Studies on Performance and Micro Structural Characteristics of Self-healing Concrete

D. J. Arpitha, A. R. Chandrashekar, and Kannam Praveen

1 Introduction

1.1 Self-healing Concrete

In recent years, a lot of advanced research is underway to heal cracks in concrete, extend concrete life, and avoid routine maintenance and concrete maintenance costs. One of the most important research projects is self- healing concrete [1].

Self-healing concrete is a type of concrete that, unlike ordinary concrete, automatically repairs or fills cracks that occur in the concrete. There are various methods of self-repair processes, mainly of two types known as intrinsic and extrinsic methods. In an inherent self-healing process, concrete or mortar repairs cracks without external additives. The presence of CaO in the material causes hydration and calcite precipitation to heal the cracks themselves [2].

Extrinsic healing process is something it requires an external enzyme to react with it and heal the cracks so a bacterium is induced into the mortar or concrete by encapsulation and direct methods. As shown in Fig. 1, the bacteria induced along with its food in the form of starch which when reacted with the water seeping from the cracks activates the dormant bacteria and chemical reaction takes place and formation of calcium carbonate takes place which fills up the cracks [3].

Selection of bacteria is the most important, not all the bacteria can be used as self-healing agents the selected bacteria must have the capabilities to survive in the worst environment and for long time as well. Mainly bacillus family bacteria are used such as *Bacillus megaterium*, *Bacillus subtilis*, *Bacillus Sphaericus*, etc.; these

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Fig. 1 Bacterial concrete

are the bacteria which can survive in extreme condition and good for self-healing process.

1.2 *Process of Self-healing*

Self-healing process, concrete is familiar for healing its own cracks through hydration of cement as cement contains calcium oxide that reacts with water in the presence of carbon dioxide leads to formation of calcium carbonate which will help in healing the cracks however not completely [4].

$$Ca\,(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$$

There are two methods of inserting bacteria in concrete one is direct method and encapsulation.

- In this research in direct method the bacteria directly inserted during mixing itself, in encapsulation method.
- The bacteria induced in the form of clay pallets or capsules which is costly than direct method.

Bacillus subtilis bacteria of 109 population of 0%, 3%, 5% is induced in M30 and M70 mix of concrete using direct method, the mechanical properties such as compressive strength, tensile strength, flexural strength of conventional concrete and bacterial concrete are compared to see the effect of bacteria on the mechanical properties of concrete and the micro structural analysis is also carried out by scanning electron microscopy (SEM) analysis where we can find the surface's topography and composition of the sample and X-Ray Diffraction (XRD) test where we can find crystalline variants and to study particle size of nanomaterial's.

1.3 The Advantages of Self-healing Concrete

- The mechanical properties of the concrete such as compressive strength, tensile strength, flexural strength increases.
- The maintenance and cost of maintenance of the cement concrete decreases.
- It can last till centuries or decades so no need to replace during life time.

1.4 The Disadvantages of Self-healing Concrete

- The initial cost of construction using bacterial will be more.
- Required skill labourers for this work.
- Still there is no particular code to standardize the self-healing concrete.

2 Materials and Methodology

2.1 General

In this chapter the various materials and methodology used in the experimental programme are described in the below paragraphs.

Cement. Cement is a binder material used for construction that mixed with aggregates and water forms concrete. Concrete produced from Portland cement is one of the most versatile construction materials available in the world [5, 6].

For high strength concrete, OPC 53 grade is used. The min. compressive strength of OPC 53 Grade Cement should not be less than 53 N/mm^2. For the concrete of grade M-30 and above 8–10% can be saved by using OPC 53. Figure 2 represents the cement sample taken for the experimentation works.

Fig. 2 OPC 53 grade

Fig. 3 Fly-ash

Fly-ash. Fly-ash also known as 'Pulverized Fuel Ash' is waste product comes out after combustion of coal, generally we get fly ash from electrostatic precipitators. By using fly ash shown in Fig. 3 as the replacement of cement helps in reducing environmental pollution. Fly-ash can significantly improve the workability of concrete.

Silicafumes. Silica fume also known as condensed silica fume or micro silica is very fine, is produced by the by products of elemental silicon or silica-alloys. The specific of silica fumes is from 2.2 to 2.3.

Silica fume is one of best replacement for cement which increases the mechanical strength of concrete and also decreases the environmental pollution.

2.2 Methodology

- Calculation of mix design for M30 and M70.
- Concentration of bacteria (population) used = CFU (109).
- Percentage (dosage) of bacteria used = (0%, 3%, 5%).

Tests Concrete

Fresh properties of Concrete. When calculating the mix design, we first mix the ingredients according to the mix design and check the fresh physical properties such as workability, consistency, settlement, bleeding, etc. There are many possible checks such as set cone test, compaction factor, V-Bee test.

Slump cone test. After the concrete has been mixed, the wet concrete is poured into the hardening cone 25 times in three layers, after filling the cone is lifted slightly, the cone is placed upside down next to it and the hardening value is measured. According to IS 1199-1959, M30 concrete has zero slump and M70 concrete has true slump.

Tests on Hardened Concrete. According to IS:516-1959 (2004), the compressive strength test was performed on concrete cube specimens measuring 150 mm. After 7 and 28 days of curing, the cubes of each mix proportion (trial mix) were evaluated in a compression testing machine with a 2000 kN capacity. According to IS:5816-1999 (1999), the split tensile strength test was performed to determine the tensile strength of concrete cylinders with dimensions of 150 mm in diameter and 300 mm in length. The concrete beams used for the flexural strength test had dimensions of 500 mm in length, 150 mm in width, and height by applying a two-point loading to the concrete beam in accordance with the specifications of IS:516-1959 (2004) in a flexural strength testing equipment with a 500 kN capacity. On specimens that had undergone a 28-day water cure, split tensile and flexural strength tests were performed.

Durability Test. To access the performance of hardened concrete following test were carried out.

- Durability test (28 days).
- Acid attack test (by immersing in 5% of H_2SO_4 solutions for 28 days).
- Micro structural analysis.
- Scanning electron microscope (SEM).
- X-ray diffraction (XRD).

Mix Design of Concrete (As Per IS 10262:2019). Mix Design was carried out using IS 10262:2019. After number trails final mix proportions were determined [7]. The mix design for M30 and M70 are presented in the tables. Table 1 shows the mix proportion for M30 and Table 2 shows the mix proportion for M70.

Table 1 Mix design for M30

Cement (kg/m^3)	Fine aggregates (kg/m^3)	Coarse aggregates (kg/m^3)	Water (kg/m^3)	Water/cement ratio
413	706	1103	186	0.45

Table 2 Mix design for M70

Cement (kg/m^3)	Fine aggregates (kg/m^3)	Coarse aggregates (kg/m^3)	Water (kg/m^3)	Chemical admixture (kg/m^3)	Water–Binder Ratio	Fly ash (kg/m^3)	Silica fumes (kg/m^3)
428	589	1219	141	2.67	0.26	80.25	26.75

3 Result and Discussion

3.1 General

In this chapter, the experimental results were discussed. After casting and curing of concrete specimens. The hardened moulds were tested for mechanical, durability and micro structural properties.

3.2 Mechanical Properties

The performance of concrete is calculated by its mechanical properties it includes fresh properties and hardened properties.

Harden properties of the concrete viz., compressive strength, tensile strength, flexural strength can be calculated after curing the specimens for 28 in water and kept outside by that time specimens get harder and can go with tests of hardened properties [8].

Compressive strength: The compressive strength of concrete is calculated by casting M30 and M70 with and without bacteria concrete cubes of 150 mm × 150 mm × 150 mm and cured for 28 days [9], it is to calculate the maximum load which the specimen can bear, based on the literature-review, dosage of the bacteria to be added is calculated and the compressive strength results are compared. In this study the conventional concrete M30 and M70 are casted and M30 and M70 with 3% of *Bacillus subtilis* bacteria (which was calculated by weight of water) and M30 and M70 with 5% of *Bacillus subtilis* bacteria (which was calculated by weight of water), three each cube of M30, M70 with and without bacteria where casted for compressive strength and compared the conventional and bacterial concrete results. By comparing the results as shown in Figs. 4 and 5, the compressive strength is increased by 15%, 20% for M30 (3%) and M30 (5%), respectively, at 28 days, and compressive strength is increased by 15%, 20% for M70 (3%) and M70 (5%), respectively, at 28 days [10].

Tensile strength: Tensile strength of concrete refers to casting a cylindrical mould with a diameter of 150 mm and a length 300 mm, curing with Bacillus subtilis for 28 days (calculated by water weight), M30, M70 with 5% Bacillus subtilis (water weight), each three cylinders M30, compared the split strength of M70 with and without bacterial injection, and compared the results for conventional concrete and bacterial concrete. By comparing the results as shown in Figs. 6 and 7, after 28 days, M30 (3%) and M30 (5%) showed 14% and 18% increases in tensile strength, and M70 (3%) showed 14% and 18% increases in compressive strength increase [11].

Flexural strength: The concrete flexural strength is a mechanical property which can be measured by casting a beam of 100 mm × 100 mm × 500mm length are casted and cured for 28 days In this study the conventional concrete M30 and M70 are casted and

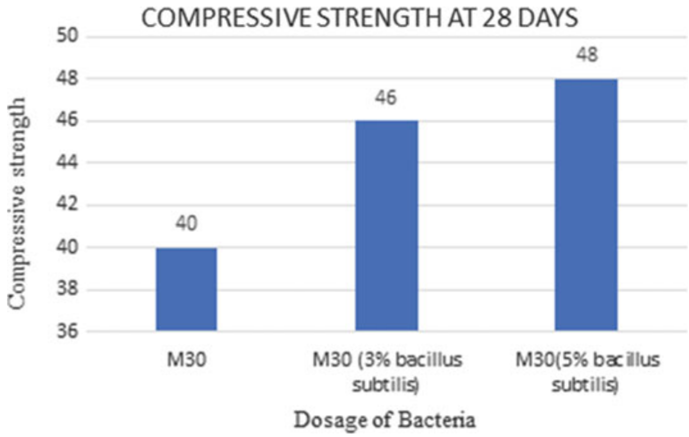


Fig. 4 M30 concrete compressive strength results at 28 days

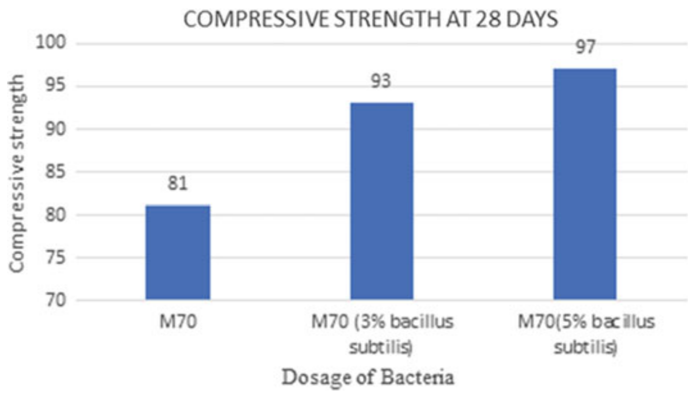


Fig. 5 M70 concrete compressive strength results at 28 days

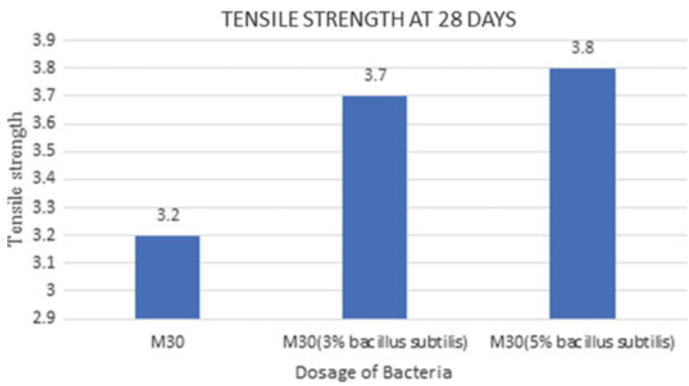


Fig. 6 M30 concrete split tensile strength results at 28 days

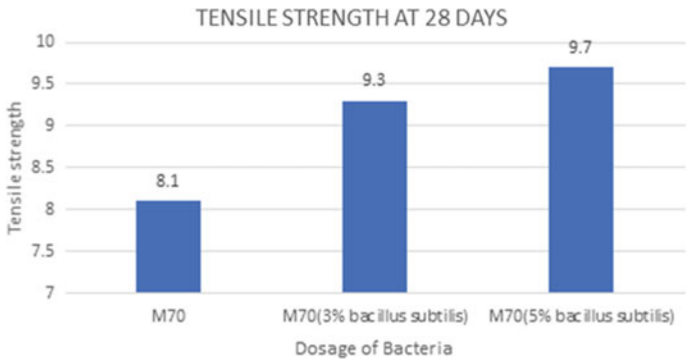


Fig. 7 M70 concrete split tensile strength results at 28 days

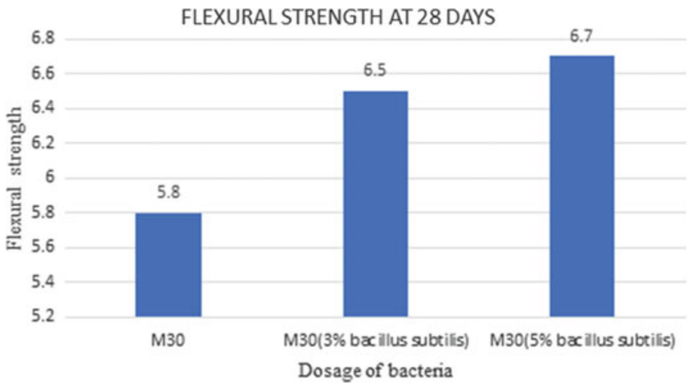


Fig. 8 M30 concrete flexural strength results at 28 days

M30 and M70 with 3% of bacillus subtilis bacteria (which was calculated by weight of water) and M30 and M70 with 5% of bacillus subtilis bacteria (which was calculated by weight of water), 3 each beams of M30, M70 with and without bacteria where casted for flexural strength and compared the conventional and bacterial concrete results. By comparing the results as shown in Figs. 8 and 9, the flexural strength is increased by 10%, 15% for M30 (3%) and M30 (5%) respectively at 28 days, and compressive strength is increased by 10%, 15% for M70 (3%) and M70 (5%) respectively at 28 days [12].

3.3 Durability Properties

Acid Attack Test: Chemical resistance of concrete cubes was tested by chemical attack by immersion in acid solution. Acid attack is caused by the reaction of the

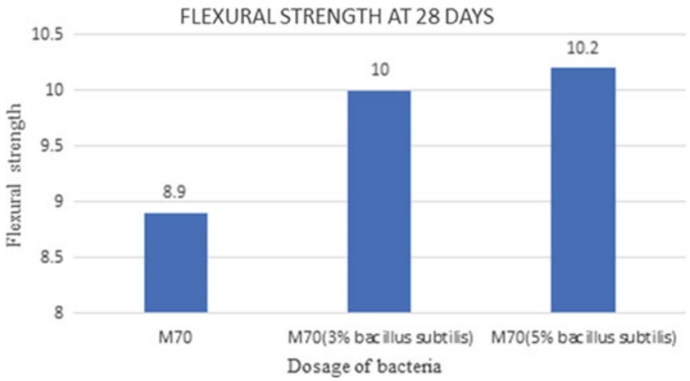


Fig. 9 M70 concrete flexural strength results at 28 days

acid with the calcium hydroxide portion of the cement paste, releasing a readily soluble by-product, the calcium salt.

These soluble calcium salts are easily removed from the cement paste, weakening the overall paste structure [13]. This fundamental reaction is

$$\text{Acid X} + \text{CH} \rightarrow \text{CX} + \text{H} \tag{1}$$

$$\text{Acid} + \text{Calcium Hydroxide} \rightarrow \text{Calcium Salt} + \text{Water}$$

The most aggressive and destructive cases of acid attack occur only when concrete is exposed to sulfuric acid (H_2SO_4). Calcium salts are formed by the reaction of sulfuric acid and calcium hydroxide to form calcium sulphate, which undergoes significant decomposition due to sulphate attack.

$$H_2SO_4 + Ca(OH)_2 \rightarrow Ca(SO)_4 + 2H_2O \tag{2}$$

At the end of the curing time, samples are taken out of the curing tank, and their surfaces are cleaned to remove any weak reaction products or loose material from the sample. Initial mass and diagonal dimension values are measured. For each batch, three samples of concrete cubes are immersed in 5% HCL and 5% H_2SO_4 solutions. At the end of 7, 14, 21, and 28 days, all through the curing time, steady acid concentration is maintained. After 28 days, each example is taken out of the tanks before testing, brushed with delicate nylon brushes and flushed in normal water. Changes in mass change, decrease of compressive strength and change in slanting aspects are observed. Acid attack results on SCC samples cured under various curing conditions are shown in the following three factors: namely, Acid Durability Loss Factor (ADLF), Acid Attack Factor (AATF) and Acid Strength Loss Factor (ASLF) are derived from ASTM C267 (2012) by reducing the durability aspects of specimens (strength, mass and geometry).

Acid Mass Loss Factor (AMLF): The Percentage Loss is estimated by placing the cubes in acid solution and finding the mass at a defined period of time. The change in mass with age compared to the initial weight of each specimen is defined as Acid Mass Loss Factor (AMLF).

$$\text{AMLF} = \frac{\text{Change in weight of specimen after placing in Acid}}{\text{Initialmass of specimen before placing in Acid}} \times 100 \quad (3)$$

Acid Attacking Factor (AAF): The continuation of deterioration at each corner of the struck face and the opposite face was measured. The change in the length of diagonal after immersion in the acid for a defined period of time is defined as Acid Attacking Factor (AAF).

$$\text{AAF} = \frac{\text{Change in dimension of diagonal after immersion in Acid}}{\text{Original diagonal dimension before immersion in Acid}} \times 100 \quad (4)$$

Acid Strength Loss Factor (ASLF): The relative strength present in concrete specimens after immersing in acid represents Acid Strength Loss Factor (ASLF). The relative strengths are always compared with respect to 28 days compressive strength values.

$$\text{ASL} = \frac{\text{Change in dimension of diagonal after immersion in Acid}}{\text{Original diagonal dimensions before immersion in Acid}} F \times 100 \quad (5)$$

To analyse the durability properties of self-healing concrete specimens, important durability properties like Acid attack are determined. The present chapter investigates the durability properties of specimens from both M30 and M70 grade concrete. Table 3 represents the loss in weight and loss of compressive strength because of acid attack test at 28 days.

From Table 4, Acid Mass Loss Factor, Acid Attacking Factor, Acid Strength Loss Factor, Acid Durability Loss Factor, the results shows that acid durability loss factor is more in M30 (5%) and M70 (5%) as the bacteria percentage increases the durability loss factor also increases.

Table 3 Weight loss due to acid attack test at 28 days

Grade	IW (kg)	FW (kg)	% of weight reduction	% of loss in CS
M30	7.53	6.92	8	58
M70	8.18	7.52	8	60
M30 (3% *Bacillus subtilis*)	8.2	7.38	10	51
M70 (3% *Bacillus subtilis*)	8.48	7.63	10	52
M30 (5% *Bacillus subtilis*)	8.76	7.70	12	54
M70 (5% *Bacillus subtilis*)	8.85	7.78	12	55

Table 4 Details of Acid Mass Loss Factor, Acid Attacking Factor, Acid Strength Loss Factor and Acid Durability Loss Factor

Grade	Acid Mass Loss Factor (AMLF)	Acid Attacking Factor (AAF)	Acid Strength Loss Factor (ASLF)	Acid Durability Loss Factor (ADLF)
M30	8.1	9.4	42	3198.247
M70	8.06	7.9	40	2549.633
M30 (3% *Bacillus subtilis*)	10.00	6.1	49	2989
M70 (3% *Bacillus subtilis*)	10.02	5.1	48	2453.774
M30 (5% *Bacillus subtilis*)	12.10	6.3	46	3506.712
M70 (5% *Bacillus subtilis*)	12.09	6.6	45	3590.847

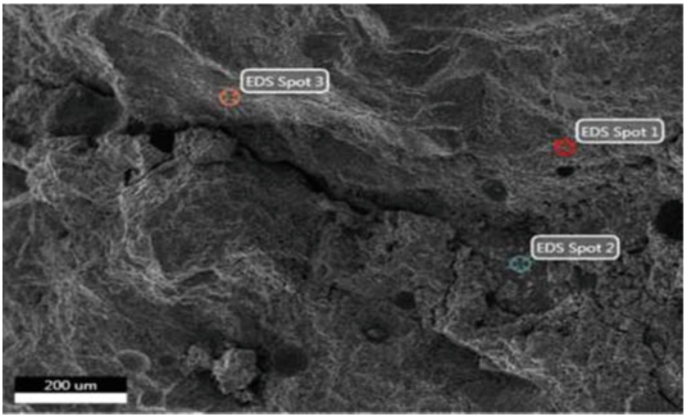
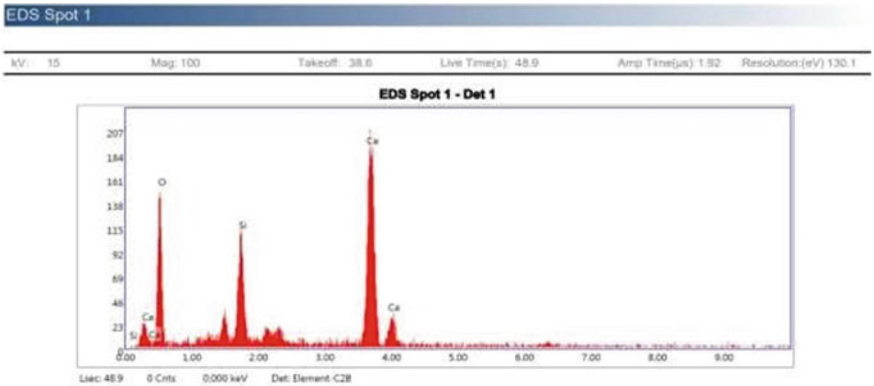


Fig. 10 SEM analysis

3.4 Micro-structural Properties

Micro structural characterization is concerned with the study of microelements and chemical composition. From Fig. 10, whether the element present is crystalline or amorphous and what it consists of, as well as its chemical composition. In this project, it is mainly used to check the contained micro cracks and confirm the formation of $CaCO_3$ that fills the bacterial morphology and micro cracks. There are various methods for micro structural analysis, such as SEM (Scanning Electron Microscope) analysis, XRD (X-Ray diffraction), and EDX (energy dispersive X-ray analysis).

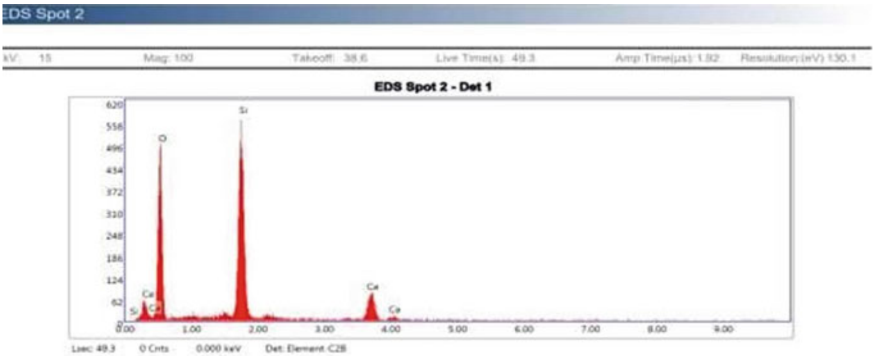
By performing EDX analysis shown in Figs. 11 and 12 at three different locations in the SEM image, different elements are obtained, including different weights and atomic percentages of SiK-OK-CaK. When this he reacts with H_2O (water), $CaCO_3$ is formed (crack healing).



eZAF Smart Quant Results

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
O K	41.50	62.70	19.57	13.66	0.0777	1.0884	0.1720	1.0000
SiK	7.83	6.74	15.98	10.20	0.0642	0.9835	0.8281	1.0061
CaK	50.68	30.56	46.92	5.28	0.4670	0.9238	0.9951	1.0023

Fig. 11 EDX at Spot 1



eZAF Smart Quant Results

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
O K	52.35	67.94	68.95	9.80	0.1889	1.0558	0.3418	1.0000
SiK	33.34	24.65	100.46	4.39	0.2782	0.9517	0.8749	1.0026
CaK	14.31	7.41	18.17	9.73	0.1247	0.8923	0.9696	1.0077

Fig. 12 EDX at Spot 2

4 Conclusion

This experiment is conducted for study of strength durability and micro structural behaviour of self-healing concrete, for M30 and M70 with and without Bacillus bacteria of 3% and 5%

1. The compressive strength for M30 (3%) and M30 (5%) at 28 days was increased by 15% and 20% respectively compared to M30.
2. The compressive strength for M70 (3%) and M70 (5%) at 28 days was increased by 15% and 20% respectively compared to M70.
3. The tensile strength for M30 (3%) and M30 (5%) at 28 days was increased by 14% and 18% respectively compared to M30.
4. The tensile strength for M70 (3%) and M30 (5%) at 28 days was increased by 14% and 18% respectively compared to M70.
5. The flexural strength for M30 (3%) and M30 (5%) at 28 days was increased by 15% and 20% respectively compared to M30.
6. The flexural strength for M30 (3%) and M30 (5%) at 28 days was increased by 15% and 20% respectively compared to M30.
7. The acid attack test shows weight reduced for M30 and M70 was 8%, for M30 (3%) and M70 (3%) was 10%, for M30 (5%) and M70 (5%) was 12%.
8. The SEM images shows us that the healing in M30 (5%) and M70 (5%) was faster compared to M30 (3%) and M70 (3%).

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