ENHANCING SELF-HEALING ABILITY OF CEMENTITIOUS MATERIALS THROUGH SUPER ABSORBENT POLYMERS (SAPS)

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ABSTRACT

Extensive research in recent years has focused on enhancing the self-healing ability of cementitious materials using Super Absorbent Polymers (SAPs).This study aims to explore the potential of SAPs for self-healing in mortar specimens. The experimental program involved casting mortar specimens with varying SAP concentrations (Control 0%, 0.2%, 0.4%, and 0.5% of the weight of Cement) combined with 3% and 5% of micro silica. The specimens were subjected to concentrated lateral load for crack initiation and then underwent wet-dry curing for 28 days. Thirteen cylindrical specimens were prepared, each cast in a mould measuring 6 inches in diameter and 6 inches in height for the permeability test. Several cylindrical specimens showed a tendency for crack healing compared to the control specimen, as observed in the permeability test. Additionally, 13 cubic specimens (2×2×2 inches) were prepared for the compressive strength test, each with a different combination of SAP and micro silica. After 28 days of curing, an increase in compressive strength was observed with an increase in SAP content, as SAPs influence the material's mechanical performance by creating macro-pores. The control specimen exhibited compressive strengths of 4.6 MPa, 6.5 MPa, and 5.7 MPa, assessed on days 3, 7, and 14, respectively, while the 0.5% SAP specimen showed significantly higher strengths of 10.4 MPa, 10.9 MPa, and 11.9 MPa on the consecutive days. In the dry shrinkage test, the control specimen displayed a higher shrinkage ratio of $59{,}200\times10^{-6}$. In contrast, the 0.5% SAP specimen demonstrated much lower shrinkage at the end of two weeks, with a shrinkage ratio of $38{,}000\times10^{-6}$. The self-healing performance was evaluated, as the incorporation of SAPs into mortar specimens showed a decrease in crack opening and a lower drying shrinkage ratio than the control specimens. The results of this study suggest that SAPs can be a promising additive for improving the self-healing ability of mortar.

Keywords: *Super Absorbent Polymer (SAP), Self-healing Ability of Concrete, Micro Silica, Macro Pores, Cement Mortar*

1. INTRODUCTION

Infrastructure plays a pivotal role in driving socioeconomic activities. Due to its advantageous mechanical properties, durability, and cost-effectiveness, concrete is the prevalent choice for civil engineering projects. Nonetheless, concrete structures degrade due to factors such as shrinkage, heavy loads, and exposure to harsh environments. Limited budgets for inspection and maintenance further compound the challenges faced by concrete structures. Cracks in concrete structures pave the way for the infiltration of harmful agents, leading to corrosion of steel reinforcement (Chindasiriphan et al., 2020). Given concrete's susceptibility to cracking, repair becomes paramount, constituting a significant portion of Europe's annual construction budget (Lefever et al., 2020). The high costs of repairs, coupled with their temporary nature, have spurred the introduction of self-healing concrete. This innovative material can autonomously mend cracks, eliminating the need for external interventions and reducing costs. Cracks in concrete structures not only curtail service life and escalate maintenance expenses but also jeopardize structural integrity, posing safety hazards in infrastructure like bridges, roads, and buildings.

Super Absorbent Polymers (SAPs), extensively utilized in agriculture, hygiene, and medicine, have garnered attention for their potential in self-healing applications. These polymers can swell several hundred to a thousand times their original size, absorbing surrounding liquids (Sidiq et al., 2019). Self-healing in cementitious materials is chiefly driven by two mechanisms: 1) the hydration of unreacted cement particles and the formation of Calcium-Silica-Hydrate (C-S-H) gel, and 2) the reaction between calcium cations (Ca^+) and dissolved carbon dioxide (CO_2), yielding precipitated calcium carbonate ($CaCO_3$) (Sidiq et al., 2020). The development of self-healing cementitious materials stems from the need to enhance durability and sustainability while reducing the environmental impact of construction. SAPs, hydrophilic polymers with a remarkable water-absorbing capacity, act as internal reservoirs, releasing water upon crack formation to initiate the healing process. Furthermore, the repetitive swelling and shrinking of SAPs throughout a structure's lifespan enable multiple healing cycles (Snoeck & Belie, 2016).

Apart from concerns about cracking during service life, matrix cracking before external loading is also a possibility. Particularly in cases of low water-cement ratio, early-age shrinkage-induced cracking demands consideration, necessitating a water-to-cement ratio of 0.42 for full hydration. SAPs offer a solution by serving as internal curing agents, absorbing hydration water during concrete mixing. This water is subsequently released during self-desiccation, facilitating continuous hydration (Lefever et al., 2020). To counter potential decreases in mechanical properties due to continuous hydration, additives like Microsilica or Silica fume can be employed in conjunction with SAPs (Du& Liu, 2014).

This experiment delves into the impact of SAPs and nano-silica on the self-healing capability of cementitious mortars. Crack initiation in cylindrical specimens was followed by continuous observation to document the progressive self-healing process. A permeability setup measured water flow through crack openings over time. Combining both methods allowed conclusions to be drawn regarding self-healing efficacy and the influence of different materials.

The primary objectives of this research are to investigate the effect of SAPs on the self-healing of mortar specimens, to examine the impact of SAPs on the compressive strength of mortar specimens, and to assess self-healing performance in terms of crack mitigation and dry shrinkage.

Cracking is a prevalent occurrence in cementitious materials used in structural construction. Minimizing crack formation has emerged as a crucial concern in the realm of Structural Engineering. The utilization of Super Absorbent Polymers (SAPs) to achieve self-healing and reduce cracks has gained momentum through recent research. Additionally, SAPs can serve as internal curing agents, absorbing water to enhance internal curing. However, excessive SAP content can lead to internal voids diminishing compressive strength. Micro silica can be introduced to address this issue and bolster specimen strength. Overall, this study underscores the potential of SAP incorporation to enhance the self-healing capabilities of cementitious materials. These findings hold significant implications for the longevity and performance of concrete structures, particularly in demanding environments prone to cracking and deterioration. Further exploration is needed to optimize SAP content and assess long-term self-healing performance under varying loads and environmental conditions. Future experimentation could also explore bond strength after SAP replacement with Cement, as well as the influence of SAPs on concrete shrinkage and creep.

2. METHODOLOGY

2.1 Materials

Super Absorbent Polymer (SAP): The SAP utilized in this study was sourced from the local commercial market, as depicted in Figure 1. The SAP is Sodium Polyacrylate, commonly known as water-lock and sodium salt of polyacrylic acid. The chemical formula of the SAP is $[\text{-CH}_2\text{-CH(COONa)-}]_n$. The SAP has an absorbent capacity of approximately 32 grams of water per gram of SAP in a concrete environment. The physical properties of SAPare as follows:

- Nominal Maximum Size: 0.38 mm
- Water Absorption:32 %
- Colour: White

Fine Aggregate (FA): Usually synonymous with sand, fine aggregate is a building material comprising small particles, typically less than 4.75 mm (0.1875 in) in diameter. Frequently utilized in construction projects like concrete and mortar, it enhances workability and strength in the mixture. Sylhet Sand, locally available, was employed as the fine aggregate, as depicted in Figure 2. The material properties of Fine Aggregate used are as follows:

- Specific Gravity: 2.6
- Water Absorption: 3.62%
- Fineness Modulus: 2.75
- Unit Weight: 1600 kg/m³

Cement: Serving as the binding agent, Cement holds together the sand and other components in mortar. Typically derived from a blend of limestone, clay, and other minerals heated to high temperatures, Cement manifests as a fine powder. Portland cement is the most commonly employed type in mortar, recognized for its robustness, durability, and resistance to moisture. The study employed Ordinary Portland Cement (OPC) and Portland Pozzolana Cement (PPC), both of which are locally accessible. Figure 3 provides an illustration of the Cement used.

Water: Integral to the mortar composition, water activates the Cement, facilitating its binding with the sand. The quantity of water used influences the strength, workability, and durability of the mortar. Inadequate water can result in a dry and crumbly mixture, while excessive water can yield weak and brittle mortar. It is imperative to use clean, contaminant-free water to ensure proper hydration of the Cement.

Figure 1: Super Absorbent Polymer (SAP)

Figure 2: Fine Aggregate (Sylhet Sand)

Figure 3: Cement

Figure 4: Micro Silica

Micro-Silica: Silica fume, classified as an acidic technolith, stands out as a highly reactive pozzolan. Through the hydration reaction, it generates calcium silicate hydrate (CSH) gel. This supplementary CSH not only enhances the flexibility of concrete but also elevates both bond strength and compressive strength. This heightened bond strength fosters improved adhesion of the mastic to the aggregate. Additionally, the micro-pores and fine intergranular voids are effectively filled, preventing their transformation into micro-cracks. Over time, a proportional amount of silica fume undergoes a reaction with all newly formed calcium hydroxide ($Ca(OH)_2$) (Frýbort et al., 2023). Figure 4 provides an illustration of the micro-silica used.

2.2 Material Properties

Unit Weight: The determination of the unit weight of fine aggregate in both compacted and loose conditions follows the procedures outlined in ASTM Test Method C-29.

Specific Gravity: The specific gravity of fine aggregates is determined in accordance with ASTM Test Method C 127. This property can be represented using either the bulk specific gravity or apparent specific gravity.

Water Absorption: The measurement of water absorption in fine aggregates is conducted as per ASTM C 566. For this test, 200 grams of fine aggregates were collected to determine their moisture content.

Fineness Modulus: The fineness modulus of fine aggregates is evaluated using ASTM C136. The fineness modulus (FM) serves as an empirical measure that provides a rapid indication of a material's coarseness or fineness.

2.3 Preparation of Cylindrical Mortar Specimen

2.3.1 Mortar Mix Design

Mortar mix design involves selecting suitable ingredient ratios for mixing mortar that aligns with the intended characteristics and performance criteria for specific applications. The three primary components of a mortar mixture are Cement, sand, and water. Additional components such as lime, fly ash, or microsilica can be incorporated to enhance workability, strength, durability, and water resistance. An appropriate mix design ratio was determined based on ACI standards and a comprehensive literature review.

For complete hydration of cement particles, a water-to-cement ratio of 0.42 is necessary. When the water content falls below this threshold, self-desiccation occurs, leading to autonomous shrinkage of the cementitious material (Lura et al., 2003). The Mortar Mix Design is as follows:

- Cement to Fine Aggregate Ratio: 1:3
- Water to Cement Ratio: 0.42
- Size of Fine Aggregate: #8 passing - #30 retained

2.3.2 Sieve Analysis

Sieve analysis is a laboratory technique used to determine the particle size distribution of granular substances, such as sand or gravel. This process involves passing the material through a series of sieves with progressively decreasing aperture sizes. Each sieve separates the material into distinct size fractions, and the amount of material retained on each sieve is measured to calculate the proportion within each size range. The collected data can provide valuable insights into characteristics like the material's homogeneity coefficient and average particle size.

2.3.3 Mixing Procedures

The mortar mixture was prepared using the mix ratio mentioned above, following ASTM C270 guidelines. Prior to blending, each component was accurately weighed. Subsequently, all components were combined and mixed manually until a uniform and consistent mixture was achieved, as shown in Figure 5.

2.3.4 Casting of Specimens

To cast the specimens, a long pipe with a 6-inch diameter was divided into 13 segments, each measuring 6 inches in diameter and 6 inches in height, as presented in Table 1. Thirteen different mixtures were prepared, incorporating varying percentages of SAP and micro silica. This included six specimens using Ordinary Portland Cement (OPC) and six with Portland Pozzolana Cement (PPC). Additionally, a control specimen was created solely using OPC, sand, and water for comparative purposes. After mixing, the specimens were cast, leaving a 2-inch free space on top of the mould for subsequent permeability testing, as shown in Figure 6. Each individual mould was lined with a smooth cardstock to provide a flat bottom surface for casting.

Figure 5: Addition of All Materials Except Water for Single Cylindrical Specimens

Figure 6: Mold and Casting of Specimens.

Table 1: Specimen and its Material Percentage

Specimen	SAP % of Cement	Micro Silica %	Cement Type
Control	0	0	OPC
S2M3	0.2	3	OPC
S2M5	0.2	5	OPC
S4M3	0.4	3	OPC
S4M5	0.4	5	OPC
S5M3	0.5	3	OPC
S5M5	0.5	5	OPC
S2M3F	0.2	3	PPC
S2M5F	0.2	5	PPC
S4M3F	0.4	3	PPC
S4M5F	0.4	5	PPC
S5M3F	0.5	3	PPC
S5M5F	0.5	5	PPC

2.4 Preparation of Cubic Mortar Specimen

2.4.1 Cubic Mortar for Wet Curing

Thirteen cubic specimens, each measuring 2x2 inches, were cast using the same percentages of SAP and micro-silica as those employed in the cylindrical specimens, as shown in Figure 7. These cubic specimens were subjected to 28 days of wet curing using a water-to-cement (w/c) ratio of 0.42.

2.4.2 Cubic Mortar for Internal Curing

Subsequently, three cubic specimens were cast for each batch with SAP concentrations of 0.2%, 0.4%, and 0.5% of cement weight, along with a control specimen, as shown in Figure 8. These specimens were sealed with a food wrapper to utilize the moisture present in the cubes for internal curing. The compressive strength of these specimens was then determined on days 3, 7, and 14. A w/c ratio of 0.36 was employed, along with additional water to account for the water absorbed by the SAP.

2.5 Preparation of Mortar Bar Specimen

Mortar bar specimens measuring 25×25×250mm were cast to evaluate the impact of SAP on dry shrinkage. Four bar specimens, each containing 0.2%, 0.4%, and 0.5% SAP of Cement, were prepared, as shown in Figure 9. An additional Control specimen bar was cast to serve as a basis for comparing shrinkage. The specimens were cast using a water-to-cement (w/c) ratio of 0.36, and OPC and Sylhet sand of #16 passing - #30 retained were utilized.

Figure 7: Thirteen Cubic Specimens for Wet Curing.

Figure 8: Cubic Specimens for Internal Curing.

Figure 9: Casting and De-molding of Mortar Bars.

2.6 Curing of Specimens

Curing is the process of allowing cementitious materials like concrete or mortar to dry and solidify after placement. This crucial procedure greatly influences the overall performance, strength, and durability of cementitious materials. Proper curing ensures optimal cement hydration by maintaining a controlled level of moisture and temperature. This facilitates the chemical reactions between water and Cement, resulting in the desired material properties.

After 24 hours of casting, the 13 cylindrical specimens, along with their pipe moulds, as well as the 13 cubic specimens, were demoulded and subjected to a 28-day curing period in water. Meanwhile, the three specimens from each SAP batch were wrapped in food wrappers for internal curing. Additionally, the four mortar bar specimens were left to cure under dry environmental conditions.

This controlled curing process aims to provide a consistent and accurate assessment of the impact of SAP on the performance and properties of the mortar specimens.

2.7 Determination of Compressive Strength of Cubic Mortar

The compressive strength of cubic mortar specimens, a crucial measure of their ability to withstand compressive forces, was determined by subjecting the specimens to a compressive load until failure occurred. ASTM C109 guidelines were followed for this test. The cubic mortar specimens were placed within a compression testing machine, and the maximum load they could bear before failure was recorded, as shown in Figure 10. The compressive strength (C) was calculated using the equation:

$$C = \frac{P}{A} \tag{1}$$

Where C is the Compressive strength in MPa, P is the Failure load in N, and A is the Contact area in mm².

2.8 Forming Cracks by Inducing Lateral Load

Following the 28-day curing period, lateral loads were applied to the cylindrical specimens that were cast within pipe moulds. The moulds were kept intact during the application of lateral loads to ensure lateral confinement and prevent the specimens from breaking apart due to fractures. As soon as a crack was observed running along the length of the specimen, the application of the load immediately ceased, as shown in Figures 11 and 12. This procedure was consistently applied to each specimen.

Precautions were taken to control the width of the cracks, as larger cracks do not exhibit self-healing characteristics in cementitious materials.

Figure 10: Compressive Strength Test Setup.

Figure 11: Inducing Lateral Load on Cylindrical Specimens.

Figure 12: Formation of Cracks in Cylindrical Specimens.

2.9 Permeability or Water Flow Rate Test

Permeability tests were conducted to assess the self-healing ability of the specimens and monitor crack closure during wet-dry curing. These tests involved maintaining a wet environment during the healing process and measuring the water flow rate through the cracks, as shown in Figure 13. A reduction in water flow rate over time indicates self-healing, as crack closure hinders water flow. The water that passed through the cracks and exited the bottom of the specimen within a 10-minute period was used to calculate the water flow rate. Although there is no specific ASTM code for this test, the flow rate was determined by dividing the weight of water (mg) by the duration of 10 minutes.

$$F = \frac{W}{T} \tag{2}$$

Where F is the Water flow rate in mg/min, W is the weight of water passing through in 10 minutes in mg, and T is the time (10 minutes).

2.10 Shrinkage Test

Dry shrinkage, characterized by the reduction in length of a cement-based mortar bar upon drying, can significantly impact the strength and durability of concrete structures, potentially leading to cracks and structural damage. To mitigate the risks associated with dry shrinkage, various methods can be employed, such as incorporating Super Absorbent Polymer (SAP) in mortar mixes. SAP acts as an internal curing agent capable of holding and absorbing water.This setup measures the expansion of the mortar bar, as shown in Figure 14. The expansion is calculated using equation (3).

$$\Delta L = \frac{L_x - L_i}{L_g} \times 100 \quad (3)$$

Where, L_x is the Comparator reading of the specimen at age x minus the reference bar comparator reading at age x, L_i is the Initial comparator reading of the specimen minus the reference bar comparator reading at the same time and L_g is the Nominal gage length, 250 mm (10 in.).

Figure 13: Permeability or Water Flow Rate Test Setup.

Figure 14: Experimental Setup for the Shrinkage Test.

3. RESULTS

3.1 Microscopic Image Analysis

After a healing period of 28 days, microscopic image processing revealed signs of healing on the exterior surfaces of several specimens, while the control specimen exhibited no healing whatsoever. It was observed that some specimens did not exhibit healing, possibly due to the initial cracks being too wide, as shown in Figures 15 and 16. Based on the literature, self-healing primarily occurs in small fissures measuring 100 to 150 micrometres in width. As a result, crack closure was only evident in regions where the initial crack width was within this range.

The healing process was documented using a cell phone camera. However, capturing healing without a microscopic perspective can be challenging.

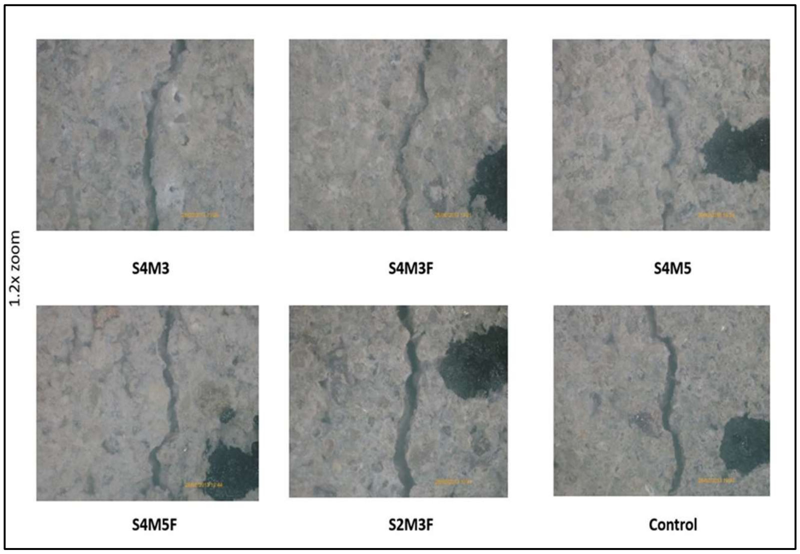

Figure 15: Microscopic Image of a Specimen Showing Evidence of Crack Healing at 1.2x Zoom.

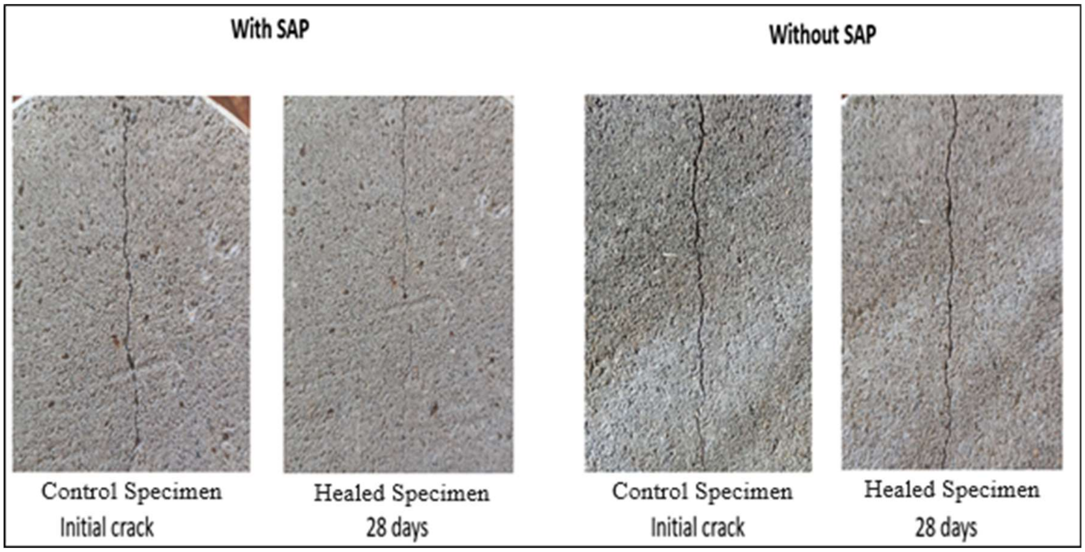

Figure 16: A Comparison between a Healed Specimen and a Control Specimen (without SAP) after 28 Days.

3.2 Compressive Strength of Cubic Specimen

Following a 28-day water curing period, the compressive strength of 13 distinct cubic mortar specimens was evaluated, as presented in Figure 17. The obtained results were plotted on a graph, revealing a gradual decrease in compressive strength as the SAP percentage increased from 0.2% to 0.5%. Additionally, higher percentages of micro-silica led to a slight enhancement in compressive strength. This effect is attributed to Micro silica counteracting the reduction in compressive strength caused by internal voids resulting from SAPs. SAPs' tendency to swell expels excess water, creating voids that reduce compressive strength. Notably, the Control specimen displayed significantly higher compressive strength than those containing SAPs. Hence, it can be inferred that a high SAP percentage in mortar mixes adversely affects compressive strength.

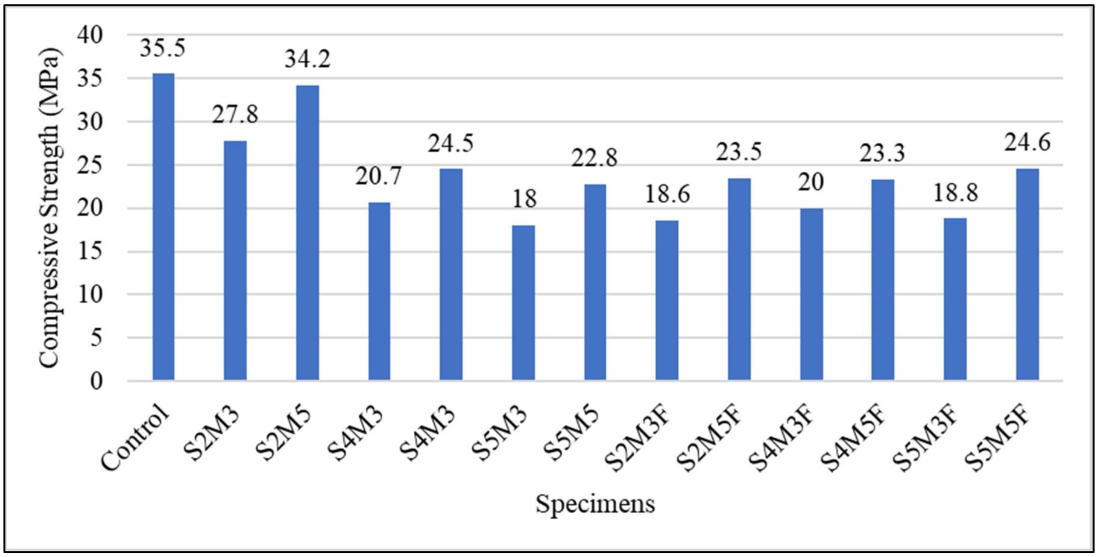

Figure 17: The Compressive Strength of Mortar Specimens Mixed with SAP.

3.3 Permeability or Water Flow Rate Test

The results of the permeability or water flow rate tests for each specimen were plotted on a graph, as presented in Figure 18. These tests were conducted on the initial day of crack formation, as well as on days 4, 8, and 18 during the healing period. Gradually decreasing water flow rates were observed, indicating internal healing along the crack surfaces.

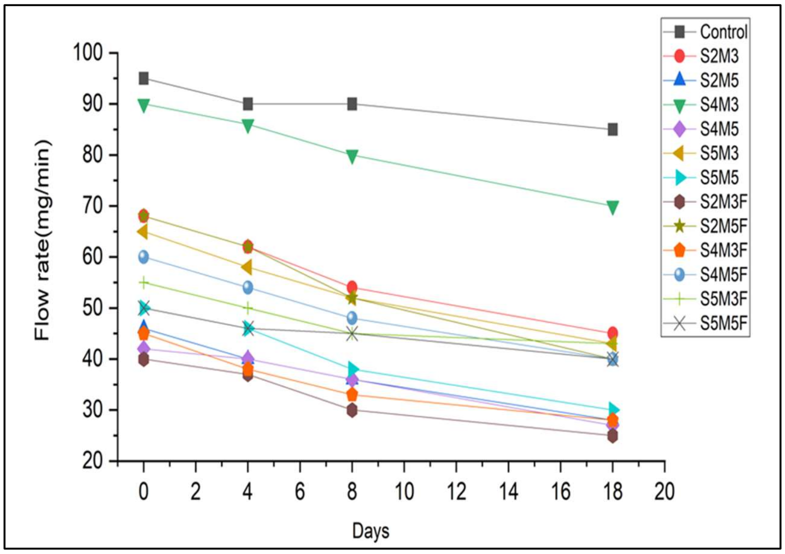

Figure 18: The results of the Water Flow Rate Test Showcase the reduction in water flow as a sign of internal healing within the cracks.

3.4 Compressive Strength of Internally Cured Cubic Specimen

Figure 19 illustrates the compressive strength of internally cured cubic specimens. It is evident from the column graph that the Control specimen exhibited a minimal increase in compressive strength, even displaying a decrease by day 14. Conversely, specimens with varying percentages of SAP demonstrated a gradual and consistent increase in compressive strength, surpassing the strength of the Control specimen. This highlights the role of SAP as an effective internal curing agent, contributing to the enhanced strength of internally cured specimens.

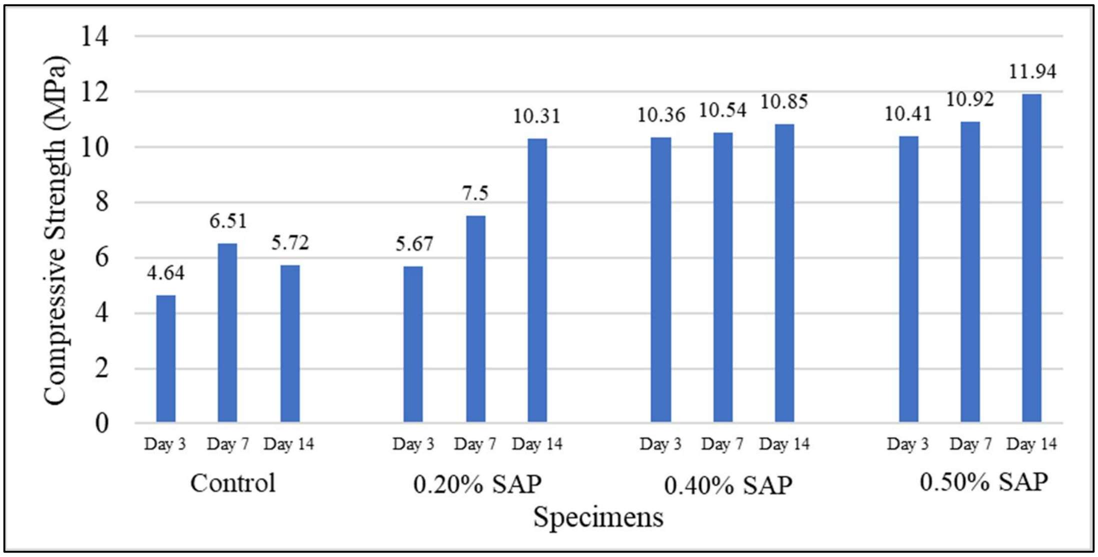

Figure 19: The Compressive Strength Test Results for Internally Cured Cubic Specimens.

3.5 Dry Shrinkage Test of SAP Mixed Mortar Bar Specimen

Figure 20 showcases the dry shrinkage rates of the specimens. Notably, the Control specimen exhibited a higher shrinkage rate compared to the specimens containing SAP. Among the SAP specimens, the one with 0.5% SAP displayed the lowest shrinkage rate. Within a span of two weeks, the shrinkage rate became consistent across all specimens. This data emphasizes the potential utility of SAP in mitigating dry shrinkage in concrete or mortar specimens. The measurements were taken continuously over a 14-day period.

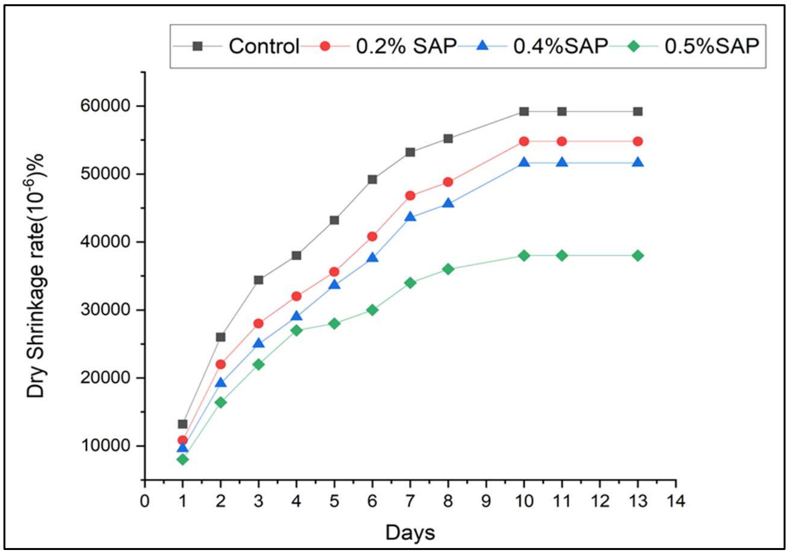

Figure 20: The Dry Shrinkage Rates of SAP Mixed Mortar Bar Specimens.

4. CONCLUSIONS

This study aimed to investigate the self-healing potential of mortar mixtures incorporating Super Absorbent Polymer (SAP) and nano-silica. Thirteen distinct mortar combinations, including a control, were prepared to examine the effects of these additives comprehensively. The self-healing efficacy was evaluated through microscopic observations, water permeability tests, and compressive strength measurements under wet-dry cycles. The following conclusions were drawn:

- Microscopic analysis revealed that cracks with widths equal to or less than 150µm demonstrated healing through SAP swelling. Although direct crack width measurements were limited, a comparison with Control specimen cracks indicated closure in SAP-treated specimens after 28 days, which was absent in the Control.
- Water permeability tests highlighted the immediate sealing effect of SAP-containing samples compared to the Control. Reduced water leakage from the healed cracks indicated ongoing self-healing in all series.
- Incorporating super-absorbent polymers led to reduced compressive strength due to macro-pore formation. Conversely, nano-silica improved mechanical properties due to its pozzolanic behaviour. Combined, the additions had no significant impact on mechanical performance, making this material viable for construction.
- Internal curing using SAPs resulted in increased compressive strength for internally cured cubic specimens over days 3, 7, and 14. Control specimens experienced strength loss due to the absence of internal curing.
- Dry shrinkage testing demonstrated that specimens with higher SAP percentages exhibited lower dry shrinkage ratios than the Control, suggesting SAP's potential to mitigate autogenous cracking.

Further investigations should explore the self-healing mechanism and the impact of SAPs on mortar shrinkage. Additional studies are warranted to determine the optimal dosage of super-absorbent polymers in concrete formulations. Utilizing a robust portable Crack-width Microscope is advisable for precise crack-width measurements and extensive micro-scale research. Given the sensitivity and time dependency of this research, along with its practical application in construction, more comprehensive and effective studies are essential. To ensure accurate dry shrinkage testing, a controlled environment maintaining constant temperature and relative humidity is crucial.

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