

Experimental Investigation on Self-Healing Concrete, Mechanical and Thermal Studies

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ABSTRACT

Self-healing concrete offers a durable solution for addressing cracks by forming calcium carbonate crystals that close microcracks autonomously. The research, conducted in five phases, begins by evaluating two super absorbent polymers (SAP) for mechanical recovery, including compressive and flexural strength, alongside crack observation, thermal analysis (TGA, DSC), and microstructural characterization. In the fourth phase, the optimal dosage of Polyvinyl Alcohol (PVA) is set at 1% with nano-silica added at 4 wt% of cement, enhancing concrete properties. Flexural behavior under four-point bending is tested for reinforced self-healing versus conventional beams. In the final phase, nano-silica is reduced to 1 wt%, with rice husk ash and sodium bentonite added for cost-efficiency, optimizing PVA at 1.5 wt%..

Keywords: Self-Healing Concrete, Polyvinyl Alcohol, Thermogravimetry Analysis, Differential Scanning Calorimetry

1. INTRODUCTION

Concrete is a durable, economical, and widely used construction material known for its compressive strength and versatility. Made from Portland cement, aggregates, and water, concrete undergoes a hydration reaction, forming a stone-like mass that binds materials together. However, its production is energy-intensive, generating 0.85 to 1.1 tons of CO₂ per ton of cement and contributing to roughly 7% of global CO₂ emissions. To improve its properties and sustainability, mineral and synthetic admixtures are added, enhancing strength, water resistance, and setting time. Despite its durability, concrete has a relatively low lifespan, leading to ongoing issues with sustainability and maintenance requirements. Cracking is a common issue in concrete structures, affecting both aesthetics and durability. Cracks can develop at any stage, compromising the structure's strength and allowing harmful substances to penetrate. The porous nature of concrete also allows for easier infiltration of water and chemicals, which can degrade concrete and corrode reinforcement over time. Fatigue, excessive loads, and environmental factors like air and water voids, improper curing, and erosion all contribute to crack formation. Crack movement under shear stress from traffic loads further accelerates deterioration at the fracture surface.

Cracks often lead to structural damage, corrosion, and chemical degradation, undermining the concrete's strength. Routine maintenance and repairs are costly, and these issues have raised the need for self-healing concrete, which could autonomously repair cracks and mitigate the high expenses of traditional maintenance. Self-healing concrete (SHC) refers to concrete materials that can autonomously repair their own microcracks, similar to a self-healing biological system. This innovative material reacts to moisture interacting with unhydrated cement particles, facilitating crack closure primarily through the formation of calcium carbonate. The self-healing process involves hydration of unreacted cement particles and crack filling as calcium carbonate precipitates. SHC typically includes water, super-absorbent polymers (SAP), Portland cement, sand, and gravel. When moisture enters a crack, the SAP swells, promoting rehydration of nearby cement particles over approximately 28 days. This self-repair process enhances concrete's durability, reduces CO₂ emissions, and increases sustainability. There are two types of self-healing: autogenic, which uses inherent materials without additives, and autonomic,

which involves engineered additives. Nano-silica further enhances the hydration process by densifying the concrete microstructure and promoting calcium silicate hydrate formation, which boosts strength.

Three main types of SHC have been identified: natural self-healing, chemical-based self-healing, and self-repair through biological means (e.g., bio-concrete). These processes help prolong concrete life, minimize maintenance costs, and improve structural stability, especially in reducing crack-induced degradation. Super-Absorbent Polymers (SAPs) are specialized polymers capable of absorbing large amounts of liquid—sometimes up to several times their weight. These "smart" materials expand upon water exposure and shrink when dry, making them valuable in concrete applications where they help manage moisture. Concrete production is energy-intensive, with cracks forming over time, often going undetected until costly repairs are required. To address this, a self-healing mechanism is introduced in concrete that uses calcium carbonate formation to seal microcracks, reducing repair costs and environmental impact. This approach not only enhances the structure's durability but also supports global sustainability efforts by lowering greenhouse gas emissions.

The study aims to identify an effective super-absorbent polymer as a self-healing admixture in concrete. It also seeks to partially replace cement with additives such as nano-silica, rice husk ash, and sodium bentonite to enhance performance. For this purpose, M40 grade concrete mixes will be designed according to IS 10262:2012, incorporating varying doses of self-healing admixture from 0 to 2.5% by cement weight. The research will assess the recovery of mechanical properties—compression, split-tension, and flexure—after the concrete undergoes self-healing of induced cracks. Flexural behavior in terms of load versus deflection for reinforced concrete beams will also be analyzed. Additionally, the study will measure hydration levels of concrete samples through thermogravimetric analysis, while microstructural properties will be examined using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS). To evaluate crack closure in concrete samples, digital image processing will be performed using MATLAB. Ultimately, the research aims to recommend an optimal dosage of self-healing admixture by weight of cement for effective self-healing properties in concrete.

2. LITERATURE REVIEW

The literature review covers recent studies on various self-healing methods, self-repairing agents, pozzolanic admixtures, microstructural characterization, and applications of materials like sodium silicate, polyvinyl alcohol, and nano-silica in concrete. Self-healing concrete is recognized for its benefits, including reduced CO₂ emissions, cost savings, and enhanced durability.

Huseien et al. (2019) highlighted self-healing concretes' environmental advantages, while Van Tittelboom and De Belie (2013) found that cracks up to 300 microns could self-heal. Research by Wang et al. (2019) indicated that replacing cementitious materials might reduce portlandite, affecting self-healing potential. Studies also show that self-healing mechanisms, such as calcium carbonate formation and hydration, help seal microcracks. The review explores the role of self-healing agents, including geomaterials and microcapsule-based polymers, which facilitate crack repair through expansion or interaction with water.

Ahn and Kishi (2010) found that silica-rich geomaterials help in water absorption and crack sealing. Additionally, crystalline chemical admixtures, such as sodium silicate and sodium carbonate, react with concrete to form sealing products like C-S-H gel. Bacterial agents are also noted for their crack healing capabilities but face challenges under extreme temperatures. Microcapsule-based agents and encapsulated microorganisms show potential for improving crack closure by allowing controlled release upon crack formation. Pozzolanic admixtures, like fly ash, silica fume, and blast furnace slag, enhance the self-healing properties of concrete by promoting C-S-H formation.

Studies by Qureshi et al. (2018) and Ache et al. (2016) demonstrated that materials like magnesium oxide and bentonite could self-heal wider cracks. Microstructural analysis techniques, such as scanning electron microscopy (SEM), thermogravimetric analysis (TGA), and mercury intrusion porosimetry (MIP), have provided insights into the pore structure, hydration levels, and mechanical properties of self-healing concrete.

Research by Zhou et al. (2016) and Ho et al. (2018) used these techniques to analyze the porosity and durability improvements in concrete with recycled aggregates.

Polyvinyl alcohol (PVA) has shown promise in enhancing concrete's durability, particularly in fiber-reinforced forms, as noted by Sclofani and Contrafatto (2013). Studies suggest that PVA fibers improve concrete's interfacial bond, creating a more durable matrix and aiding in self-healing through hydrophilic interactions.

Sodium silicate, identified as an effective self-healing agent by Li et al. (2019), releases upon crack formation to form C-S-H gel, sealing cracks.

Additionally, research by Shaikh and Joshi (2015) and Prabahar et al. (2017) demonstrated that incorporating sodium silicate improves long-term durability by protecting reinforcing bars from corrosion.

Applications of pozzolanic materials, such as nano-silica and rice husk ash, have also been explored for their self-healing

potential and enhanced concrete strength. Meddah et al. (2020) emphasized the benefits of nanoparticles like TiO₂ and Fe₂O₃ for strengthening concrete. Nano-silica's high surface area and reactivity improve the concrete matrix by filling pores and reinforcing the interfacial transition zone.

Researchers like Raman et al. (2011) and Singh and Rai (2001) have also studied rice husk ash, which, through pozzolanic reactions, enhances compressive strength and reduces porosity, further supporting self-healing properties in concrete.

3. MATERIALS AND METHODS

3.3.1 Cement

Portland Pozzolana Cement (PPC), based on fly ash, serves as a binding agent in concrete. Key properties based on BIS standards, including a standard consistency of 32%, initial and final setting times of 145 and 350 minutes respectively, fineness of 2%, specific gravity of 3.15, and compressive strengths of 16, 22, and 33 MPa at 3, 7, and 28 days.

3.3.2 Fine Aggregate

Manufactured sand (M-Sand) with a fineness modulus of 3.06 was used as the fine aggregate. After cleaning, screening, and sieving as per IS 383-2016 standards, M-sand had a specific gravity of 2.6 and water absorption of 3.34%.

3.3.3 Coarse Aggregate

Coarse aggregate, primarily crushed stones, complies with IS 383-2016 standards. It has a maximum size of 20 mm, specific gravity of 2.73, and water absorption of 0.35%, with crushing and impact values of 20% and 8.82%, respectively.

3.3.4 Super Absorbent Polymers (SAP)

SAPs function as hydrogels, absorbing water to aid the hydration process. Acting as self-healing agents, they prevent crack propagation and contribute to durable concrete structures. Sodium silicate and polyvinyl alcohol (PVA) were used as self-healing agents in this study.

3.3.5 Water

Potable soft water with a pH of at least 6, conforming to IS 456:2000, was used in concrete production and curing.

3.3.6 Rice Husk Ash (RHA)

RHA, a byproduct of rice husk combustion, contains high reactive silica. RHA is pozzolanic and enhances concrete strength. Pulverized RHA has specific gravity of 0.45, particle size distribution with a maximum size of 45 μ m, and significant silica content (92.26%). A 10% replacement of OPC with RHA accelerates setting and improves strength.

3.3.7 Expansive Clay

Sodium bentonite, an expansive clay, is used to enhance the mechanical strength and durability of concrete. Properties include silica content of 0.4-0.45%, magnesia 0.06-0.07%, and swelling capacity of 5 cm³ in 24 hours. It acts as a partial substitute for cement, increasing durability under specific conditions.

3.3.8 Nano-Silica

Nano-silica improves concrete's density and compressive strength. Studies show adding 3-5% nano-silica enhances strength and reduces permeability. It's a high-cost material but significantly improves performance under severe environmental conditions.

Phases of Experimental Investigation

Phase I: Self-Healing Concrete Using Sodium Silicate

M40 grade control concrete and superabsorbent polymer (SAP) concrete were mixed according to IS 10262:2009, with ratios of 1:1.80:2.93 for control and 1:1.76:2.91 for SAP. Sodium silicate (2% by weight) was incorporated. A total of 45 specimens (15 cubes, 15 cylinders, 15 prisms) were cured and tested for compressive, split tensile, and flexural strengths on the 7th, 14th, 28th, and 56th days. Pre-cracks were induced on the 28th day, followed by 28 days of healing to observe self-healing capabilities.

Phase II: Self-Healing Concrete Using PVA (1%)

Control and polymer-modified concrete (PMC) were formulated to achieve a target compressive strength of 40 MPa at 28 days, using 1% PVA. A total of 24 specimens were prepared, and compressive strength tests were performed on the 7th, 21st, and 28th days. Pre-cracked samples were allowed to heal for 28 days, with microstructural analysis conducted using SEM and XRD on samples that failed on day 56.

Phase III: Self-Healing Concrete Using PVA (1.5%)

In this phase, PVA was added at 1.5% of cement weight. Similar testing and pre-cracking procedures were followed as in Phase II, with additional strength tests on the 56th day to evaluate healing effectiveness.

4. EXPERIMENTAL INVESTIGATION

4.1 INTRODUCTION

This section focuses on the usefulness and mechanical properties of new concrete through various tests: compressive, split tensile, and flexural strength tests, along with the four-point bending test for reinforced concrete beams. The study also includes crack analysis using MATLAB, microstructural evaluation via Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD), and thermal analysis through Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC).

4.2 WORKABILITY TEST

The workability of fresh concrete is assessed using the slump cone test, following IS 7320: 1974. The slump cone, a 300 mm-high frustum of a cone with a 200 mm base and a 100 mm top, is filled with concrete in three layers, each compacted with a standard 16 mm steel rod. The cone is then lifted, and the slump is measured as the vertical drop from the top of the cone to the highest point of the slumped concrete, recorded to the nearest 5 mm.

4.3 PROPERTIES OF HARDENED CONCRETE

The mechanical properties of hardened concrete are determined through compressive, split tensile, and flexural strength tests. Each test involves three samples for hydration ages of 7, 14, and 21 days, with six samples collected for testing between 28 and 56 days.

4.3.1 Compressive Strength Test

Conducted according to IS 516 (1959), this test determines the material's ability to withstand compressive loads. Concrete samples are typically 150 mm cubes, and the compressive strength is calculated using the formula:

 $F_c = P/A$

Where:

P = maximum load (kN),

A = cross-sectional area (mm²)

The test is performed by applying a load at a rate of 14 N/mm² per minute using a compression testing machine.

4.3.2 Split Tensile Strength Test

This test measures the resistance of concrete to tensile stress, following IS 5816 (1999). The specimen is a cylinder, and the load is applied using a tensile testing machine at a rate of 1.2 to 2.4 N/(mm/min). The split tensile strength is calculated using:

 $f_{cr} = 2P \: / \: \pi \: L \: d$

Where:

P = maximum load (kN), L= length of cylinder (mm), d= diameter of cylinder (mm)

4.3.3 Flexural Strength Test

According to IS 516 (1959), this test determines the flexural strength of concrete. The sample is supported on two rollers, and a load is applied at the center until failure occurs. The flexural strength is calculated using:

Flexural strength = PL/bd²

Where:

P = maximum load (kN), L = length of prism (mm), b= breadth of prism (mm), d = depth of prism (mm)

Sample dimensions are $100 \text{ mm} \times 100 \text{ mm} \times 700 \text{ mm}$.

4.3.4 Restrained Flexure Strength

Modified concrete prisms ($100 \text{ mm} \times 100 \text{ mm} \times 500 \text{ mm}$) are restrained using steel plates and a central bar. After curing for 7 and 28 days, flexural strength is tested using a four-point bending test at a rate of 1.4 kN/s. The specimens are submerged for an additional 28 days post-cracking before testing their recovery strength, comparing them with control samples.

5. RESULTS AND DISCUSSIONS

5.1 PHASE I: SELF-HEALING CONCRETE USING SODIUM SILICATE

5.1.1 Workability

The slump cone test results indicate that the control concrete (CC) exhibited a slump of 115 mm, calculated as 300 mm minus 185 mm. In contrast, the Very Permeable Polymer Concrete (SAPC) showed a slump of 82 mm, calculated as 300 mm minus 218 mm. This demonstrates that SAPC is less workable compared to CC, as sodium silicate tends to form a gel by absorbing mixing water.

5.1.2 Mechanical Properties

The mechanical properties, including compressive, split tensile, and flexural strengths, were evaluated at 7, 14, 28, and 56 days. The compressive, split tensile, and flexural strengths of SAPC were found to be slightly lower than those of CC at 7, 14, and 28 days, primarily due to the slower rate of hydration and the presence of more voids caused by sodium silicate in SAPC. This slower hydration resulted in a higher relative presence of unhydrated concrete particles compared to CC. However, by the 56th day, the properties of the SAPC specimens improved compared to CC, indicating that the hydration process between unhydrated concrete particles and water continued effectively in SAPC. Furthermore, the strengths of the pre-cracked SAPC specimens were higher than those of the CC specimens at the 56th day, attributed to increased calcium hydroxide formation in the cracked regions of SAPC.

5.1.3 Determination of Degree of Hydration

The results of TGA, detailing weight loss percentages and peak temperatures for both concrete types. For control concrete (CC), the degree of hydration was 27.5%, while for self-healing concrete (SHC), it was significantly higher at 45.3%. This indicates that SHC had 65% higher hydration levels than CC. The moisture loss during TGA showed that PVA absorbed more moisture through shrinkage cracks in SHC than in CC. The presence of more unhydrated particles in SHC suggests an enhanced self-healing capacity. The higher degree of crystallization of calcium hydroxide in SHC correlates with more effective healing of microcracks compared to CC. The data indicate that SHC exhibited a 78% greater heat flow during dehydroxylation, leading to more effective crack healing due to increased hydration products.

PHASE II: SELF-HEALING CONCRETE USING POLYVINYL ALCOHOL AT 1.5%

5.2.1 Workability

The workability of the concrete was measured using the slump cone test. Results indicated that the workability of the polymer-modified concrete (PMC) was lower than that of the control concrete (CC) due to PVA's absorption properties.

5.2.2 Compressive Strength Test

The compressive strength was evaluated at various ages using 15 cm³ cubical molds. PMC reached 80%, 65%, and 95% of CC strength on days 7, 21, and 28, respectively. Initially, the presence of PVA led to slower hydration and lower strength due to its high porosity, capturing cement particles. After 28 days, PMC exhibited 5% greater strength than CC due to better hydration response during the self-healing phase in fig 4.

5.2.3 Self-Healing Efficiency

The self-healing efficiency was calculated, showing PMC at 118% and CC at 87%, with PMC outperforming CC by 35% post-healing.

5.2.4 Formation and Healing of Micro Cracks

Micro and macro cracks were formed in both mixes before loading. The induced cracks occurred at the same pre-cracking stress, with visual evidence of crack formation and healing documented.

6. CONCLUSIONS

In Phase I of the study, it was found that the mechanical properties of superabsorbent polymer (SAP) concrete improved significantly compared to control concrete after 28 days of self-healing, specifically at the 56-day mark. The self-healing efficiency of the mechanical properties in SAP concrete surpassed that of the control sample, indicating that the self-healing mechanism was activated by the addition of sodium silicate as SAP. Moving to Phase II, analysis utilizing TGA, DTG, and DSC revealed that the self-healing capacity of control concrete (CC) was substantially lower than that of self-healing concrete (SHC) of the same grade (M40). The incorporation of polyvinyl alcohol (PVA) into the concrete further enhanced the independent self-healing capability of SHC, with increased compressive strength observed in later stages after 28 days. In Phase III, the introduction of SAP significantly improved the performance of both fresh and hardened concrete. SAP contributed to the quality of fresh concrete by enabling the gels formed during the curing process to act as treatments for cement particles and aggregates, thus facilitating the internal curing process and minimizing autogenous shrinkage. This

delayed the final setting time in polymer-modified concrete (PMC). Additionally, higher porosity in younger concrete led to a more pronounced reduction in mechanical strength after the addition of SAP, while reduced porosity facilitated internal curing, resulting in strength gain at later ages due to gradual hydration.

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