standards. These parameters except for gradation are listed in Table 3.1.

Table 3.1. Properties of aggregates

Materials	Specific Gravity	Absorption, %	Moisture Content, %	DRUW, lb/ft^3	Fineness Modulus
Limestone Coarse Aggregate	2.7	0.62	0.04	92	-
River Sand	2.63	1.61	0.76	-	2.99

The sieve analysis for the gradation of coarse aggregate and sand in a 1:1 ratio is shown in Table 3.2 and Figure 3.1.

Table 3.2. Particle size distribution of coarse aggregate and sand

Change the Aggregate Gradation	**1 in. Aggregate (Limestone)**		**River Sand**		**Combination Gradation**	
Sieve Size	**% Retained**	**% Passing**	**% Retained**	**% Passing**	**% Retained**	**% Passing**
#200	0.11%	0.00%	1.80%	0.20%	0.96%	0.10%
#100	0.01%	0.11%	6.00%	2.00%	3.00%	1.06%
#50	0.00%	0.12%	37.00%	8.00%	18.50%	4.06%
#30	0.01%	0.13%	23.00%	45.00%	11.50%	22.56%
#16	0.01%	0.13%	16.00%	68.00%	8.00%	34.07%
#8	0.29%	0.14%	8.00%	84.00%	4.14%	42.07%
#4	11.99%	0.43%	8.00%	92.00%	9.99%	46.21%
0.375 in.	23.21%	12.42%	0.00%	100.00%	11.61%	56.21%
0.5 in.	39.01%	35.63%	0.00%	100.00%	19.51%	67.81%
0.75 in.	24.66%	74.64%	0.00%	100.00%	12.33%	87.32%
1 in.	0.70%	99.30%	0.00%	100.00%	0.35%	99.65%
1.5 in.	0.00%	100.00%	0.00%	100.00%	0.00%	100.00%
Coarse Sand % (#8 to #30) =					23.65%	**YES**
This range amount is a minimum of 15%.						
Fine Sand % (#30 to #200) =					33.97%	
This allowable range for slipform paving is between 24% and 34%.						**YES**
This allowable range for pumping is between 25% and 40%.						**YES**

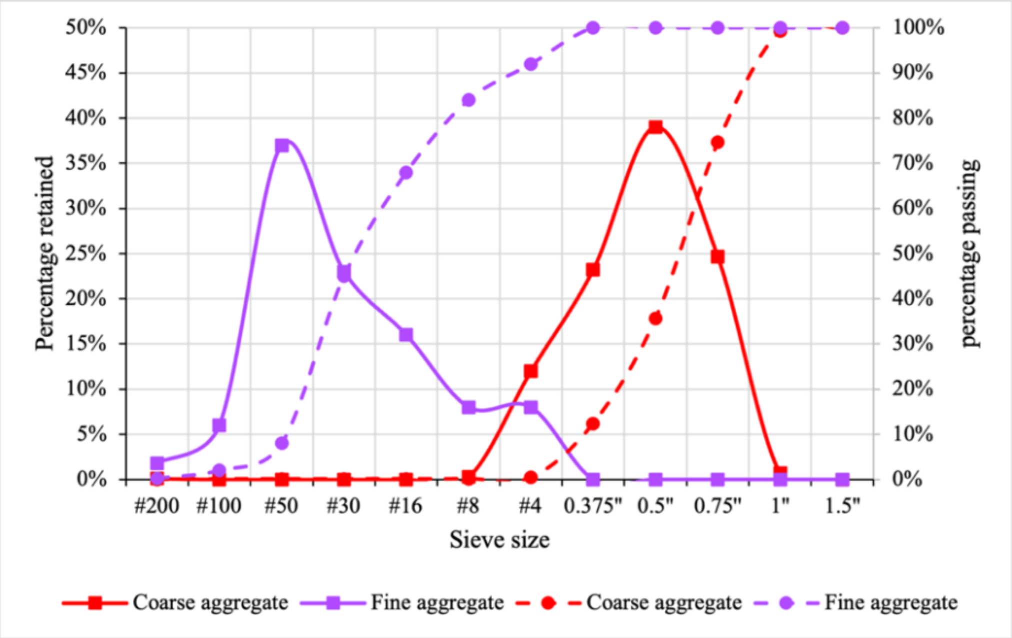


Figure 3.1. Aggregate gradations

The combined gradation curve of the coarse aggregate and sand was developed, and it fell within the Tarantula Curves, as shown in Figure 3.2.

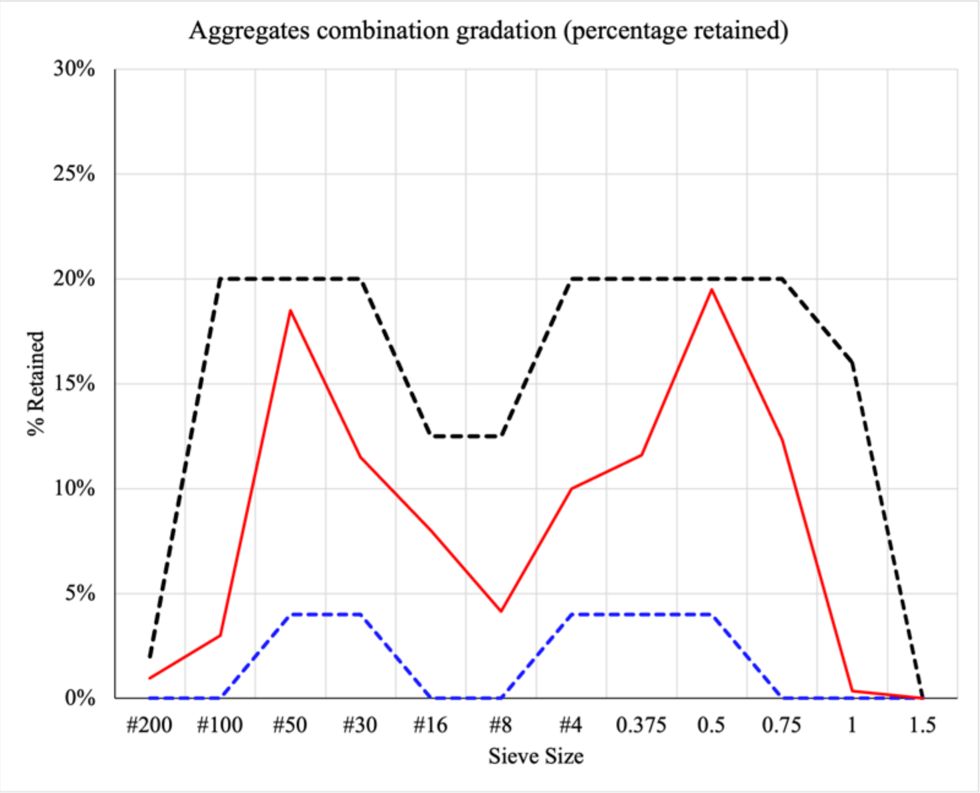


Figure 3.2. Tarantula curve of aggregates

Chemical Admixtures

A high-range water reducer (HRWR), MasterGlenium 7920 (US), was used in concrete. The dosage was varied to obtain an initial slump of 1.5 to 2.5 in. An air-entraining admixture, MasterAir AE90, was utilized to achieve the target air volume of 5% ±1% in the concrete.

Mixtures and Samples

Paste mixtures were based on ratios similar to those used in Phase 1.

Concrete Mix Proportions

The target w/c ratio of the mixtures was 0.42. The dosage of SAP added to the concrete mixtures was determined based on the method shown in equation (5) (Bentz and Snyder 1999).

$$M_{SAP} = \frac{C_f CS \alpha_{max}}{\Phi_{SAP,30} W_{sap}} \tag{5}$$

where M_{SAP} is the mass of the dry SAP (kg/m^3), C_f is the cement content of the mixture (kg/m^3), CS is the chemical shrinkage of the cement (mL water/g cement), α_{max} is the expected maximum degree of hydration (0 to 1) (unitless), Φ_{SAP} is the absorption of the SAP in pore solution, here at 30 minutes absorption (kg of solution/kg of dry SAP), and W_{sap} is the desorption rate of SAP upon equilibrium at 94% RH expressed as a fraction of oven-dry mass. The values of CS and α_{max} in this research were set as 0.07 and 0.91, respectively.

Therefore, combined with the measured results of absorption and desorption, the required amount of SAP in concrete was calculated as 7%, 4%, and 3% for Hydromax, BASF, and PX-1A-TA, respectively.

To evaluate the effects of using SAP on the workability and other properties of concrete mixture designs, three approaches can be used for the modification of concrete mixture designs: Approach I, which adds water only to offset SAP absorption without using water-reducing admixture (WRA); Approach II, in which only WRA is used; and Approach III, which adds both water and WRA. In this study, Approach III was utilized to achieve the target slump. The mixture proportions are shown in Table 3.3.

Table 3.3. Concrete mixture proportions

	Materials (lb/yd^3)							
Items	**Cement**	**Sand**	**Coarse Agg.**	**Water**	**Extra Water (wt.%)**	**AEA (wt.%)**	**SAP (wt. %)**	**WRA (wt. %)**
Control	593	1518	1532	249.06	0	0.05%	0	0.4%
Hydromax					7%		0.7%	0.5%
BASF					7%		0.4%	0.4%
PX-1A-TA					7%		0.3%	0.35%

Items	**Cement**	**Sand**	**Coarse Agg.**	**Water**	**Extra Water for Preparing the Presoaked SAP (wt.%)**	**AEA (wt.%)**	**SAP (wt. %)**	**WRA (wt. %)**
Presoaked-Hydromax	593	1518	1532	249.06	7%	0.05%	0.7%	0.4%
Presoaked-BASF					7%		0.4%	0.3%
Presoaked- PX-1A-TA					7%		0.3%	0.25%

Furthermore, concrete with presoaked SAP was prepared to compare its mechanical properties against those of concrete with dried SAP. The presoaked SAP included 7% water by mass of cement. The dosage of the WRA was adjusted to achieve the desired slump. Specifically, 0.4%, 0.3%, and 0.25% of WRA by weight of cement was required for concrete with Hydromax, BASF, and PX-1A-TA, respectively.

Mixing Procedure

The concrete mixtures were prepared in the laboratory using a drum mixer with a capacity of 3.5 ft^3 according to the following steps:

1. Add coarse aggregate and half of the batch water with AEA and mix for 1 minute to build air voids.
2. Add fine aggregate and mix for 1 minute.
3. Add cement (this is the initialization point for hydration) and mix for 30 seconds.
4. Add the remaining half of the batch water gradually while maintaining mixing for 2.5 minutes (add some WRA to the water if applicable).
5. Allow the concrete to rest for 3 minutes.
6. Mix for 2 minutes and verify the slump and air content if needed.
7. Remix for 3 minutes (add WRA or AEA to adjust slump or air content if needed).

All samples were covered with plastic sheets after casting. After staying for 24 hours in the laboratory, all specimens were then demolded and cured in the moist-cured room at 72°F and >95% RH.

Experimental Methods

The various test methods used in Phase 2 are discussed below.

Calorimetry

In order to investigate the influence of SAP on the cement hydration process, the heat flow and rate of heat generation of cement paste were determined during the first 7 days of hydration at a constant temperature of 20°C in an isothermal calorimeter (TAM-air).

The heat flow measured was analyzed based on the four phases proposed by Bullard et al. (2011): (I) initial formation, (II) period of slow reaction (dormant period), (III) acceleration period, and (IV) deceleration period.

Thermogravimetric Analysis

TGA analysis was also conducted on hardened paste samples to detect the various hydrate phases. Powder samples processed from the cement paste after curing for 7 days were dried at 50°C before testing. The testing temperature ranged from 35°C to 800°C, with the heat rate set at 10°C/minute under air in a Netzsch STA 449 F1 Jupiter device (Figure 3.3). The amount of mass loss/water release, denoted as H (g/100 g cement), was determined at the specific temperature intervals according to the equation (6), which was proposed by Beuntner and Thienel (2022).

$$H\ or\ CO_2 = \frac{\Delta m_{T_1-T_2}}{f_c * m_s} \tag{6}$$

where $\Delta m_{T_1-T_2}$ is the difference in mass between two temperature stages, f_c indicates the cement content in the sample being analyzed, and m_s represents the mass of the testing sample.

The evaluation of mass loss was partitioned into the following discrete temperature intervals:

- H_I (T = 20°C to 140°C): C–S–H and AFt (Bremmer and Ries 2009)
- H_{IIa} (T = 140°C to 320°C): AFm phases (AFm-Hc, Mc, Ms (De La Varga et al. 2012) and C_2AH_8 (Van et al. 2014)
- H_{IIb} (T = 320°C to 400°C): AFm phases (Henkensiefken et al. 2009), C_3AH_6 and C_4AHx (Van et al. 2014)
- Portlandite (CH) (T = 450°C to 530°C) (Van et al. 2014) according to the Marsh and Day (1988) method (Justs et al. 2015)

Figure 3.3. TGA test equipment

Fresh Concrete Properties

The following fresh concrete properties were determined: slump, setting time (see Figure 3.4b), air content, unit weight, and VKelly index (as shown in Figure 3.4a) according to ASTM C143/C143M, ASTM C231/C231M, ASTM C138/C138M-13, and AASHTO T403, respectively.

Figure 3.4. Test setup for fresh concrete: (a) VKelly, (b) penetrometer device

Compressive Strength

Cylindrical specimens with a size of 4 by 8 in. were prepared for compressive strength testing at 3, 7, 28, and 56 days following ASTM C39, as shown in Figure 3.5.

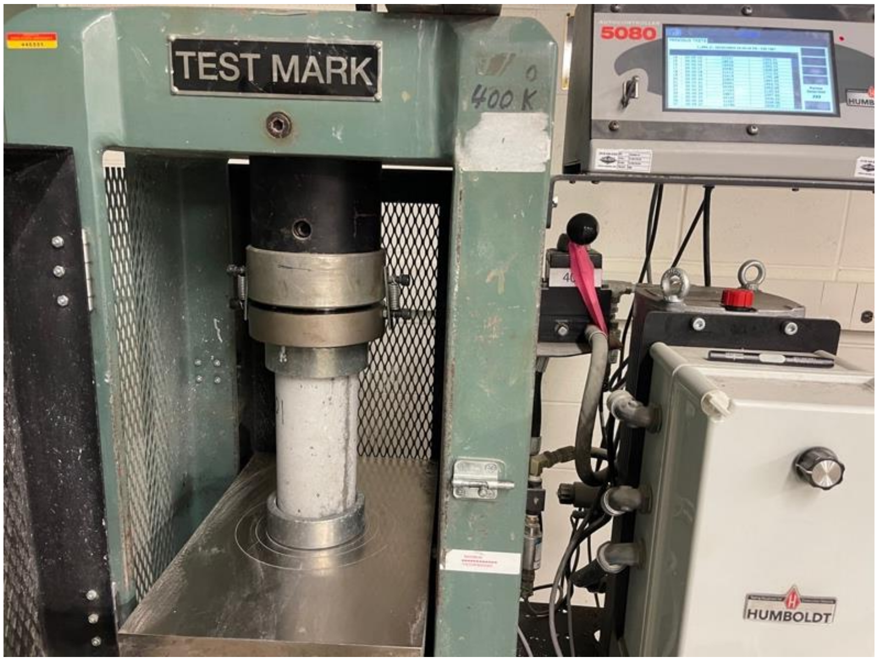


Figure 3.5. Test setup for concrete compressive strength

Resistivity

The bulk and surface electrical resistivity tests, used to evaluate permeability, were conducted at 3, 7, 28, and 56 days. These tests correspond to the direct two-electrode method and four-point Wenner probe method, conducted in compliance with ASTM C1760 and AASHTO 95-11, respectively. The electrical resistivity was calculated using equation (7):

$$\rho = R \times k \tag{7}$$

where ρ is the electrical resistivity of the specimen and R and k are the measured resistance and geometry correction factors, respectively. For bulk conductivity and surface resistivity, the measurement device is shown in Figure 3.6, and the correction factors (k_b and k_s) were determined using equations (8) and (9):

$$k_b = \frac{2\pi a}{1.1 - \frac{0.73}{d/a} + \frac{7.82}{(d/a)^2}} \tag{8}$$

$$k_s = \frac{A}{L} \tag{9}$$

where the parameters d, a, A, and L are the diameter, cross-sectional area, probe spacing, and length of the measured specimen, respectively.

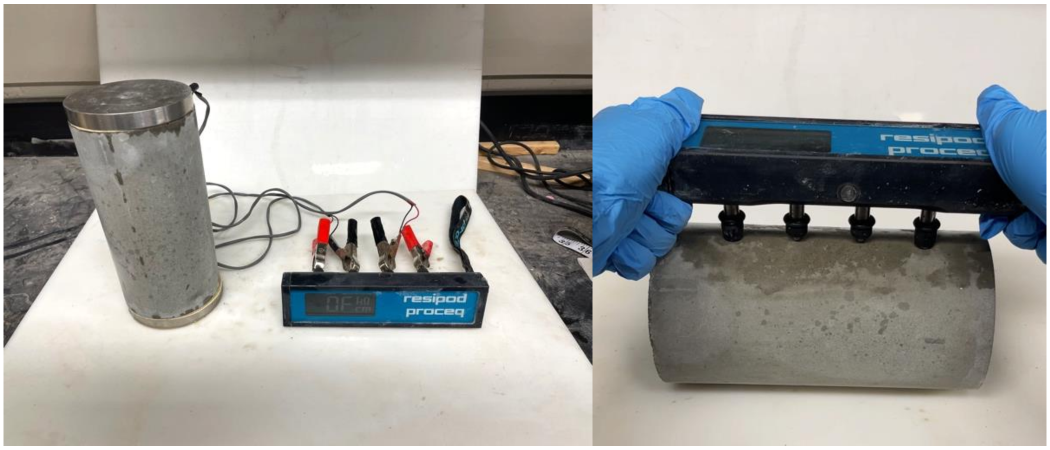


Figure 3.6. Measurement of bulk resistivity (left) and surface resistivity (right)

Water Absorption/Degree of Saturation

The water absorption test was performed after a 29-day curing period in accordance with ASTM C1585, Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic-Cement Concretes (see Figure 3.7).

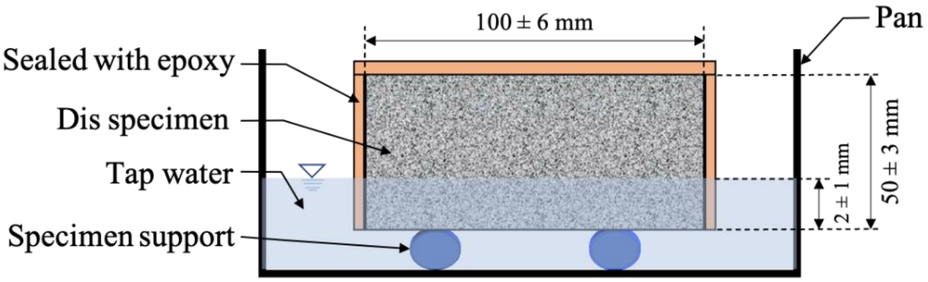


Figure 3.7. Schematic of the absorption test setup

The absorption was computed as described in equation (10):

$$I = \frac{m_t}{a * d} \tag{10}$$

where I is the absorption with a unit length in mm, d is the density of the water in g/mm^3, a is the exposed area of the specimen in mm^2, and m_t is the change in specimen mass in grams at time t.

Optical Image Analysis

Disc specimens subjected to the same treatment as that used in the water absorption test were used to observe the surface microstructure through an optical microscope. The dry specimens were photographed and then rewet with a specific amount of water to observe whether the particles would swell again.

Restrained Shrinkage

Ring tests for restrained shrinkage in this research were conducted per the ASTM C1581 method. The top surface of the concrete sample was sealed and exposed at 23ºC ± 2.0ºC and 50% ± 5% RH. The concrete rings were placed with strain gauges inside the steel rings. The strains were recorded every 10 minutes by a data acquisition system.

Results

This section presents the results and discusses the absorption and desorption kinetics of different kinds of SAP, the optimized selection of SAPs as internal curing materials in concrete, the effects of SAPs on cement hydration, and the influence of SAPs on the performance of internally cured concrete.

Calorimetry

The heat flow and cumulative heat generated by all of the mixtures are shown in Figure 3.8.

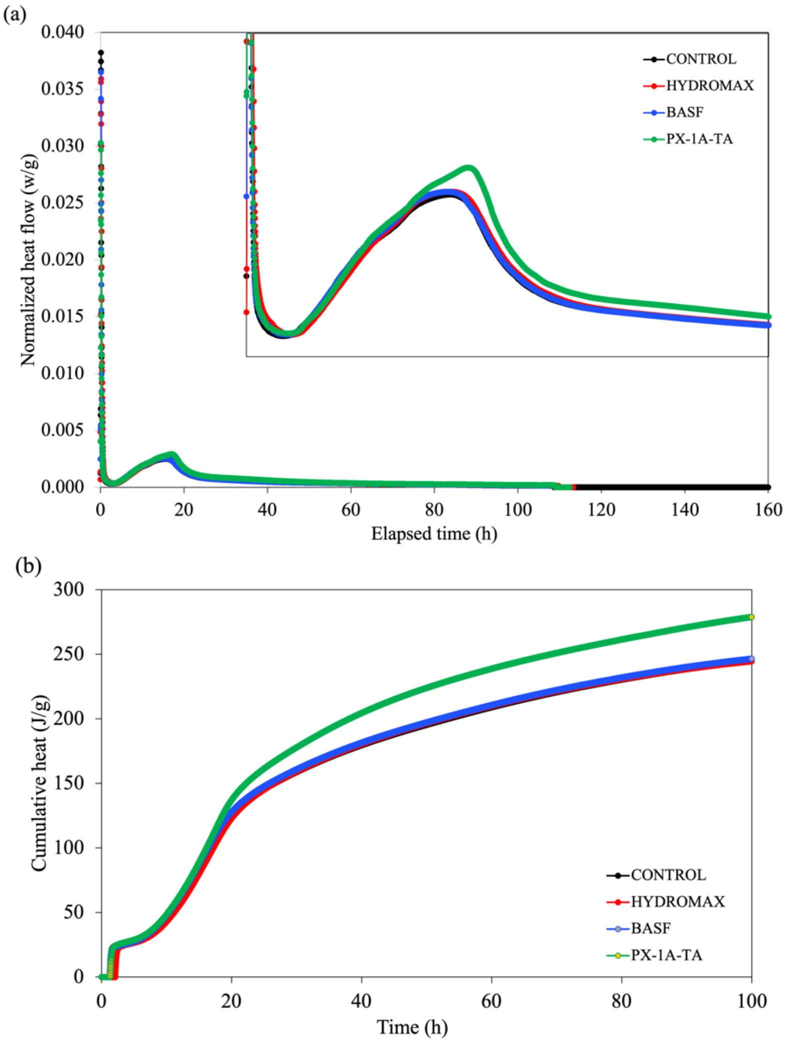


Figure 3.8. Heat of hydration of cement pastes: (a) hydration heat release rate and (b) cumulative heat

Thermogravimetric Analysis

The TGA test results of the four samples at 7 days are presented in Figure 3.9.

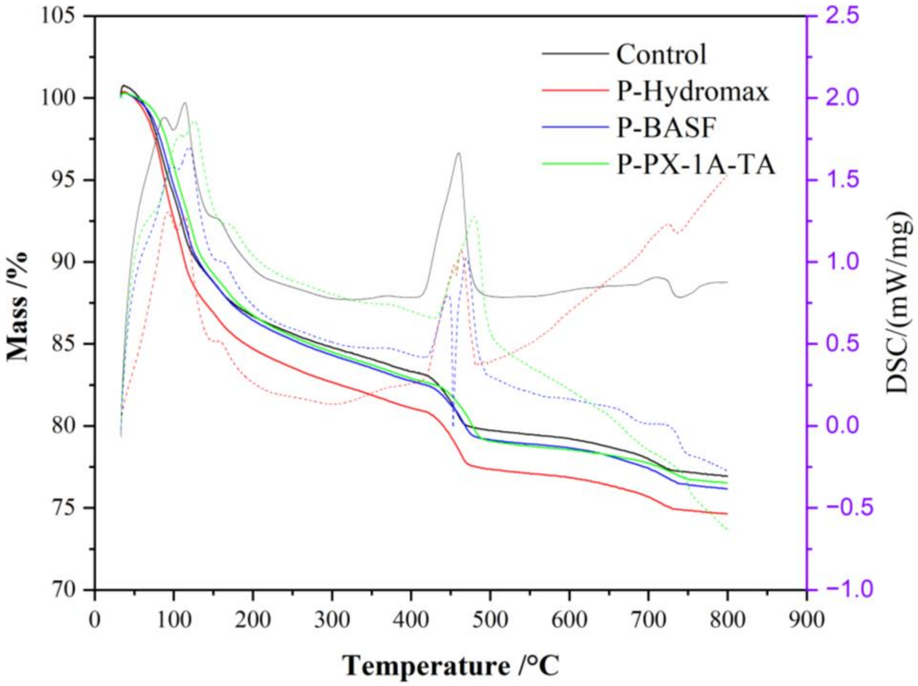


Figure 3.9. TGA results of cement paste samples at 7 days

As shown in Figure 3.9 and Table 3.4, the samples with SAP had a higher value of bound water than the control mixture. This suggests that the moisture distribution in cementitious composites was improved due to the addition of SAP, indicating an increase in cement hydrate products. This was in good accordance with the results of the isothermal calorimetry test.

Table 3.4. Quantification of bound water

Mixture	H_I (wt%)*	H_{Iia} (wt%)	H_{Iib} (wt%)	$H_I + H_{Iia} + H_{Iib}$ (wt%)	CH (wt%)	CO_2 (wt%)
Control	16.2	6.9	1.7	24.8	4.5	3.3
P-Hydromax	18.3	7.3	1.8	27.4	4.8	3.2
P-BASF	15.5	7.7	1.8	25.0	4.7	3.6
P-PX-1A-TA	10.4	5.8	1.3	24.8	3.6	2.9

* H_I at T = 35°C –140°C, H_{IIa} at T = 140°C –320°C, and H_{IIb} at T = 320°C –400°C, the release of CO_2 occurring between 600°C and 800°C, and the determination of CH content in hardened cement paste mixtures at 7 days.

Fresh Concrete Properties

VKelly, Air Content, and Unit Weight

The results of the VKelly, air content, and unit weight tests are shown in Table 3.5.

Table 3.5. Fresh concrete properties

Mixture	VKelly Index (in/$\sqrt{s}$)	Slump Measured by VKelly Test (in.)	Air Content (%)	Unit Weight (lb/ft^3)
Control	0.43	1.9	5.0	148.2
Hydromax	0.57	1.9	5.8	148.4
BASF	0.49	1.8	5.6	148.9
PX-1A-TA	0.51	2.0	5.2	149.2

Workability Retention

The change in the slump of the mixtures over time is summarized in Table 3.6 and Figure 3.10.

Table 3.6. Slump of fresh concrete over time (inches)

Time (min)	0	15	30
Control	1.90	0.50	0.25
Hydromax	2.00	1.66	1.21
BASF	1.84	1.38	1.00
PX-1A-TA	2.13	1.83	1.43

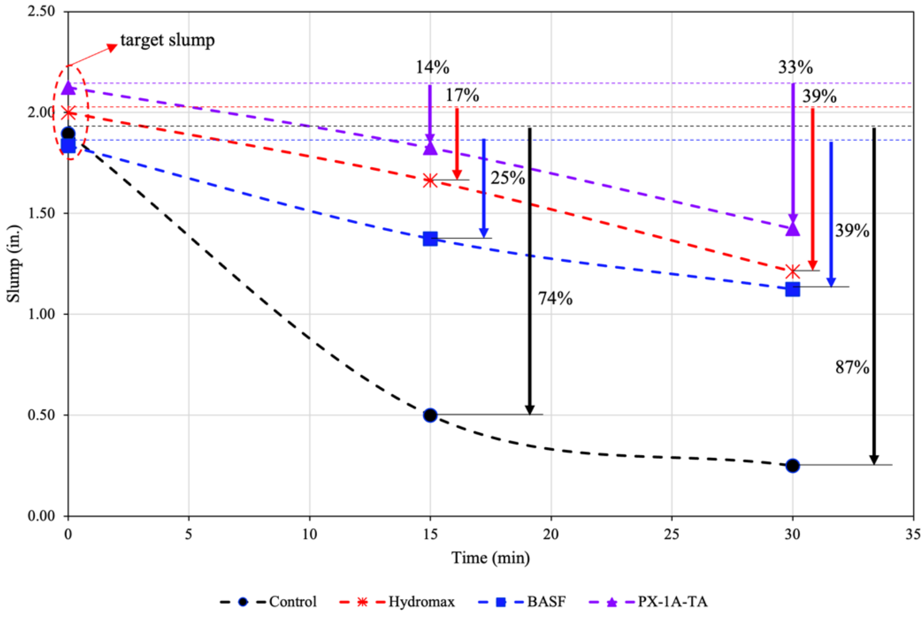


Figure 3.10. Slump of four mixtures over time

Setting Time

The penetration resistance (setting time) results are shown in Figure 3.11.

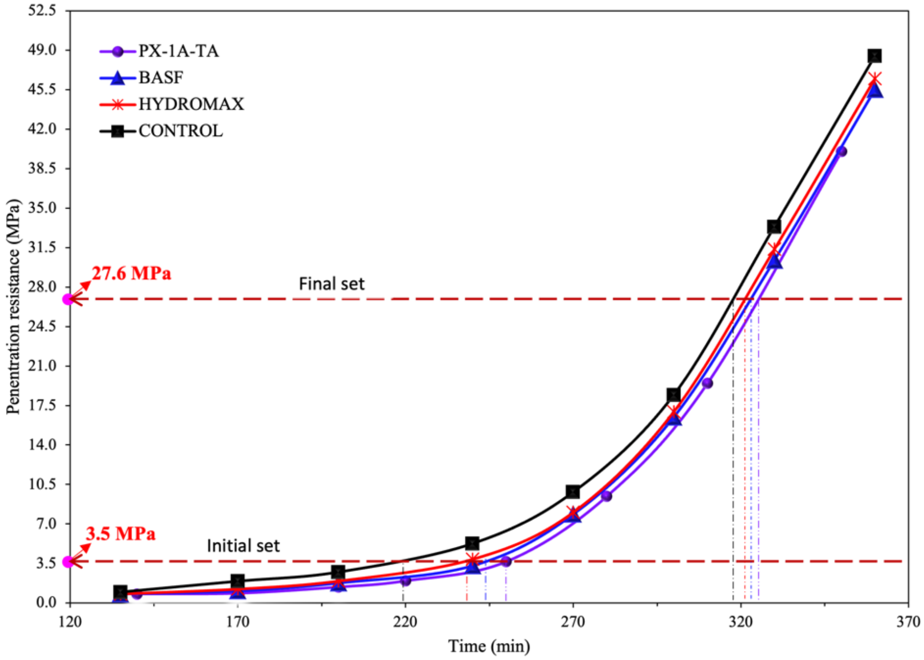


Figure 3.11. Penetration resistance values of mortars over time

Hardened Concrete Properties

Compressive Strength

Figure 3.12 shows the compressive strength results of all concrete mixtures made without and with SAPs at various ages.

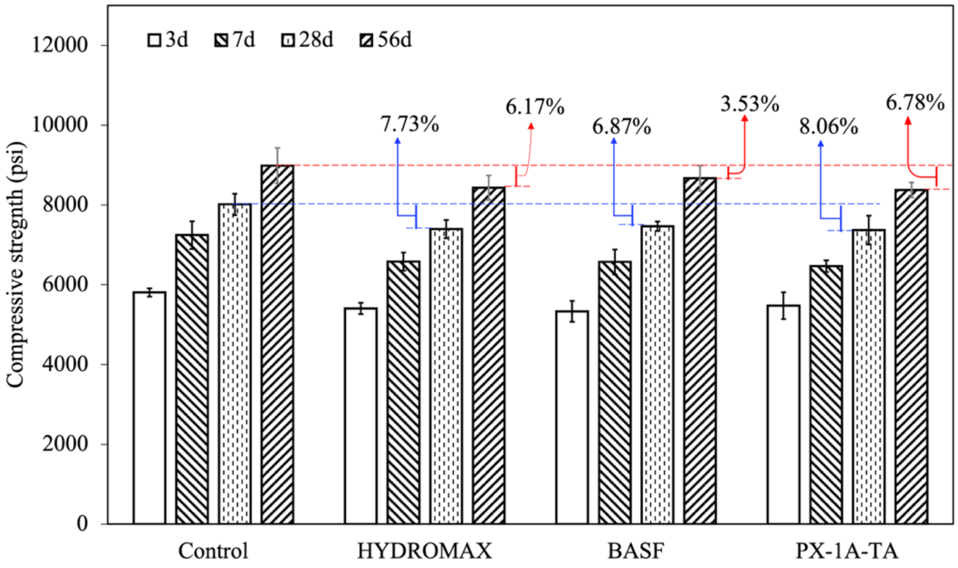


Figure 3.12. Compressive strength development of concrete

Resistivity

Figure 3.13 displays the surface resistivity development for all of the concrete mixtures. Table 3.7 provides a means of assessing the reported values according to AASHTO T 358.

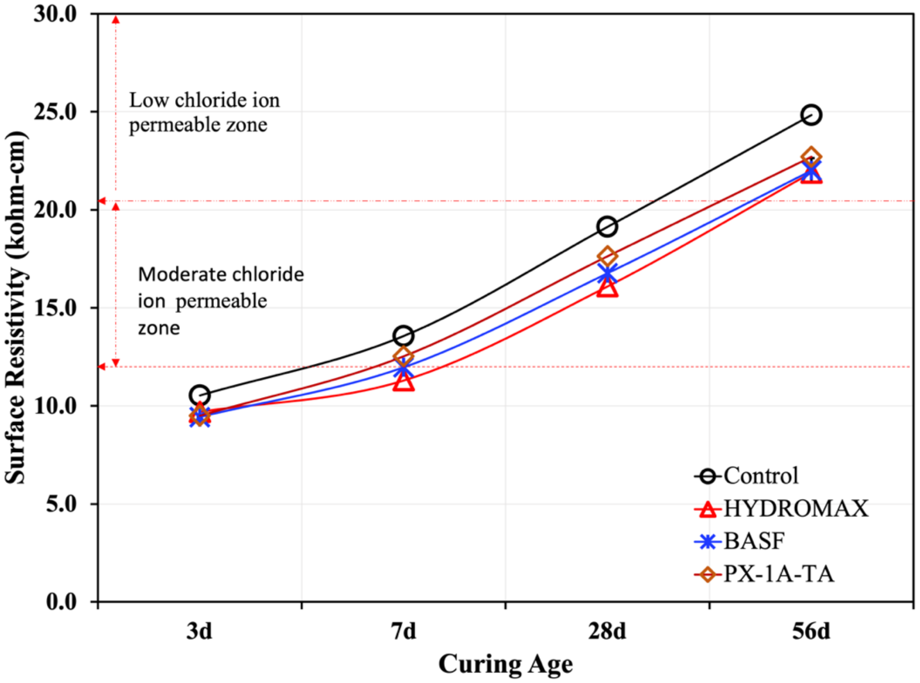


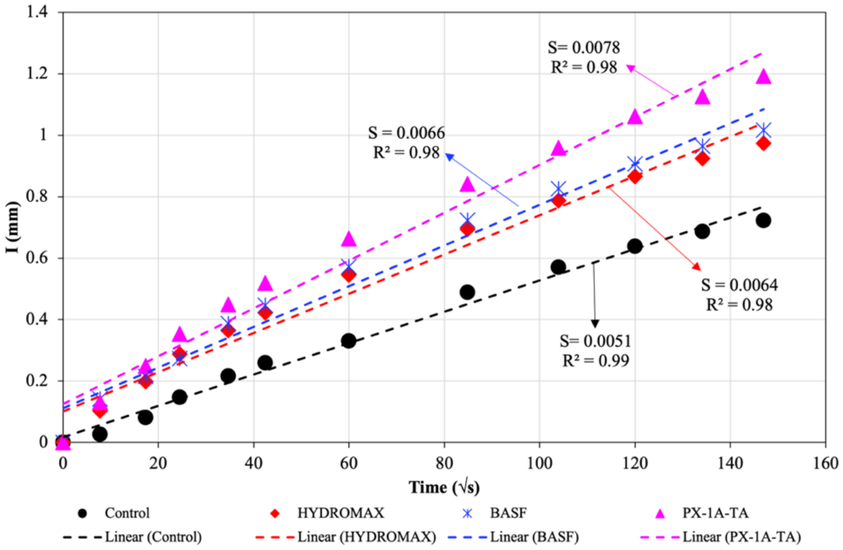
Figure 3.13. Change in electrical surface resistivity over time

Table 3.7. Comparison of rapid chloride penetrability test (AASHTO T 358) and surface resistivity

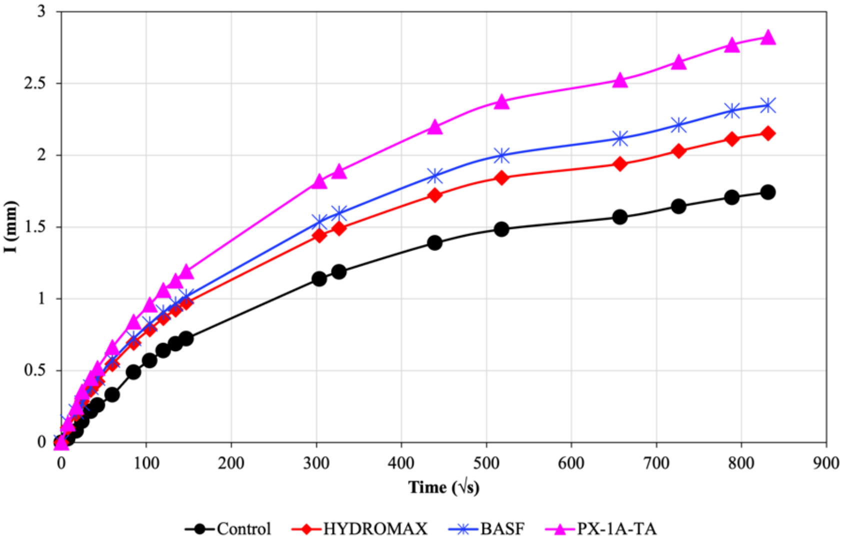
Chloride Ion Penetrability	Resistivity (kΩ·cm)
High	<12
Moderate	12 to 21
Low	21 to 37
Very Low	37 to 254
Negligible	> 254

Water Absorption/Degree of Saturation

Measuring water sorptivity is a simple and reliable way to assess the durability of concrete. The rate of water absorption through capillary suction in concrete provides vital information on the permeability characteristics, pore structure, and durability. All concrete specimens before being processed were cured for 28 days. It can be observed from Figure 3.14 that the amount of water absorbed by the disc specimens normalized by the cross-sectional area in contact with water is a function of the square root of time according to ASTM C1585. To put it simply, the sorptivity is calculated as the gradient of the line that best fits the data points (see Figure 3.14a). The correlation coefficients of all specimens are not less than 0.98, indicating that the initial water absorption index can be used to evaluate the water absorption characteristics of the concrete surface. The control specimen and specimens treated with Hydromax, BASF, and PX-1A-TA have initial water absorption indexes of 0.0051, 0.0064, 0.0066, and 0.0078 mm/$s^{0.5}$, respectively. As can be seen from Figure 3.14b and Table 3.8, the secondary water absorption ability of all disc specimens with SAPs (ranging from 1 to 7 days) is higher than that of the control concrete, reflecting the fact that their permeability is greater than that of the control specimens. This may be mainly correlated to the porosity, pore connectivity, and swelling capacity of the SAPs in their respective cement matrixes.



(a) Initial water absorption test data points



(b) Water absorption rate over time

Figure 3.14. Relationship between absorption versus time ($s^{1/2}$) for concrete at 28 days

Table 3.8. Secondary index for different concretes

Name	Secondary Absorption Rate (mm/s$^{0.5}$)	Correlation Coefficient
Control	0.0011	0.98
Hydromax	0.0013	0.98
BASF	0.0015	0.98
PX-1A-TA	0.0018	0.98

Optical Image Analysis

It can be seen from the typical images in Figure 3.15 that the dry SAP particles are situated within the voids that are created as a result of the mixture being formed around wet, swollen particles. It is also evident that the SAP particles swell again and become visible with rewetting, indicating that the structure of the SAPs in the disc specimens remains intact during drying at 50°C. The SAP particles did not exhibit any agglomeration.

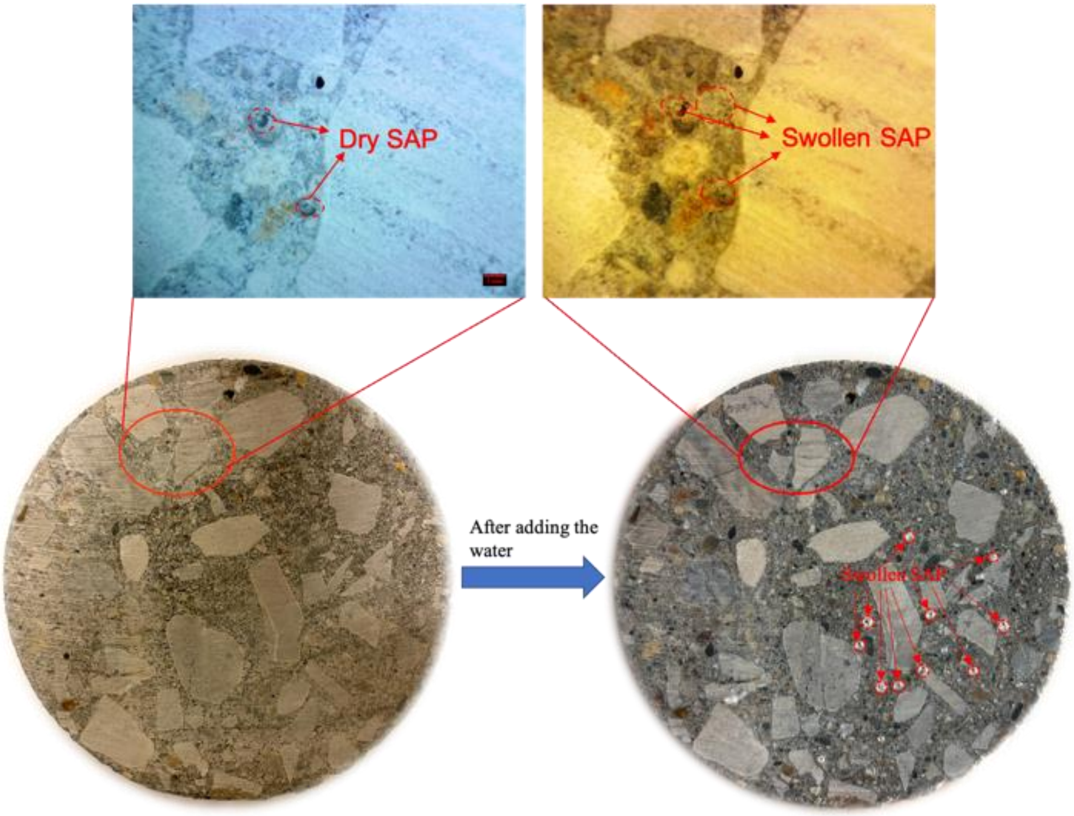


Figure 3.15. Optical images of a concrete cross section

Restrained Shrinkage

Restrained ring shrinkage was monitored for 33 days. The average strain value from three specimens per mixture was calculated. Figure 3.16 illustrates the strain behavior over time, showing a gradual increase. Typically, the appearance of a crack results in a sudden strain release, which the gauges should record. However, none of the concrete specimens exhibited cracking, even though cracking is a common issue with the ASTM method. Notably, concrete specimens containing SAP demonstrated lower strain levels than the control specimens. Hydromax shows the most significant reduction in shrinkage, followed by PX-1A-TA, and then BASF. The control sample without SAPs shows the highest shrinkage.

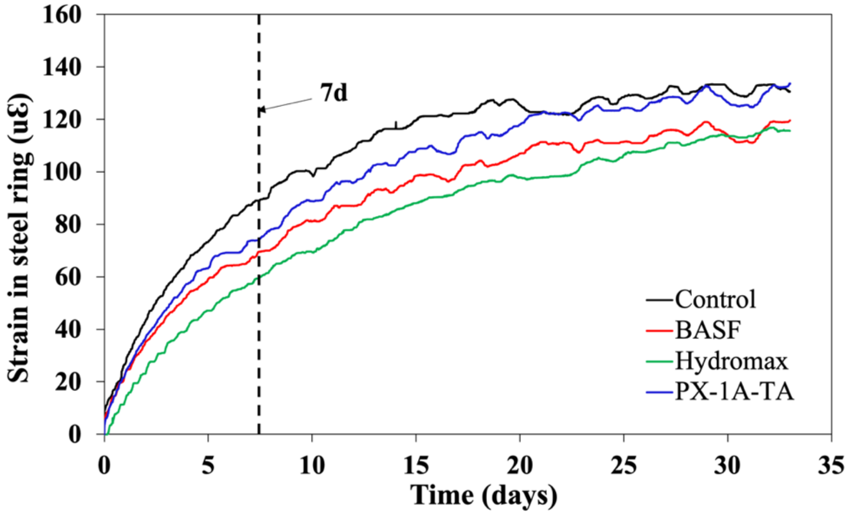


Figure 3.16. Restrained shrinkage of concrete mixtures

Impact of Dried and Presoaked SAP on Concrete Mechanical Properties

Figure 3.17 shows the compressive strength results of all concrete mixtures at 28 days, including those made with dry and presoaked SAPs.

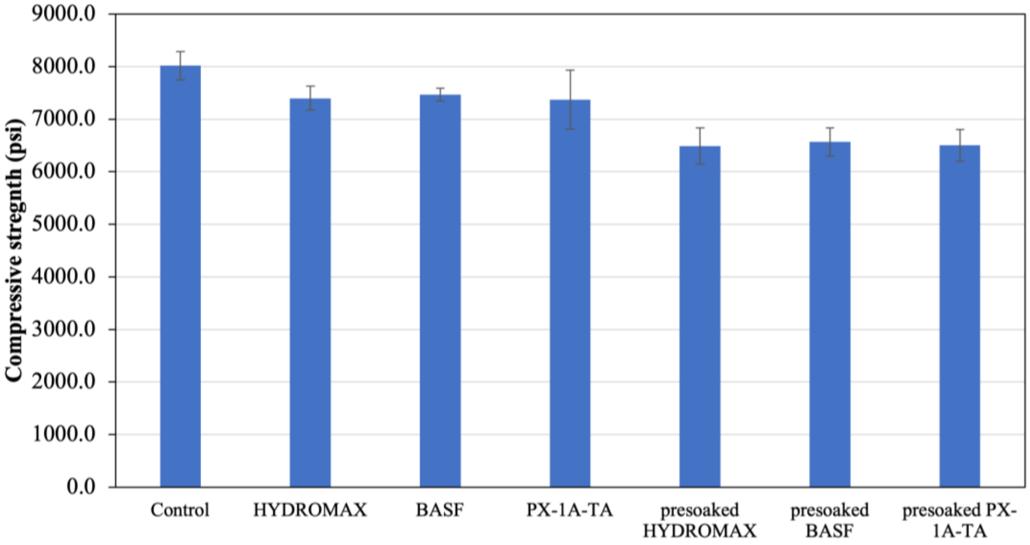


Figure 3.17. 28-day strength of concrete mixtures

Figure 3.18 shows the water absorption of concrete cylinder specimens that were cured for 14 days and then immersed in tap water after processing.

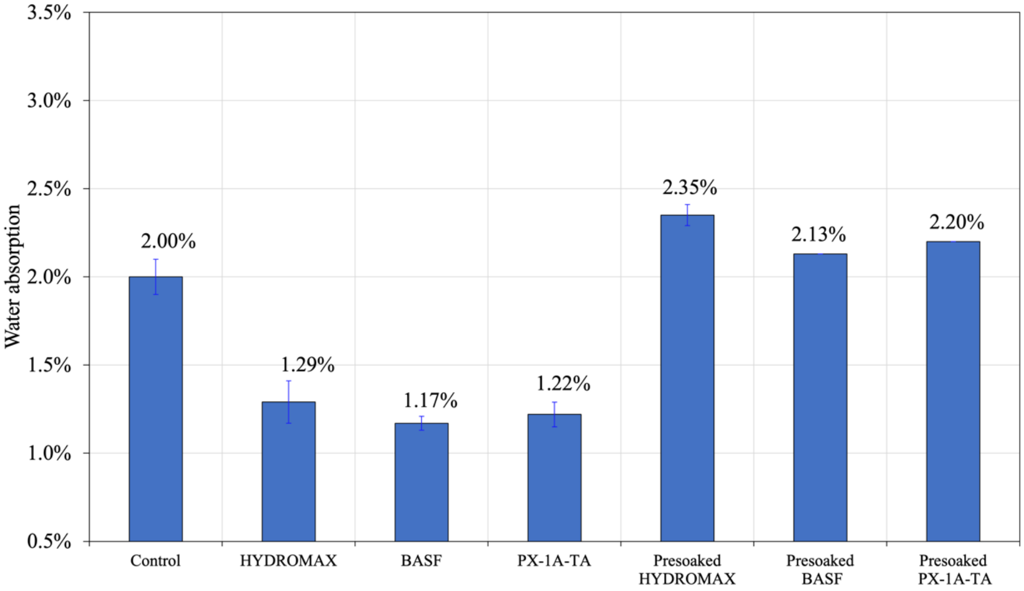


Figure 3.18. Water absorption of concrete mixtures

Discussion

Figure 3.8 illustrates that the primary hydration peaks in the samples containing PX-1A-TA were slightly higher than those of the control, indicating enhanced hydration. At 7 days, the degree of hydration of this paste increased somewhat, suggesting that the water released promoted hydration and could improve overall performance.

The TGA results showed that the mass losses (main phases in hydrated products) for all mixtures occurred at the same temperature ranges. The first peak appeared from 110°C to 140°C, which is commonly attributed to the loss of chemically bound water caused by the dehydration of C-S-H and ettringite (Van et al. 2014). The second peak occurred between 150°C and 170°C, which was associated with carboluminates (AFm) (Jensen 1999). The most prominent decrease in mass recorded was within the temperature range of 430°C to 530°C, which can be attributed to the decomposition of CH. Based on Marsh and Day's (1988) methodology, Table 3.4 presents the quantification of bound water and the corresponding mass of CH for all samples within the respective temperature intervals. Moreover, an additional peak was observed within the temperature interval of 600°C to 800°C, which could be attributed to the decarbonization of crystalline $CaCO_3$.

As shown in Figure 3.9 and Table 3.4, the samples with SAP had a higher value of bound water than the control mixture, which suggests that the moisture distribution in cementitious composites was improved due to the addition of SAP, indicating an increase in cement hydration products.

Although the same slump was achieved for fresh concrete in all groups, the SAP contents added to the concrete differed due to their sorption kinetics, as discussed in the water absorption/desorption section of this report. The amount of SAP and WRA required to achieve similar slumps was inversely related to the product absorption. Selection of materials should therefore also consider the costs of the amount of material required to achieve performance.

Table 3.5 indicates that all of the mixtures exhibited similar workability at initial mixing. The air content of all concrete mixes was in the range of 5.0% to 5.8%. The mixtures made with SAP had slightly higher initial air contents than the control, which may be related to the addition of extra water, the SAP, or both.

The initial slump measured by the VKelly test generally aligned with the results of the slump test, as shown in Table 3.5 and Table 3.6. However, the VKelly index of the concrete mixtures with SAPs was higher than that of the control, which may be attributed to the release of water from the swollen SAP particles under vibration.

Table 3.6 and Figure 3.10 show that the SAP mixtures experienced less slump loss than the control over time. The PX-1A-TA mixture seemed to retain slump the best, which can possibly be attributed to its higher cross-linking compared to other SAPs, resulting in its larger absorption capacity.

Figure 3.11 demonstrates that the incorporation of SAP and additional water into the concrete slightly delayed both the initial and final setting times relative to the control mixture. This delay may be attributed to a potential increase in the effective w/c ratio, likely resulting from insufficient water absorption by the SAP during mixing.

The strength results (Figure 3.12) indicate that incorporating SAPs into concrete caused a decrease of about 8% in compressive strength at 28 and 56 days compared to the control mixture, which was consistent with the results from other studies presented in Table 1.2. The observed behavior may be ascribed to the formation of pores left by SAP particles after releasing their stored water (Justs et al. 2015, Savva and Petrou 2018). The size of SAP particles may play a role in this reduction, as larger SAP particles tend to leave larger pores. Notably, the compressive strength may improve if the advantages of internal curing surpass the drawbacks associated with increased porosity. Otherwise, a decline in strength may be observed (Piérard et al. 2006, Karthikeyan et al. 2018).

Similarly, the 28-day resistivity of all mixtures containing SAP was lower than that of the control, as illustrated in Figure 3.13. It is likely that the presence of SAPs caused additional pores and voids in the concrete matrix.

Figure 3.14 illustrates that the SAP-containing samples exhibited higher permeability compared to the control. This increase may be attributed to the incomplete absorption of additional water by the SAP, which could increase the effective w/c ratio and lead to a more porous matrix.

Figure 3.15 shows the distribution of SAP particles in the hardened concrete. When rewetted, the SAP particles regained their swollen state, indicating that their structure had remained intact. Additionally, SAP particles did not appear to agglomerate, promoting effective internal curing.

The restrained shrinkage results in Figure 3.16 show that all types of SAP could effectively reduce the early shrinkage of concrete. This is due to the release of the stored water in the SAP, enhancing the internal moisture of the concrete and thus minimizing shrinkage stresses. The reduction in shrinkage appears to be related to the absorption of the product.

Figure 3.17 illustrates a further slight decline in 28-day compressive strength for mixtures with presoaked SAP compared to the control. This may be due to insufficient water being released by the SAP during mixing, effectively increasing the w/cm ratio of the mixture.

Figure 3.18 shows the results of water absorption testing. In comparison to the control, samples treated with dry SAPs demonstrated a significant reduction in water absorption. Conversely, samples modified with presoaked SAP demonstrated higher water absorption values, consistent with the strength results.

SUMMARY AND CONCLUSIONS

The use of SAPs can improve some of the properties that affect the performance of concrete. However, consistently controlling water absorption and desorption has proved to be a challenge, potentially leading to detrimental effects on performance.

Based on the laboratory test results presented in this investigation, the following conclusions can be made:

- Slump loss tests indicated that the SAPs used in this study were absorbing some water over at least the first 30 minutes.
- Calorimetry and TGA tests confirmed that the mixtures containing SAPs experienced increased cement hydration, likely due to the water provided by the SAPs.
- Hardened concrete with SAPs had lower compressive strength and resistivity values than the control concrete, likely because not all of the added water was absorbed into the SAPs.
- The concrete mixtures containing SAPs exhibited lower shrinkage up to 7 days, likely due to retention of water in the SAPs.
- All three SAP products tested performed similarly to each other.
- The addition of dry SAP was less detrimental to performance than the use of presoaked SAP.

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