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Mechanical and microstructural properties of self-healing concrete based on Hay Bacillus

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Abstract. The experimental investigation delves into assessing the influence of varying ratios of calcite (Cc) and sand on the mechanical and microstructural characteristics of self-healing concrete (SHC). This study employs Hay Bacillus as a catalyst for initiating calcite precipitation within the concrete matrix. The proportions of calcite under scrutiny encompass 5%, 10%, and 15% of the cement's weight. Additionally, two distinct types of sand, crushed stone sand (CSS) and river sand (RS) are juxtaposed for comparative analysis. The primary focus of this research is on evaluating the compressive and flexural strengths of the SHC, with particular emphasis on the utilization of a 10% bacterial solution. This proportion emerged as the optimal dosage for enhancing concrete strength. To gain a comprehensive understanding of the underlying mechanisms, the microstructure of the concrete is probed through scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques. These tests allow elucidating the impact of varying calcite and sand ratios on the formation of calcium lactate, as well as the production of calcium silicate hydrate (CSH) gel and non-expanding ettringite within the concrete matrix. This investigation contributes valuable insights into the development of self-healing concrete with improved mechanical properties, underpinned by a deeper comprehension of its microstructural transformations.

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1. Introduction

Concrete, apart from liquids, is one of the most widely used materials on the planet. However, it is susceptible to developing small cracks and holes, which can serve as pathways for water and harmful substances, resulting in reinforcement rust and concrete deterioration. It can lead to a loss of concrete strength and durability, resulting in significant expenses for repairing and maintaining concrete structures globally. Although several methods exist for repairing cracks, most conventional restoration techniques involve chemicals, require significant labor, are costly, and pose environmental and health risks. Recently, a new, efficient method for repairing microcracks and pores in concrete using microbiologically induced calcium carbonate precipitation has been proposed [1–3]. This bacterial remediation method is superior to

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other methods because it is bio-based, environmentally friendly, cost-effective, and sustainable [4–8]. Moreover, it was found that locally isolated Bacillus Sonorensis bacteria from Iraqi soil samples, with their high urease-producing abilities, can be used to increase cementation between soil particles and improve the undrained shear strength of soft clay soil by utilizing the MICP technique. This sustainable technique can be used for biocementation in the construction industry [9].

Recent discoveries have revealed that bacteria that can produce the enzyme urease can significantly impact CaCO₃ precipitation. The enzyme facilitates the hydrolysis of urea to produce CO₂ and ammonia, which increases pH and promotes the precipitation of CaCO₃ in bacterial settings. This innovative and eco-friendly technique has been successfully used to repair concrete cracks and prevent channel leaching. Bacillus pasteurii and Bacillus sphaericus are highly effective in inducing CaCO₃ precipitation, remediating concrete cracks, and improving compressive strength. These findings hold significant potential for developing new approaches for repairing and strengthening concrete specimens, the durability of concrete specimens treated with Bacillus pasteurii and subjected to alkaline, sulfate, and freeze-thaw environments improved [13].

Strength is one of the most important characteristics of concrete. It can be defined as the ability of concrete to withstand loads [14]. Concrete can be accepted or rejected for structure use based on its strength properties. High strength in concrete is desirable for many applications, such as bridges, tall buildings, and other massive structures. High-strength concrete can be made by adding admixtures [15]. The addition of any material to concrete should not hamper its strength. Another crucial factor in concrete construction is durability. The ability of a concrete structure to last for an extended period without significant deterioration is referred to as its durability. Because of its longevity, a durable construction helps conserve resources, as there is no need to produce new building materials [16].

Additionally, it lessens the production of building trash, thus decreasing environmental pollutants. From an economic point of view, it lowers repair and maintenance costs. High strength alone does not guarantee the durability of concrete [4]. Concrete contains microcracks and pores, which increase the material's permeability. The increased permeability allows the passage of water and other substances. Water entry leads to reinforcement corrosion, which is the primary cause of deterioration and loss of durability in concrete [17].

The bacteria, capable of healing apertures and micro-blows in concrete by precipitating Cc, may be used for the bioremediation of concrete. These bacteria can attract Ca²⁺ from the surrounding environment and produce a urease enzyme. This urease enzyme catalyzes urea and produces carbon dioxide and ammonia. This process causes a rise in pH and Ca²⁺, which combine with carbon dioxide to form Cc. The bacteria for use in concrete should be capable of creating endospores. Endospores can withstand the harsh concrete environment and remain dormant for up to [12, 18]. The formation of cracks, water, and air seeps inside the concrete, creating a favorable environment for the endospores to germinate. After germination, the bacteria will start precipitating C_c to seal the pores and cracks in the concrete. Adding cementitious materials made from byproducts to concrete provides manifold advantages as it supports ecological and energy maintenance and advances the strength and toughness of concrete [14].

The cost of SHC, when compared to predictable concrete, would be high. The substrate for growing bacteria is the primary factor contributing to the bacterial treatment's cost. The inclusion of cheaper substrates will thus reduce the cost of SHC. The SHC will also lead to the economy over the long run because of reduced costs for the repair of cracks and maintenance [19]. Microbial mineral precipitation arising from the metabolic operations of beneficial concrete microorganisms has recently enhanced the general concrete behavior. The procedure may be carried out within or outside the microbial cell or within the concrete uniform some distance away. Bacterial operations often merely cause a shift in the chemistry of solutions that mains to over-saturation and precipitation of minerals. The use of these ideas of biomineralogy in concrete mains to the possible creation of a fresh solid named SHC.

2. Materials and Methods

2.1. Materials

2.1.1. Cement

53-grade Portland cement cast-offs in this research. This Portland cement was verified as per IS 4031-1996 [20], and the physical belongings are presented in Table 1.

Table 1. Properties of Portland cement.

Property	Specific gravity	Blaine's fineness	Soundness	7-d CS	28-d CS
Value	3.02	283 m²/kg	2.2 mm	42 MPa	55 MPa

2.1.2. Fine aggregates

Resident-offered crushed stone sand (CSS) and river sand (RS) are utilized as fine aggregates. For the fine aggregate, the distribution of the gradation curve is presented in Figure 1. The specific gravities of CSS and RS are 2.76 and 2.67, respectively.



Figure 1. Grading curve of fine aggregate.

2.1.3. Coarse aggregates

Wrinkled granite cracked stones of nominal size 20 mm are cast off as coarse aggregate. Table 2 presents the properties of coarse aggregate. The grading curve of coarse aggregate is illustrated in Figure 2.



Table 2. Test Properties of coarse aggregate.



2.1.4. Culturing of Hay Bacillus

Refined Hay Bacillus microscopic organisms were used to achieve the aim of the research. Ca(LaC)₂ was utilized with the bacteria Hay Bacillus and nutritious broth. It is available in powder form with white coloring. 500ml of distilled water, 2.5 gm of peptone, 2.5 gm of sodium chloride, and 1.5 gm of beef extract concentrate were combined to create the nutritious broth. A silver thimble and a cotton plug for security protected this flask. Afterward, the arrangement was preserved in an autoclave for 20 minutes at 121 °C and 150 lbs. The arrangement was orange in color, and the clarity of pollution after this treatment. The cup opened in the laminar wind current chamber, and the Hay Bacillus were introduced to this configuration. Afterward, the arrangement hatched in an orbital shaker at 125 rpm and 37 °C. This arrangement turned into white-yellow turbidity after one day, as presented in Figure 3, illustrating the development of Hay Bacillus.



Figure 3. Culturing of Hay Bacillus.

2.1.5. Calcium lactate Ca(LaC)₂

Calcium lactate, also known as Ca(LaC)₂, was used along with Hay Bacillus as nutrient broth, as shown in Figure 4, to ensure successful bacterial growth and to obtain accurate and reliable experimental results.



Figure 4. Calcium lactate and Hay Bacillus.

2.2. Methods

2.2.1. Mix Design

The mix proportions for M40-grade concrete are designed using IS 10262-2009 [21]. Resources essential per one m³ of concrete are presented in Table 3.

Type of Fine aggregate	Mixture Designation	Cement (kg/m ³)	RS (kg/m³)	CSS (kg/m ³)	Coarse Aggregate (kg/m ³)	w/c ratio	Bacterial Cells (CFU/ml)	Percent of bacterial- sol
River sand (RS)	RSHC00			-	1261	0.45	105	00
	RSHC05		642	-				05
	RSHC10			-				10
	RSHC15			-				15
Crushed sandstone (CSS)	CSHC00	390	-					00
	CSHC05		-	642				05
	CSHC10		-					10
	CSHC15		-					15

Table 3. The volume of materials to one cum of concrete.

2.2.2. Samples preparation and test methods

2.2.2.1. Samples preparation

Before placing concrete, the cast iron molds of the cylinder, cube, and prism are entirely cleaned and oiled on the inside surfaces. The molds are filled with evenly mixed SHC.

2.2.2.2. Compressive strength (CS) test

According to IS 516-1959 [22] requirements, CSs on SHC specimens with 150×150×150 mm dimensions were performed. According to IS 456-2000 [23] criteria, these specimens were produced and cured at 7, 14, 28, and 90-d.

2.2.2.3. Flexural strength (FS) test

According to IS 516-1959 [22], concrete prism specimens with bacteria were subjected to an FS.

2.2.2.4. Microstructural analysis test

For better visualization, SEM analyses are done on the SHC aimed at which the concrete is made into powder by crushing it into small pieces. The powdered specimen is placed on nerve ends with the help of carbon tape, and before they are analyzed at 20 kV, they are coated with gold. As is customary, the specimens are tested after curing for 28-d.

3. Results and Discussion

3.1. Compressive strength (CS)

The results of the M40 grade concrete experiment of RS and CSS mixtures show the influence of bacteria on the compressive strength of the modified concrete, as shown in Figure 5. The obtained data indicate that the sand mixtures' compressive strength is higher than that of all ages of RS mixtures, irrespective of the amount of bacterial sol. This can be attributed to the fact that CSS, which is cubic and has sharp edges, helps to achieve higher compressive strength than RS mixtures.

The CS of the control mixture concrete was compared with the CS of SHC-5%, SHC-10%, and SHC-15% at 28-d and 90-d. The results showed that the percentage increase in CS for SHC-5%, SHC-10%, and SHC-15% at 28-d was 8.98%, 17.02%, and 4.65%, respectively. Similarly, CS at 90-d was found to increase by 8.49%, 12.27%, and 2.59%, respectively.

In the CSS mixtures, the CS at 28-d for SHC-5%, SHC-10%, and SHC-15% increased by 6.94%, 14%, and 2.28%, respectively. While at 90-d, the percentage increase in CS of the CSS mixture was 9.0%, 14.9%, and 3.09%, respectively. Furthermore, it was observed that the increase in CS at 90-d was higher than the increase at 28-d. It is justified by the role of hay-Bacillus bacteria and Ca(LaC)₂ in enhancing CS at ages beyond 28-d for both RS and CSS mixtures [24].

The CS was found to increase as the percentage of bacterial sol in concrete increased from 0% to 10%, but at 15%, it decreased. This is because the hydration products were saturated at 10% bacterial sol, and further increase in bacterial sol did not enhance the strength, resulting in a reduction in CS.

The bacteria used in this technology are typically spore-forming, which means that they remain dormant until activated by moisture. When the concrete cracks and water enters, the bacteria are activated and start to produce calcium carbonate, which fills in the cracks and restores the strength of the concrete. However, when the bacterial solution is added to the concrete in concentrations higher than 10%, it can have adverse effects on the compressive strength of the concrete. This is because the bacteria consume some of the nutrients in the concrete mixture, which can weaken the concrete matrix and reduce its strength. Additionally, higher concentrations of bacterial solution can also increase the water content of the concrete, which can negatively affect its strength, which are other factors justifies the decreases of the CS at concentrations higher than 10%

Moreover, Figure 6 (a and b) shows the relationship between the volume of the hay-Bacillus bacteria solution and compressive strength with River sand (RS) and Crushed sandstone (CSS), respectively. Strong R2 values were found for SHC samples with CSS more than RS.



Figure 5. Result of compressive strength of SHC.





(b)

Figure 6. Relationship between the volume of hay-Bacillus bacteria-solution and compressive strength with (a) River sand (RS) and (b) Crushed sandstone (CSS).

2.3. Flexural strength (FS)

Figure 7 shows how the FS of SHC varies with curing time for mixtures made from RS and CSS. As the curing age of SHC grows, so does its FS. According to these findings, CSS mixtures have stronger FS than RS mixtures at all ages, regardless of the amount of bacterial sol. At 28-d, the percentage increases for RS mixtures with SHC-5%, SHC-10%, and SHC-15% are 4.97%, 9.04%, and 2.26%, respectively. Like this, at 90-d, the percentage increase in FS is 5.5%, 12.71%, and 2.54%, respectively. The percentage increases in FS for CSS mixtures at 28-d for SHC-5%, SHC-10%, and SHC-15% are 3.8%, 6.84%, and 1.14%, respectively. Like this, at 90-d, the FS percentage increase was 4.65%, 10.03%, and 1.43%, respectively. Additionally, as the percentage of SHC increased from 0% to 10%, the FS also increased. However, at 15%, the FS decreased due to the saturation of the hydration products with 10% bacterial-sol, which prevents further increases in bacterial-sol from increasing FS [25].

Moreover, Figure 8 (a and b) shows the relationship between the volume of hay-Bacillus bacteria solution and flexural strength with River sand (RS) and Crushed sandstone (CSS), respectively. Strong R2 values were found for SHC samples with CSS more than RS. Besides, Figure 9 shows a relationship between compressive strength and flexural strength. R2 indicates a strong relationship between them in both RS and CCS.



Figure 7. Result of flexural strength of SHC.



Figure 8. Relationship between the volume of hay-Bacillus bacteria-solution and flexural strength with: (a) River sand (RS) and (b) Crushed sandstone (CSS)



Figure 9. Relationship between compressive strength and flexural strength with: (a) River sand (RS) and (b) Crushed sandstone (CSS).

3.2. Microstructural analysis

3.3.1. Scanning electron microscope (SEM)

SEM analyses are used to imagine the occurrence of Cc crystals precipitated by bacteria inside concrete specimens.

The findings of the SEM analyses for regular concrete are considered at different amplifications. Subacceleration voltage and pixel sizes have been presented in Figures 10 and 11, which show the microstructure of normal concrete. SHC analyses are carried out in a similar setting. SEM analyses demonstrated the emergence of Cc prepetition in the SHC. It shows that the reduction in permeability and rise in the strength and curing of crashes in concrete are due to the formation of Cc [7,8]. In contrast, pores, calcium silicate hydrate (CSH), and CH, have occurred in almost all the specimens.

This method was utilized to investigate bacteria-treated and untreated concrete specimens. The SEM images are displayed in Figures 10 and 11. The concrete specimens had identifiable Cc crystals, according to the SEM analyses. The presence of Cc in the form of CaCO₃ was verified by the high calcium concentrations in all the bacterial specimens. Bacteria acted as nucleation sites during mineralization since crystalline Cc is associated with bacteria.

Calcite crystal growth was examined in both the treated and untreated bacteria specimens. While the matrix of the concrete specimens treated with the microbe seems crystalline, and individual crystals could be distinguished, the untreated specimens (those without bacteria) appear amorphous and show no trace of crystal formation. The degree of crystallization in the treated specimens' matrix is relatively diverse. Concentrations of somewhat large crystals can be found at the boundaries between the sand particles and the matrix. This textural setting implies that preferential crystallization at the concrete-matrix interfaces is likely enhancing the coherence between cement particles and the matrix at the microscale.







Figure 10. SEM Micrograph for SHC with river sand (RS).



Figure 11. SEM Micrograph for SHC with crushed sandstone (CSS).

3.3.2. X-ray diffraction (XRD)

As shown in Figure 12, the crests are seen at various stages when the recordings are obtained at a wavelength of 1.54 Å. From the observed highest peak at the respective theta, the value obtained is 27.914, representing pure Cc. These peaks can be seen for some other specimens, which represent a few other materials like calcium silicate hydrate (CSH), quartz (Q), calcite (Cc), ettringite (E), and Larnite (L). All these results are shown in Figure 12. Compared to the non-bacterial samples, the composition of Cc is visually peaked for bacteria after the quantitative analysis. The existence of amorphous content with the addition of the crystalline phase of Cc, portlandite, and larnite has been shown in the form of a hump in Figure 12.

The presence of CSH and Cc in concrete samples with Bacillus subtilis was identified using XRD analysis. The presence of CSH peaks demonstrates the cause for the strength development of concrete specimens [8]. The presence of Cc peaks will confirm the Cc precipitation by bacteria which is the reason for the increased strength and durability of concrete specimens.

Each crystalline solid has a distinct XRD powder pattern that can be used as a "fingerprint" for identification. XRD is also used to characterize fingerprints and determine the structure of crystalline minerals such as CSH and Cc. Broken cube specimens collected from compressive strength tests and precipitates in cracks of concrete beam specimens were used to evaluate crack healing [26]. XRD analysis was performed on the fraction that passed through a sieve size of 5µm.



Figure 12. XRD diffraction of SHC.

4. Conclusions

The conclusions drawn from this study are as follows:

1. The CS of SHC of Grade M40 is improved with a 10% increase in bacterial sol for both RS and CSS mixtures due to the formation of $Ca(LaC)_2$ in concrete. However, the strength is condensed beyond 10% bacterial sol due to the saturation of hydrated compounds. As a result, it is estimated that 10% of the bacterial sol is used in concrete applications.

2. The FS results also show similar patterns of increase in strength up to 10% bacterial sol at all curing ages.

3. Due to the cubical particles and increased hardness of CSS mixtures, they outperformed RS mixtures in CS and FS. To create SHC for structural usage, CSS can be utilized.

4. After 28-d, all SHC mixtures showed evidence of the creation of Cc, which is CaCO3, according to microstructure analyses.

5. SEM and XRD studies revealed that the enhanced CSH gel and non-expanding ettringite production confirms the improved CS at 10% bacterial sol.

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