Effect of microbial solution on compressive strength, water absorption and sorptivity of cement mortar incorporated with metakaolin

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Abstract: Recent researches in literature have established that the overall life of mortar used in plastering works can be improved by the self-healing processes. Cracking of mortar plasters is a common hurdle due to the intrinsic brittleness of the material. This will result in severe loss of durability and water tightness. There are various methods to prevent this problem, such as adding glass fibers to the mortar mix which prevents shrinkage cracks is one among them and another way to do it is with the help of microorganisms that precipitates calcium carbonate which fills the cracks. But not all kinds of bacteria will precipitate calcium carbonate. Even if it does it should survive under extreme conditions present in mortar. The species called bacillus is a kind which fulfills those conditions is used in this project. The two bacteria namely “Bacillus Subtilis” and “Bacillus Megaterium” were isolated by taking 10g rhizosphere soil and they are cultured. The objective of this project is to compare the productivity of two bacteria. Along with this, a material called metakaolin is also used in combination with bacteria and its efficiency is also checked. The use of metakaolin (kaolinite) in this project increases compressive strength, decreases the heat of hydration which in turn increases the efficiency of calcium carbonate (CaCO₃) precipitation along with bacteria. Mortar cubes are cast in different combinations to observe the compressive strength by Compression Test, healing capacity of mortar through Ultrasonic Pulse Velocity (UPV) Test and the precipitated amount of calcium carbonate via X-Ray Diffraction Test. Also, the durability of mortar cubes has been identified using Water Absorption Test and Sorptivity Test.

Keywords: Bacillus Megaterium, Bacillus Subtilis, Metakaolin, Ultrasonic Pulse Velocity Test, X-Ray Diffraction Test

1. INTRODUCTION

In the field of construction industries, mortar is a broadly used material. It is a mixture of cement, sand and water and mainly used for holding bricks or stones together. Generally, in most of the constructed structures, cracks are formed after plastering which is usually applied at the finishing stage of the work. These cracks are formed due to various factors which include temperature change, shrinkage effect and due to load applied on it etc. Therefore, cracks get easily formed which in turn leads to the reduction in durability. Thus, repairing of cracks becomes a mandatory process and also maintaining frequently is very important. But practically repairing cracks is very costly and also it requires high manpower. The best alternative comes in the idea of self-healing of cracks which can be
achieved using bacteria [1,7,13]. The precursor mineral compound is converted into calcium carbonate mineral by bacteria which is known as limestone [6,18]. Fastening and plugging of the cracks on the crack surface can be done by precipitation of limestone. Self-healing mortar is used to cure cracks that appear on the plastering surface by producing limestone biologically. Specially chosen type of bacteria named genus Bacillus, accompanying Calcium Chloride, a calcium-based nutrient and Urea are added to the mortar when it is being mixed. For about 200 years, these self-healing agents can lie inert within the mortar. Though, the plastering surface would be damaged and water starts to percolate through the crack that occurs in the mortar, the spores of the bacteria develop a contact with the water and nutrients. Calcium Carbonate precipitated by Bacillus is being effective in crack remediation [1,5]. Bacillus is a type of bacteria which acts as a filling material to decrease the pore size of cement paste, mortar and concrete specimens to improve its service life in terms of durability [8,9]. The microorganisms that exist in the soil is used for the sealing of cracks in concrete. The durability of building materials is enhanced by bacterial concrete formation. Since the pH value of mortar is extremely high, the so-called alkaliphilic bacteria are able to last. Bacterial spores can withstand very high mechanical forces and are designated by a long-term viability of up to 200 years even under dry conditions [14]. Oxygen consumption measurements provide evidence for the bacterial activity in concrete materials for several months after concrete casting [15]. Hence by adding calcite-precipitating bacteria, it is possible to create mortar which have self-healing ability and enhance the strength by reducing sorptivity and water absorption capacity. Therefore, the two bacteria Bacillus Megaterium and Bacillus Subtilis was used as they satisfy all the requirements. By using significant amount of Supplementary cementitious materials reduce the cement content thereby limit the material costs also need less energy and CO\textsubscript{2} emission is less than that of cement [10,11]. Addition of metakaolin resulted with lesser micro cracks and shrinkage formation [2,16].

2. EXPERIMENTAL

2.1. Microbial Culture Details

2.1.1 Isolation and Culture of bacteria by Serial Dilution Technique

The bacteria selected for the study should be harmless to humans and should have self-healing property with the ability to resist high alkaline environment. Considering these factors, the bacteria selected for crack remediation is of Bacillus species having cell concentration \(10^5\) cells/ml as shown in figure 1, isolated from the rhizosphere soils of healthy coconut palm growing in Tumkur region of Karnataka. The bacteria were isolated by taking 10g rhizosphere soil in 90 ml sterile water blank, by keeping the flask in a water bath that is at 80°C for 20 mins after thorough mixing and serial dilution had been done. From the 103 and 104 dilutions, the 100μl sample was incubated at 30°C until the colonies of the Bacillus species grew after spreading the sample and then plated it on a nutrient agar plate [22]. Polyphasic identification using BIOLOG and 16srRNA sequencing was carried out to identify the bacteria to species level [23].

![Figure 1 Diluted Bacterial Solutions of 105 cells/ml concentration](image-url)
2.2 Specimen Preparation

The mortar mix comprises of Ordinary Portland Cement according to IS 12269-1987 (grade 53), easily available well-graded river sand according to IS 383-1970 is utilized as fine aggregate. Water used has properties as per IS: 3025 – 1964 part 22, part 23 conforming to standards specified in IS 456-2000 are taken as per the mix calculation. Cement to Sand ratio is 1:3 with a water cement ratio between 0.4 and 0.48, which is identified from the consistency test and the calcium chloride - 50 gram/litre of water and urea-20 gram/litre of water is added directly during the casting process for urease activity. Metakaolin conforms to IS: 1727 (1967) was added to the amount of 10% in replacement with cement. For bacteria integrated mix for different range of bacterial solution 5 ml of 105 cells/ml concentration to total amount of water added includes the particular range of bacterial solution [17, 19, 20]. Different cube samples of size 70.6 mm were casted for the mix proportion as shown in table 1.

|                | Cement (kg) | Fine Aggregate (kg) | Metakaolin (kg) | Water + Bacillus Megaterium (ml) | Water + Bacillus Subtilis (ml) |
|----------------|-------------|---------------------|-----------------|----------------------------------|-------------------------------|
| CM             | 0.232       | 0.696               | -               | 92.8 ± -                         | -                             |
| MG             | 0.232       | 0.696               | -               | 92.8 ± 0.464                    | -                             |
| SB             | 0.232       | 0.696               | -               | 92.8 ± 0.464                    | -                             |
| MGM            | 0.207       | 0.696               | 0.018           | 109.45 ± 0.55                   | -                             |
| SBM            | 0.207       | 0.696               | 0.018           | 109.45 ± 0.55                   | -                             |

2.3 Characterization methods

2.3.1 Compressive Strength

The compressive strength test has been done to determine the compressive strength of mortar cubes of size 70.6mm. All the cubes were tested under dry condition. For each mix proportion, three cubes were tested on 7th, 14th and 28th day using Compressive Testing Machine and the final results have been shown in figure 1. The compressive strength of particular mix cube at failure load has been calculated by dividing the cross-sectional area of cube specimen from ultimate load.

\[ F_c = \frac{P}{A} \]

Where, P- Load in N, A- Cross sectional area in sq. mm

2.3.2 X-ray Diffraction

X-ray diffraction, is a method based on the constructive interference of the monochromatic X-rays and the crystalline sample. Cathode ray tube produces X-ray, which in turn produce monochromatic radiation and is being focused to the sample. By contact of incident rays with the specimen the constructive interference is produced, which satisfy Bragg’s Law: \( n\lambda=2d \sin \theta \) Bragg’s law gives the relation between wavelength of the electromagnet radiation to diffraction angle and the lattice spacing in crystalline samples. These diffracted X-rays are then sensed, processed and measured. Due to random orientation of powdered material, the sample could be scanned at an angle of 20 that offers all probable directions of X-diffraction of the lattice [24].

2.3.3 Ultrasonic Pulse Velocity Test

This method is a non-destructive method used for assessing the sample in terms of pore structure, uniformity, absence of internal flaws, segregation, cracks and also to analyze self-healing efficiency of samples. Comparing the time taken by the ultrasonic pulses to pass through the sample being tested and confirmed and the quality is determined in terms of density of sample, cracks or flaws, segregation, healing efficiency and the workmanship employed [3].
2.3.4. Sorptivity Test
The durability of mortar can be assessed by Sorptivity test. It was measured by rate of absorption of capillary rise on reasonably consistent materials [4]. Coefficient of sorptivity will determine the durability of the specimen. Mortar cube samples of dimension 70.6 mm were cured for 56 days. After 56th day, samples were taken out and oven dried at temperature of 100 to 110°C for 24 hours, dry weight was weighed after oven drying was done. Samples were kept in water at a depth of not more than 5mm above the base of the sample flow from outer surface was capped by fastening it with nonabsorbent coating [21]. Weight of samples were noted down at a time interval of 5, 10, 30, 60, 120, 240, 1440 minutes as shown in below table 3.

\[
\Delta W = W_2 - W_1 = \text{Change in weight}
I = S \times t^{0.5}
I = \Delta W / (A \times d)
\]

Where, 
- \(W_1\) – Oven dry weight
- \(W_2\) – Weight of specimen after particular time interval
- \(A\) – Surface area of specimen through which water penetrated
- \(D\) – Density of water

2.3.5. Water Absorption Test
This test is done for assessing the durability of the mortar specimen according to ASTM committee C09. This method is conducted by finding the percentage of water absorption found by measuring dry and wet weight of the specimen. Mortar cube samples of size 70.6 mm were cured for 56 days. Specimens was oven dried at temperature of 100 to 110°C for 24 hours. After removing each sample from the oven, it was made to cool at a temperature of 20 to 25°C, and then the first dry weight (W1) was determined. All samples were kept in the oven for second time to find the second dry weight. At a temperature of 21°C all the samples were saturated in the water for 24hrs, wet weights (W2) of the samples were taken. Each specimen’s water absorption was calculated by using the formula,

\[
\text{Percentage of absorption} = \left( \frac{W_2 - W_1}{W_1} \right)
\]

3. RESULTS AND DISCUSSIONS

3.1 Compressive Strength
From the experiments, it is clear that the percentage increase in strength for MG, SB, MGM, SBM are 50%, 35%, 22.5%, 42.5% respectively when compared to conventional mortar for seventh day. Similarly, a significant percentage increase in the strength for 14th day compared to 28th day has illustrated in figure 2.

![Figure 2. Compressive strength versus Samples (7th, 14th and 28th days)](image_url)
For MGM specimen strength increment is about 43.3% and 6.61% to that of conventional mortar and SBM specimens.

3.2 X-Ray Diffraction
Phase identification of a crystalline material is generally done by X-ray powder diffraction (XRD) method. Finely grounded, homogenized material is analyzed by this method. Sample is taken from the interior portion of the mortar cube specimens of about 2g to confirm the presence of CaCO3 in the sample. This test is done for 14th Day and it produced the following results as shown in table 4. Through XRD test it confirms the precipitation of CaCO3 when conditions satisfy Bragg’s Law: (n\lambda=2d \sin \theta) for all bacterial samples illustrated in figure 3-7. From table 2, it is proved that for conventional mortar at 2\theta angles the conventional specimen doesn’t show any CaCO3 precipitation values [24].

Table 2: XRD Score for Conventional Mortar

| SAMPLES | COMPOUND NAME(CACO3) | SCORE |
|---------|----------------------|-------|
| CM      | Absent               | 0     |
| MG      | Present              | 18    |
| SB      | Present              | 12    |
| MGM     | Present              | 17    |
| SBM     | Present              | 13    |

Figure 3. X-Ray Diffraction Pattern for Conventional Mortar
Figure 4. X-Ray Diffraction Pattern of Megaterium (Without Metakaolin) Mortar

Figure 5. X-Ray Diffraction Pattern for Subtilis (Without Metakaolin) Mortar
Figure 6. X-Ray Diffraction Pattern for Megaterium (With Metakaolin) Mortar

Figure 7. X-Ray Diffraction Pattern for subtilis (With Metakaolin) Mortar

3.3 Ultrasonic Pulse Velocity

It is very evident that the Ultrasonic pulse velocity increases with increase in number of days. On seventh day, it is to be noted that the velocity reached more than 4000m/s for all samples. Therefore, it is obvious that all mortar cubes show good quality. At both twenty-eighth and fifty sixth day, it is clear that the velocity is less for conventional mortar when compared to other samples. On comparing the velocities of Megaterium and Subtilis samples, Megaterium sample shows higher velocities than Subtilis sample, which shows that Megaterium had precipitated more CaCO3 than subtilis. Even though on 56th day all the sample except conventional mortar sample shows almost the same velocities, on comparing all the five samples we can see that Megateriummetakaolin Shows higher...
velocities in 14th, 28th, 56th days of tests as shown in figure 8.

![Figure 8 Pulse Velocities versus Days](image)

3.4 Sorptivity
From figure 8 it is observed that sorptivity coefficient for conventional mortar was higher than other specimens, however bacteria have some effect on decrease in sorptivity, metakaolin has greater effect on it. Especially, for Megaterium with metakaolin shows a significant decrement in sorptivity values when compared to other specimens in 240 mins and 1440 mins [25]. This is shown in table 3.

| TIME (mins) | CM | MG | SB | MGM | SBM |
|-------------|----|----|----|-----|-----|
| 5           | 8.97 | 7.17 | 5.38 | 5.38 | 5.38 |
| 10          | 6.34 | 5.08 | 3.81 | 3.81 | 3.81 |
| 30          | 4.39 | 2.93 | 2.93 | 2.19 | 2.19 |
| 60          | 3.11 | 2.59 | 2.07 | 1.55 | 2.07 |
| 120         | 2.56 | 2.19 | 1.65 | 1.47 | 1.83 |
| 240         | 1.81 | 1.81 | 1.55 | 1.04 | 1.29 |
| 1440        | 1.06 | 1.06 | 1.06 | 0.53 | 0.85 |

3.5 Water Absorption
Absorption test was done for a time period of 24 hours [25]. It shows that conventional mortar has more absorption capacity than all other specimens but Metakaolin samples shows less percentage of water absorption as shown in table 4. As stated in [12], existed substantial difference in the water absorption values of Bacterial solution subjected specimens.

| WATER ABSORPTION % | CM | MG | SB | MGM | SBM |
|--------------------|----|----|----|-----|-----|
| 6.66               | 5.4 | 5.7 | 3.41 | 3.5 |

4. CONCLUSION
The Compressive Strength (7 & 14 Day) of bacterial mortar shows about 21-50 % increase when
compared to Conventional Mortar. The Compressive Strength of 7th Day for sample with Megaterium showed 50% increase in strength than that of Conventional Mortar. At 28th Day, Compressive strength increment for all the bacterial samples and metakaolin embedded bacterial samples was almost similar. At 7th Day of UPV Test, all samples attained good quality, i.e., velocity was more than 4000 m/s. At 28th and 56th Day, the velocity was less for Conventional Mortar than that of other samples. Megaterium sample showed more velocity than Subtilis which implies that Megaterium had precipitated more CaCO3. Addition of Metakaolin with Bacteria had decreased the sorptivity of samples. Samples with Metakaolin and Bacteria showed less water absorbing capacity. XRD score confirms the precipitation of CaCO3 by both Megaterium and Subtilis. Bacillus Megaterium solution incorporated specimen show greatest strength improvement and durability properties incorporated with and without metakaolin compared to bacillus subtilis.

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