CRACK HEALING IN CONCRETE USING VARIOUS BIO INFLUENCED SELF-HEALING TECHNIQUES

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thesis titled

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has been accepted towards the partial fulfillment

of the requirements for the degree

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DEDICATED

TO

MY PARENTS AND SIBLINGS

ACKNOWLEDGEMENTS

In the name of Allah, the most merciful, the most compassionate all praises be to Allah, the lord of the worlds and prayers and Peace be upon Muhammad his servant and messenger.

The completion of this project was only possible due to unlimited blessings of almighty Allah and collaboration of many people, to whom I wish to express my gratitude.

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ABSTRACT

Concrete is most widely used engineering material in construction industry due to its strength, durability and low cost as compared to other construction materials. However, crack formation and progression under tensile stress is a major weakness of concrete. These cracks can make concrete vulnerable to deleterious environment due to ingress of harmful compounds, hence compromising its durability resulting in deterioration of concrete. For a durable and good structural concrete control over microcracks development and propagation of these cracks is necessary and crack healing can be helpful in mitigation of development and propagation of cracks in concrete.

Self-healing concrete involves the crack repairing in concrete by use of compounds, bacteria and resins, added in concrete during the mixing stage. Bio concrete or bio influenced self-healing concrete is a product which has the ability to fill the cracks by producing chemical products from intentionally incorporated micro-organism in the concrete. In this kind of concrete micro-organisms along with a precursor compound are introduced in the concrete during mixing phase. When cracks are produced, water seeps in the cracks and activates the already present dormant micro-organisms. These micro-organisms later produce minerals to fill the cracks hence inhibiting strength loss and increasing durability of concrete.

An experimental program was conducted to study the self-healing mechanism in concrete under various conditions. Bacteria were introduced in concrete by different techniques such as directly incorporating in mix, by immobilizing it in light weight aggregate and in combination with graphite nano platelets. In all the techniques, calcium lactate was used as an organic precursor and replaced 5% of cement. Specimens were made for each mix to compare the change in compressive strength of each mix. In addition to that, concrete specimens were also subjected to pre cracking at 3,7,14 and 28 days to determine the crack healing efficiency of each mix.

Results show that bacteria immobilized in graphite nano platelets were more effective in samples pre cracked at 3 and 7 days while bacteria immobilized in light weight aggregates were more efficient in samples pre cracked on 14 and 28 days. In addition, results of compressive strength depict that self-healing concrete made with light weight aggregate incorporating immobilized bacteria had significant enhancement in compressive strength of concrete.

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CHAPTER 1

INTRODUCTION

1. General

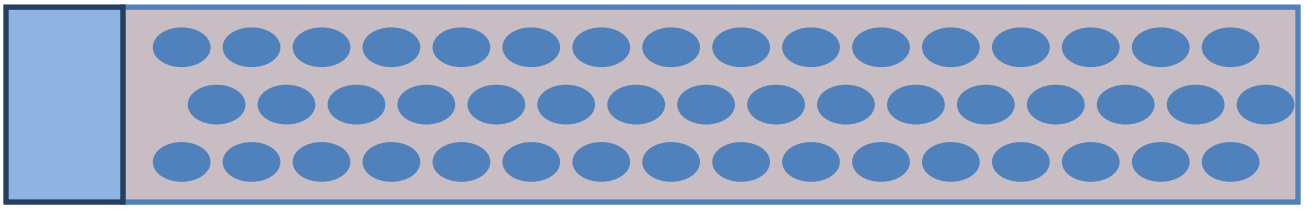
Self-healing concrete is a product which has the ability to fill the cracks by producing chemical products from intentionally incorporated bacteria, compounds or resins present in it. One of these methods of self-healing, through the production of minerals by using mineral producing bacteria in the concrete, is known as bio-influenced self-healing concrete or bio concrete. Bio concrete is a product which heals the cracks produced in it by producing the mineral compounds to fill the cracks. During earlier studies bacterial solution was sprayed on the cracks formed in a structure which was a less effective and impractical procedure. However, in recent years bacteria along with organic mineral precursor compound are incorporated in the concrete during the mixing phase.

Concrete is most widely used engineering material in construction industry due to its strength, durability and low cost as compared to other construction materials. However, it has certain drawbacks as well. A major drawback of concrete is its low tensile strength which makes it susceptible to progression and coalescence in microcracks resulting in low strength and durability. These tensile stresses can be due to tensile loading plastic shrinkage and expansive reactions (Mehta and Monteiro, 2006). This liability to cracking not only results in strength reduction of concrete but also makes concrete vulnerable to deleterious environment. Cracking in concrete is a major concern as these microcracks permit the ingress of harmful chemicals in concrete structures. This entry of harmful chemicals may result in concrete deterioration and can also cause corrosion of steel reinforcement (Reinhardt and Jooss, 2003). This corrosion leads to increase in crack width and length ultimately resulting in loss of strength and stiffness of concrete structures (Hewlett, 2003). To get a more durable structural concrete propagation of cracks must be controlled. This deterioration of both concrete and reinforcement results in high maintenance cost. According to report of Federal Highway Administration (2001) United States of America spends 4 billion dollars annually in terms of direct cost of maintenance of concrete highway bridges. De Rooij and Schlangen (2011) stated that UK spends 45 % of its annual construction cost on maintenance of already constructed concrete structures.

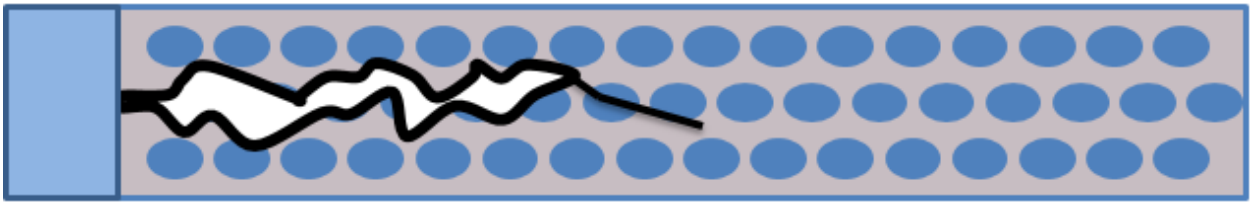
These drawbacks made scientists and researchers to look for an optimum solution to increase the durability of concrete by finding a solution to counter these cracks propagation in concrete. Scientists has made use of several materials, such as epoxy systems, acrylic resins and silicone based polymers to control this problem. However, most of these materials were found to be non-compatible with concrete, hazardous to environment and expensive (Vekariya and Pitroda, 2013).

As a result of current studies, bio concrete or bio influenced self-healing concrete is emerging as a viable solution the tensile crack formation in concrete structures. Bio concrete involves the incorporation of two component healing system in the concrete mixture, comprising of suitable bacteria and calcium based nutrient compound. Autonomous healing through this process increases the structure durability and on the other hand reduces the manual maintenance required for structures through the process of bio-mineralization. This self-healing technique reduces the use of environmental unfriendly repair materials and increases compressive strength of concrete, hence saving the environment and economy (Vekariya and Pitroda, 2013).

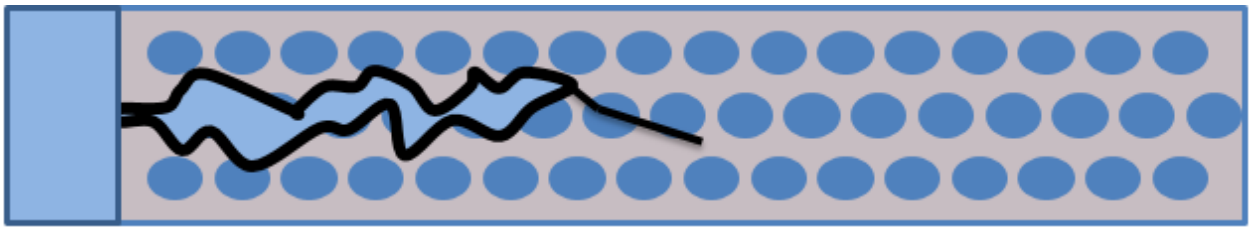
Generally, bacteria are incorporated in dormant form along with the nutrient compound which stays inactive until cracks are produced allowing water penetration in the concrete. Bacteria become active when come in contact with water and begin to feed on nutrient compound. Calcium carbonate is produced as the result of metabolic action of bacteria on calcium lactate and seals the crack, restricting the water penetration in concrete hence increasing durability of concrete.



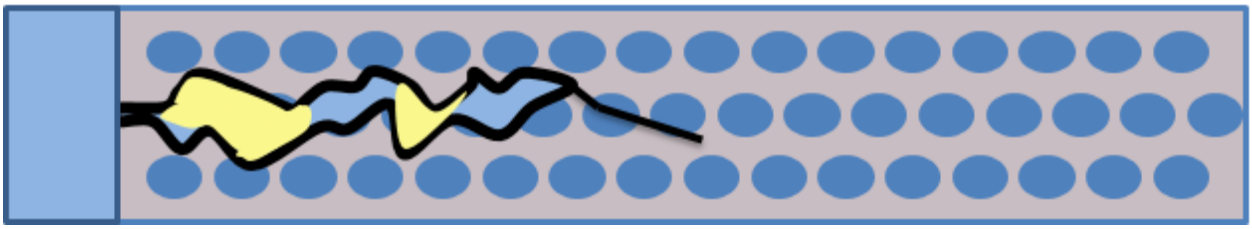
(a)- Bacteria incorporated in concrete mix



(b)- Crack produced in concrete due to tensile stresses



(c)- Water is deliberately entered in the cracks



(d)- $CaCO_3$ is produced which heals the cracks

Fig. 1.1 Process of self-healing of crack through bacteria

Self-healing in concrete is dependent on certain parameters including dissolved inorganic carbon, pH of concrete, nucleation sites and presence of calcium ions (Hammes et al., 2003). Additional factors include In addition, other variables such as type of bacteria, their varying concentrations, various curing procedures and material used for incorporation of bacteria also contribute towards efficient self-healing of concrete

Many species of Bacillus family have been proved to meet these standards and are used in this process. These bacteria can form spores when face harsh environments and become dormant. However, when concrete cracks and water is introduced in the cracks, alkalinity of concrete decreases and these bacteria become active again and start producing calcium carbonate by feeding on the nutrient compound.

In addition to the choice of suitable bacteria, selection of nutrient compound is also of great significance as bacteria yield calcium carbonate by feeding on it. Most commonly two type of nutrient compounds are used i.e. calcium lactate ($CaC_6H_{10}O_6$) and urea ($CO(NH_2)_2$). However, while using urea calcium rich environment is also required for the development of calcium carbonate.

2. Objectives

The main objective of this study is to investigate the self-healing response of concrete when bacteria are introduced using various techniques. This will be done in the following manner:

- Introduction of bacteria samples in the concrete mix without any carrier compound and study its efficiency in self-healing process.
- Comparing the self-healing process in concrete through different carrier compounds and identifying the optimum technique of bacteria introduction.
- Studying the viability of graphite nanoplatelets (GNP) as a new carrier compound for bacteria in concrete.
- Studying the effect of using bacteria and nutrient compound (in this case calcium lactate) on the strength of concrete.

Three different techniques were used for incorporation of bacteria in concrete which involve direct incorporation of bacteria during mixing, by incorporation of light weight aggregate containing immobilized bacteria and by incorporation of graphite nano platelets containing immobilized bacteria. Out of these, effect of introducing bacteria through graphite nano platelets (GNP) has not been studied before. Compressive strength of concrete was measured by subjecting the concrete specimen to compression tests whereas; self-healing process was monitored by visual inspection and SEM analysis of pre-cracked samples.

To accomplish the above mentioned objectives several tasks were undertaken. Literature review was first conducted. Testing and data acquisition setup was designed to acquire the required results. Samples configuration was determined and specimen were made on basis of this configuration. Compressive tests were conducted to determine the compressive strength. For pre-cracking, specimens were subjected to compressive load carefully. These pre-cracked samples were then placed in curing tank and crack width was measured on regular intervals to determine

the efficiency of self-healing process. On basis of data gathered comparison was made between efficiency of various self-healing techniques and their effect on compressive strength of concrete specimen.

3. Organization of the report

Chapter 1 is an introductory chapter about bio influenced self-healing concrete, objective of the study and thesis overview. A brief literature review on bio-concrete is discussed in chapter 2. Chapter 3 represents the procedure and materials of test setup, the testing facility and construction of specimens. The testing apparatus is also elaborated in Chapter 3. Chapter 4 discusses the tests carried out, the observations, test results and evaluation of test results. The conclusions based on findings of this research and recommendations for further studies are presented in Chapter 5.

CHAPTER 2

LITERATURE REVIEW

1. General

Bio-influenced self-healing concrete is expected to be a viable solution to the environmental concerns of carbon dioxide production by cement industries as well as durability of concrete structures. Due to these advantages it has been the focus of several research works and studies during the past few years. This chapter contains notable studies carried out on bio-influenced self-healing concrete. These research works have taken into account different variables contributing towards self-healing of concrete. Some of these variables are type of bacteria, pre-cursor compound and techniques used for incorporation of bacteria.

2. Previous studies on bio influenced self-healing practice

A number of research studies have been carried out on bio influenced self-healing process. Few of important studies are presented here:

Ramachandran et al. (2001) studied the role of micro-organisms in remediation of cracks and their effect on compressive strength in mortar samples. Micro-organisms used in this research were Bacillus Pasteurii. For this purpose Portland cement mortar beams and beams of dimensions 25 x 25 x 150 mm and 50 x 50 x 50 mm respectively were made and 3.175 mm wide cracks with varying depths were produced. Cracks were filled with sand mixed with bacterial specimen of 3×10^8 cells/cm^3. Samples were placed in solutions of urea and $CaCl_2$ for 28 days. Compressive tests were performed to find out the compressive strength of samples. In addition to compressive tests, SEM and XRD tests were also conducted to find out the crack healing efficiency of bacteria. Results showed that at lower concentrations B.Pasteurii increased the compressive strength of mortar sample. Microbiological remediation is more effective in shallow cracks as compared to deep cracks. Fig. 2.1 depicts the results of compressive tests as observed by Ramachandran (2001) at various concentrations. Fig. 2.2 indicates that living B.Pasteurii at the concentration of 7.6×10^3 cells/ml showed maximum strength at the age of 28 days.

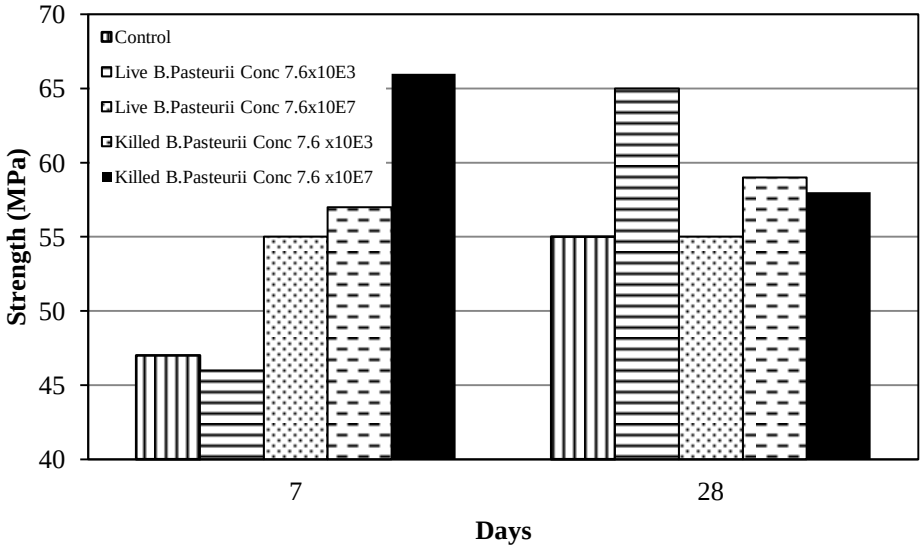


Fig. 2.1 Compressive strength of cement based mortar under various self-healing conditions

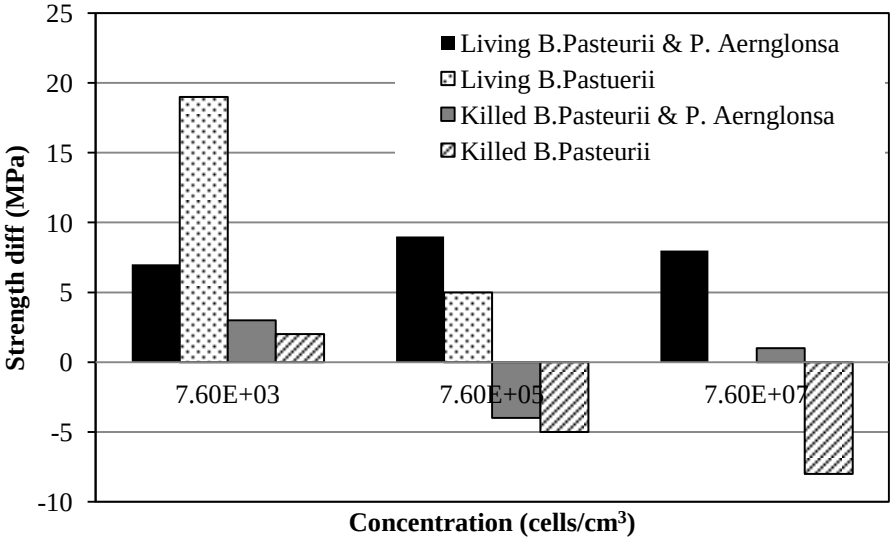


Fig. 2.2 Difference in 7 and 28 day compressive strength of mortar in various self-healing conditions

Ghosh et al. (2005) studied the effect of anaerobic bacteria of Shewanella species and Escherichia Coli on the compressive strength of cement mortar. For this purpose mortar cubes specimens of 70.6 mm were studied. Different cell concentrations of both bacteria were used in these samples ranging from zero to 10^7 per ml. It was observed through results that Shewanella bacteria had a positive effect on compressive strength at a cell concentration of 10^5 per ml and increased compressive strength by 25%. However, by increasing its concentration further caused a decrease in compressive strength. On the other hand, addition of E.Coli bacteria had no

significant effect on the compressive strength of mortar samples. In addition to compressive strength, SEM and MIP tests were also conducted on the samples. Results of these tests showed an improvement in pore size distribution at Shewanella cell concentration of 10^5 per ml. increase in compressive size was attributed to this improved pore size distribution in samples. Improvement in compressive strength at various concentrations throughout the curing period can be seen in the Fig. 2.3.

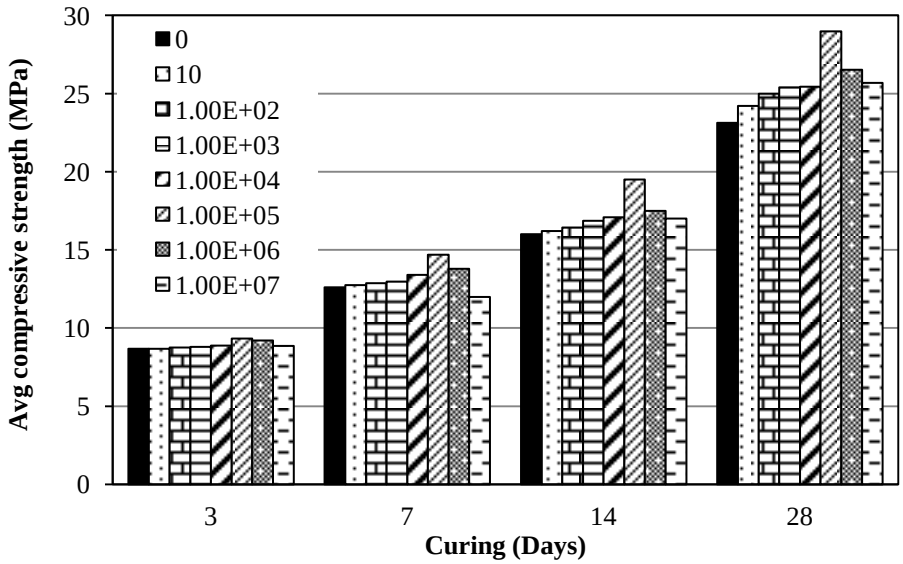


Fig. 2.3 Effect of bacteria (Shewanella) addition on mortar strength

Jonkers et al. (2010) studied the direct incorporation of bacteria along with nutrient compound in the concrete. Two different species of bacillus family were added (Bacillus Cohnii and Bacillus Pseudofirmus) in the concrete together with four different nutrient compounds including calcium acetate, yeast, peptone and calcium lactate. Compression tests were conducted and results exhibited that calcium acetate and yeast lowered the strength of concrete to half to that of control specimen. Peptone addition resulted in complete detrimental effect on concrete. Only the incorporation of calcium lactate increased compressive strength of concrete in 28 days cured samples as shown in Fig. 2.4. Crack surfaces of both control and bacterial concrete samples was inspected and a difference was noted among them in 7 day cured samples but surfaces of both samples were not substantially different in 28 days cured samples. Copious amount of particles with size 20-80μm was found on crack surface of 7 days cured bacterial sample which was

absent in 28 days cured samples. This along with results from most probable number (MPN) techniques proved that viability of bacterial spores decreases significantly over time.

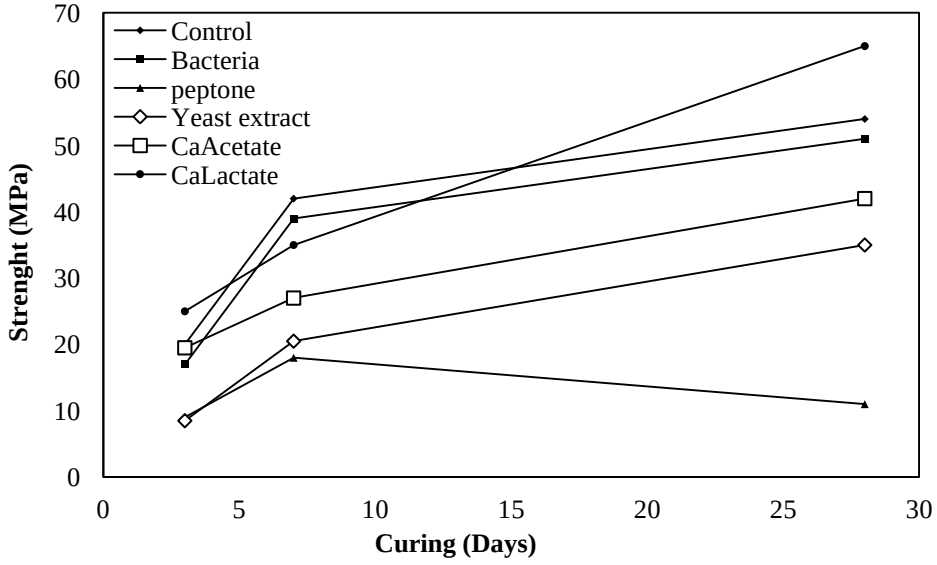


Fig. 2.4 Development of compressive strength of control and organic compound incorporated mortar

De Belie and De Muynck (2008) studied the effect of crack healing using bio deposition. Bacillus Sphaericus culture was used in this study. In order to study it standardized cracks of 0.3 mm were created by incorporating thin copper plates in concrete and pulling them out one day after casting or by performing split tensile tests on samples wrapped in fiber reinforced polymer. Cracked samples were cured in nutrient solution containing $CaCl_2$ or $Ca(NO_3)_2$. Bacteria were immobilized in silica gel and introduced in the concrete samples for protection. Visual inspection, ultrasound testing and water permeability tests were conducted. Visual inspection and ultrasound tests confirmed crack healing up to 0.3 mm in width and 10 mm in depth. Water permeability tests showed that 0.6 mm wide cracks were healed through the process of bio deposition. In addition to that water permeability test also depicts that epoxy, BS+sol-gel+$CaCl_2$ and BS+sol-gel+$Ca(NO_3)_2$ were most efficient in reducing water permeability as shown in Fig. 2.5.

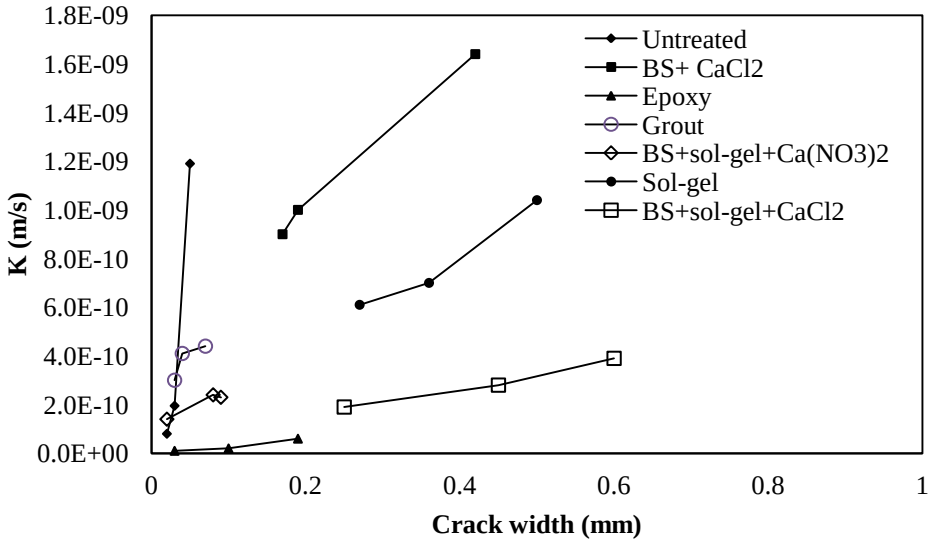


Fig. 2.5 Water permeability results of concrete samples against various crack widths

Van Tittelboom et al. (2010) investigated the efficiency of bacteria to repair cracks in concrete in comparison with traditional crack repairing methods. During this study water permeability tests, ultrasound transmission measurements and visual examinations were conducted to determine the crack healing efficiency of various crack repairing techniques. Cracks were created by means of split tensile methods and by creating grooves using copper wire. Crack treatment with B.Sphaericus immobilized in silica gel showed increased ultrasonic pulse velocity. Fig.s 2.6 and 2.7 show the results of water permeability and ultrasonic pulse velocity tests respectively. As it can be seen, bacterial solution incorporated in silica gel and treated with $CaCl_2$ had the least value of water permeability coefficient (K) and shows great reduction in water permeability as compared to untreated samples. In the same way, results for ultrasonic pulse velocity tests at the crack width of 10 mm show that maximum reduction in transmission time is observed in samples containing bacterial solution incorporated in silica gel and treated with $CaCl_2$. Through this increase in ultrasonic pulse velocity together with visual examination and water permeability tests, it was concluded that enhanced crack repair can be obtained by treating crack with biological mix containing B.Sphaericus culture incorporated in silica gel along with calcium source.

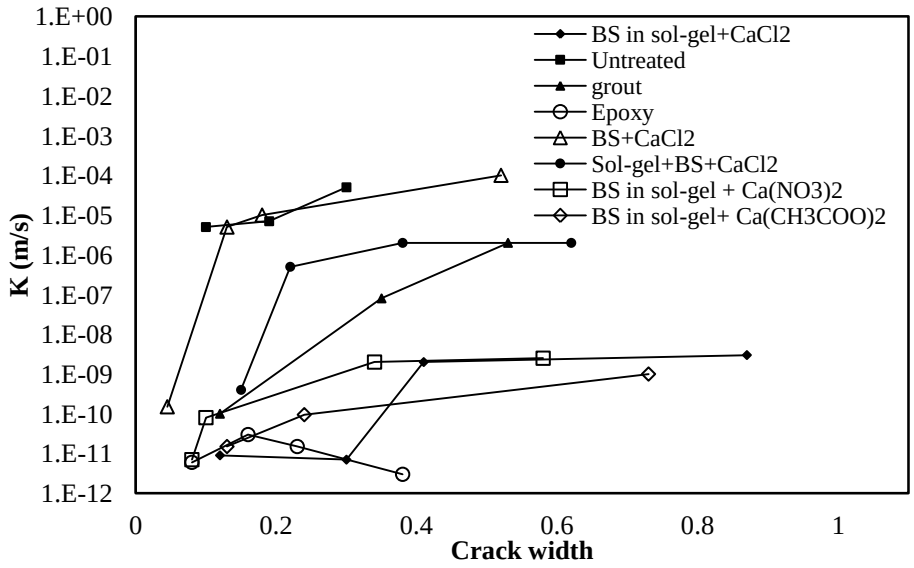


Fig. 2.6 Water Permeability results of concrete samples against various crack widths

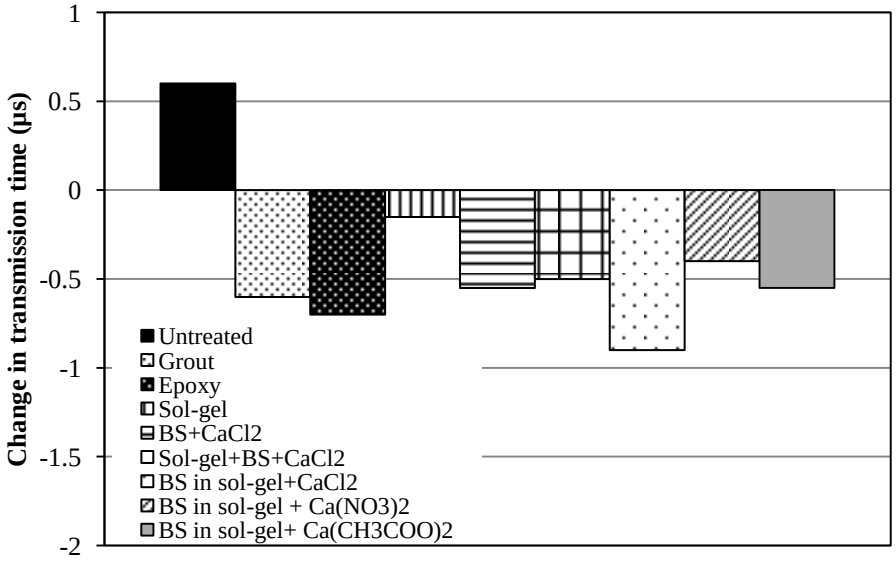


Fig. 2.7 Comparison of change in transmission time under various crack filling techniques at crack depth 10 mm

Wang et al. (2012) studied the possibility of using silica gel (SG) and polyurethane (PU) as a carrier for bacteria into the concrete mix. Bacteria used for this purpose was B.Sphaericus along with urea as the nutrient precursor compound. In order to identify the efficiency of bacteria, two different suspensions were prepared and incorporated with SG and PU. The bacterial suspensions

containing bacteria in alive form were represented by “BS” and those containing dead bacteria were named “BSA”. Bacteria together with nutrient and other compounds were encapsulated in glass tubes. When cracking occurred, glass tubes broke with it causing the encapsulate solution to release and heal the cracks. Fig. 2.8 and 2.9 demonstrates the results of conductivity tests at different intervals during the curing period and the strength regain observed in the prepared specimens.

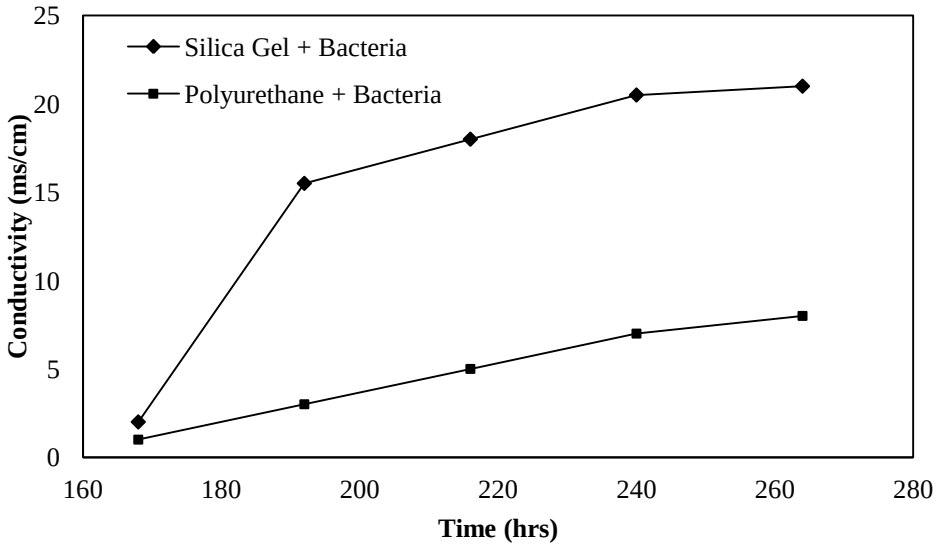


Fig. 2.8 Comparison of conductivity in different carrier compounds

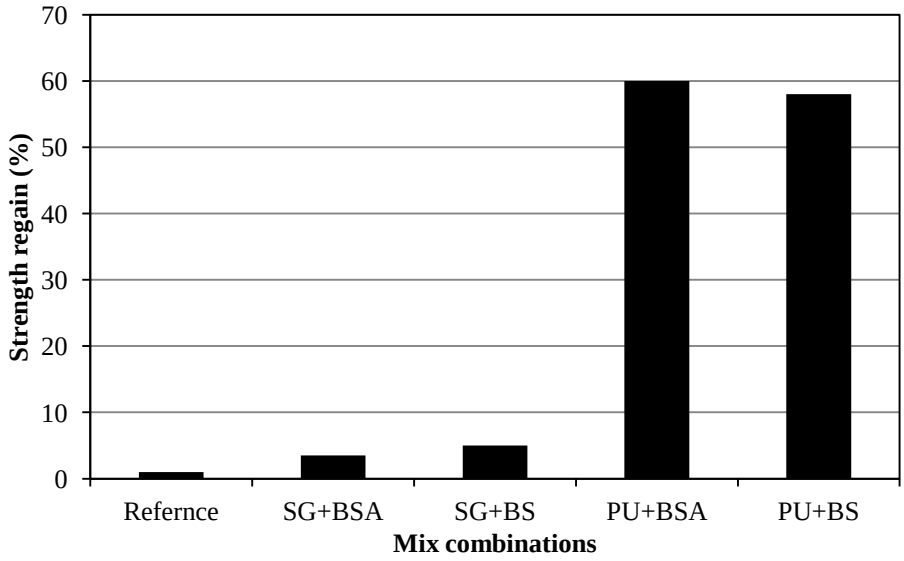


Fig. 2.9 Strength regain percentage for various mix combinations

It can be seen that specimen having bacteria incorporated in silica gel showed better conductivity as compared to those incorporated in polyurethane. Results showed calcium carbonate precipitation of 25% by mass in silica gel as compared to 10% by mass in polyurethane. Fig. 2.9 shows the results of strength regain in both samples. Strength regain in case of silica gel was relatively low i.e. 5% as compared to 60% strength regain in polyurethane. Based on these conclusions polyurethane was nominated a better option as bacterial carrier.

Wiktor and Jonkers (2011) carried out tests on cement mortar samples. During their research study bacteria were made inactive by heating them at 80^{o}C for 30 minutes. Bacteria along with nutrient precursor were protected by infusing them in light weight aggregates (LWA). Reinforced mortar specimen were made by using ordinary Portland cement, fine aggregate and LWA either soaked with bacteria and calcium lactate in case of bacterial specimen or non-impregnated in case of control specimen. After 56 days of curing, cracks were created and were re-cured. Fig. 2.10 shows threshold crack width at various healing days.

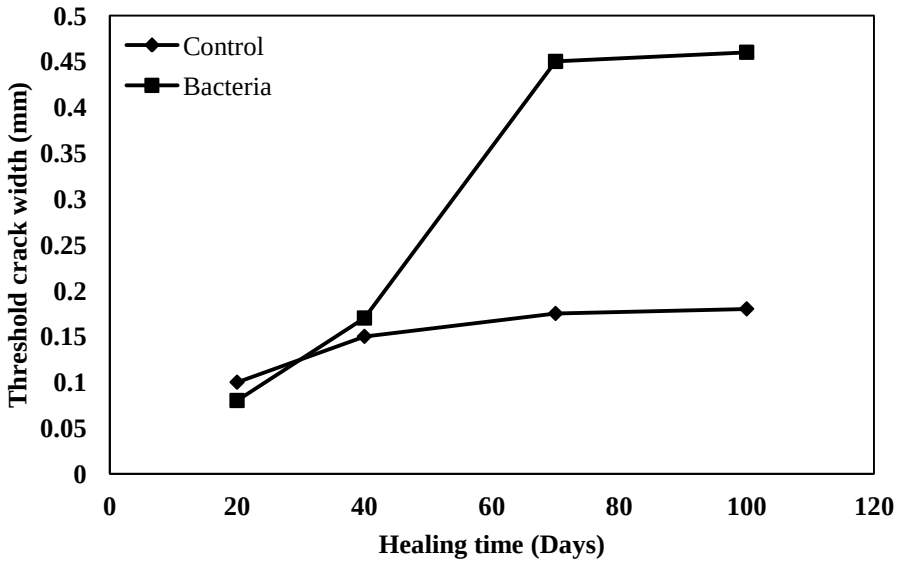


Fig. 2.10 Threshold values of maximum crack healed

In the light of observations made by them, they concluded that width of completely healed cracks was more than double in bacterial specimen (46 mm) as compared to that in control specimen (18 mm).

Sierra-Beltran and Jonkers (2012) discussed the effect and use of bio-based mortar for concrete repair. Initially, in this research four different types of ECC minerals were studied and depending upon their drying shrinkage and mechanical properties two of them were selected for further testing as bio based mortar. In these selected two material fillers, either sand or limestone was replaced with LWA having bacteria on them. Calcium lactate was used as nutrient precursor compound. The incorporation of healing agent had a negative impact on flexural strength and deflection but caused increase in compressive strength. However, all the test results exhibited that mortars having self-healing agent lied in the acceptable limit described in European standard EN 1504-3

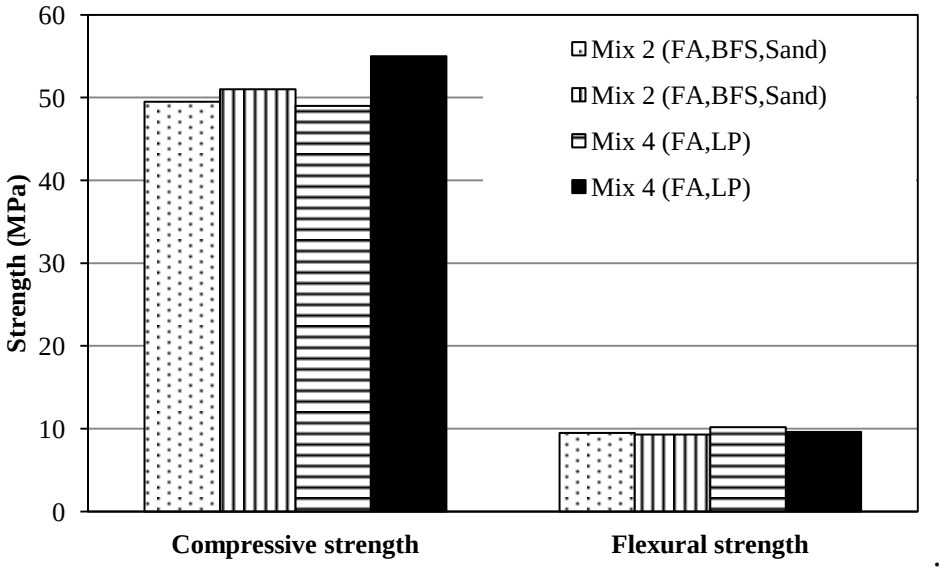


Fig. 2.11 Comparison of 28 day strength of mixes with and without incorporation of LWA

Wang et al. (2014) conducted research study on self-healing efficiency of microencapsulated bacteria. During this study B.Sphaericus was used as a healing agent. Six different series of samples were prepared containing control specimen, specimens containing nutrients, with microcapsules, having both microcapsules and nutrients, containing nutrients and 3% microencapsulated bacterial spores (NCS3%) and containing nutrients and 5% microencapsulated bacterial spores (NCS5%). Cracks were produced in these specimens and after that five different incubation techniques were used to check role of water in self-healing process. These incubation techniques included placing specimen in air conditioned room,

immersion in water, immersion in deposition medium, wet-dry cycles with water and wet-dry cycles with deposition medium.

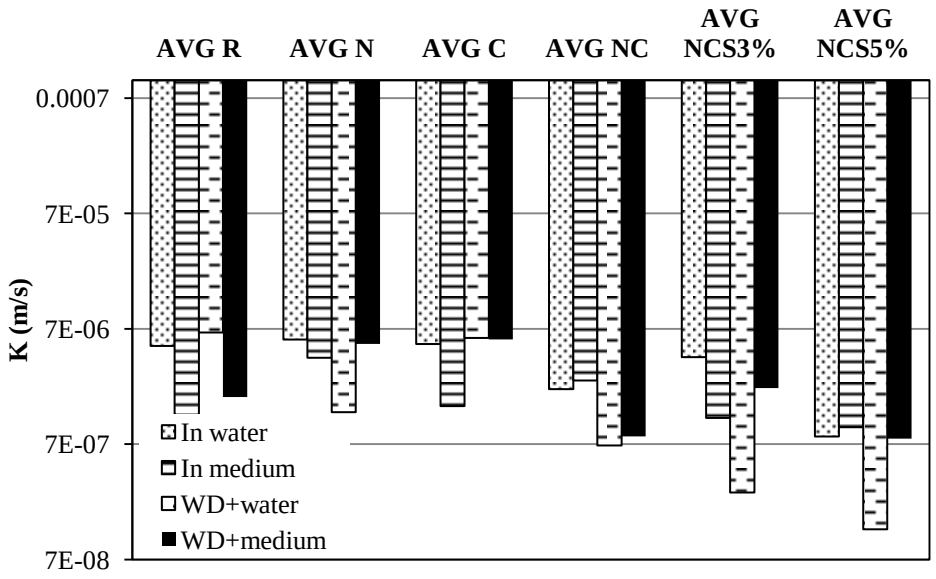


Fig. 2.12 Water permeability coefficient of samples in different curing conditions

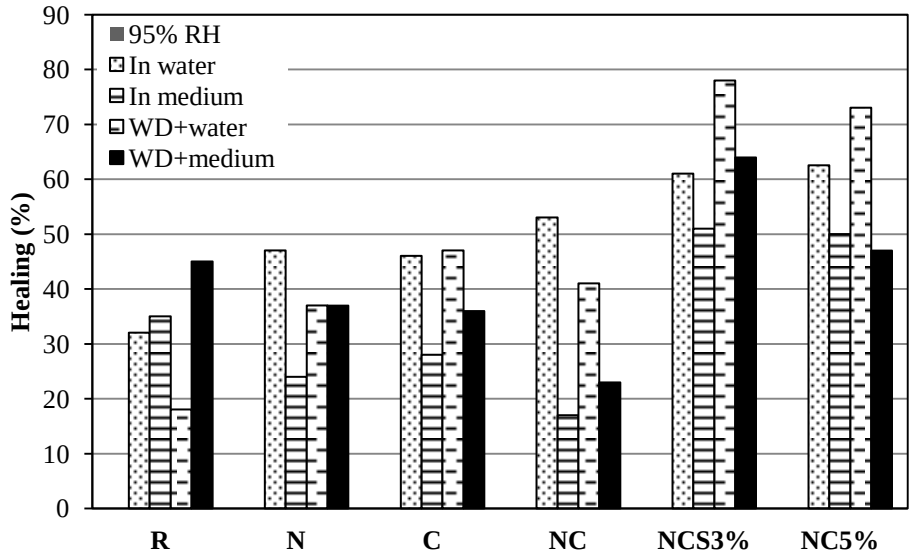


Fig. 2.13 Healing ratio of samples in various curing conditions

Observation were made 56 days after cracking, it was concluded that specimen in series NCS3% showed maximum crack healing while those of NCS5% gave lowest water permeability results because of the reason that greater amount of inert microencapsulation caused water-proofing

effect and didn't allow contact of bacteria with water, hence decreasing self-healing ration and permeability. While observing effect of incubation techniques it was evident that wet dry cycle with water was the most effective technique as during wet cycle specimen absorbed water and during dry cycle it got more oxygen from atmosphere.

In addition to type of bacteria, selection of carrier is also a matter of great significance. When it comes to strength of concrete and viability of bacteria, carrier material is an important factor in the manufacturing of self-healing concrete Therefore, studies related to the carrier materials are also presented.

Gadea et al. (2010) studied the use of polyurethane foam wastes (PFW) in making lightweight cement based mortar. During his research he grounded the PFW to the size less them 4 mm and gradually replaced fine aggregate with grounded PFW. Two types of cement were used in this study i.e. Cem I 42.5 R and Cem IV 42.5 N. mechanical properties including workability, permeability and strength were checked for each replacement level. It was found that inclusion of PFW increases the workability of mix and at the replacement level of 100% it increases the workability of mix by 120 min. on the other hand, effect of PFW on the flexural and compressive strength of mix at various checking days was devastating.

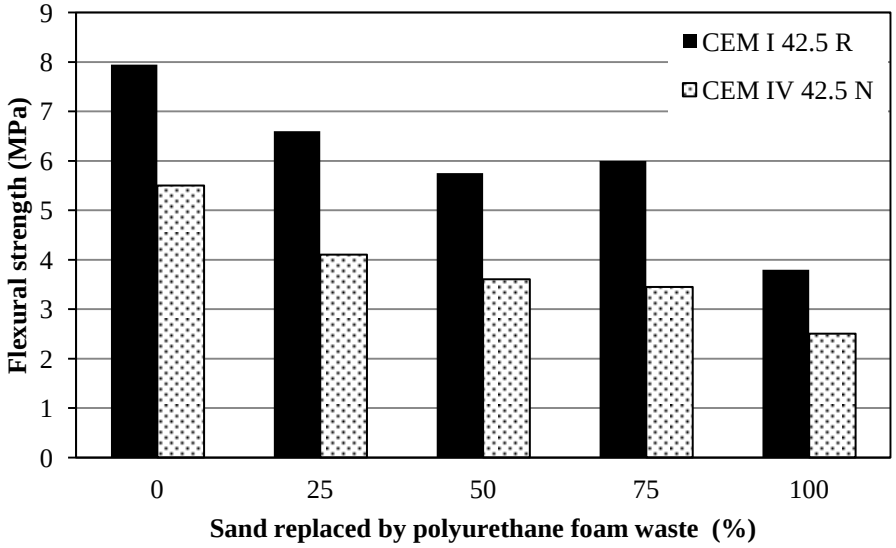


Fig. 2.14 Flexural strength at various replacement levels of polyurethane foam wastes

Sixuan (2012) studied the possibility of using graphite nano platelets (GNP) in cement based construction materials. During his study, GNP particles were added in the cement mix and both mechanical and electrical properties of cement mix were studied. To study the compressive and flexural strength of cement mix two different kind of samples were made. 50 x 50 x 50 mm cubes were made to study the compressive strength of mortar and 40 x 40 x 160 mm prisms were made to study the flexural properties. During this experimental work it was found that increasing the amount of GNP in mortar has a better effect on the compressive and flexural properties of mortar. Three different percentages of 0.5, 1 and 1.5% were used. It was seen that mix with 1.5 % GNP had maximum compressive strength where mix with 1% GNP had maximum flexural strength and flexural strength started to decrease when percentage was increased to 1.5%.

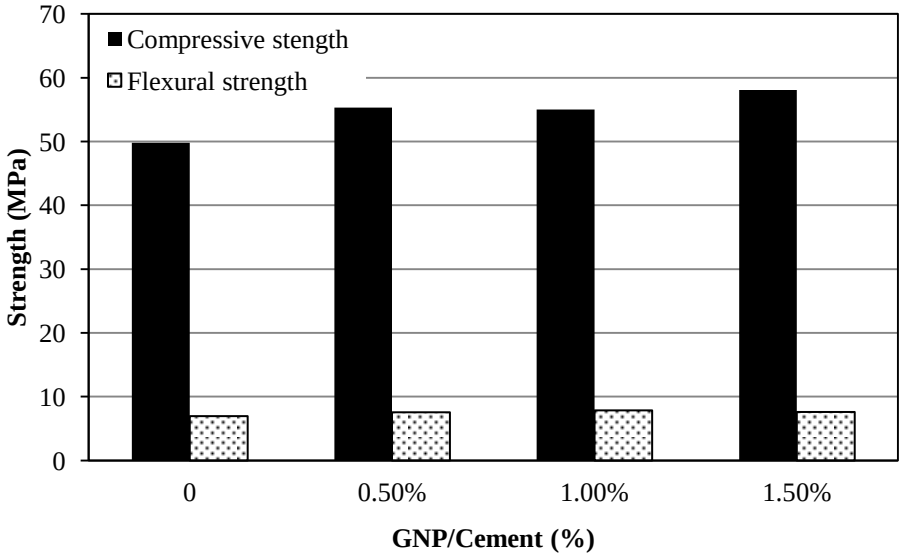


Fig. 2.15 Effect of GNP on strength of cement based mortar

This difference in healing ratio, threshold crack healing and difference in compressive strength in various studies can be attributed to a number of factors such as type of bacteria, their varying concentrations, various curing procedures, nutrient compounds and material used for incorporation of bacteria. To identify most practical self-healing techniques in concrete, there is a need to determine the most effective bacteria along with its optimum concentration, a host precursor and activation process.

It is evident from the researches presented here that there is still not enough data to determine the most effective bacteria and its optimum concentration. As we compare the results of various

researches there is a considerable difference in self-healing capacity displayed by the procedures. These results are for the cracks in specimens with B.Sphaericus as healing bacteria, incorporated in mix using various techniques. Comparison of the results shows a considerable variation in permeability of self-healing concrete. Technique of incorporating bacteria with micro-encapsulation exhibits better healing as compared to others, however, micro-encapsulation is a comparatively difficult process and needs extra care. Considering the variation displayed by different techniques, it is hard to recognize an optimum carrier with better incorporation techniques.

Owing to the low tensile strength of concrete, it is desired that introduction of carrier compounds in self-healing concrete give beneficial effects by providing protective cover to bacteria as well as enhancing the flexural tensile strength of concrete. Fig. 2.16 shows the comparison of flexural strength provided through polyurethane (PU), light weight aggregate (LWA) and graphite nano platelets (GNP). It can be seen that PU and LWA cause decrease in the flexural strength of mortars instead of increasing it. GNP on the other hand provides better flexural strength to the mortar and at 1% addition of GNP maximum flexural strength was observed. It is evident, out of three presented methods only GNP increases the flexural strength of concrete, therefore GNP is considered helpful in reducing crack formation in concrete.

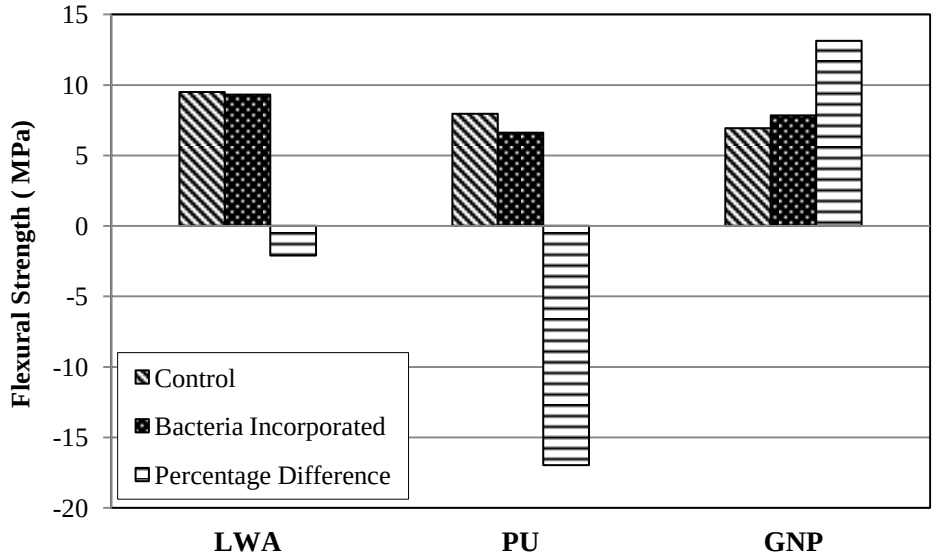


Fig. 2.16 Comparison of flexural strength in PU, GNP and LWA incorporated concrete

Not only the choice of carrier compound affects the self-healing capacity but choice of bacteria selection also plays a great role in the self-healing process. Results on compressive strength by

Ramachandran et al. (2001) show that using bacteria, B.Pasteurii, 28 days strength of concrete increased by 18% at concentration of 7.6 x 10^3 cells/cm^3, whereas the research work done by Ghosh et al. (2005) shows that bacteria, Shewanella, at the concentration of 10^5cells/cm^3 results in 25% increase in 28 day strength than the control samples which is greater as compared to 18% increase as reported by Ramachandaran and 2% increase in strength as measured in case of E.Coli. In the case of B.Pseudofirmus, it can be see that at a concentration of 6 x 10^8 cells/cm^3 results in a 10% decrease in the strength of mortar. in addition to that, research work done by Wang et al. (2012) shows that compressive strength of mortar decreased with the increasing replacement level of B.Sphearicus and show an approximately 35% decrease in 28 day compressive strength at the replacement level of 5%. Fig. 2.17 shows a comparison of effect of different bacteria on strength of mortars.

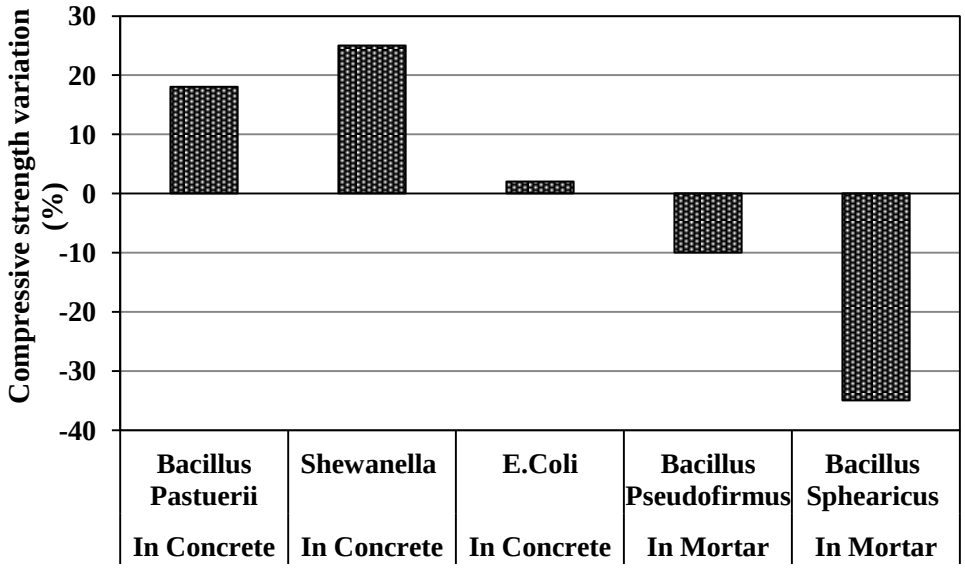


Fig. 2.17 Effect of various bacteria on compressive strength of self-healing concrete

These results depict that there is still uncertainty about appropriate bacteria and their optimum concentration that can have a better impact on compressive strength of concrete. Therefore, work is required to study the impact of new bacteria and incorporation techniques on the compressive strength of concrete to determine optimum conditions.

3. Summary

A review of previous studies on self-healing techniques has been presented. It is seen that quite limited type of bacteria and carrier compounds have been used in the previous studies. The

purpose of this research is to collect sufficient data on the efficiency of Bacillus Subtilis and use of graphite nano platelets to check the possibility of an alternate bacteria and carrier compound for self-healing studies that can lead to most conducive and favourable outcome in self-healing of concrete.

CHAPTER 3

EXPERIMENTAL PROGRAM

1. General

In recent years, several studies have brought the attention towards the importance and significance of the emerging technique of bio-influenced self-healing concrete. These studies were, however, limited to a small group of bacteria and protective carrier compounds. Therefore, there is a need to carry out in-depth study on new alternatives in the form of both bacteria and protective carrier compounds. Evaluating the best suited bacteria and ideal protective carrier materials can be helpful in achieving an optimum self-healing process in bio-influenced concrete. Compressive strength tests and pre-cracking techniques were applied to concrete specimen incorporated with precursor materials and carrier compounds containing bacteria. Description of specimen for these tests is provided in the Table 3.1.

Table 3.1 Description of test specimens

Specimen	Sample dimensions for compressive strength tests		Sample dimensions for pre-cracking	
	Dia (in)	Height (in)	Dia (in)	Height (in)
Mix 1	6	12	6	4
Mix 2	6	12	6	4
Mix 3	6	12	6	4
Mix 4	6	12	6	4

Specimen names indicate the techniques and methods used for the incorporation of bacteria in them. Control specimens containing neither bacteria nor organic pre-cursor compound are denoted by “Mix 1”, whereas, “Mix 2” shows that bacteria were incorporated in those specimens directly without the use of any protective carrier compounds. In the same way, those incorporated with bacteria by the use of light weight aggregates (LWA) as protective carrier are represented by “Mix 3” and specimen containing graphite nano platelets as a mean of bacteria inoculation are termed as “Mix 4”.

The tests for compressive strength were conducted according to criteria defined in ASTM C-39 (revised 2014) and ASTM 2809 standard was followed during the process of scanning electron microscopy. Crack widths were measured by the use of crack width measuring microscope with a least count of 0.02 mm. This chapter describes the experimental program including testing procedures. Details about material properties, sample preparation and testing methodology have also been included in this chapter.

2. Materials

The important feature of this study is the comparison of various techniques of bacterial incorporation in concrete. It is being used to compare the results achieved by use of both GNP and LWA and determine the better choice. The constituent materials used in this study were tested conferring to their relevant standards and to ensure consistency in their supply, periodical quality assurance tests were also carried out. Tests conducted on different materials used in this study are described below.

2.1 Micro-organism

In order to be used as a healing compound in the concrete samples, the micro-organism had to possess certain features. It must be able to withstand alkaline atmosphere of concrete in order to carryout production of calcium carbonate, it should produce copious amount of calcium carbonate without being affected by calcium ion concentration, it must be able to withstand high pressure and should be oxygen brilliant to consume much oxygen and minimize corrosion of steel (Gupta et al., 2013; Jonkers et al., 2010; Rao et al., 2013). Bacteria namely, Bacillus Subtilis, was selected in this study as it fulfilled the necessary criteria for survival in harsh environment. It is gram-positive bacteria having an ability to form spores when subjected to unfavourable conditions. This spore formation provides its protection against high mechanical pressure and alkaline environment, making it ideal selection. Members of genus bacillus can produce spores which can lay dormant for over 200 years (Schlegel, 1993).

Bacterial solution (bacteria in nutrition bath), specially prepared and treated to ensue spore formation in controlled microbiology laboratory, in usually used to introduce these bacteria in different incorporation techniques in concrete. Fig. 3.1 shows the bacteria solution containing spores of B.Subtilis bacteria. The quantity of solution required in the mix was calculated on the basis of concentration found by optical density test using a spectrophotometer. For this purpose,

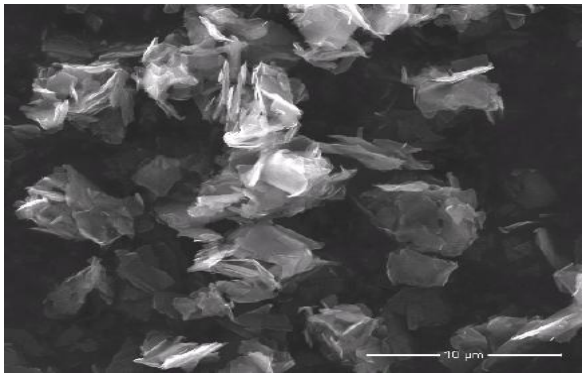
medium in which bacteria was growing in was selected as blank. This blank solution was used as a reference, on the basis of which optical density of bacterial solution was measured. Initially 0.5 ml of blank solution was placed in spectrophotometer and 600 nm wavelength was selected. After the machine had read the blank solution, 0.5 ml of bacterial solution was placed in spectrophotometer and wavelength of 600 nm was selected to carry out the optical density test. On the basis of this test, concentration of bacteria in the solution was measured using the expression $Y=8.59 \times 10^{7} X^{1.3627}$ (Ramachandran et al., 2001). Where Y is the bacterial concentration per mL and X is the reading at OD_{600}. With spectrophotometer, the bacterial concentration was found to be $2.8x10^{8}$ cells/ml. Based on these results, spore concentration in samples was kept equal to $3x10^{8}$ cells/cm^{3} of concrete mixture.

Fig. 3.1 Bacteria solution in a cylinder

2.2 Graphite nano platelets

Studying the effect of graphite nano platelets (GNP) as carrier material and its comparison with other carrier compounds is the core objective of this research. Therefore, evaluating the properties of GNP is of prime importance. Physical appearance of GNP is shown in Fig. 3.2.

(a) Graphite nano platelets



(b) Microstructture of raw graphite nanoplatelets

Fig. 3.2 Graphite nano platelets

The material properties of GNP provided by supplier are given in the Table 3.2

Table 3.2 Properties of GNP

Properties of GNP	
Grade	Nano 25
Primary particle size	≤ 100nm
Composition	99 % carbon
Surface Area	180 m^2/g
Lamella thickness index (LTI)	14-15

Absorption tests were also conducted on GNP to find their maximum absorption capacity. This test helped to determine the amount of GNP required as a carrier compound for bacteria. Graphite nano platelets were submerged in water for 24 h and were filtered by the help of filter paper. After measuring the saturated weight of GNP, it was oven dried and weighted again to calculate water absorption. On the basis of this test water absorption of GNP was found to be 62%

2.3 Light weight aggregate

Using light weight aggregates (LWA) was studied as a carrier material and was compared with GNP to evaluate the optimum method of bacteria transfer in the mix. Fig. 3.3 shows the LWA used in this study for the incorporation of bacterial spores.

Fig. 3.3 Light weight aggregates

Like GNP, water absorption test was also conducted on LWA to determine the exact amount of LWA to be used. To evaluate the water absorption in light weight aggregate 500 gm of LWA were submerged in water to make it saturated. After 24 h they were surface dried and their saturated weight was measured. These aggregates were later placed in oven and were dried for 24 h and dry weight was measured. On basis of this test water absorption of LWA was found to be 16.6%

3. Mix proportion

Four different types of mix were used for the study. The mix proportion for these four different categories of specimens contained ordinary portland cement (OPC) type – I conforming to ASTM C 150-07 as 370 kg/m^3, fine aggregate as 840 kg/m^3, coarse aggregate as 990 kg/m^3 and calcium lactate of 18 kg/m^3 with a water to cement ratio of 0.4 for all the mixes for concrete. The mix was designed for a compressive strength of 4000 psi. Sikament® - 520 set-retarding admixture was used as 1% by weight of cement for producing free-flowing concrete in hot climates. ASTM C 191-11 and ASTM C 187-11 codes were conformed for normal consistency test and initial and final setting time respectively for cement. Control specimens were named “Mix 1” in which no bacterial spore specimens were added. In “Mix 2” specimens, bacteria were incorporated directly by mixing the bacterial solution in water during mixing of concrete without use of any protective carrier compound. In the same way, those incorporated with bacteria by use of LWA as protective carrier were labelled as “Mix 3”. In order to incorporate bacteria, LWA were kept soaked in bacterial solution for 24 h till they were saturated prior to their mixing in

concrete. Specimens containing GNP as a mean of bacteria introduction were termed as "Mix 4". GNP was also soaked with bacterial solution before mixing in concrete. However, in order to ensue uniform distribution of GNP in concrete, superplasticizer (Sikament® - 520) was added to GNP soaked bacterial solution. Addition of superplasticizer in GNP prior to mixing in concrete ensures uniform distribution of GNP particles throughout the concrete mix (Sixuan, 2012).

The mixing procedure and time were kept constant for all the concrete mixes investigated and details of mixing proportions for different batches of concrete are presented in Table 3.3.

Table 3.3 Mix design of different sets of specimens

Specimens		Mix 1	Mix 2	Mix 3	Mix 4
Cement	kg/m^3	370	370	370	370
Fine Aggregate	kg/m^3	840	840	840	840
Coarse Aggregate	kg/m^3	990	990	990	990
Water cement Ratio		0.4	0.4	0.4	0.4
Super Plasticizer	(%)	1	1	1	1
Calcium Lactate	kg/m^3	18	18	18	18
Bacteria with Spore concentration (2.8×10^8 cells/ml)	$liter/m^3$	0	6.33	6.33	6.33
Bacteria incorporation technique		None	Direct	By LWA	By GNP

4. Test specimens

Specimens were removed from moulds after 24 h of casting and were placed to be cured in controlled conditions. For all mix types, samples for two different dimensions were made. For pre-cracking specimens of 150 mm dia and 100 mm height were prepared and five specimens pre-cracked at 3,7,14 and 28 days for each mix were studied for healing measurements. For the compressive strength tests cylindrical specimen of diameter 150 mm and height of 300 mm were prepared and an average of three specimens were utilized to determine 3,7,14 and 28 days compressive strength. Fig. 3.6 shows prepared specimens for different tests to study the material properties. Test specimens were made in accordance to ASTM C 39. Proper tamping was carried out to ensure the compaction of specimens. Moreover, samples were also subjected to scanning electron microscope (SEM) analysis to monitor microstructural changes due to mineral

formation. In addition, samples of mineral produced during the process of self-healing was taken and was subjected to X-Ray Diffraction analysis (XRD) to identify its chemical composition.

Casting of samples for pre-cracking

Casting of samples for compressive strength measurement

Fig. 3.4 Casting of samples

5. Test procedure

The tests for compressive strength were performed on 3, 7, 14 and 28 days cured samples and an average of three samples was taken. At the appropriate age the specimens were removed from water and surface water was wiped off. In order to ensure uniform distribution of load on the face of sample, proper capping was done prior to placing sample in the compression machine as shown in Fig. 3.5. Sample was placed in testing machine and load was applied on permissible rate.

However, for pre-cracking of samples, samples were monitored carefully under controlled compressive load. Application of load was stopped immediately after the development of visible cracks. The crack widths were measured at different points on the specimens and the cracks with a width around 1 mm were selected and marked for further observation of self-healing. Initial crack width was measured at the time of crack development by the help of crack width measuring microscope, accurate up to 0.02 mm.

Capping of samples

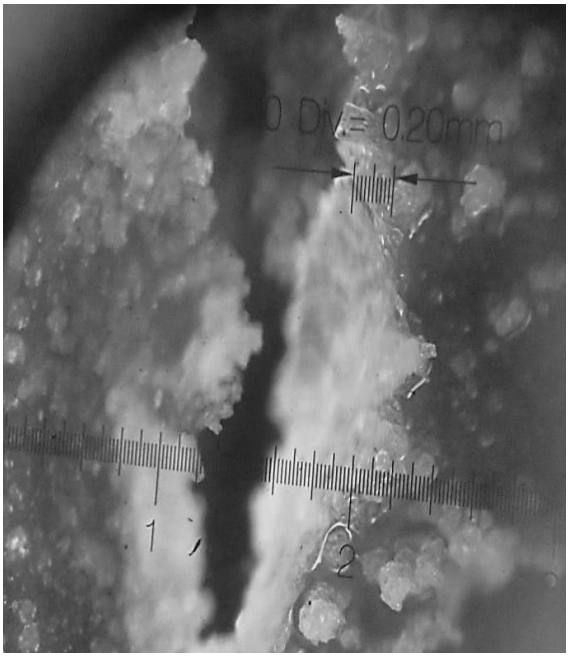
Compression testing machine

Cylinder after compresssive strength test

Fig. 3.5 Capping of cylinders, compression machine and cylinders after compressive strength tests

Fig. 3.6 shows the crack measuring microscope. Cracks were marked to ensure that crack widths are measured at the same location every time as shown in Fig. 3.7. After measuring the initial crack width, the pre-cracked specimens were continued to cure under controlled curing conditions. At appropriate age of 3,7,14 and 28 days, these pre-cracked specimens were taken out and crack width was measured again by using crack width measuring microscope. However, during this study visual inspection was carried out for this purpose as shown in Fig. 3.8 and difference between crack widths measured initially and on later stages was calculated as the measure of self-healing efficiency of concrete. An average of three tests on specimens was taken for compressive strength, while for self-healing measurement an average of five test specimens was accounted.

(a) Crack measuring microscope



(b) Crack width measurement by crack measuring microscope

Fig. 3.6 Crack measuring microscope and measurement of crack by it

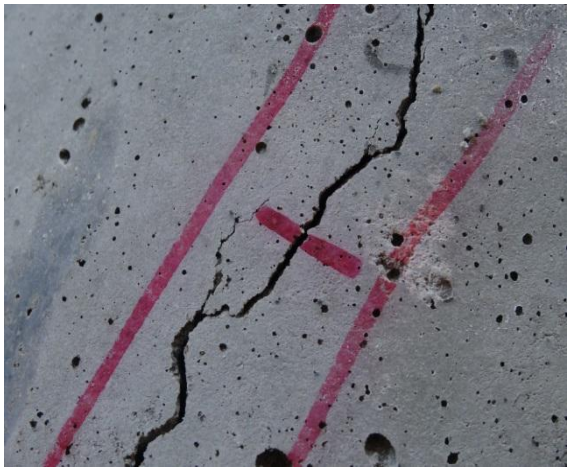


Fig. 3.7 Marking of cracks on pre-cracked specimens to ensure that multiple reading are taken from the same point

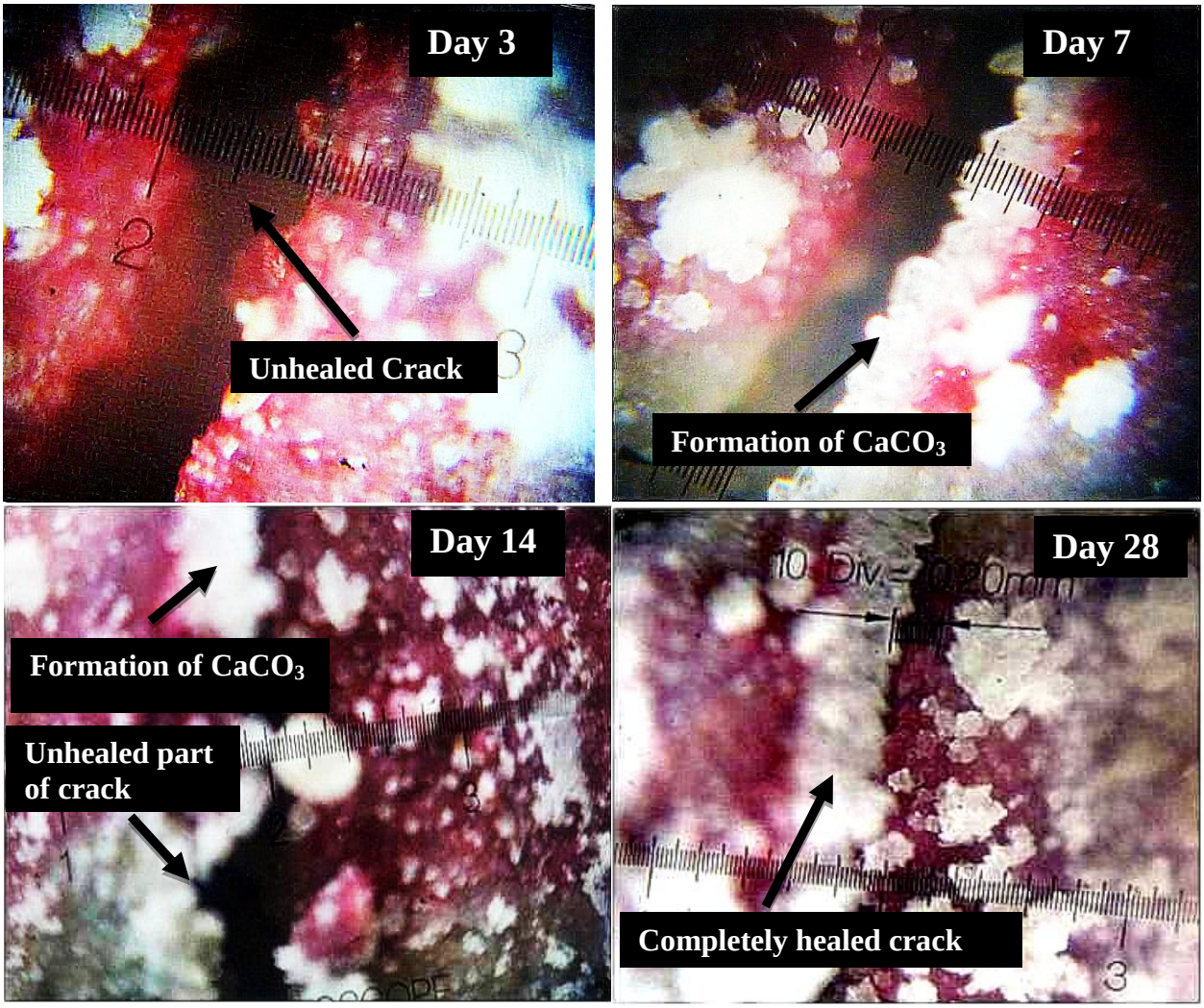


Fig. 3.8 Closer view of cracks showing self-healing process with calcium carbonate formation

In addition to these tests, samples were collected form the cracked samples at the age of 7 and 28 days after pre-cracking and were subjected to scanning electron microscopy (SEM).

Results from compressive strength tests, visual inspection of crack healing, SEM and XRD were recorded. These results were later analyzed and compared to reach the conclusion regarding comparison of techniques. Comparisons of these results along with related discussion are presented in chapter 4.

CHAPTER 4

RESULTS AND DISCUSSIONS

1. General

Results from previously mentioned tests are presented and discussed here to determine the efficiency of self-healing process of all mixes. These results include the crack width measurements, visual inspection of cracks, compressive strength of self-healing concrete samples and micro structural study through SEM and mineral composition of healing compounds through XRD analysis. Furthermore, relationships of self-healing at different curing days and compressive strength with self-healing techniques are elaborated.

1.1 Self-healing analysis

Pre-cracked specimens were monitored at specified time of 3,7,14 and 28 days to determine the efficiency of self-healing process by use of crack measuring microscope. These cracks showed significant self-healing specially in specimens of Mix 2, Mix 3 and Mix 4. Production of calcium carbonate crystals ($CaCO_3$) in significant amount was observed in these cracks as shown in Fig. 4.1. The healing compound, $CaCO_3$ crystals, was the main factor in reducing the cracks width in bacteria incorporated specimens. This formation of healing compound, as a result of bacterial conversion of calcium lactate into calcium carbonate, was also observed and discussed by Jonkers (2010).

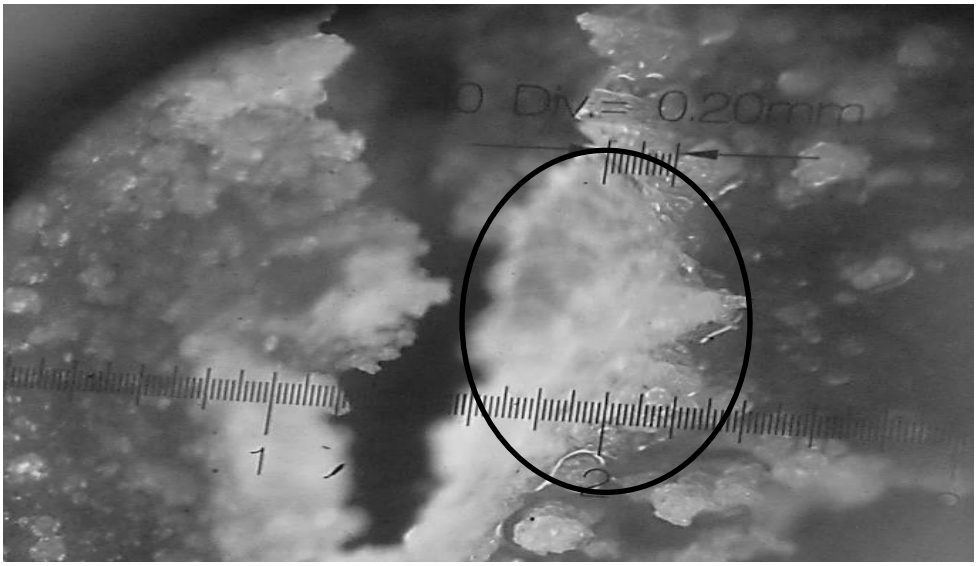


Fig. 4.1 Crack width measurement by crack measuring microscope

The measure of healing is obtained in millimetres as the difference of initial crack width and healed crack width in different predefined times. Fig. 4.2 illustrates the efficiency of crack healing of each mix as a function of time. While observing specimens of all incorporated techniques pre-cracked on 3 days of curing, healing of cracks was prominent especially after 7 days of curing. However, as shown in Fig. 4.2 healing efficiency of Mix 4 samples with GNP as a carrier compound show the maximum healing as a function of time on all crack measuring days. This increase in healing in Mix 4 is due to the particle size of GNP. The small size of GNP enables it to act like a filler material (Sixuan, 2012) and assures its uniform distribution throughout the mixture. GNP particles are saturated with bacteria medium, resulting in uniform distribution of bacteria through colloidal dispersion in the mix and ensuring the availability of bacteria medium at the crack site for formation of healing compound. These self-healing bacteria when come in contact with water, ingressed through cracks, gets activated and carry out the conversion of calcium lactate into water insoluble calcium carbonate which results in crack healing. Mix 4 specimens are followed by specimens containing LWA as a carrier material. While LWA has particle size greater than that of GNP, therefore it becomes difficult to ensure the distribution of LWA in the mix as uniformly as GNP. Bigger size of LWA makes it harder for LWA to get in the inter particle spaces where GNP can penetrate easily owing to its nano sized particles. This feature of LWA hinders the equal and even distribution of bacteria in the mix hence decreasing the efficiency of self-healing process in concrete. Fig. 4.2 illustrates that all the samples incorporating bacteria had showed improvement in healing results as compared to control samples. Controlled concrete samples, without any bacteria, showed small crack healing as well, which can be credited to a number of reasons. Some cement particles are not completely hydrated during the initial mixing process and at early age. These particles go through continued hydration resulting in production of expansive hydration products which eventually lead to healing of cracks. Another factor for crack healing in controlled samples is the carbonation process of calcium hydroxide. This carbonation process leads to conversion of calcium hydroxide to calcium carbonate.(Homma et al., 2009; Jonkers and Schlangen, 2009; Wiktor and Jonkers (2011)).

Calcium carbonate production in controlled specimens is mainly dependent on the availability of carbon dioxide (CO_2) dissolved in the ingressed water. However, the amount of carbon dioxide dissolved in water is limited, which in turn limits the production of calcium carbonate due to

carbonation process. Furthermore, portlandite ($Ca(OH)_2$) is soluble in water therefore, the ingressed water not only provides carbon dioxide for the carbonation process but it also dissolves the precipitated calcium hydroxide formed in controlled samples, leaving less calcium hydroxide in concrete samples for conversion into calcium carbonate.

According to Ter Heide (2005) healing in controlled samples can also be attributed to the swelling of cement matrix.

Contrary to that, the process of self-healing in bio concrete is different due to the presence of two component healing system i.e. calcium lactate and self-healing bacteria. After coming in contact with the water ingressed through cracks dormant bacterial spores get activated. As a result of bacterial activity, calcium lactate present in bio concrete is converted directly into calcium carbonate, which is insoluble in water. This bacterial activity also results in the production of CO_2 within the specimens, as a by-product of metabolic reactions, hence ensuring the availability of more carbon dioxide for the process of calcium hydroxide carbonation leading to production of calcium carbonate. Both of these process take place simultaneously within the bio concrete samples resulting in two fold production of healing compound (Schlangen et al., 2010).

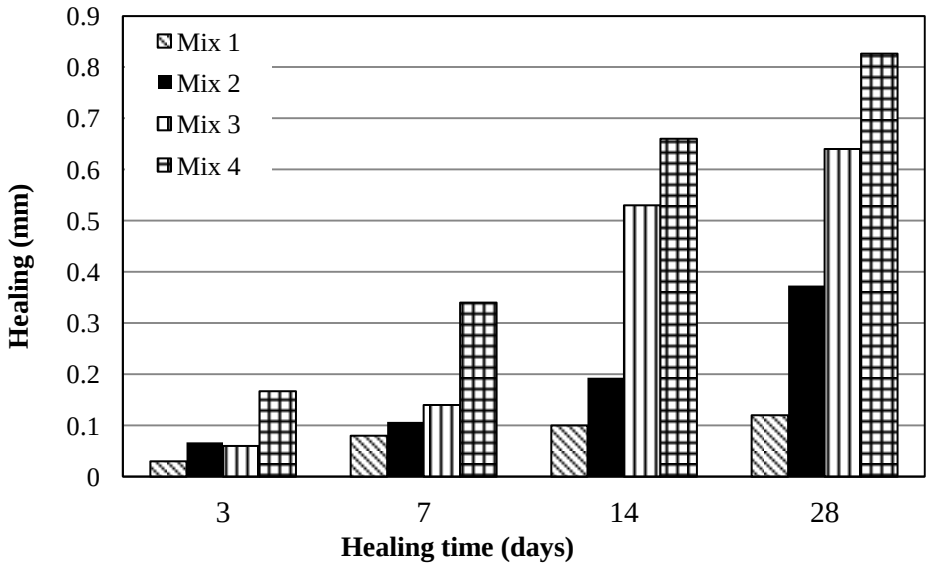


Fig. 4.2 Crack healing in specimens pre-cracked at 3 days

Crack healing in specimens pre-cracked on 7 days of curing show similar trend observed in specimens pre-cracked at 3 days as shown in Fig. 4.3. Fig. shows that Mix 4 specimens, having

GNP as carrier compound, show much more healing as compared to all other techniques. As previously discussed, this efficient behaviour of GNP is due to its small size which ensures thorough and even distribution of particles carrying bacteria in the mix. This small size allows GNP particles to enter the inter-particle spaces and make the availability of bacteria possible for the process of self-healing. LWA, being bigger in size as compared to GNP, cannot penetrate in the micro sized gaps which limits the capacity of LWA to evenly distribute self-healing bacteria in concrete matrix. The maximum healing of cracks observed in Mix 4 after 28 days was 0.81 mm. Whereas, crack healing of 0.61 mm was observed in Mix 3 specimens, comprising of LWA. Mix 2 samples, prepared by direct introduction of bacteria in the concrete mix, showed healing of 0.37 mm. However, comparison of results achieved from 3 and 7 days pre-cracked specimens indicates that there has been a slight decrease in self-healing observed in specimens of all mix types. This decrease in self-healing is because of an overall reduction in further production of hydrated products in concrete as significant amount of cement is already hydrated. Another reason for this overall reduction in bio-concrete specimens is due to slight decrease in viability of bacterial survival in concrete as development of denser micro-structure in concrete is started. Mix 3 and Mix 4 are least affected by denser micro-structure at this stage, because at this stage pressure exerted by microstructure development is not significant and carrier compounds are able to provide better cover to bacteria against the exerted pressure.

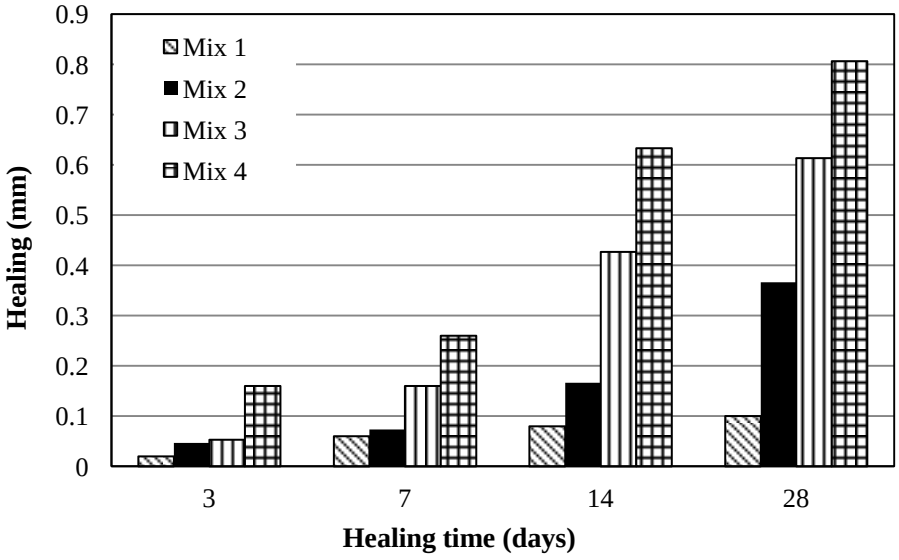


Fig. 4.3 Crack healing in specimens pre-cracked at 7 days

Healing observed in specimens pre-cracked after 14 days of curing is expressed in the Fig. 4.4. Mix 3 specimens show maximum healing in samples pre-cracked at 14 days of curing. Mix 4, which displayed maximum healing in samples pre-cracked at 3 and 7 days of curing, proved less efficient than Mix 3 when pre-cracked at 14 days of curing.

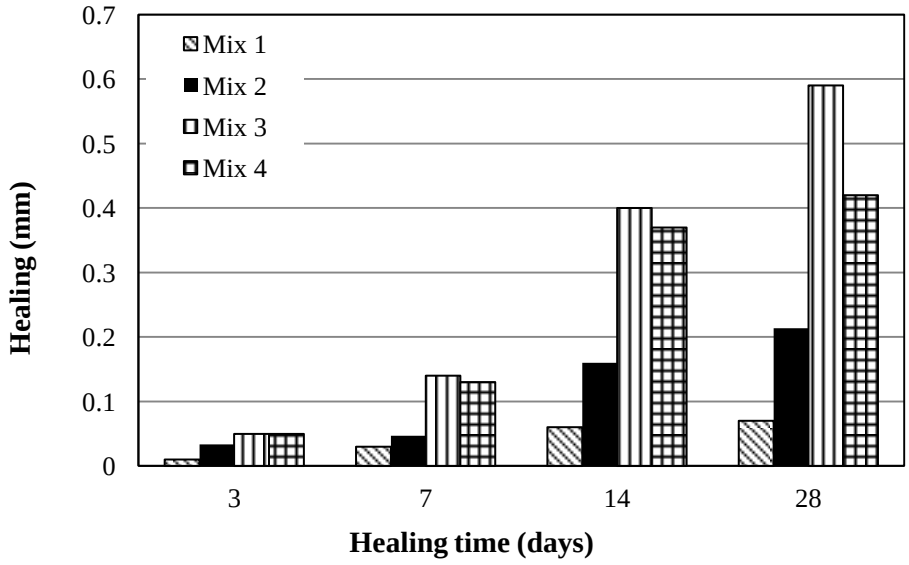


Fig. 4.4 Crack healing in specimens pre-cracked at 14 days

This change in behaviour of Mix 4 can be due to continued development of dense micro structure resulted by continued hydration reactions in concrete. This dense micro structure in concrete creates a pressure on the carrier compounds containing incorporated bacteria. This pressure was not resulting in a significant affect over the viability of bacteria survival in specimens pre-cracked at 7 days. However in specimens pre-cracked at 14 days of curing this pressure has been increased enough to have a considerable effect on the self-healing capacity of all specimens. The effect of micro-structural pressure is most significant in specimens of Mix 2 and Mix 4. On the other hand, LWA is seen to be least affected by this pressure. GNP is weak when subjected to multi axial loading (Sixuan, 2012), hence, it is unable to provide better cover to bacteria as compared to that provided by LWA. This leads to elimination and annihilation of bacteria in Mix 4, therefore healing process observed in Mix 4 was decreased than it was in samples pre-cracked at 3 and 7 days. This decline in self-healing can be attributed to underdeveloped microstructure which is not fully matured till only 7 days of casting and becomes more compact and mature at 14 days. The change in self-healing process can also be

observed in the Mix 2 specimens, with directly incorporated bacteria, with the value of 14 days healing decreased from 0.37 mm to 0.21 mm. This decreases in crack healing of Mix 2 samples is due to decrease in the viability of bacteria survival in concrete under the pressure applied during the mixing phase and that developed due to formation of dense micro structure (Jonkers et al., 2010).

Jonkers and Schlangen (2009) attributed this decrease in self healing activity to disintegration of calcium lactate in the concrete mix. However, small amount of calcium lactate in his research was used (1% of cement weight) and in this research 4.86% of cement weight has been used to ensure enough availability of calcium lactate to the bacteria. Furthermore, this drastic decrease in bacterial activity was only observed in Mix 2 specimens, which shows that this decrease is the result of elimination of bacteria and not because of calcium lactate disintegration..

Fig. 4.5 depicts the results obtained through measurements of self-healing in samples pre-cracked after 28 days of curing. It can be seen that Mix 3, with LWA, is showing maximum healing of 0.52 mm, higher than all other mixes. Mix 4 specimens, with GNP, shows much less healing than it showed at 3 and 7 days of pre-cracking. The healing exhibited by Mix 4 specimens was 0.38 mm, which is higher than healing of 0.15 mm showed by Mix 2 specimens.

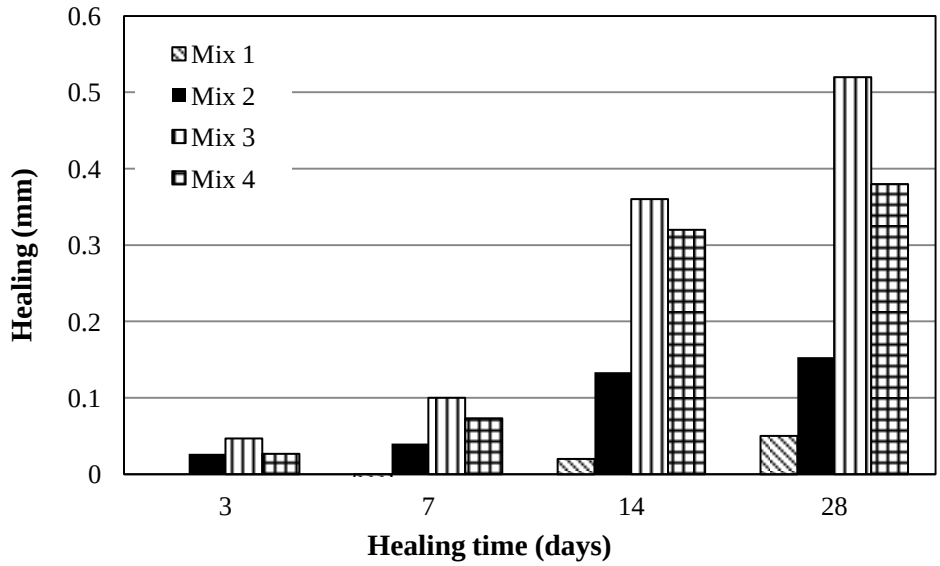


Fig. 4.5 Crack healing in specimens pre-cracked at 28 days

This shows that Mix 4 still provides a significant improvement in healing with GNP incorporated bacteria but this improvement in healing is lower than that exhibited by Mix 3. This reduction in healing of Mix 4 specimens at 28 day pre-cracking can again be attributed to the dense microstructure formed in the concrete after 28 days of curing similar to samples pre-cracked at 14 days of curing.

1.2 Microstructure analysis

In addition to the results achieved by visual inspection of concrete samples, specimens of all four mixes were subjected to scanning electron microscopy (SEM) analysis to study changes in concrete microstructure due to self-healing. For comparison of self-healing process SEM analysis was conducted at 7 and 28 days of healing.

Production of calcium carbonate based crystals were the main focus of this study as it expresses the crack healing efficiency of respective mix. Calcium carbonate crystals are developed in three different forms which are named calcite, aragonite and vaterite (Rao et al., 2013). Out of the three, calcite is most stable form of calcium carbonate.

Fig. 4.6 shows SEM images of all four mixes at 7 days pre-cracked specimens with 2 µm resolution. Fig. shows the development of calcite crystals which are orthorhombic in nature (De Yoreo and Vekilov, 2003). Mix 4 containing GNP as carrier compound showed maximum calcium carbonate ($CaCO_3$) formation as compared to mixes with other techniques.

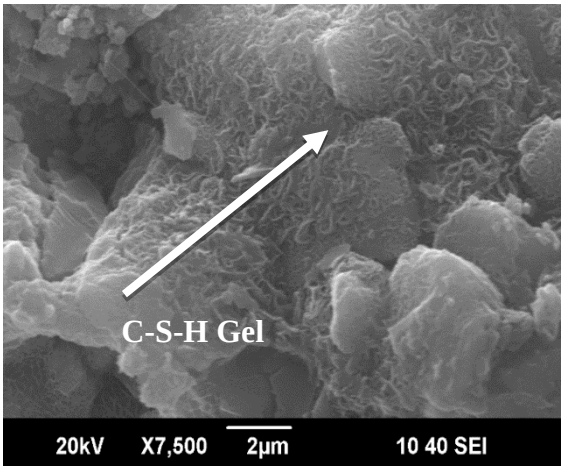
This $CaCO_3$ formation results from presence of two component, bacteria and calcium lactate, based healing system. Although $CaCO_3$ is also formed in controlled samples, however presence of bacteria along with calcium lactate catalysis the production of $CaCO_3$ crystals. $CaCO_3$ crystals formation in higher quantities and similarities in shape is identical and conform to those reported by Jonkers et al. (2010) and Wang et al. (2012). The chemical process of calcium carbonate formation by bacterial activity is presented in equation given below.

$$CaC_6H_{10}O_6 + 6O_2 \rightarrow CaCO_3 + 5CO_2 + 5H_2O \qquad (1)$$

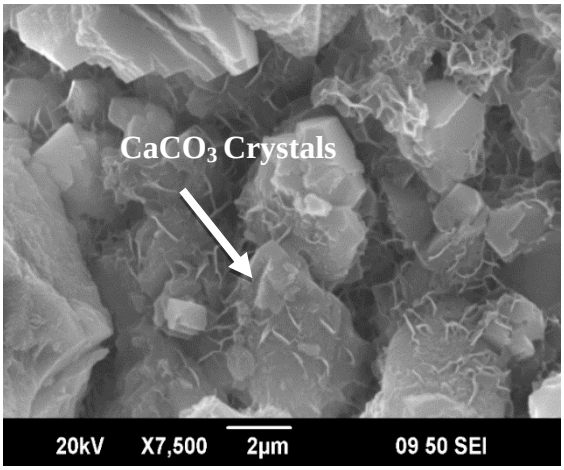
As stated earlier that production of CaCO3 is not only limited to concrete with bacteria incorporated in them. The presence of CaCO3 is also evident in controlled concrete specimens. However, the process of CaCO3 crystals formation in controlled specimens is quite different to that in bacteria incorporated specimens. Formation of $CaCO_3$ in Mix 1 specimens is due to the

carbonation of calcium hydroxide, as given in equation 2, which is one of the major hydration products of cement.

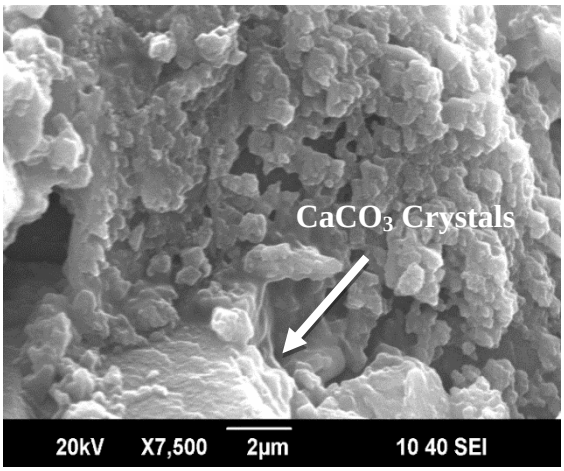
$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \quad (2)$$



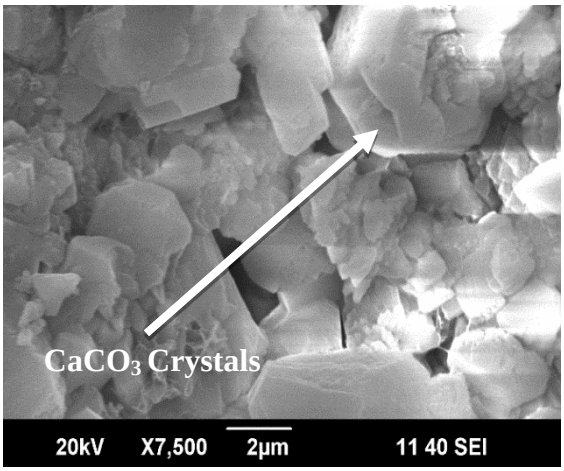
A : Mix 1



B : Mix 2



C : Mix 3



D : Mix 4

Fig. 4.6 Scanning electron microscope analysis of 7 days pre-cracked samples

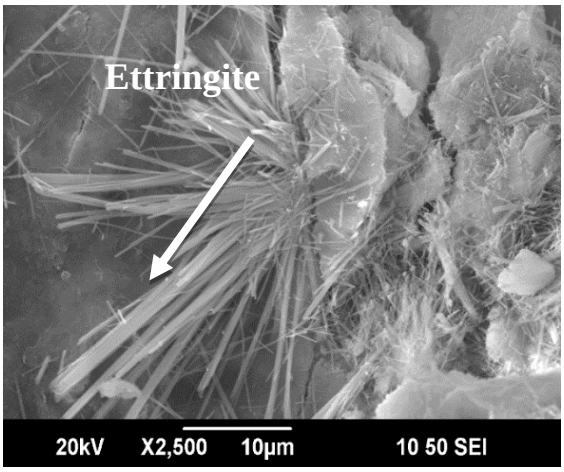
However, this production of $CaCO_3$ crystals in Mix 1 due to carbonation process is very slow as compared to those produced by bacterial activity. In addition, as calcium carbonate production in controlled specimens is due to the availability of carbon dioxide (CO_2) dissolved in the ingressed water; therefore, less amount of CO_2 is available for carbonation process. Furthermore, as portlandite ($Ca(OH)_2$) is soluble in water so whenever it comes in contact with ingressed water it gets mixed in it, leaving less calcium hydroxide on the contact surface to convert in $CaCO_3$. On the other hand, in bio concrete, the process is different due to the presence of calcium lactate and

bacteria. Bacteria converts calcium lactate directly into calcium carbonate which is insoluble in water and as result of this metabolic reaction CO_2 is produced which reacts with calcium hydroxide on spot and does not let it wash away. Hence, producing more calcium carbonate (Schlangen et al., 2010).

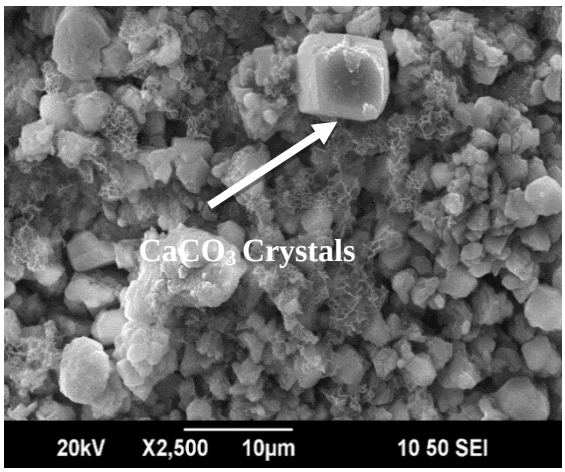
Fig. 4.7 shows SEM analysis of 28 days pre-cracked specimens. It can be seen that calcium carbonate crystal formation is higher in Mix 3, observed in specimens pre-cracked at 28 days compared to Mix 2. The amount of $CaCO_3$ in Mix 2 seems even less as compared to those produced in samples pre-cracked at 7 days of curing. Curing for 28 days improves the development of microstructure within the concrete samples, making it denser. This results in decreasing the pore sizes and application of pressure on self-healing bacteria. This leads to elimination of bacteria, leaving less number of bacteria in specimens for the production of calcium carbonate.

Fig. 4.6 shows that Mix 4 displayed much more crystal formation in 7 days pre-cracked samples as compared to Mix 3. However, as shown in Fig. 4.7, GNP is no longer able to provide effective cover to bacteria and therefore, calcium carbonate crystal formation in Mix 4 is reduced significantly compare to $CaCO_3$ crystal produced in Mix 3. These results depict that in samples pre-cracked at 28 days of curing; LWA provides the best cover to bacteria. As described by Sixuan (2012), GNP are weak when it comes to multi-axial load application and does not provide better cover to bacteria. Thus, with the increase in completion of hydration reaction and decrease of pore size, healing efficiency of Mix 4 samples decreases. However, LWA provides cover during the mixing phase and provides better protection to spores in the samples as it provides resistance against the pressure developed in samples due to microstructure development. The variation in $CaCO_3$ formation with and without carrier compound conforms the trends seen in the results, in the study carried out by Wiktor and Jonkers (2011). The crystals formation observed in SEM images are also similar to those observed and presented by Wang et al. (2012) and Wang et al. (2014) which confirms the formation of calcium carbonate with similar crystalline structure.

A : Mix 1



B : Mix 2



C : Mix 3

D : Mix 4

Fig. 4.7 Scanning electron microscope (SEM) analysis of 28 days pre-cracked samples

1.3 X- Ray diffraction analysis

For better understanding of self-healing process and to verify the formation of calcium carbonate in the samples, the healing compounds developed in cracks were subjected, to XRD analysis. In order to get the sample, the healing product formed inside the cracks was scratched with great care and was placed in the XRD apparatus. Copper (Cu) was selected as a X-ray target because it can be kept cool easily, due to its high thermal conductivity, and which produces strong Kα and Kβ lines. The readings were recorded at a wavelength of 1.54 Å and different representative peaks were obtained as shown in Fig. 4.8. It can be seen from the Fig. that no sharp needle like peaks were obtained during XRD. This is due to the reason that sample was scratched from the crack surface and contained a mixture of compounds from concrete surface as well.

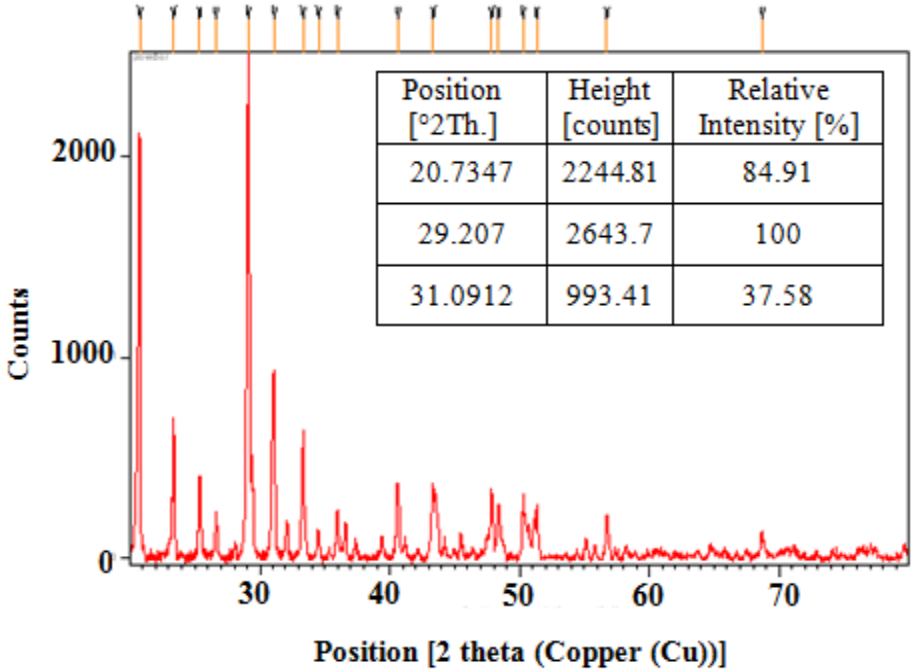


Fig. 4.8 XRD analysis of healing compound produced in the cracks.

Bacillus Subtilis is calcite forming bacteria (Rao et al., 2013), therefore, the results of XRD were compared with the reference cards of calcite. A highest peak was obtained at the 2theta (2θ) value of 29.2070^{o} which is quite close to 2θ of 29.455^{o} of pure calcite as observed by (Herrington, 1927). The slight difference in 2θ value can be due to the impurities in the powder resulted from the scratching off process. This shows that the material produced in the cracks is calcium carbonate in nature and is in harmony with the results obtained from previous studies.

1.4 Compressive strength analysis

Measured compressive strength of self-healing specimens is presented in Fig. 4.9. It can be seen that all bacterial incorporation techniques result in increased compressive strength of the mix.

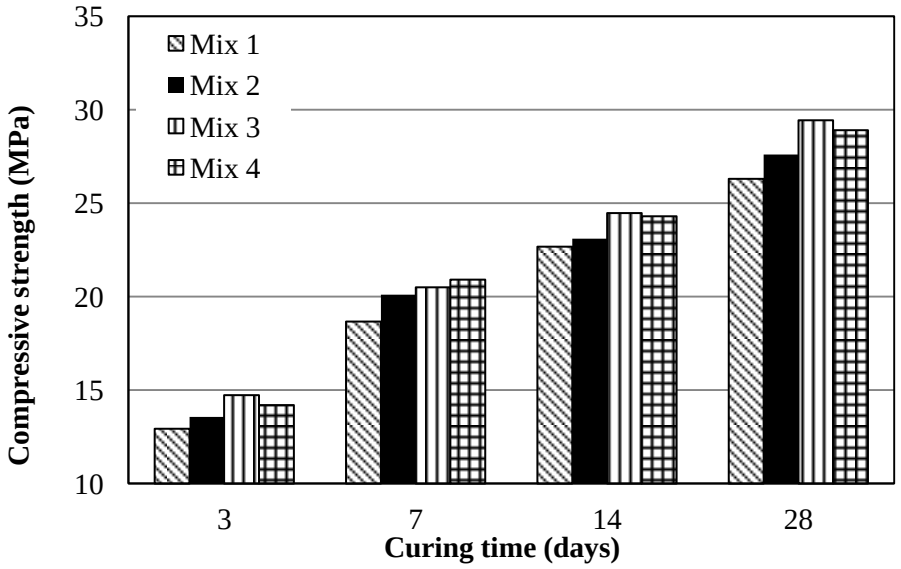


Fig. 4.9 Compressive strength development with different bacteria incorporation techniques

Samples having LWA as a carrier compound for bacteria incorporation showed maximum strength of 29.43 MPa and improvement of 12 % in compressive strength as compared to controlled concrete specimens. The increase in compressive strength are in accordance with the results as recognized in the study in self-healing carried out by Sierra-Beltran and Jonkers (2012) and confirms that self-healing is a cause of increase in compressive strength. This increase in compressive strength can be attributed to smaller size of LWA in comparison to regular sized coarse aggregates. This allowed better packing and compaction of concrete matrix around them which gave these specimens much higher strength than controlled specimens.

Specimens containing GNP showed an increase of 9.8% in compressive strength. This improvement in compressive strength can be attributed to the addition of GNP. GNP being a nanosized material acts like a filler material with even and uniform suspension in the mix. Small size of GNP also decreases the formation of weak interfacial transition zone (ITZ) in concrete by allowing filling of porous and crystalline microstructure within ITZ. Decrease in ITZ makes the mortar matrix denser and more compact resulting in higher compressive strength. GNP particles also act as crack arrestors and block and divert crack formation and propagation (Sixuan, 2012). GNP therefore, acts in many ways to enhance the compressive strength of concrete.

Direct incorporation of bacteria also showed an increase in compressive strength of concrete. This improvement is because of the presence of calcite producing bacteria in the mix. These

calcium carbonate continuously manufactured by the bacteria and calcium lactate provided as organic precursor makes the internal structure of concrete more compact, therefore, results in increase of compressive strength. This improvement seen by the direct introduction of bacteria is in consistence with the results achieved by Ghosh et al. (2005). However, after careful comparison of the results achieved by Ramachandaran (Ramachandran et al., 2001) by direct introduction of bacteria, it is evident that there is no difference in strength by introduction of Bacillus Pasteurii at a rate of 7.2×10^7 cell/cm^3. This shows that as far as compressive strength is related B.Subtilis is a better choice as compared to B.Pasteurii as its addition significantly improves the compressive strength of concrete.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

1. General

The aim of this study is to compare the efficiency of different self-healing techniques in terms of their crack healing capacity. Efficiency of these techniques was studied in terms of their effects on compressive strength, micro structure, production of calcium carbonate and healing of cracks. SEM analysis was carried out to observe changes in micro-structure because of various techniques. In addition, healing compound observed in specimens was subjected to XRD in order to determine its chemical composition. Results required from experimental work were used to develop the relationships of observed crack healing and compressive strength with bacteria incorporation techniques.

2. Conclusions

Based on the results achieved during this study following conclusions are drawn:

- Specimens incorporated with graphite nanoplatelets (GNP) as carrier compound displayed uniform distribution and protection of bacteria at samples pre-cracked at early age of 3 and 7 days, resulting in maximum crack healing efficiency. However, when pre-cracked at later days, such specimens presented a significant decrease in healing of cracks.
- Although specimens incorporated with lightweight aggregate (LWA) as carrier compound, were not as efficient as GNP at early age pre-cracked specimens, they showed consistency in their crack healing efficiency in specimens pre-cracked at later days.
- Specimens incorporated directly with bacteria did not show any effects in crack healing of concrete.
- Compressive strength trends of all mixes suggest that, addition of bacteria “Bacillus Subtilis” resulted in slight increase in compressive strength, irrespective of the incorporation technique, with significant improvement through LWA technique.

3. Recommendations

While this research has provided significant understanding about self-healing mechanism and the effect of newly introduced carrier compound on crack healing capacity, further research is

required to completely characterize the self-healing mechanism. Given below are some of the recommendations for further research in this area.

- There is still a need of further research in order to determine the optimum concentration of bacteria to maximize the production of biologically produced $CaCO_3$ and increase compressive strength.
- Further study is required for the detailed understanding of activation process of bacteria and to find the methods to control them.
- Experiments should be carried out to determine the durability of bacteria based self-healing concrete.

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