`

STUDY OF SELF-HEALING BEHAVIOR OF RIGID PAVEMENT(CONCRETE) USING NANO GRAPHITIC INSERTIONS

FINAL YEAR PROJECT UG 2015

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2019

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This is to certify that the

Final Year Project Titled

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has been accepted towards the requirements
for the undergraduate degree
in
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DEDICATION

This thesis is dedicated to our parents, teachers and friends who have been encouraging, motivating and mentoring us throughout our entire life.

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ACKNOWLEDEMENTS

In the name of Allah, the most merciful, the most beneficent, all praise to Him, the lord of the worlds and all prayers and Hazrat Muhammad (S.A.W), his servant and final messenger.

We are, first of all, grateful to Almighty Allah, without Whose willingness and blessings, we would have never achieved what we have ended up ultimately accomplishing. Praise be to Almighty ALLAH, Most Gracious for bestowing us with health and knowledge to complete our thesis.

We would like to thank our supervisor Dr.Arshad Hussain for providing us the oppourtunity wo work under his supervision and utilize the state-of-art testing facilities in National Institute of Transportation Laboratory in National University of Sciences and Technology. We would also like to thank our co-supervisor Dr.Rao Arsalan Khushnood, Head of Structure Department, for constantly mentoring us throughout our research journey and allowing us to utilize the modern equipment in Structure Laboratory in National Institute of Civil Engineering. It has been an honour for us to have such qualified, reputed and exalted people to supervise throughout our research phase and we are unable to express in words our gratitude towards our mentors for investing their precious time and knowledge. Our sincerest and heartfelt thanks to them for helping us in becoming hard working, knowledgeable and a better person.

We would like to express our gratitude to the lab staff of transportation lab and structure lab for helping us out in the lab testing throughout our research.

We are grateful to the UG student of ASAB Ms. Marjan Gul (Batch:2016) for carving out time from her own work to prepare bacterial culture and perform identification tests for several consecutive weeks and providing technical support throughout our project.

Special thanks to Mr.Ibrahim Sarwar (Batch:2015,NICE) and Mr.Hassan Ali Subhani (Batch:2015,NICE) for providing us their valuable support throughout the research journey.

At the very last we would like to thank the most important people in our lives, our parents, our sibling and our friends, for supporting and believing in us throughout the whole project.

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ASTM	American Society of Testing and Materials
SHM	Structural Health Monitoring
ECC	Engineered Cementitious Composites
MICP	Microbial Induced Calcite Precipitation
PSA	Particle Size Analysis
SEM	Scanning Electron Microscopy
EDS	Energy Dispersive Spectroscopy
OM	Optical Microscopy
UVS	Ultraviolet Spectroscopy
DDF	Ductility Deformation Factor
MoR	Modulus of Rupture
CWMA	Crack Width Measurement Analysis
SRI	Strength Recovery Index
UPV	Ultrasonic pulse velocity

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND:

Concrete is an important building material. Concrete is the most widely used construction material in the world due to its versatile characteristics like formlessness, plasticity, hydraulicity, strength and toughness and is relatively cheap. It is widely used in the infrastructure industry for construction of different facilities e.g. buildings, roads, bridges, tunnels etc. It is durable having higher upholding strength against weather conditions and is easy to maintain. It is a cost-effective material with a longer service life. A building made with concrete have life of about 50 years but due to cracks it service life gets decreased. Concrete is good in compressive strength but its strength gets decreased due to the appearance of cracks.Concrete is mostly reinforced in buildings ,while due to appearance of cracks water propagates and corrode the reinforcement in the conctrete material. Concrete is the main building material in the construction of rigid pavements. It acts more like a bridge in pavements i-e load exerts less pressure on material under rigid pavement as compared to asphalt pavement.Concrete is used in bridges where it needs to be highly compressive strength material.

However, since it is overwhelmingly brittle in nature and has a low tensile strength, it is vulnerable to movement and mixture in small scale cracks which renders low durability and strength (Khaliq et al., 2016). Movement and subsequent increment in width of cracks can make them harmful substances that can likewise be inconvenient to solid strength and concrete durability. It is assessed that harm because of corrosion of concrete in the US is $276 billion (Koch et al., 2002) with yearly expense in fixes to be $18 – 21 billion every year (Emmons and Sordyl, 2006). And prior to the repair applied, structures also require periodic maintenance i.e. Structural Health Monitoring (SHM), which also carries a significant cost. There is a compelling economic incentive to develop a multipurpose Engineered Cementitious Composite (ECC) that can repair the damage all by itself and subsequently increase its longevity.

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1.2 SELF-HEALING:

Self–healing is an autonomous repair of cracks produced in structures due to deterioration produced over the course of their service life while self-sensing involves microstructural modification of cementitious matrix by addition of conductive filler whose conductive properties can be utilized for monitoring of such deteriorations produced at nano and micro levels, also termed as Structural Health Monitoring (SHM). Structural Health Monitoring in such type of smart modified cementitious matrix with self-sensing properties can be applied to check the depth of cracks produced inside the structure by keeping the record of its resistivity with respect to current (Lu, Xie, Feng, & Zhong, 2015).

Cementitious materials that exhibit autonomous biological self-healing and smart intrinsic self-sensing are inspired by natural phenomenon of proliferation and somatosensation of the human skin respectively. Different techniques have been used to autonomously heal microstructural deficiencies produced in concrete. Bio influenced self-healing has lately been the most suited methodology since it is a natural process and environmental friendly. In brief, it was hypothesized that once moisture enters through freshly formed cracks, dormant but viable bacterial spores immobilized in the cementitious matrix become metabolically active. Then, these cracks will be healed through microbial calcite precipitation, hindering further ingression of water and other deleterious materials (Bhaskar, 2016).

Carbonaceous media has been the most widely used nano-composite for introducing self-sensing characteristics in the cementitious matrix (Kashif et al., 2017; Yoo et al., 2017). Graphene Nano Platelets (GNP), Short-cut carbon fibers (CFs), carbon black, steel fibers and steel slag have been added into cement-based materials to fabricate multifunctional smart engineered cementitious composites (ECC). These not only have improved the flexural strength, fracture toughness, and anti-shrinkage ability but also serve as stress and temperature sensitive sensors within the matrix (Zuo, Yao, Liu, & Qin, 2013).

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1.3 PROBLEM STATEMENT:

- Autonomous self-healing using different carrier compounds has been studied in the past to increase survival potential of microbes. However, varying concentration of single carrier type has not been studied. Immobilizers act as direct encapsulators of the bacterial spores, therefore, there can be variation in healing results obtained due to varying carrier concentration. Moreover, past studies have confined self-healing to densification of cementitious matrix by filling of micro and Nano cracks, mostly below 1 mm width (W. Khaliq et al., 2016). Effectiveness of bio-based self-healing in macro cracks (>0.7 mm) (Wang et al., 2014) has yet to be studied in detail.

- Similarly, nano composites have been used in the past to develop Engineered Cementitious Composites (ECC), developing improved mechanical properties and also forming an effective conductive network within the cementitious matrix for selfsensing purposes by translating the induced strains in the form of electrical resistivity. However, most of these nano composites, like CNTs are expensive, therefore, limit their broad spectrum use in industry which demands economically viable products. So a material similar in characteristics to past used nano-composites that is cheap as well needs to be introduced and studied to see whether it is equally effective in enhancing the mechanical properties and developing an effective sensing network within the cementitious matrix.

- Furthermore, there have been no studies where a correlation has been established between the immobilizer used for microbial preservation in self-healing, and nanocomposite material used for self-sensing purposes.

- In building construction, cracks develop in concrete which results in propagation of water through them. Due to this reinforcement in concrete gets corroded and tensile strength also decreases with the decrease of compressive strength due to concrete cracks.

- Pakistan is moving towards becoming a developing country. One of the major reasons is the introduction of CPEC in our economy. CPEC involves movement of heavy weight vehicles which requires the construction of rigid pavements. These vehicles tend to damage the pavements. Hence introduction of self-healing pavements would help in reducing the maintenance cost as well as increasing the service life of these pavements.

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1.4 OBJECTIVES:

In light of components detailed out in the problem statement, the objectives of our thesis are enlisted as follows:

- To analyze the bio-influenced self-healing behavior for the repair of nano/micro-cracks.
- To study the impact of nano graphitic insertions on compressive strength of rigid pavement.
- To study the impact of nano graphitic insertions on flexure strength of rigid pavement.
- To study correlation and effectiveness of utilizing different concentrations of cheap raw graphite as; immobilizer for bacterial spores and overall enhancement of mechanical properties of engineered cementitious multipurpose grout and studying the feasibility of graphite as an immobilizer.

1.5 Thesis Structure

Following the introductory **Chapter 1**, a literature review on the self-healing materials is presented in **Chapter 2**.

Chapter 3 describes the experimental research methodology, testing procedures and materials used in casting. Mix design, casting regime and curing has also been discussed in this chapter.

Chapter 4 includes tests conducted in different samples casted of our multipurpose grout and their results. It outlines a detailed analysis of fracture mechanics, self-healing of different samples. Micro-structural and micro-analytical investigation and its results are also discussed in this chapter.

Conclusions based on findings of this research and future recommendations are presented in **Chapter 5**.

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CHAPTER 2

LITERATURE REVIEW

2.1 Self-Healing

2.1.1 Characteristics of self-healing behavior:

Self-healing materials are such types of materials that are used to aid damaged materials in regaining their lost strength. The extent of damage over the course of a structure's service life greatly governs the potential of strength recovered. Van der Zwaag (2010) suggests that the phenomenon of self-healing is specifically useful in composite materials, since their potential of damage detectability is low and is susceptible to sudden failure. With increasing time, materials with better mechanical properties are being manufactured. However, materials are lacking self-healing characteristics. So, any minor damage induced can aggravate strength loss unless appropriate human intervention has taken place (Ghosh, 2005). All the materials require self-repair for long time service.

2.1.2 Classification based on self-healing behavior:

Chemical process undertaken by the self-healing material can be categorized into two separate sorts; non-autonomic and autonomic (Hager et al., 2010). Process which involves presence of an external stimulus, e.g. heat or light for healing process to take place is known as Non-autonomic self-healing. In autonomic self-healing, the damage induced is itself the triggering component for repair to be undertaken (Hager et al., 2010). Another type is the intrinsic and extrinsic self-healing. As the name suggests, healing process in extrinsic self-healing is based on external healing components e.g. capsules, that are deliberately embedded inside the cementitious matrix and material present inside these capsules performs healing action (Van der Zwaag, 2010). In intrinsic self-healing, no separate material is required to be added.

2.1.3 Biological Healing

Bio-influenced self-healing includes using the precipitation capability of specific kinds of microorganisms to fill in the microstructural lacks created through the span of service life of a structure. Scientists have directed investigations to monitor the utilization of bacterial calcite in improvement of cementitious matrix (Ramachandran et al., 2001; Jonkers et al., 2007). This technology conveys a specific microbial plugging process, in which microbial metabolic activities advance precipitation of calcium carbonate as calcite. Numerous investigations have been accounted for to evaluate the use of microorganisms such as bacteria in concrete for the remediation of splits (Jonkers, 2007; Khaliq et al., 2016). On account of high alkalinity inside the cementitious framework, bacterial spores successfully precipitate sealant for example calcite since they can get by in this high alkaline condition. Cooperation of bacterial spores immobilized in concrete with outer moisture going through the crack line and with the vital supplements either included concrete amid throwing or conveyed in by the water and the encompassing pH being brought down to around 10 to 11.5 outcomes in the activation of bacterial spores.

2.1.4 Non-Biological Techniques of Self-Healing

Many researchers have conducted detailed studies to develop self-healing concrete in the past (Tittelboom et al., 2011; Yang et al, 2007). Most of these studies involved extrinsic self-healing, whereby the healing agent was encapsulated inside a microcapsule. This external layer of microcapsule is sensitive to some triggering process which can either be change in pH or moisture. The ruptured capsule oozes out the healing agent inside and ultimately healing the crack. Healing agents like epoxy resins (Nishiwaki et al., 2006), cyanacrylates (Li et al., 2009; Dry et al., 2001; Joseph et al., 2007), and alkali-silica solutions (Mihashi et al., 2000) were studied and their respective results showed that the strength regained post healing action is nearly same to that usually recovered by manual healing mechanisms. The biggest disadvantage of microencapsulation was the permanent cavity or void left at capsule site when it was consumed, thereby reducing the mechanical properties of concrete (Tittelboom et al., 2011). Capillary tubes used in blood testing can also be used as encapsulating tubes in concrete (Joseph et al., 2007)

2.1.4.1 Biological processes of Microbial Induced Calcite Precipitation (MICP)

Type of bacteria specie as well as other factors like salinity, surrounding pH, temperature and available nutrients govern the precipitation potential of bacterial spores in precipitating calcium carbonate (calcite). Four key factors which govern the MICP are: (i) the calcium concentration, (ii) the concentration of dissolved inorganic carbon, (iii) the pH (iv) the availability of nucleation sites (Hammes and Verstraete, 2002). The urease enzymes produced by the bacteria decompose urea into ammonium and carbonate ions. The chemical reaction is given below (Ng et al., 2012):

$(NH2)\ +2 + 2H2O \rightarrow 2NH+4 + CO3\text{-}2$

$Ca+2 + CO3\ \text{-}2 \rightarrow CaCO3$

The ammonium ions (NH4+) yielded as a result of hydrolysis of urea makes the surrounding environment more alkaline i.e. it increases local pH and since bacterial spores are alkalinity sensitive, they instigate the precipitation of calcium carbonate. The high localized pH helps spores in serving as nucleation site for calcite crystal.

2.1.4.2 Process

Figure 1 below shows the basic principle of self-healing of different materials. The step by step procedure is as follows (Hager et al., 2010):

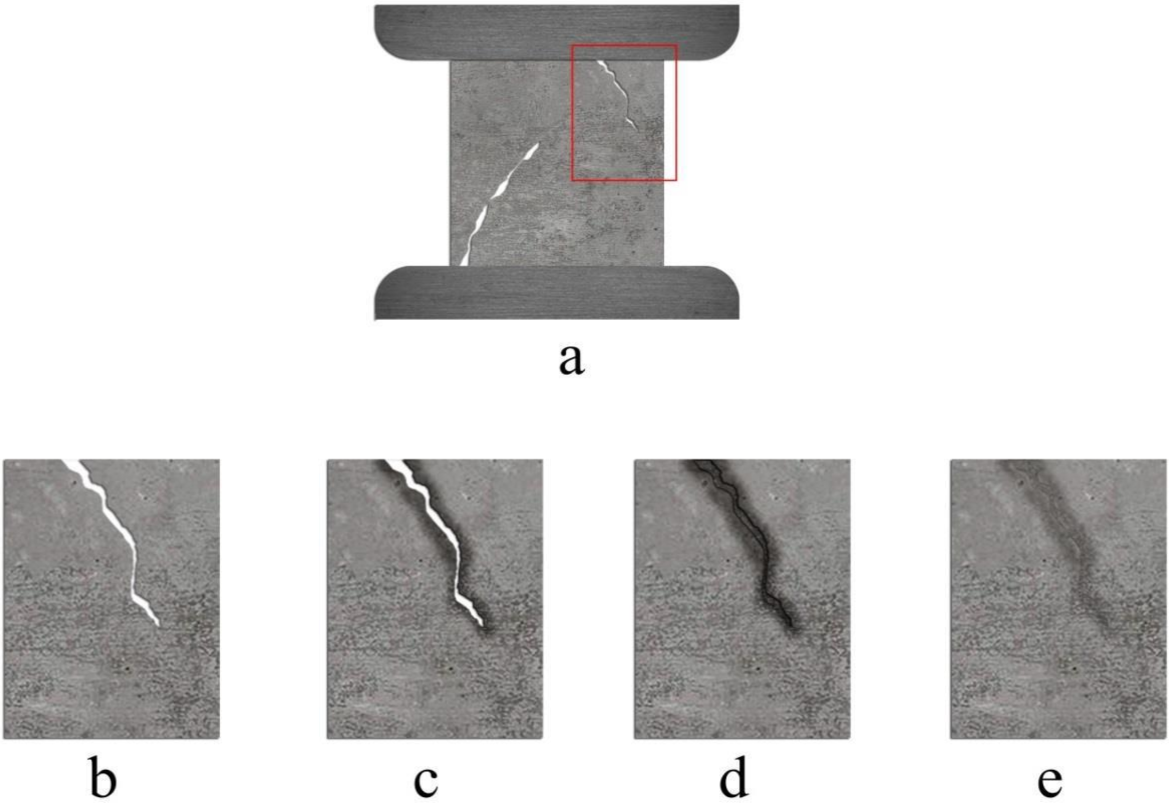

Figure 2.1 : Stages of healing of cracked specimen

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a. At the outset, as shown in Figure 1.1a, crack is formed under mechanical load.

b. As per the general principle, a "mobile phase" is generated as shown in Figure 1.1c which is triggered either by the occurrence of damage or by external stimuli.

c. Consequently, mass transport takes place towards the damaged site and due to the following local mending reaction, the damage can be removed as shown in Figure 1.1d. This is achieved by adhering of the crack planes by physical interactions or chemical bonds.

d. Once the healing is attained with fully restored mechanical properties, the previously mobile material is again immobilized which is shown in Figure 1.1e.

2.1.4.3 Influence of the type of microorganisms

The hydrolysis process of urea is the most suitable pathway for the production of carbonate ions since it has the ability to alkalinize the surrounding environment. Therefore, the bacteria should be able to act as a catalyst in the urea hydrolysis and they are usually urease positive bacteria. (Bhaskar, 2016). Most urease positive bacteria belong to genera *Bacillus, Sporosarcina, Clostridium* and *Desulfotomaculum* (Kucharski et al, 2008). Past researchers have used urea-utilizing bacteria like *Sporosarcina pasteurii* (Bang et al., 2001 and Ramakrishnan et al, 2005), *Bacillus sphaericus* (Ramachandran et al., De Muynck et al., 2008; Wang et al., 2011) and *Bacillus Subtilis* (Khaliq et al., 2016) as the most commonly used bacteria in cementitious materials and concrete while *Bacillus psuedofirmus*, *Bacillus cohnii* (Jonkers and Erik Schlangen, 2014; Jonkers et al., 2010) and *Bacillus alkalinitrilicus* (Wiktor and Jonkers et al., 2014) have also been tested to a certain extent. This presents a wide range of microorganisms that have been used in the past for self-healing. The prime governing factor in effective calcite precipitation is the alkalinity. Fresh concrete has high alkalinity with pH values between 11 and 13. It was found that strains of the bacteria genus *Bacillus* were able to survive most effectively in the high-alkaline environment. These bacteria typically form spores, which are specialized cells able to resist high mechanically and chemically induced stresses (Sagripanti and Bonifacino 1996). Also, they have rates of metabolic activity and extremely long dormancy duration and lifetimes where some species are known to produce spores which are viable for up to 200 years (Schlegel 1993, Jonkers 2007).

Therefore, most promising bacterial spores appear to be alkaliphilic spore-forming bacteria of genus *Bacillus* (Jonkers et al, 2010).

However, it has been reported that the bacteria *Escherichia coli* imparts no improvement in healing property. From this, it can be inferred that the selection of appropriate micro-organism plays a fundamental role in the improvement of compressive strength (Jonkers, 2007).

2.1.4.4 Bacterial Concentration

Apart from the bacteria type, its concentration in cells/ml also plays a vital role in performing healing action. Ghosh (2007) has suggested *E. coli* with 105 cells/ml as ideal to enhance mechanical properties. Ramachandran (2005) has suggested 7.6x103 cells/ml for *Bacillus Pasteurii* and 6x108 cells/ml for *Bacillus Psuedofirmus*. Nugroho (2015) has suggested 105 and 106 cells/ml as ideal bacterial concentrations for *Bacillus Subtilis* for both effective self-healing and enhancement of mechanical properties. Similarly, De Belie, Jonkers and Beltran have also given different ranges of optimum bacterial concentration for different bacteria types.

2.1.4.5 Carrier materials

Mere introduction of bacterial spores without appropriate protection can greatly retard the healing action and subsequent survival duration of the bacterial spores. This can be addressed by introducing carrier particles for bacterial spores which will keep them protected in concrete environment by serving as effective immobilizers. Polyurethanes (PU) and silica gel have been widely used as a vehicle for immobilization of enzymes (Wang et al., 2011). Jonkers et al. (2010) used clay pellets as carrier material for the bacteria. Diatomaceous earth (DE) consists of diatom skeletons which are highly porous, light in weight and chemically stable and inert, was used as a suitable carrier material for aerobic and anaerobic bacteria in a high-pH concrete environment (Wang et al., 2012). Graphene Nano Platelets (GNP) are known to serve as effective carriers for bacteria genus *Bacillus Subtilis* with substantial healing action post 7-day curing while Light Weight Aggregate (LWA) has yielded better healing action under prolonged durations of curing (Khaliq et al., 2016).

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2.1.4.6 Influence of Nutrients

Bacterial spores need an organic precursor to precipitate calcite (Khaliq et al, 2016). Generally, calcium lactate has been reported to be a good choice because it starts to dissolve during the mixing process and does not interfere with the setting time of concrete (Jonkers et al, 2010). However, it can impart degrading effect on concrete due to presence of calcium chloride since it releases chloride ions that maybe harmful for the concrete reinforcement (Jonkers and Schlangen, 2009).

2.1.4.7 Improvement in Properties

It has been reported that bacteria genus *Bacillus Subtilis* resulted in slight increase in compressive strength when incorporated with LWA and GNP (Khaliq et al., 2016). Jonkers (2007) selected *Bacillus psuedofirmus* and *Bacillus cohnii* to be added in concrete specimens and found a 10% increase in the compressive strength. Achal et al. (2009) selected the bacterial species *Sporosarcina pasteurii* with mortar cubes and noticed a 17% increase in compressive strength.

Flexural strength of bio-based the ECC materials were evaluated by Sierra-Beltran et al. (2014). Samples with healing agent post cracking and subsequent healing action showed a slightly better recovery of both flexural strength and deflection capacity from control mixtures without bio-based healing agent.

Achal et al. (2011) observed a considerable reduction in water permeability in cement mortar cubes by using Sporosarcina pasteurii. It is believed that this lower permeability of bacteria incorporated cubes may be due to the presence of a denser interfacial zone formed between the aggregate and the concrete matrix by calcite precipitation (Bhaskar, 2016). Also, nearly six times reduction in water absorption in mortar cubes was observed in comparison to untreated specimen up on incorporation with *Bacillus sp. CT-5* (Achal et al., 2011). Also, a 68 % reduction in water permeability was reported by Wang et al (2014) by using hydrogel encapsulated *Bacillus sphaericus* spores in mortar cubes.

Dispersion is the process of breaking down clusters formed due to the development of electrostatic attractive forces between individual particles. Proper dispersion of carbon Nano-materials in cementitious matrices is a major concern which directly affects the properties of cement-based Nano-composites. It is not feasible to directly disperse Nano/micro composites within cement paste. Since the physical dispersion technique i.e. water bath sonication is not sufficient alone for dispersion, we had to use a chemical dispersant as well. Out of the options available to us as shown in table below we selected GA (Gum Arabic), as it is readily available and is known to be used for dispersing Nano-graphite.

2.1.5 Dispersion of carbon Nano-composites in cementitious matrices

Dispersion is the process of breaking down clusters formed due to the development of electrostatic attractive forces between individual particles. Proper dispersion of carbon Nano-materials in cementitious matrices is a major concern which directly affects the properties of cement-based Nano-composites. It is not feasible to directly disperse Nano/micro composites within cement paste. Since the physical dispersion technique i.e. water bath sonication is not sufficient alone for dispersion, we had to use a chemical dispersant as well. Out of the options available to us as shown in table below we selected GA (Gum Arabic), as it is readily available and is known to be used for dispersing Nano-graphite.

Type of dispersants	Acetone
	Ethanol,
	Nitrite acid,
	Sulphate acid
	Gum Arabic

Table 1: Different types of surfactants

Table 2.1 Different types of surfactants

2.1.6 Percolation Threshold Theories

Percolation threshold is a mathematical term related to percolation theory, which identifies the minimum content of filler (GNMPs) required to be added to form a desired conductive network inside the cementitious matrix. Theoretical evaluation of the percolation threshold range will be evaluated using different mathematical models. However, to verify these theoretical percolation threshold ranges and establishing an experimental percolation threshold range, samples have to be casted and their electrical conductivity has to be tested via 4-probe test. In literature, there are some relations derived on the basis of empirical methods and numerical simulations which have been used to estimate the percolation threshold of GNMPs in cement composites.

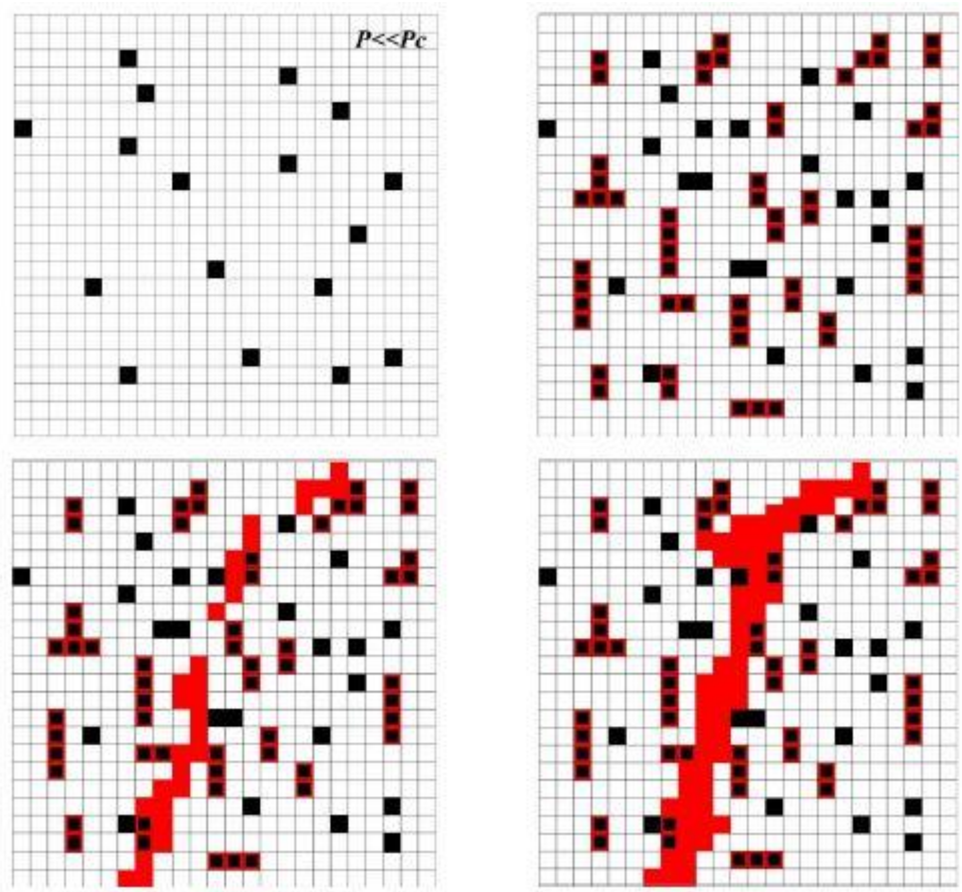

Figure 2.2: Formation of effective conductive network with increasing filler concentration

2.1.6.1 IPD (Inter particle distance) approach

It is a mathematical model that utilizes IPD and aspect ratio of individual particles of disc shaped nano sized platelets in polymer composites proposed by Jing li et. al.. The equation for the model is: $Pc=27\pi D2t4(D+IPD)$

Mechanism of electron hopping is initiated if IPD is less than or equal to 10nm. Since IPD is very small in comparison with the average effective diameter D of Nano platelets, it may be ignored and finally equation reduces to: $Pc=27\pi t4D=21.195\alpha$ $\alpha=Dt$

However, this model tends to give exaggerated values based on observation of related studies leading to uneconomical results.

2.1.6.2 Celzard approach

Celzard.et al. proposed a relation for percolation threshold related to aspect ratio of conductive fillers which is based on excluded volume approach and is as follows; $Pc=1-\exp(-3.6t\pi D)\leq\emptyset c\leq1-exp(-5.6t\pi D)$

Where D denotes the diameter of individual GNMP and t is its thickness (lesser dimension)

2.1.6.3 Ratio approach for percolation threshold

Celzard.et al. also proposed a relation for percolation threshold based on C:S(cement–sand ratio) and water-cement ratio. The resultant mathematical equation for the model is as follows: $Pc=0.36*(c+s+wc+s)$

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CHAPTER 03

RESEARCH METHODOLOGY

3.1 Materials

For the development of bio-healable concrete, following materials were used. The characterization tests of these materials are also shown here. The overall experimental program is also discussed later in the chapter.

3.1.1 Raw Graphite Nano/Micro Platelets (GNMPs):

Graphite was procured from Habib Rafique Pvt .Source of graphite was Swat and was in raw powdered form. Since graphite is being added in addition to cement, they can effectively serve the purpose of enhancement of mechanical properties and also forming an effective conductive network within cementitious matrix for self-sensing purposes, by having as fine the particle size as possible. Graphite is relatively inexpensive as compared to other carbon alternatives but is similar in characteristics in comparison with other carbon alternatives. It also serves as an effective carrier for microorganisms and acts as an effective barrier for bacteria, keeping the bacteria preserved for post crack healing.Also it is non-corrosive in nature.Another important property of graphite is its ability to resist crack propagation inside the concrete matrix.Thereby increasing the durability of concrete.

Carbon Nanotubes (CNTs) have known to be effective in enhancing mechanical properties. Li et al. found that addition of 0.5% multiwalled CNTs increased both compressive and flexural strength of Portland cement composite. Since they are nano particles, such a nanoreinforcement would delay the nucleation and growth of cracks on the nanoscale. If cracks are controlled at nano-scale level their propagation can be prevented to the micro level (Babak et al., 2014). So our initial requirement was nano-sized graphite.

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Figure 3.1 Graphite

3.1.1.1 Scanning Electron Microscopy (SEM):

Carbon content in graphite greatly affects the overall performance of graphite.Thus in order to study the morphology of graphite, scanning electron microscopy was performed in CASEN-NUST.Results are shown in Table 3.1 :

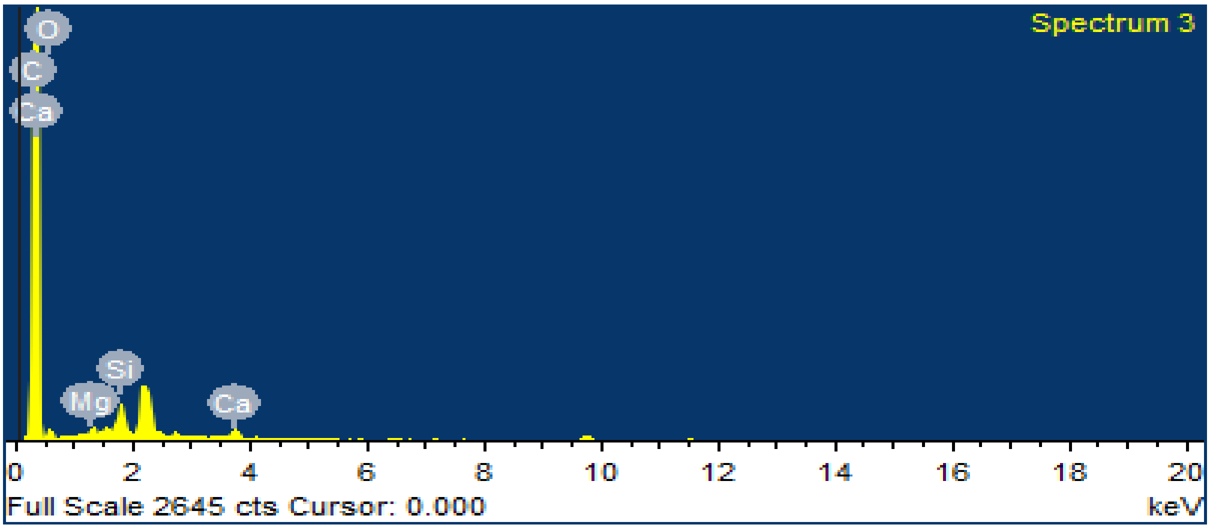

Figure 3.2: EDS Spectrum of raw graphite

ELEMENT	WEIGHT%	ATOMIC%
C	91.09	93.79
O	7.04	5.44
Mg	0.29	0.15
Si	0.96	0.42
Ca	0.63	0.19

Table 3.1 Elemental Composition of Graphite

From table 3.1, we see that graphite contains about 94% of carbon which is a good percentage.Thus the graphite we procured was suitable to be used.

SEM images of graphite were used to find the size of the particles.This data was used in Perculation Threshold Analysis (PTA) to find the optimum percentage of graphite that should be used for preparing samples.SEM images are shown below:

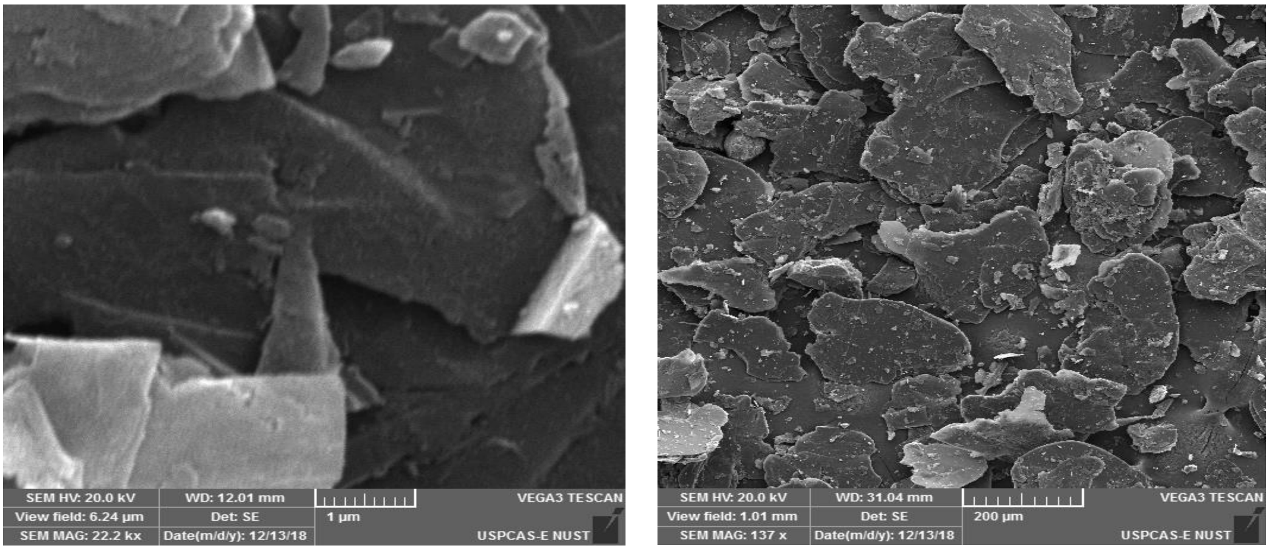

Figure 3.3: SEM images of raw graphite at a) 1 micron and b) 200 microns resolution

3.1.2 Bacteria (Bacillus Subtilis):

Bacillus Subtilis is a gram positive bacteria, having peptidoglycan layer and is commonly found in soil.It is non-pathogenic and is vastly used for research purposes. It has rod-shaped structure, can form protective endospores in the alkaline environment and endure extreme pH, temperature and high salinity. It is known to enhance structural properties of concrete. The increase in strength is directly proportional to cell concentration.

Bacillus Subtilis used has American type culture collection (ATCC) number 11774. ATCC genuine culture,is manufactured under ISO 9001:2008 certification.

3.1.2.1 Preparation of Bacteria(Bacillus Subtilis):

Bacteria was prepared in microbiology lab ASAB, NUST. Following steps were adopted for preparation of bacterial solution:

LB media preparation (Autoclave):

- Lysogeny broth (LB), a nutritionally rich medium, is primarily used for the growth of bacteria.
- 500ml LB broth contains, 5g tryptone (a mixture of peptides formed by the digestion of casein with the pancreatic enzyme, trypsin), 2.5 g yeast extract (an autolysate of yeast cells), and 5g NaCl and 500ml distilled water
- Bacillus subtilis grows in the mesophilic temperature range. The optimal temperature is 25-35 °C.

Purpose of autoclave:

- Its function is similar to sterilization. It is a machine that uses pressure and steam to reach and maintain a temperature that is too high for any microorganisms or their spores to live.
- To be effective, the autoclave must reach and maintain a temperature of 121 °C for at least 30 minutes by using saturated steam under at least 15 psi of pressure.

Figure 3.4: Autoclaved bacterial spores

Figure 3.5: Autoclave

Inoculation:

- The act of introducing microorganism or suspension of microorganisms (e.g. bacteria) into a culture medium.
- Inoculation of culture media is used to obtain reproducibility of quantitation results.

Figure 3.6: Inoculated bacterial spores

Incubation/Growth of Bacillus Subtilus:

- Bacillus subtilis grow in the mesophilic temperature range. The optimal temperature is 25-37 °C.
- Principal component that is necessary to be seen is the concentration in cell/ml of the bacterial spores that are required to be added in our samples. As mentioned in literature review, we have fixed our required bacterial concentration to about $5x10^6$ cell/ml.

Figure 3.7: Incubator

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Measurement of optical density:

- Optical density, measured in a spectrophotometer, can be used as a measure of the concentration of bacteria in a suspension. As visible light passes through a cell suspension the light is scattered. Greater scatter indicates that more bacteria or other material is present.
- OD for Bacillus Subtilus is measured at 600 nm wavelength.
- In order to ensure the concentration of bacterial solution, medium in which bacteria grow i.e. nutrient broth is selected as blank. This blank solution was used as a reference, based on which absorbance of bacterial solution was measured in the spectrophotometer. A quantity of 0.5 ml of blank solution was placed in spectrophotometer with a selected wavelength of 600 nm. After the machine had read the blank solution, it was replaced by 0.5 ml of bacterial solution and again the same wavelength of 600 nm was used. The obtained absorbance (Optical Density OD) was put in Ramachandran equation i.e. $Y = 8.59 \times 10^7 \times X$ 1.3627 in place of "X" to determine cells/ml concentration "Y". Whenever this concentration was found to be 5×10^6 cells/ ml, solution was used for casting.

Figure 3.8: Spectrophotometer

Figure 3.9: Prepared Bacterial Solution

3.1.3 Surfactant Gum Arabic:

The main problem with graphite being used as immobilizer for bacteria is its non-dispersive nature. The non-dispersive nature of graphite is due to lack of oxygen in graphite in purest form. Oxygen facilitates the dispersion like in Graphene Oxide, which has oxygen functional groups that penetrate into the graphite interlayers that lead to weakening the interaction between the layers. This helps in easy dispersion of GO into aqueous solutions and form a stable suspension. (Cai et al., 2012).

In order to ensure proper dispersion of graphite in microbial solution,a surfactant known as Gum Arabic is added. It was commercially purchased and is of DAEJUNG (CAS # 900-01-5) company (SG=1.35 and D50 = 98 um). Its basic function is to lower down the surface tension and allowing easier dispersion of carbon composites.

Ratio of GNMPs to that of Gum Arabic was kept as 1:0.6.

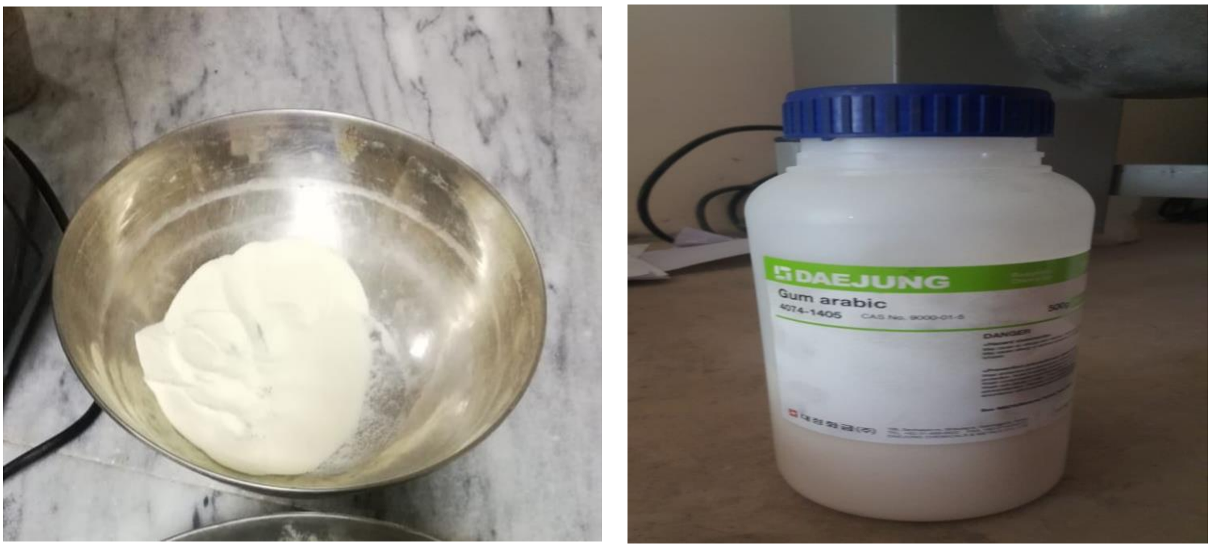

Figure 3.10: Gum Arabic

3.1.4 Calcium Lactate:

The microbes incorporated require mineral substrate to precipitate limestone. It serves as the food for the bacterial microbes. So, a suitable mineral substrate is to be provided along with bacteria during casting. Calcium Lactate is selected as the mineral substrate due to its successful use in past in developing self-healing concrete. Calcium Lactate was commercially purchased and is of DAEJUNG (CAS # 5743-47-5, CAT # 2513-4405) company.Following are some of the properties of calcium lactate as provided by the manufacturer :

- **Linear Formula** C6H10CaO6 · 5H2O; Ca(CH3CHOHCOO)2 · 5H2
- **Melting Point** 240 °C
- **Assay** 98%
- **Molecular Weight** 308.3

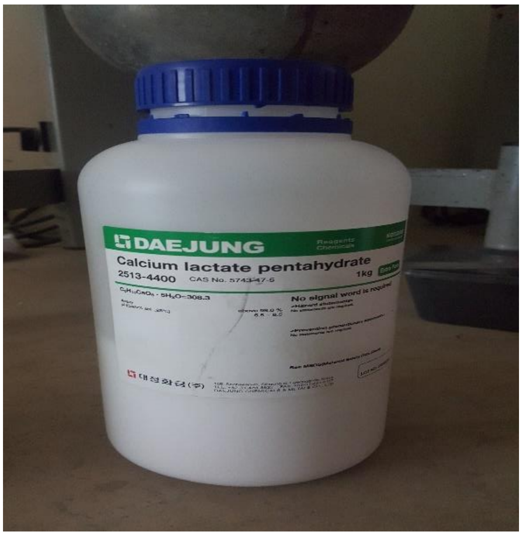

Figure 3.11 Calcium Lactate

3.1.5 Cement:

Cement used in casting was of Best Way Grade 53.Different tests were performed to determine the physical properties of cement.Results are shown in table 3.2 below :

Initial Setting Time	35 mins
Final Setting Time	165mins
Specific Gravity	3.14
Consistency	7mm penetration (5-7mm permissible)
Soundness	4mm (10 mm permissible)
Median Size (D_{50})	5.5 Microns

Table 3.2 Properties of cement

3.1.6 Sand:

Sand was procured from Lawrencepur. It is slightly fine to medium sized sand. Sieve analysis of the sand in accordance to ASTM C33 was done and the gradation curve was plotted as shown in figure 3.6. Gradation curve came out to be with the envelope specified by ASTM C33.

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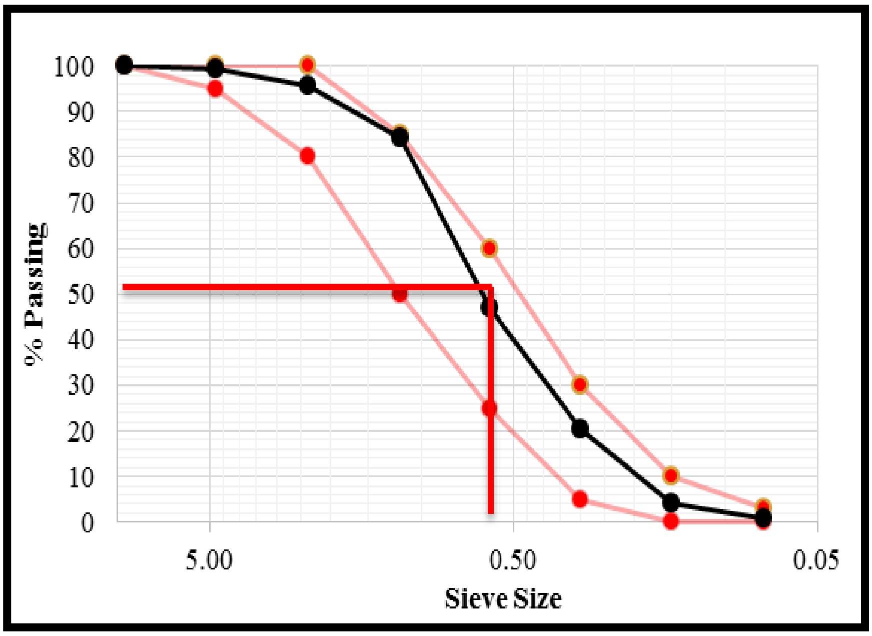

Figure 3.12 Particle size distribution of sand

Different tests were performed to determine the physical properties of sand.Results are shown in table 3.3 below :

Median Size (D_{50})	0.47 mm
Fineness Modulus	2.48
Specific Gravity (kg/m^3)	2.66
Absorption	1.6%

Table 3.3: Properties of sand

3.1.7 Water:

Water available in Structural lab was used in casting of the samples.

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3.1.8 Aggregate:

Aggregate is obtained from Margalla Quarry Site. Aggregate resists deformation in pavements so it should have sufficient strength, texture so that it can withstand its purpose in pavement.Aggregates plays a vital role in defining the strength,durability of pavements.It takes maximum load of pavements.These strength related properties of aggregate are greatly influenced by the texture and shape.Generally,more angular and rough textured the aggregate is,more it is capable to resist stresses in pavement due to application of repeated loads.Seaveral tests are performed per ASTM and BS to check properties of aggregate affecting pavement.

3.1.8.1 Shape Test of Aggregate:

This test determines the percentage of flaky and elongated particles in aggregate. Flaky particles are defined to be those having their least dimensions lesser than 0.6 times their mean dimension. While elongated particles are defined to be those having their greatest dimension greater than 1.8 times their mean dimension. For better interlocking of aggregate particles angular shape is preferred. The flakiness index should be less than 15% while elongation index should be less than 15%.

3.1.8.2 Water Absorption and Specific Gravity Test:

Specific gravity is defined as the ratio of given volume of aggregate to weight of equal volume of water at 23 °C. This test was performed only on coarse aggregate per ASTM C 127-88. Three weights were determined for calculating specific gravity i.e weight of oven dried aggregate, weight of aggregate completely submerged in water, and saturated surface dry weight of aggregate. Specific Gravity of Fine aggregate and water absorption was determined using ASTM C 128.

Figure 3.13 Performing Specific Gravity test

3.1.8.3 Los Angeles Abrasion Test:

This test is used to check resistance of aggregate to wear and tear due to heavy traffic load. More the abrasion value of aggregate more the performance of pavement is adversely affected. The equipment used was LOS angles machines, sieve set, balance steel balls. NHA Gradation B was selected for this test. About 5000gm of sample, containing 2500gm retained on sieve ½” and 2500gm retained on 3/8”, was placed in Los Angles Machine along with 11 steel balls and drum was rotated at speed of 30-33 rpm for 500 revolutions. After that material from machine was passed through 1.7mm sieve weight (W2) of sample passing it was noted. The abrasion value is defined to be = W2/W1 * 100

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Figure 3.14 Performing Los Angeles Abrasion test

3.1.8.4. Impact Value of Aggregate:

The impact value of aggregate gives its relative strength against impact loading. The equipment required was impact testing machine, temping rod, sieves of sizes 1/2” 3/8” and No 8. About 350 grams of sample passing sieve ½” and retained on 3/8” was transferred impact testing machine cup in three layers while each layer was temped with the help of temping rod. After that it was subjected to 25 blows from rammer of impact machine weighing 14 kg and free fall of 38cm. After that material was taken out from cup and passed through sieve No 8. The percent passing through sieve No 8 gives impact value of aggregate.

Figure 3.15 Performing Impact Value test

Results of all the tests performed on aggregate are shown in table 3.4 :

Type of Test		Results %	Specification	Standards
Los Angeles Abrasion		23	40% (Max)	ASTM C 131
Flakiness Index		13	15% (Max)	ASTM D 4791
Elongation Index		3.76	15% (Max.)	ASTM D 4791
Aggregate Impact Value		15	30% (Max.)	ASTM D5874
Water Absorption	Coarse Aggregate	0.73	3% (Max.)	ASTM C 127
Specific Gravity	Coarse Aggregate	2.63	-	ASTM C 127

Table 3.4: Tests results of Aggregate

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3.2 Experimental Methodology

To perform distinctive sorts of tests on bio healable concrete, diverse kind of molds were casted. These included 100x100x400 mm short beams which were 12 in number and cylinder 100 x200 mm too which were 36 in number. For the compelling dispersion of GNMP'S, Gum Arabic was included and their expansion just in microbial arrangement does not ensure successful dispersion. So extraordinary dispersion methods can be conveyed to scatter GNMPs viably. We have utilized sonication procedure for scattering. Be that as it may, sonication includes expanded temperature which can change properties of our microbial arrangement. Organisms are brooded at around 37 °C. So expanded sonication temperature can change cell/ml convergence of microorganisms. Too high temperature presentation to unprotected microorganisms can yield dead spores too.

Moreover, Gum Arabic can possibly fill in as a natural forerunner to microorganisms also. Both these elements can modify our planned microbial concentration of $5x10^6$ cells/ml even before they are included the blend. So, to avoid this both GNMPs and Gum Arabic are first sonicated in water and after that the sonicated arrangement is added to microbial arrangement, after which hand blending is completed, ensuring that microorganisms get adequately typified by the successfully scattered GNMPs and afterward are added to the blend.

Figure 3.16 Dry mixing

Figure 3.17 Sonication

3.3 Mix Design

Since there was no reference of mix design developed to study self-healing of concrete, therefore we were using, trial casting and based on the results obtained, mix proportions shown in table 8 were finalized for all mold types. GNMP concentrations were selected based on Percolation Threshold Analysis. Plain Control Sample (PCS) and Bacterial Control Sample (BCS) are two control samples. Plain Control Sample (PCS) has neither bacteria nor graphite while Bacterial Control Sample (BCS) has microbes poured in it without any immobilizer/carrier particle. Super plasticizer was used in casting. Mix design of 1:2:2.2 was finalized.Table 3.5 shows the casting regime followed during preparation of samples.

Sample Type	Mode of control	Mix Design	W/C Ratio	Graphite Concentration	Bacterial Concentration	Calcium Lactate	Gum Arabic	Super Plasticizer
PCS	Plain	1 : 2 : 2.2	0.4	No Graphite	No Bacteria	No Lactate	No Gum arabic	1% of Cement
BCS	Bacterial			No Graphite	5 x 10^6	3% of Cement	6% of Graphite	
GNMP-1.0	Graphitic			1% of cement				
GNMP-1.25	Graphitic			1.25% of cement				

Table 3.5 Casting Regime

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CHAPTER 4

EXPERIMENTAL TESTS AND RESULTS

4.1 Compressive Strength:

Compressive strength is the capacity of a material or structure to withstand loads tending to reduce size, as opposed to tensile strength, which withstands loads tending to elongate. Compressive strength is often measured on a Compression Testing Machine; these range from very small table-top systems to ones with over 53 MN capacity. Measurements of compressive strength are affected by the specific test method and conditions of measurement.

Figure 4.1: Compression testing machine

Compression test was performed at a stress rate of 0.25 MPa/sec as per ASTM C 39. Specimens were tested after 3,14 and 28 days of curing. Testing was carried out on all the sample types i-e PCS, BCS, GNMP-01 and GNMP-1.25. Graph was plotted to compare the results.

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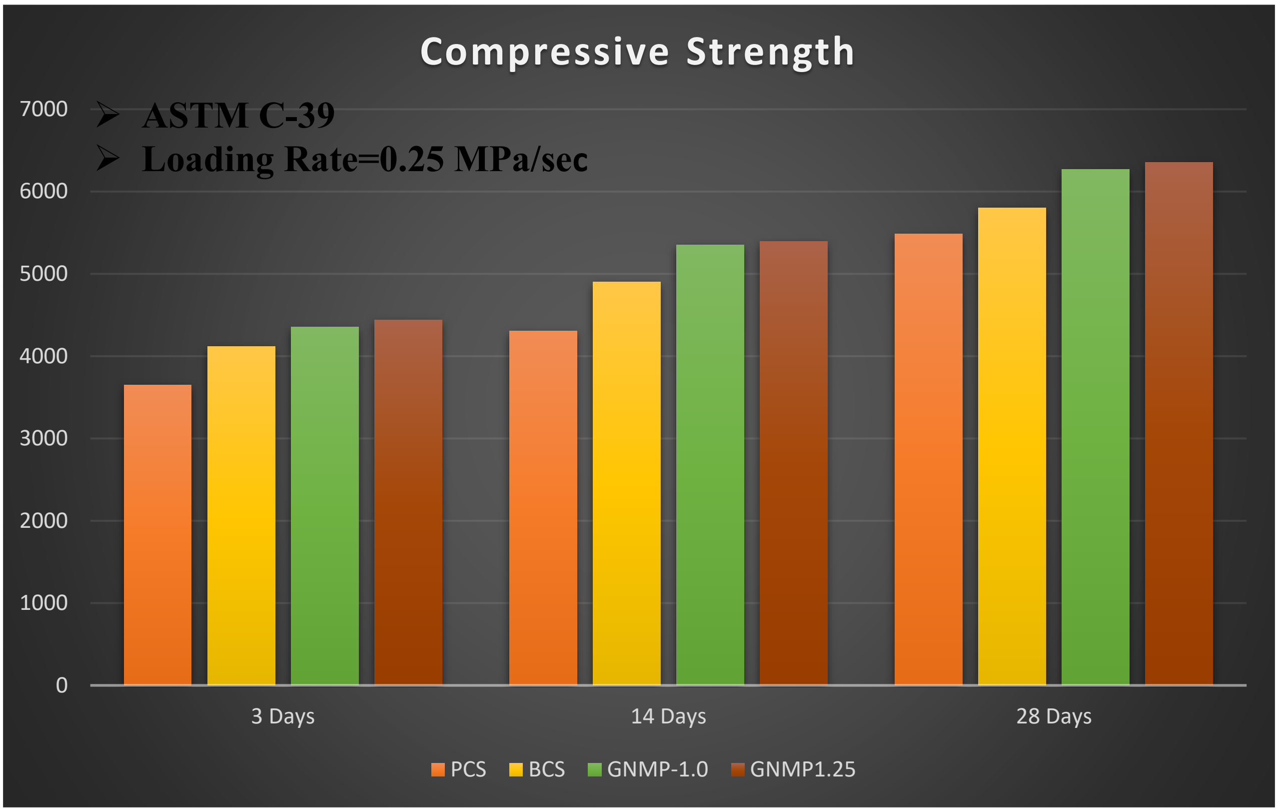

Figure 4.2: Compressive strengths of different samples

Figure 4.2 shows that the highest strength was obtained for the GNMP-1.25 sample which concludes that the addition of bacteria causes a significant increase in the compressive strength of the concrete. Another reason of the increased compressive strength of GNMP samples is the ability of graphite to inhibit propagation of crack which is opposite to PCS, which lacks crack arrest mechanism.

4.2 Fracture Mechanics:

Fracture mechanics is the study of the propagation of cracks inside the concrete. In order to study the fracture mechanics, short beams of size (100 x 100 x 400) mm were casted and 3-point bend test was performed at Comsats Wah.

4.2.1 3-point bend test:

A three-point bend test consists of the sample placed horizontally upon two points and the force applied to the top of the sample through a single point so that the sample is bent in the shape of a "V".We selected lower strain rate of 0.3mm/min because the concrete is brittle material and is quite sensitive to higher strain rates and since we need to study the crack propagation arrest ability of graphite as it acts as micro reinforcement inside the concrete. Thus, the test was performed on Universal Testing Machine since it can accommodate lower strains. This also reflects slow crack propagation in structures in real life. Short beams were tested after 28 days of curing. The data obtained from the test gave the values of force and stroke which were converted to stress and strain value using formulas. Graph between stress and strain was plotted using Origin software. Graph is shown in figure 4.5.

Figure 4.3: Universal Testing Machine at COMSATS Wah.

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Figure 4.4: 3-point bend test on short beam

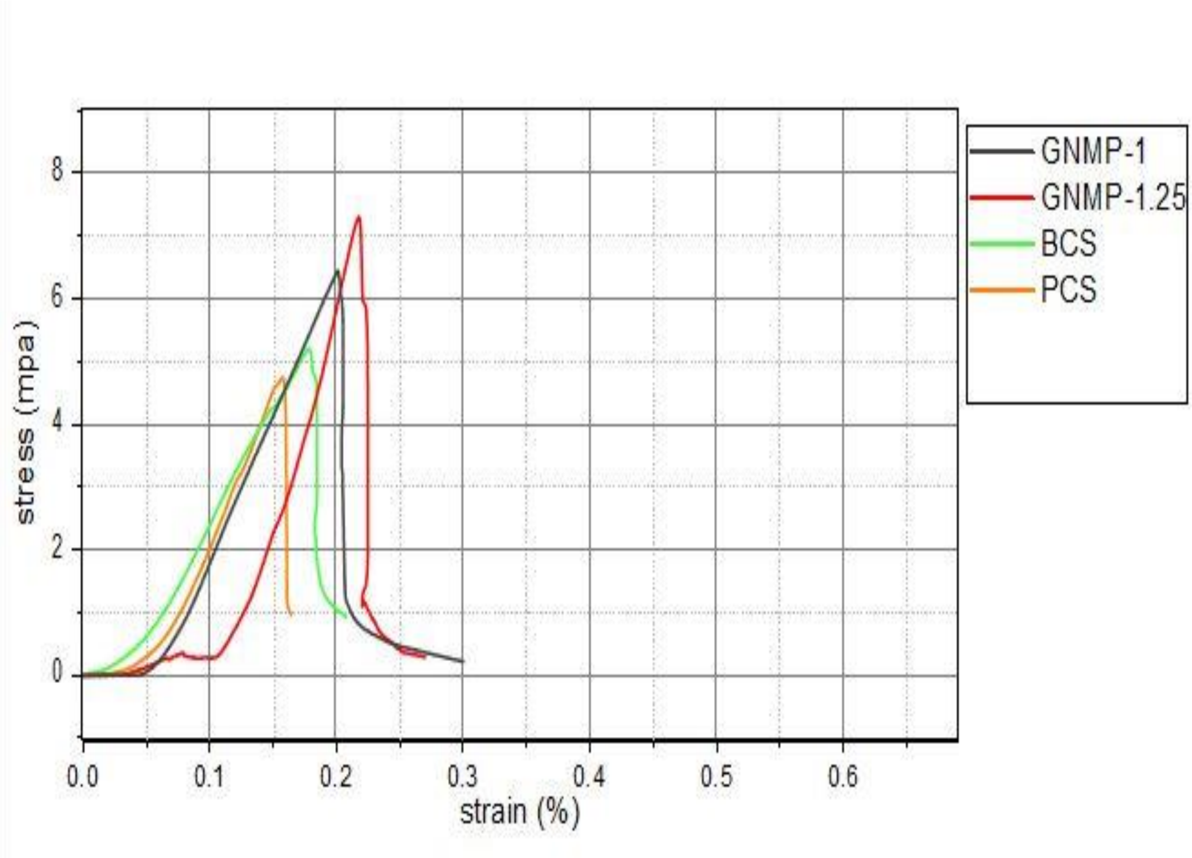

Figure 4.5: Graph between Stress (Mps) and Strain (%)

From figure 4.5, it can be seen that the addition of only bacteria (BCS) does not have significant effect on the modulus of elasticity but the addition of GNMPs along with bacteria have a significant effect on the modulus of elasticity. As the concentration of GNMP increases, a

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linearly increasing trend in stress values can be seen. This increase in stress value is primarily due to the crack propagation arrest ability of GNMPs which acts as micro reinforcement inside the concrete, thereby delaying the crack nucleation and propagation. Also, the bacteria precipitates inside the voids present in the matrix of concrete, thereby densifying it and making the concrete more durable.

4.2.2 Flexure Strength or Modulus of rupture:

Flexural strength or MoR is a measure of the tensile strength of concrete beams or slabs. Flexural strength identifies the amount of stress and force an unreinforced concrete slab, beam or other structure can withstand such that it resists any bending failures.It shows the strength of material before it undergoes complete rupture.Flexure strength for each type of concentration was determined by taking the maximum value of stress for each concentration from the stress vs strain graph shown in figure 4.5. or it can be calculated by using the following formula:

$$MoR = 3PL/2bd2$$

Following results were obtained as shown in figure 4.6:

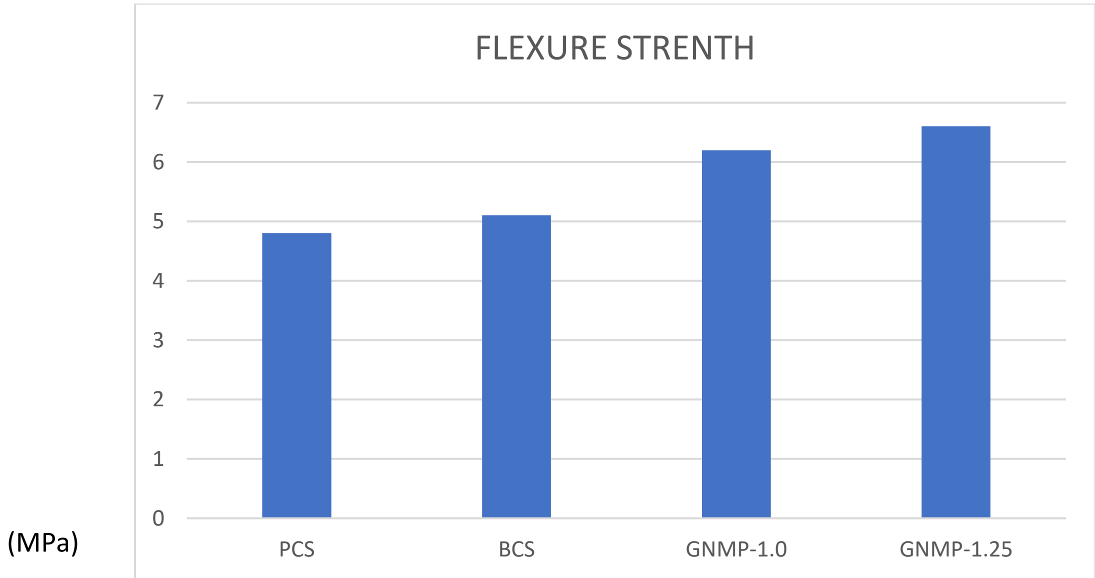

Figure 4.6 Flexure strength of different samples

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Figure 4.6 shows flexure strength values of each concentration. No significant difference between flexure strength of PCS and BCS samples is observed since bacteria does not enhances the flexure properties. However, significant increase in flexure strength of GNMP samples is observed. This means that the addition of graphite causes this enhancement since graphite resists the propagation of cracks and the concrete yields at higher stress. Secondly, linearly increasing trend in flexure strength values was obtained in GNMP samples with increasing concentrations as compare to PCS since calcite precipitation in GNMP samples depend upon the content of surface exposed bacteria, so with increasing GNMPs concentration, surface exposed bacteria increases, which results in greater calcite precipitation before cracking which densifies the matrix and induces improved microstructural properties. Also, GNMPs themselves enhances the strength values as well. So improved stress values are a combination of both bacteria and GNMPs.

4.2.3 Ductility Deformation Factor (DDF):

The fracture mechanics can be effectively understood by studying in detail the change in ductility (DDF).DDF shows us the behavior of concrete after it yields. As we know that due to addition of steel reinforcement in concrete, the post yield energy dissipation capacity of concrete is enhances, and the GNMPs also acts as micro reinforcements, thus their effect on post yield behavior of concrete is necessary to study. Since presence of post yield potential classifies the material under study as ductile, hence DDF is a measure of overall ductility introduced in heterogeneous concrete matrix due to presence of GNMPs, and maybe microbes as well. Ductility deformation factor is calculated by the following formula:

Ductility Deformation Factor (DDF) = (Du / Dy) *100

Where values of ultimate Du and yield strain Dy values of each concentration were determined from the stress vs strain graph shown in figure 4.5.

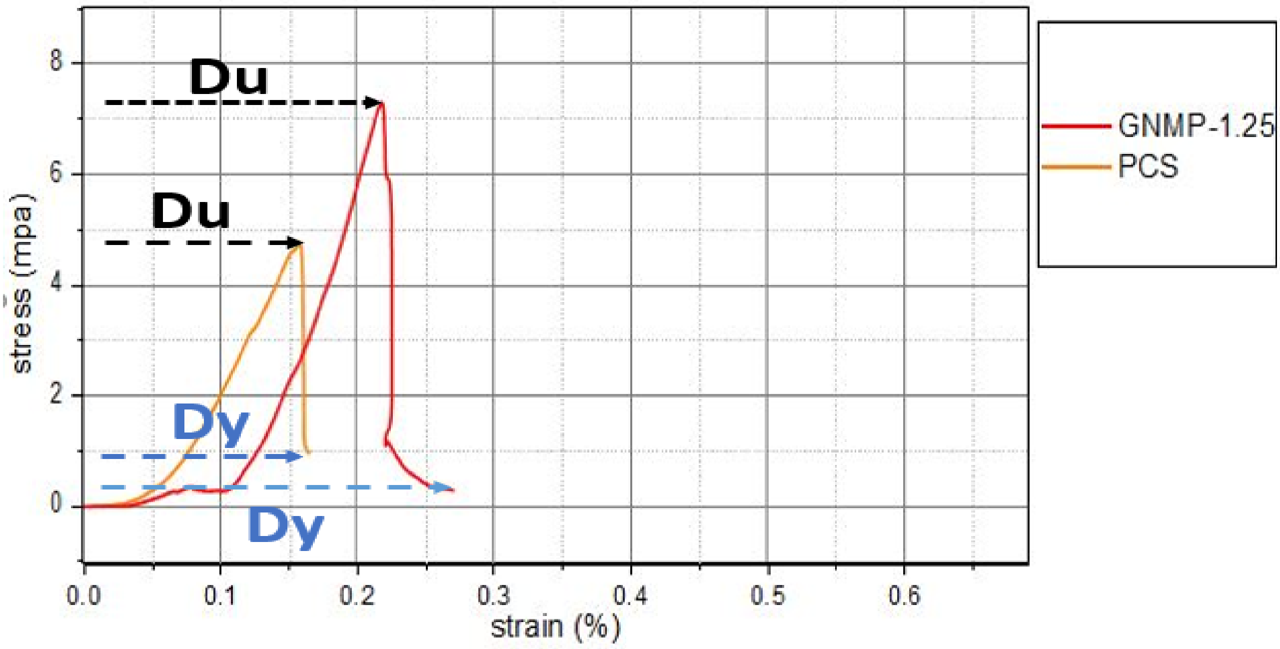

Figure 4.7: Determining Du and Dy for PCS and GNMP-1.25 samples

Results obtained are shown in figure 4.8.

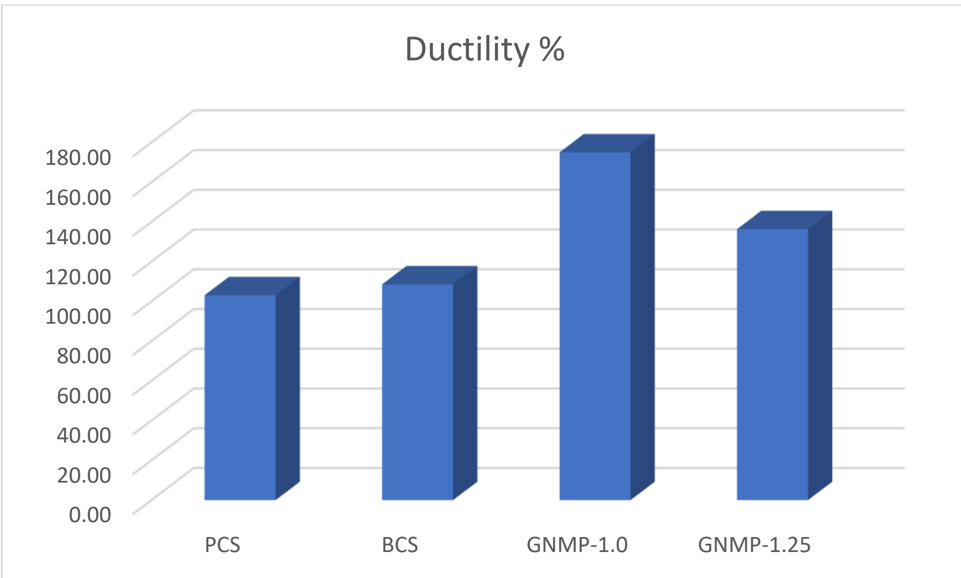

Figure 4.8: Ductility(%) for different samples

As evident from the graph, mere introduction of bacteria in BCS samples has caused no change in post yielding behaviour in comparison to PCS. It means that bacteria is certainly not a governing factor in exhibiting post-yield behaviour. This hypothesis is complemented by the subsequent GNMP concentrations and comparison of ductility of GNMP concentrations with PCS and BCS where introduction of GNMPs has generated post yielding behaviour. However, post yield behaviour is reduced in higher GNMP concentrations. This is due to the fact that higher GNMPs can cause ineffective dispersion, causing micro reinforcement to not effectively resist crack propagation and subsequently causes agglomeration inside the cementitious matrix, therefore forming weak pockets.

4.3 Self-Healing Analysis:

4.3.1 Crack Width Measurement Analysis (CWMA):

Crack width measurement analysis is used to check the healing of surface cracks, healed by bacterial precipitation. The cracks are artificially generated on the surface of the concrete samples by applying compressive load lesser that the ultimate compressive strength of the sample. The healing is checked by marking the cracks on the sample and then measuring its width by using Crack Width Measuring Microscope. After the healing has occurred, the width of the precipitation occurred in the crack is measured and the amount of healing is calculated.
3 cylinders of size 100mm x 200mm were casted for each concentration. Cylinder 1 was cracked at full compressive strength while cylinder 2 and 3 were cracked at 80% and 70 % of the original compressive strength respectively. The reason to crack the samples at lesser load is to artificially induce micro cracks at the surface of the samples. Healing was checked for 3,14 and 28 days wet cured samples. Then for each day, each specimen was recurred for 28 days. Reason behind keeping 28 days was to check whether bacterial precipitation occurs in the same duration in crack healing as is the natural hydration period of concrete to gain 99% of strength. After curing, significant healed crack widths of each specimen were measured using Crack Width Measuring Microscope.

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Figure 4.9: Crack width measuring microscope

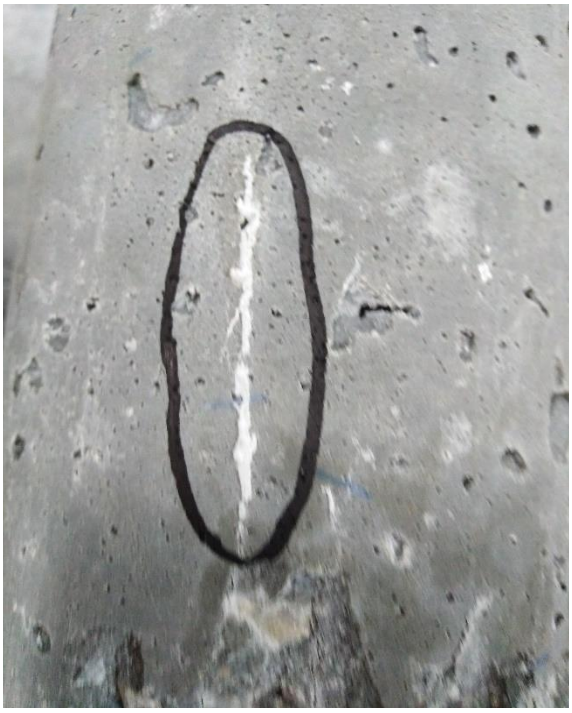

Figure 4.10: Marking of cracks

Figure 4.9 shows the microscope which measures the width of the cracks. Figure 4.10 shows how cracks are marked and the white colour indicates the precipitation that has healed the crack.

Results of crack width healed are summarized in table 4.1.

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CRACK WIDTH MEASUREMENT					
SAMPLE	**DAYS**	**CYLINDER 1 Healing(mm)**	**CYLINDER 2 Healing(mm)**	**CYLINDER 3 Healing(mm)**	**Average Healing (mm)**
PCS	**3**	No Healing	No Healing	No Healing	No Healing
	14	No Healing	No Healing	No Healing	No Healing
	28	No Healing	No Healing	No Healing	No Healing
BCS	**3**	Partial Healing	Partial Healing	Partial Healing	Partial Healing
	14	Partial Healing	Partial Healing	Partial Healing	Partial Healing
	28	Partial Healing	Partial Healing	Partial Healing	Partial Healing
GNMP-1.0	**3**	1.70	2.4	2.2	2.1
	14	1.8	1.8	2.2	1.93
	28	1.4	2	1.6	1.67
GNMP-1.25	**3**	1.9	1.9	1.8	1.87
	14	1.3	1.6	1.5	1.47
	28	2	1.3	1.8	1.7

Table 4.1: Crack widths healed

As evident from the results, the PCS samples has shown no healing since it has no bacteria. Another important deduction here is that the natural hydration process of concrete does not heal cracks at all. Microscopic image of crack in PCS sample after 28 days healing period is shown below:

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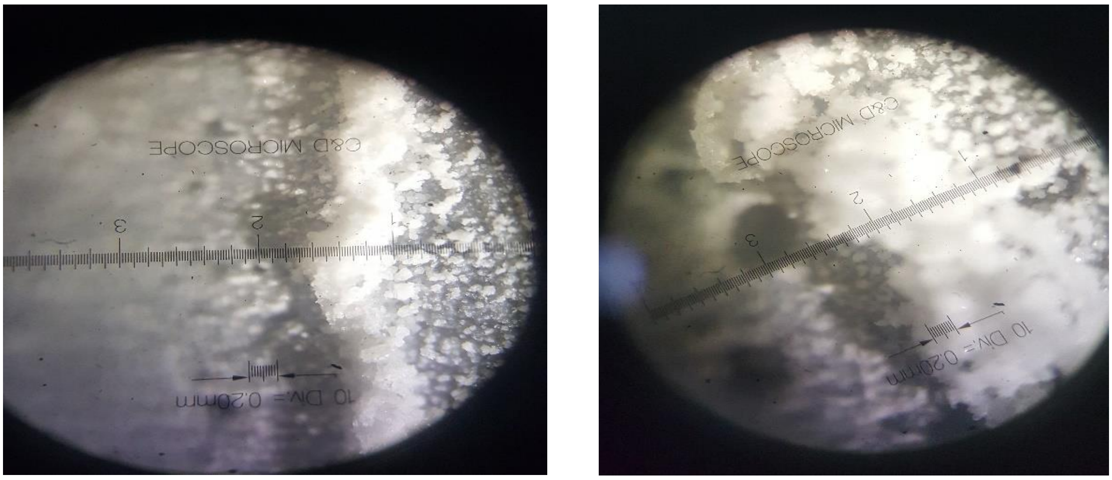
Figure 4.11 No healing in PCS

From table 4.1, we see that in BCS samples only partial healing has occurred. This is because in BCS samples water acts as immobilizer for bacterial which is not an effective carrier and cannot serve the purpose of long-term survival of most bacterial concentration. That's why full healing has not been observed in BCS samples. Microscopic images of partially healed cracks in BCS samples after 28 days healing period are shown below:



`Figure 4.12 Partially healed cracks in BCS samples seen from microscope

From table 4.1, it is evident that complete healing of cracks has occurred in the GNMPs samples. This means that graphite is an effective carrier or immobilizer for the bacteria and bacterial precipitation occurs more efficiently in GNMP samples. Among GNMP samples, we see that maximum crack healed in GNMP-1.0 samples is of 2.4mm width while the maximum crack healed in GNMP-1.25 samples is of 1. 9mm.Ideally, we should follow the same trend that we have followed in fracture mechanics i.e. to determine optimum GNMP concentration that yield greatest crack width healing. However, since pre-cracking is not a controlled process, therefore it is not necessary that cracks having same widths have been produced in every sample. It means that even though GNMP-1.0 has shown maximum crack width healing of 2.4 mm and GNMP-1.25 has shown only 1.9 mm, it doesn't conclude that GNMP-1.0 is a better concentration because from literature, we know that greater carrier concentration yields greater healing. This trend is not seen here since cracks produced were not uniform. Maybe if GNMP-1.25 had a crack of 2.4 mm width, it would have healed as well.

From this we conclude that this particular range of GNMPs (1.0-1.25) is quite ideal for effective crack width healing. The optimum concentration can be determined from EDS test. Maximum crack healed is of 2.4 mm corresponding to GNMP-1.0. This extends our healing capacity form non-structural to structural cracks, a result not quoted before in literature. In past, maximum crack width healed reported using Bacillus Subtilis is around 0.81 mm (Khaliq et al., 2016). So, we have improved on this value significantly. Average crack width healing of GNMP concentrations ranges from 1.47 to 2.1 mm.
Microscopic image of completely healed cracks in GNMPs samples after 28 days healing period are shown below:

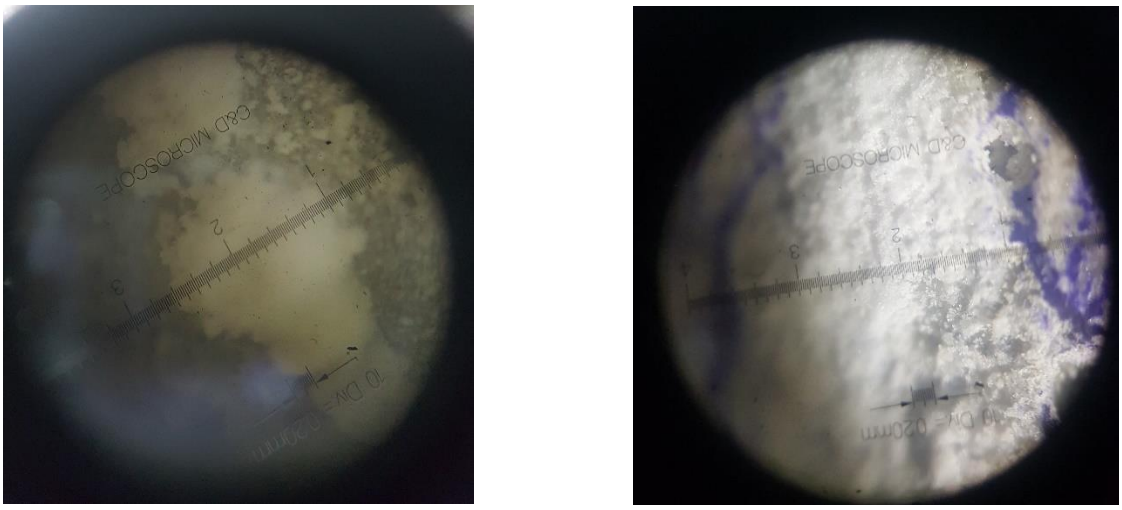


Figure 4.13 Completely healed cracks in GNMPs samples seen from microscope

4.3.2 Ultrasonic Pulse Velocity (UPV):

An ultrasonic pulse velocity (UPV) test is an in-situ, nondestructive test to check the quality of concrete. We performed this test in order to find out the amount of precipitation occurred inside the concrete matrix or whether significant healing do occur inside the concrete matrix. Equipment used for UPV test is known as PUNDIT-PL 200.It consists of 2 probes.One probe is connected to one end of the concrete cylinder and other probe is connected to the other extreme of the cylinder. An ultrasonic pulse is generated by one probe which is received by the other probe .Time taken by the ultrasonic pulse from point of generation of pulse by one probe to the point at which it is received by another probe is noted and shown on the screen. This time is then used to find the velocity of the ultrasonic pulse using the following formula :

Pulse Velocity = Width of concrete cylinder / Time taken by Ultrasonic pulse

Velocity of the pulse depends upon the voids/cracks present inside the concrete. If more cracks are present in the concrete matrix, the ultrasonic pulse takes longer time to travel from one probe to the other. Thus, the velocity of ultrasonic pulse is lower.

Figure 4.14 Performing UltraSonic Pulse Velocity test

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Cylinder 2 and 3 cracked at 80% and 70% of the original compressive strength respectively, for each concentration type, were used for the test. Time of ultrasonic pulse was noted for 3 different conditions i-e

i) Uncracked (Before subjecting to compressive load or induction of cracks)

ii) Cracked (After subjecting to compressive load or induction of cracks0

iii) Healed (After 28 days healing period)

Time obtained for each condition and sample type were used to find the ultrasonic pulse velocity. Results were compared by plotting bar graph shown in figure 4.15

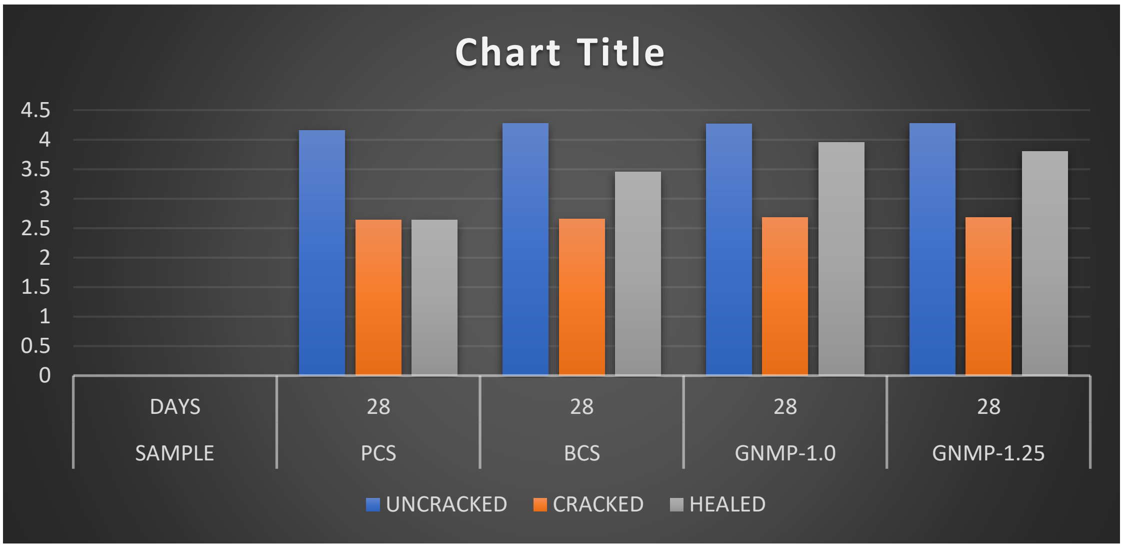

Figure 4.15 UPV of different samples under different cracking condition

From graph, we can see that the maximum values of ultrasonic pulse velocity are obtained for uncracked condition for each sample type. In uncracked condition, concrete is in prime form having minimum number of voids/cracks. Thus, the ultrasonic pulse passes at higher speeds through the densified concrete matrix. Minimum values of ultrasonic pulse velocity are obtained for cracked condition for each sample type. Since concrete had undergone compressive stresses at this point, thus cracks are produced inside the concrete matrix due to which ultrasonic pulse takes longer time.

For the healed condition, we can see that no increase in the velocity has occurred for PCS samples. Thereby confirming that the hydration process of concrete does not causes healing. In BCS samples, increase in the velocity is observed which confirms that precipitation do occur

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inside the concrete matrix but still lots of cracks remain unhealed because bacteria does no perform well when immobilized with water. In GNMP samples, we see significant increase in the velocity of ultrasonic pulse ,thereby confirming the bacterial precipitation inside the concrete matrix. Comparing the UPV values of BCS and GNMP samples for healed condition, we see that GNMP samples have higher UPV values which concludes that in the presence of graphite as immobilizer for bacteria, precipitation occurs more efficiently.

Thus, graphite is much better carrier material for bacteria as compared to water. The concrete matrix is densified more in GNMP samples as compared to BCS samples.

4.3.3 Strength Recovery Index (SRI):

Healing of cracks not only prevents the entrance of undesirable impurities into concrete, but also results in the recovery of mechanical strength of the cracked specimens after curing for some days. In order to study this behavior, Strength Recovery Index (SRI) has been developed, which describes in percentage the amount of strength recovered due to post healing action after curing. Initial strength is noted for fresh sample and the regained strength is noted after 28 days of healing period.SRI is calculated by the following formula:

SRI = (1 – (Initial Strength −Regained Strength) / Initial Strength) *100

Strength recovery index was carried out for three cylinders, one of them was cracked at full compressive strength, the other was cracked at 80% of original compressive strength while the third cylinder was cracked at 70% of original compressive strength. Specimens were cured for 3,14- and 28-days wet curing. Following results were obtained:

Below 40 %	41-50%	51-60%	61-70%	71-80%	80-90%	90-100%

Figure 4.16 Strength Recovery Index (SRI) scale

CYLINDER 1 (30D)	**3D**	PCS	BCS	GNMP-1	GNMP-1.25
	7D	PCS	BCS	GNMP-1	GNMP-1.25
	28D	PCS	BCS	GNMP-1	GNMP-1.25

CYLINDER 2 (30D)	**3D**	PCS	BCS	GNMP-1	GNMP-1.25
	7D	PCS	BCS	GNMP-1	GNMP-1.25
	28D	PCS	BCS	GNMP-1	GNMP-1.25

CYLINDER 3 (30D)	**3D**	PCS	BCS	GNMP-1	GNMP-1.25
	7D	PCS	BCS	GNMP-1	GNMP-1.25
	28D	PCS	BCS	GNMP-1	GNMP-1.25

Figure 4.17 Tabulated results of percentage strength recovered

Firstly, if we observe this index it is clearly identified that there is a significant difference in percentage strength recovered between samples having GNMPs as carrier media for bacteria and samples having no GNMPs. This is because in plain controlled samples, strength recovered is not due to calcite precipitation but as a result of natural hydration of cement which is not a contributing factor in healing which we are studying. Similarly, in case of Catamount of preserved bacteria is less for healing purposes before cracking due to which calcite is not efficiently precipitated in cracks of the sample after cracking which results in less strength recovered. Similarly, in case of samples having graphite as immobilizer for bacteria, more strength has been recovered because amount of preserved bacteria left is more before cracking

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for healing action due to which after cracking, more bacteria will be in action for precipitating calcite which ultimately results in greater strength recovery.

Secondly, if we compare the amount of percentage strength recovered in samples having different amount of GNMPs concentrations, we can conclude from this index that with increase in GNMPs concentrations, greater amount of strength is recovered. This is because samples having more bacterial carrier act better and effectively as a protective layer, resulting in more preserved bacteria before cracking which results in more calcite precipitation after cracking.
Third, the cylinders that were cracked at 70% of original compressive strength were found to have most healing among all. The reason being the fact that it had of cracks of lesser widths due to which better healing took place within 28 days of curing after cracking, as compared to other cylinders which had cracks having greater widths. Cylinders that were cracked at 80% of original compressive strength had considerably less healing while that of 100% was healed the least amongst all.

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CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

- Bacillus Subtills is an effective bacterial media for inducing self-healing abilities in concrete.
- GNMPs (graphite) is an effective immobilizer and carrier material for bacteria and further enhances the working of microbes. Thus, better results of healing are evident in GNMP samples where full healing has occurred as compared to the BCS samples in which only partial healing took place.
- Maximum healing was noted for GNMP-1.0 samples with cracks up to 2.4mm were healed. In GNMP-1.25 samples, cracks up to 1.9mm were healed.
- Healing of internal cracks is also observed as evident from the ultrasonic pulse velocity results. Thus, the overall quality of concrete is enhanced after healing and concrete become more durable.
- GNMPs also acts as micro reinforcement in concrete and resists the propagation of cracks due to their crack arrest ability. Hence adds to the flexure strength of concrete. Increase in flexure strength up to 40% writ to PCS is observed.
- Significant increase in compressive strength is observed in BCS and GNMP samples as compared to Pushiest compressive strength was noted for GNMP-1.25 sample having 28 days strength of 6350 Psi.
- Higher concentration of GNMPs yields better compressive strength. Highest compressive strength was noted for GNMP-1.25 sample having 28 days strength of 6350 Psi.
- Due to healing action, concrete undergoes strength recovery, Maximum strength recovered was 92%, observed in GNMP-1.25 sample cracked at 70% of original compressive strength.

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5.2 RECOMMENDATIONS

- Develop bio-healable RCC concrete and study its corrosive resistance ability.
- Develop a mechanism to detect the absorption of carbon dioxide by bio-healable concrete as well as determine the amount of CO_2 absorbed.
- Develop carbonadoes immobilizer from waste materials and study its effect in bio-healable concrete.
- Study the effect of healing under continuous or sustained loadings.
- Investigate whether a controlled healing triggering mechanism can be developed to preserve microbes and enhance efficiency of healing.
- Study the fatigue response of bio-healable concrete in a field trial by preparing a rigid pavement patch.

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APPENDICES

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APPENDIX:01 COMPRESSIVE STRENGTH TEST RESULTS

		COMPRESSIVE STRENGTH					
		Cylinder 1		Cylinder 2 (0.8xCylinder 1)		Cylinder 3 (0.7xCylinder 1)	
SAMPLE	DAYS	KPa	Psi	Kpa	Psi	Kpa	Psi
	3	25.2	3656.02	20.2	2930.62	17.6	2553.41
PCS	14	29.7	4308.88	23.9	3467.41	20.8	3017.66
	28	37.8	5484.02	30.3	4395.92	26.5	3844.62
	3	28.4	4120.27	22.7	3293.32	20	2901.60
BCS	14	33.8	4903.70	27.3	3960.68	23.5	3409.38
	28	40	5803.20	32	4642.56	28.2	4091.26
	3	30	4352.40	24	3481.92	21.8	3162.74
	14	36.9	5353.45	27.7	4018.72	26	3772.08
GNMP-1.0	28	43.2	6267.46	34.5	5005.26	30.8	4468.46
	3	30.6	4439.45	24.5	3554.46	21.5	3119.22
	14	37.2	5396.98	29.8	4323.38	26	3772.08
GNMP-1.25	28	43.8	6354.50	35	5077.80	30.6	4439.45

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APPENDIX:02 ULTRASONIC PULSE VELOCITY TEST RESULTS

ULTRASONIC PULSE VELOCITY							
		CYLINDER 3 (Cracked at 70% of original compressive strength)					
		UNCRACKED		CRACKED		HEALED	
SAMPLE	DAYS	Time (µs)	Velocity(µ/mm)	Time (µs)	Velocity(µ/mm)	Time (µs)	Velocity(µ/mm)
PCS	3	47.4	4.22	75.4	2.65	75.4	2.65
	14	46.5	4.30	75.1	2.66	74.8	2.67
	28	46.7	4.28	76.2	2.62	76.2	2.62
BCS	3	47.7	4.19	76.4	2.62	52.8	3.79
	14	48	4.17	76.6	2.61	52.5	3.81
	28	47.8	4.18	75.2	2.66	50.6	3.95
GNMP-1.0	3	46.4	4.31	75.5	2.65	51.3	3.90
	14	46.8	4.27	74.1	2.70	49.8	4.02
	28	47.3	4.23	73.8	2.71	48.8	4.10
GNMP-1.25	3	46.5	4.30	74.8	2.67	51	3.92
	14	47.5	4.21	74.3	2.69	50.2	3.98
	28	47.3	4.23	73.9	2.71	49.2	4.07

APPENDIX:02 ULTRASONIC PULSE VELOCITY TEST RESULTS

ULTRASONIC PULSE VELOCITY							
		CYLINDER 2 (Cracked at 80% of original compressive strength)					
		UNCRACKED		CRACKED		HEALED	
SAMPLE	DAYS	Time (µs)	Velocity(km/sec)	Time (µs)	Velocity(km/sec)	Time (µs)	Velocity(µ/mm)
PCS	3	47	4.26	75.6	2.65	75.4	2.65
	14	47.8	4.18	76.5	2.61	76.3	2.62
	28	48.1	4.16	75.7	2.64	75.7	2.64
BCS	3	47.4	4.22	76.6	2.61	52.8	3.79
	14	47.8	4.18	76	2.63	52	3.85
	28	46.8	4.27	75.4	2.65	50.6	3.95
GNMP-1.0	3	47	4.26	75	2.67	50.4	3.97
	14	46.8	4.27	74.8	2.67	49.8	4.02
	28	46.9	4.26	74.6	2.68	49.4	4.05
GNMP-1.25	3	47.1	4.25	75.6	2.65	51.3	3.90
	14	47.5	4.21	75.2	2.66	50.7	3.94
	28	46.8	4.27	74.5	2.68	49.4	4.05

APPENDIX:02 ULTRASONIC PULSE VELOCITY TEST RESULTS

STRENGTH RECOVERY INDEX				
		Cylinder 3 (Cracked at 70% of original compressive strength)		
SAMPLE	DAYS	Initial strength(psi)	Regained strength(psi)	Strength recovery index(%)
PCS	3	3656.02	1619.90	44.31
	14	4308.88	1711.57	39.72
	28	5484.02	2091.24	38.13
BCS	3	4120.27	2643.90	64.17
	14	4903.70	3018.61	61.56
	28	5803.20	2971.74	51.21
GNMP-1.0	3	4352.40	3692.76	84.84
	14	5353.45	4123.97	77.03
	28	6267.46	4676.41	74.61
GNMP-1.25	3	4439.45	4092.34	92.18
	14	5396.98	4491.43	83.22
	28	6354.50	5322.40	83.76

APPENDIX:03 STRENGTH RECOVERY INDEX RESULTS

		STRENGTH RECOVERY INDEX		
		Cylinder 1		
SAMPLE	DAYS	Initial strength(psi)	Regained strength(psi)	Strength recovery index(%)
PCS	3	3656.02	1114.67	30.49
	14	4308.88	1278.90	29.68
	28	5484.02	1283.16	23.40
BCS	3	4120.27	1945.54	47.22
	14	4903.70	2394.67	48.83
	28	5803.20	2256.49	38.88
GNMP-1.0	3	4352.40	2352.12	54.04
	14	5353.45	2341.83	43.74
	28	6267.46	2845.00	45.39
GNMP-1.25	3	4439.45	2496.37	56.23
	14	5396.98	2613.56	48.43
	28	6354.50	3359.70	52.87

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