

A study on the self-healing and self-sensing capabilities of graphene-oxide (GO) nano reinforced bacterial cement mortar



Supervised By

Dr Taimur Mazhar Sheikh

Group Members

| | |
|------------------------|------------------|
| Muhammad Tauqeer Zaman | UW-19-CE-BSc-036 |
| M. Tauqeer Ahmad | UW-19-CE-BSc-034 |
| Maaz Ullah | UW-19-CE-BSc-037 |
| Shehryar Ahmad | UW-19-CE-BSc-R49 |

Department of Civil Engineering

Wah Engineering College

University of Wah

Wah Cantt, Pakistan

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

Final Year Design Project Thesis Report

| | | | |
|-------------------|----|--------------------------|----|
| Project ID | 02 | Number Of Members | 04 |
|-------------------|----|--------------------------|----|

| | |
|--------------|---|
| Title | A study on the self-healing and self-sensing capabilities of graphene-oxide (GO) nano reinforced bacterial cement mortar. |
|--------------|---|

| | | |
|------------------------|--------------------------|---|
| Supervisor Name | Dr. Taimur Mazhar Sheikh | Assistant Professor & Civil Engineering Department) |
|------------------------|--------------------------|---|

| Group Members Name | Reg. No. | Email Address |
|-------------------------------|------------------|--------------------------------------|
| <u>Muhammad Tauqeer zaman</u> | UW-19-CE-BSC-036 | <u>UW-19-CE-BSC-036@WECUW.EDU.PK</u> |
| <u>Toqeer Ahmed</u> | UW-19-CE-BSC-034 | <u>UW-19-CE-BSC-034@WECUW.EDU.PK</u> |
| <u>Maaz Ullah</u> | UW-19-CE-BSC-037 | <u>UW-19-CE-BSC-037@WECUW.EDU.PK</u> |
| <u>Shehravar Ahmed</u> | UW-18-CE-BSC-R49 | <u>UW-18-CE-BSC-R49@WECUW.EDU.PK</u> |
| | | |

Supervisor Signature

Chairperson CED Signature

CHECKLIST

- | | |
|--|---|
| <ul style="list-style-type: none"> Number of pages attached with this form I/We have enclosed the softcopy of this document along with the codes and scripts created by ourselves. My/Our supervisor has attested the attached document. I/We confirm to state that this project is free from any type of plagiarism and misuse of copyrighted material. | <div style="border: 1px solid black; width: 40px; height: 20px; margin: 0 auto;"></div> <p>Yes / No</p> <p>Yes / No</p> <p>Yes / No</p> |
|--|---|

Undertaking

It is declared that the work entitled “**A study on the self-healing and self-sensing capabilities of graphene-oxide (GO) nano reinforced bacterial cement mortar**” presented in this report is an original piece of our own work, except where otherwise acknowledged in text and references. This work has not been submitted in any form for another degree or diploma at any university or other institution for tertiary education and shall not be submitted by us in future for obtaining any degree from this or any other University or Institution.

| Group Members Name | Signatures |
|---------------------------|-------------------|
| 1.M. Tauqeer Zaman | |
| 2.Toqeer Ahmed | |
| 3. Maaz Ullah | |
| 4. Shehrayar Ahmed | |

Acknowledgement

We are thankful to Allah Almighty for successful completion of our project. Our special thanks to our supervisor [Dr. Taimur Mazhar Sheikh] for giving his supervision. We are also grateful to Co-supervisor [Dr. Habil Ahmad] who gave his precious time and guiding us in project completion and all faculty members who helped us with their advices and comments. We are thankful to the administration of Electrical Department for allowing us to use the lab equipment as per requirement.

| Group Members Name | Signatures |
|---------------------------|-------------------|
| 1.M. Tauqeer Zaman | |
| 2.Tauqeer Ahmed | |
| 3. Maaz Ullah | |
| 4. Shehrayar Ahmed | |

Abstract

The development of cracks in concrete is inevitable, however increased crack propagation leads to reduced service life of concrete due to ingress of harmful compounds and accelerated rebar corrosion. By implementing a highly oxidized graphene oxide (GO) and *Bacillus subtilis* bacteria, this research aims to improve durability of concrete through self-healing and self-sensing abilities. Results indicate significant crack healing in early age concrete at 0.04% GO by weight of cement. Additionally, GO nano reinforced samples exhibit greater sensitivity to electrical changes under compressive loading. These findings hold great promise for development of sustainable infrastructure, contributing to resilient practices and long-term durability of concrete structures.

Contents

| | |
|--|-----------|
| 1. Introduction..... | 1 |
| 1. Problem Statement..... | 2 |
| 2. Statement of Project..... | 2 |
| 3. Aims & Objectives | 2 |
| 4. Methodology..... | 3 |
| 5. Explanation..... | 4 |
| 2. Literature Review | 6 |
| 2.1 Introduction..... | 6 |
| 2.2 Chemical attack..... | 6 |
| 2.3 Rebar corrosion..... | 7 |
| 2.4 Moisture and Oxygen..... | 8 |
| 2.5 Graphene oxide | 8 |
| 2.6 Autogenous healing..... | 12 |
| 2.7 Bacteria-based self-healing concrete | 13 |
| 2.8 Bacteria | 15 |
| 2.9 Nutrient substance: -..... | 15 |
| 2.10 Carrier Compounds..... | 16 |
| 2.11 Impact of environmental factor on the effectiveness of bacterial concrete..... | 17 |
| Relevant Work..... | 18 |
| 3. Research Methodology | 20 |
| 3.1 Materials..... | 20 |
| 3.1.1 Formation of Graphene oxide..... | 20 |
| 3.1.2 Extraction of Bacillus Subtilis..... | 23 |
| 3.2 Mould Selection and Mix Tables..... | 26 |
| 3.2.1 Mould Selection..... | 26 |
| 3.2.2 Mix Tables..... | 26 |
| 3.3 Tests..... | 28 |
| 3.3.1 Optical microscopy test. | 29 |
| 3.3.2 Resistivity Test..... | 30 |
| 3.3.3 Compression test | 34 |
| 4. Results..... | 36 |

| | | |
|------------|--|-----------|
| 4.1 | Results of self- healing | 36 |
| 4.1.1 | Result of GO Samples | 36 |
| 4.1.2 | Result of Bt Samples | 38 |
| 4.1.3 | Results of Bacteria with calcium lactate | 39 |
| 4.1.4 | Results of Simple Bacteria..... | 41 |
| 4.1.5 | Results of Bacteria with GO..... | 43 |
| 4.2 | Self-Sensing Results | 44 |
| | Results of Compression Sensing test | 46 |
| 5. | Conclusion and Recommendation | 49 |
| 5.1 | Conclusion: -..... | 49 |
| 5.2 | Recommendation..... | 49 |
| | References:..... | 50 |

List of Figures

| | |
|--|------------------------------|
| Figure 1.1 Show how conductive fillers conduct electric current | Error! Bookmark not defined. |
| Figure 1.2 Show a Methodology of Research..... | 3 |
| Figure 2.1 comparison of GO and CNTS transformation..... | 9 |
| Figure 2.2 show the layer view of graphite | 9 |
| Figure 2.3 Show the layer view of graphene oxide | 10 |
| Figure 2.4 Demonstrate Autogenous healing..... | 13 |
| Figure 2.5 Show the culture of Bacteria..... | 15 |
| | |
| Figure 3.1 Adding H ₂ SO ₄ in beaker | 20 |
| Figure 3.2 Adding H ₃ PO ₄ to H ₂ SO ₄ | 20 |
| Figure 3.3 Add 1 g graphite..... | 21 |
| Figure 3.4 Add kmno ₄ to above solution..... | 21 |
| Figure 3.5 Add hydrogen peroxide | 22 |
| Figure 3.6 Sonicate the above solution | 22 |
| Figure 3.7 Show serial dilution method | 23 |
| Figure 3.8 Show culture media | 23 |
| Figure 3.9 Spread bacteria to plate | 24 |
| Figure 3.10 Incubation of plates | 24 |
| Figure 3.11 Observe the colony..... | 25 |
| Figure 3.12 show gram staining method | 25 |
| Figure 3.13 Observing samples by Optical microscope | 29 |
| Figure 3.14a Crack at day 1 Figure 3.14b Crack after healing..... | 29 |
| Figure 3.16 Arduino Based resistivity monitoring and its code | 30 |
| Figure 3.18 Compression testing for sensing | 34 |
| | |
| Graph 4.1 Shows the result of self-healing GO samples..... | 37 |
| Graph 4.2 Shows the result of self-healing Bentonite samples..... | 38 |
| Graph 4.3 Shows the result of bacteria with calcium lactate samples | 40 |
| Graph 4.4 Shows the result of self-healing of simple bacteria sample | 42 |
| Graph 4.5 Shows result of self-healing bacteria with GO samples..... | 43 |
| Graph 4.6 Shows the results of self-sensing samples | 45 |
| Graph 4.7 Compression Sensing Result of GO 0.02% | 46 |
| Graph 4.8 Compression Sensing Result of GO 0.03%..... | 46 |
| Graph 4.9 Compression Sensing Result of GO 0.04%..... | 47 |
| Graph 4.10 Compression Sensing Result of GO 0.05% | 47 |
| Graph 4.11 Compression Sensing Result with no GO | 48 |
| Graph 4.12 Compression Sensing Result of GO 0.07% | 48 |

List of Tables

| | |
|--|-----------|
| Table 3: 1 Mix for GO Sample | 26 |
| Table 3: 2 Mix for Bentonite samples | 26 |
| Table 3: 3 Mix for simple bacteria sample | 27 |
| Table 3: 4 Mix for bacteria with calcium lactate sample | 27 |
| Table 3: 5 Mix for bacteria with GO sample | 27 |
| Table 3: 6 Mix for self- sensing samples | 28 |
| Table 3: 7 Mix for self-sensing compression samples | 28 |

List of Acronyms

| | |
|------|-------------------------------|
| GO: | Graphene Oxide |
| Bt: | Bentonite |
| BC: | Bacteria with Calcium Lactate |
| BS: | Simple Bacteria |
| BG: | Bacteria with Graphene Oxide |
| SSC: | Self Sensing Concrete |
| rGO: | Reduced Graphene Oxide |
| CNT: | Carbon nano tube |
| GNP: | Graphite nano platelets. |
| LWA: | Light weight aggregate. |
| PU: | Poly Urethane |

Chapter 1

1. Introduction

Concrete is the most common construction material because of its durability, strength and low cost. Low tensile strength is the main issue in concrete, which is the main reason to microcracks more likely to develop and coalesce, reducing strength and durability. These tensile stresses may result from expanding chemical reactions, plastic shrinkage, or tensile loading.[1] Aggregates, cement, such as Ordinary Portland Cement (OPC), and water are the main components of concrete. When cement powder comes into contact with water, it reacts to create a cementitious matrix that keeps the coarse and fine aggregates together and effectively transfers compressive loads to the stronger aggregates across the whole mix. Concrete is a strong and affordable building material as a result. Concrete, however, has a weak tensile and flexural capacity because of the cementitious nature of its bonding, which causes fast failure via cracking and necessitates the use of steel reinforcement (rebars) or, at the micrometer level, fiber reinforcement and/or high mix packing density. Concrete's susceptibility to cracking not only reduces its strength but also leaves it open to the effects of the environment[2]. When dangerous substances enter the concrete through these fissures, the concrete may deteriorate chemically and the steel reinforcement may corrode. The loss of strength of concrete structures is caused by this corrosion, which also causes an increase in fracture damage. The process of functioning of conductive fillers in concrete is shown in figure 1.1 below.

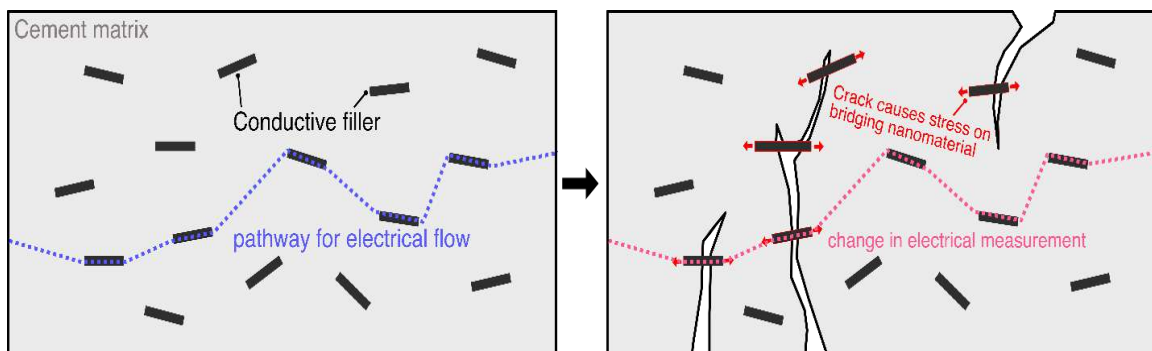


Figure 1.1 Show how conductive fillers conduct electric current

Although this raises the cost of concrete for construction and serviceability, it has long been accepted in the construction industry as an unavoidable expense. The United States spends 4 billion dollars yearly on the direct costs of maintaining concrete roadway bridges, according to a Federal roadway Administration assessment spends 45% of its yearly construction budget on maintaining its concrete infrastructure. Concrete's ability to self-heal allows for the creation of concrete with a dense microstructure while also reducing the occurrence and spread of cracks. As a result, structural concrete that is more durable and requires less upkeep can be created [1]. One of the main reasons why concrete deteriorates and becomes less durable is due to cracks. Both the plastic and the hardened phases are capable of forming cracks. In the plastic state, cracks form as a result of formwork movement, plastic settlement, and plastic shrinkage caused by the rapid loss of water from the concrete surface, whereas the hardened state is characterized by weathering, drying shrinkage, thermal stress, design and detailing errors, chemical reactions, constant overload, and external loads. Additionally, concrete structures have relatively low tensile and ductile strengths [3]. Concrete is frequently reinforced with embedded steel bars to address issues with poor tensile strength and ductility. Strengthening bars have beneficial.

1. Problem Statement

Crack monitoring and mitigation are expensive and time consuming. A bacterial GO cement /concrete that is both self-sensing and self-Healing has not been researched to another knowledge.

2. Statement of Project

A study on the self-healing and self-sensing capabilities of graphene-oxide (GO) nano-reinforced bacterial cement mortar.

3. Aims & Objectives

The aims will be undertaken by: -

- To find the optimum mix of bacteria and GO for self- sensing cement.
- To find the optimum mix of bacteria and GO for self- healing cement
- To find a mix of bacteria and GO of Self-Healing and self -sensing cement / concrete.

4. Methodology

The research methodology is outlined in Figure 1.2 as follows.

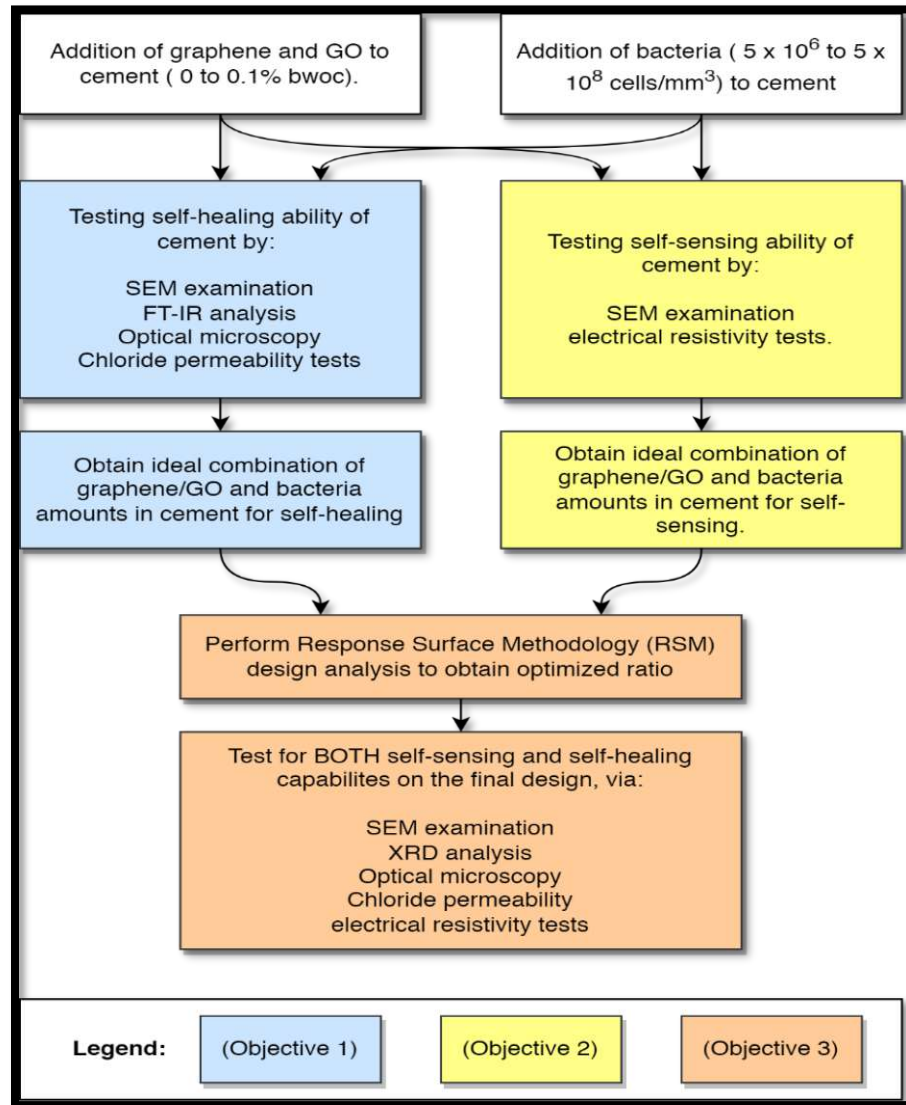


Figure 1.2 Show a Methodology of Research

5. Explanation

Crack control with bio concrete or bio-influenced self-healing concrete is becoming a practical option. Use of bio concrete or the increasingly common self-healing concrete with bio influences can be used to manage cracks. Using microbial activity within the concrete, a product known as "bio concrete" creates mineral compounds that aid in the healing of fractures. Through this process of autonomous healing, the structural durability is increased through a decrease in cracks, and the repairing needed for structures is decreased. The creation of calcium carbonate, which is influenced by a number of variables such as the concrete's pH, the amount of dissolved inorganic carbon, and nucleation sites, is directly tied to the self-healing process. Concrete's ability to cure on its own is further influenced by the type of bacteria present, their varying concentrations, the curing method, and the chemical used to incorporate the bacteria. For better activity at deep in the concrete matrix and to keep bacteria conveniently accessible, these bacteria and organic mineral precursor chemicals are blended into the concrete during the mixing process rather than applied externally.[1]. Concrete buildings could be sensed and monitored by self-sensing concrete, improving their dependability, serviceability, and reliability. Intrinsically self-sensing concrete (ISSC), which has the capacity to detect damage and stress/strain, is created by mixing functional fillers such carbon black (CB), carbon fibers (CF), graphite powder (GP), carbon nanomaterials, and others with normal concrete. Numerous research studies were conducted to assess the efficacy of SSC, including its use for traffic monitoring and corrosion monitoring.[3], strain sensing, and seismic damage monitoring. As a natural, eco-friendly technique that increases the compressive strength of broken concrete, bio mineralization is preferred. The creation of calcium carbonate, which is influenced by a number of variables such as the concrete's pH, the amount of dissolved inorganic carbon, the presence of calcium ions throughout the mixture, and nucleation sites, is directly tied to the self-healing process. Additional factors include the different bacteria., The chemical used to integrate microorganisms, the different concentrations, and curing methods all contribute to the concrete's efficient self-healing. For better activity at deep in the concrete matrix and to keep bacteria readily available, these bacteria and organic mineral precursor chemicals are blended into the concrete during the mixing process as opposed to being applied externally. It is necessary to verify the efficiency of the bacterium "Bacillus

subtilis" implanted in concrete using various incorporation strategies among the many bacteria capable of crack healing. It is also crucial to consider how these methods will affect the size of fracture healing and how much they will affect the compressive strength of concrete. Because of its potential to provide a workable solution for the structural health monitoring of concrete projects, self-sensing concrete (SSC) has attracted a lot of attention in practical applications. In-depth details on self-adhesive concrete components, dispersion methods, mix design, and recent industry breakthroughs are provided in this study document. Information and latest research discoveries on autosensing materials for smart compounds are discussed, including their characteristics, the measurement of the autosensing signal, and the behaviour of autosensing concrete under various loading conditions. The electrical resistance of self-sensing concrete is influenced by a variety of elements, such as the dry-wet cycle, the freeze-thaw cycle, the frequency of the current, etc.[3]

Chapter 2

2. Literature Review

2.1 Introduction.

This chapter offers an extensive examination of the degradation of concrete structures. It covers the utilization of nanomaterials for crack sensing and explores the phenomenon of autogenous healing in mortar, where cracks can repair themselves.

2.2 Deterioration mechanism of concrete.

Although concrete is a strong and long-lasting building material, it can deteriorate over time for a variety of reasons, including environmental exposure, chemical attack, and physical damage. Some of the processes that cause concrete to deteriorate include[4]:

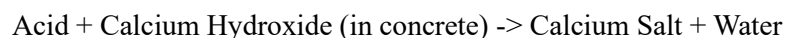
One of the most frequent reasons for concrete deterioration is corrosion of steel, which happens when chloride and sulphate ions enter the concrete and interact with the steel. The concrete structure may crack, spall, or become less sturdy as a result[5].

Freeze-Thaw Concrete can be damaged by frequent freezing and thawing cycles in colder climates. Water expands when it freezes, causing pressure that can cause cracks in the concrete and other damage.

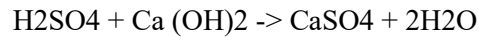
2.3 Chemical attack

Chemicals react with the components of concrete in a process known as "chemical attack," which causes the concrete to deteriorate and lose strength. Acids, alkalis, salts, and sulphates are a few of the typical compounds that can harm concrete.

Concrete is vulnerable to acid damage from substances including cleaning products, industrial chemicals, and acid rain. Concrete's calcium hydroxide dissolves when acidic solutions interact with it, leaving holes and fissures behind [4]. This may weaken the concrete's framework over time and lead it to degrade.



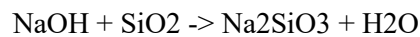
As an example, calcium hydroxide and sulfuric acid can react in concrete to produce calcium sulphate and water.



Alkali Attack: Alkalis like sodium and potassium hydroxide can attack concrete and result in a reaction known as the alkali-aggregate reaction. The expansion of the concrete's aggregates as a result of this reaction results in structural weakness and cracking.



For example, an alkali-silica gel may form when concrete containing reactive silica aggregates is exposed to an alkali, such as sodium or potassium hydroxide [6]:

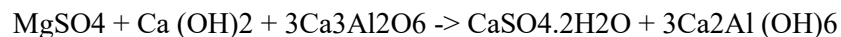


The gel may then take in water and expand, weakening and cracking the concrete as a result.

Attack by Sulphates: Sulphates can interact with calcium hydroxide and aluminium compounds in concrete, forming expansive compounds that can lead to cracking and the destruction of the structure.

Sulphate (such as magnesium sulphate), calcium oxide (found in concrete), and aluminates (found in cement) can be combined to create calcium sulphate and aluminium oxide.

When calcium hydroxide and magnesium sulphate react, for instance, aluminates in the cement cause the creation of calcium sulphate and aluminium hydroxide.



Concrete may expand as a result of the development of calcium sulphate, which can cause cracking and other damage [7].

These reactions show how chemical substances can interact with concrete's constituent parts to weaken and deteriorate the material. In order to avoid and reduce chemical attack on concrete, it is crucial to understand these responses.

2.4 Rebar corrosion

Reinforced concrete constructions' resilience and safety are significantly impacted by rebar corrosion. Steel reinforcing bars (rebars) in concrete that are exposed to corrosive substances including moisture, oxygen, and chloride ions may rust and lose their strength, causing the structure to crack, spall, or even collapse. Rebar corrosion is a complicated

process that depends on a number of variables, such as the type and consistency of the concrete[4], the environment in which the structure is placed, and the maintenance and repair procedures. The following are some of the major causes of rebar corrosion.

2.5 Moisture and Oxygen

Water and oxygen are essential components for the corrosion of rebars in concrete. When water penetrates the concrete and reaches the reinforcing steel, it forms iron oxide, commonly known as rust by reacting with iron. The rust occupies a larger volume than the original steel, causing it to expand and crack the surrounding concrete. As a result, the concrete becomes more porous and more susceptible to further corrosion.

2.6 Graphene oxide

Graphene is a 2D sheet structure comprising carbon atoms, whereas GO is an oxidized version of graphene. usually grouped in a honeycomb-like hexagonal pattern. GO is created when oxygen atoms are added to graphene sheets to form the compounds hydroxyl (C-OH), epoxy (C-OC), carbonyl (C=O), and carboxyl (O=C-OH). GO is now polar and easily dispersible in water thanks to the additional functional groups. Since cement needs to have access to water molecules in order to hydrate, GO's polarity concentrates a lot of water molecules nearby, giving cement particles "seeding" locations to adhere to. This enables a quicker and better overall hydration, which results in a denser microstructure and a cement-reinforcing effect[2]. Initially, GO was thought to be a necessary intermediary step in order to produce reduced graphene oxide (rGO), a material that resembles graphene and is employed in a variety of engineering and biological applications. Similar to CNT functionalization, exfoliation of graphite flakes is followed by oxidation with potent acids to create GO. The carbon sheet's structure is enhanced by functional groups like carboxyl (-COO), carbonyl (-C=O), epoxy (C-O-C), and hydroxyl (C-OH). It should be noted that a carbon atom is necessary to attach the carboxyl group. Figure 2.1 elaborate the biproducts form during preparation of graphene oxide.

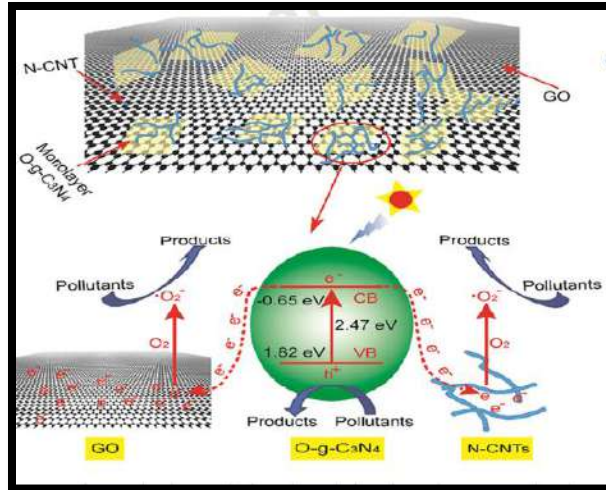


Figure 2.1 comparison of GO and CNTs transformation

atom, in an O=C-O-H configuration, both once to a hydroxyl group and again to another oxygen. As a result, the carbon has only one accessible bond, which is why the carboxyl groups are primarily found towards the borders of the GO sheets. The sheet's "wavy" appearance is caused by the hydroxyl and epoxy groups, which are present at the sheet's edges and in the basal planes (perpendicular, protruding from the sheet). The figure 2.2 show the bonding and closely packed layers of graphite.

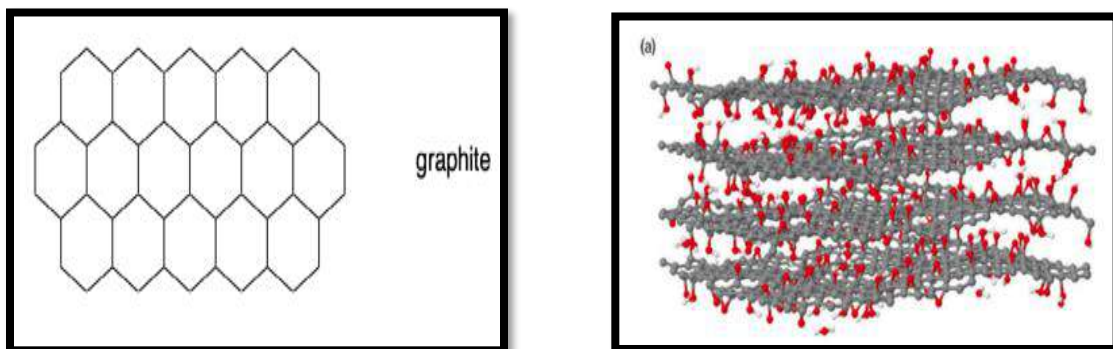


Figure 2.2 show the layer view of graphite

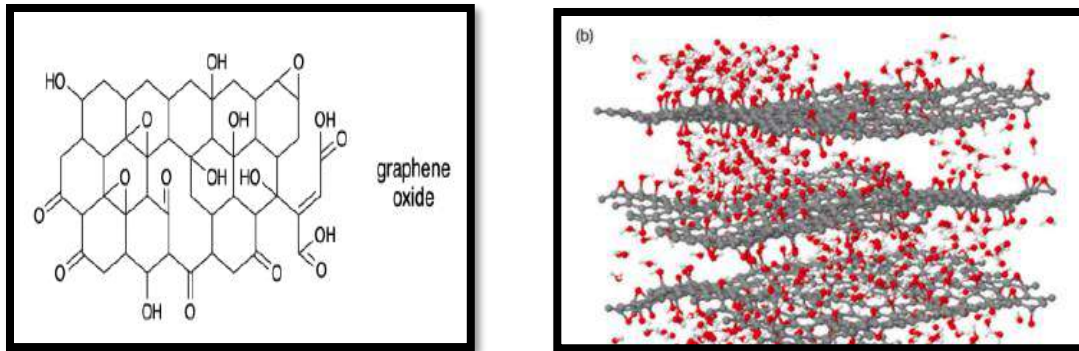


Figure 2.3 Show the layer view of graphene oxide

As a result, the carbon has only one accessible bond, which is why the carboxyl groups are primarily found towards the borders of the GO sheets. The spacing between GO layer is shown in fig 2.3. The sheet's "wavy" appearance is caused by the hydroxyl and epoxy groups, which are present at the sheet's edges and in the basal planes (perpendicular, protruding from the sheet). As previously mentioned, pristine graphene is anticipated to contain layers that are spaced roughly 0.33 nm apart [2]. However, GO can have layers separated between 0.6 and 1.2 nm because to the basal functional groups. According to Medhekar's simulations, the separation between layers can be increased to 0.51 nm with a water concentration of 0.9% and to 0.9 nm with a water level of 25.4%. The hydroxyl and other functional groups' polar nature causes polar water molecules to be drawn to them (thus the term "hydrophilicity") and combine to generate hydrogen. b) A water content of 25.4% [2]. There are 3.4 nm by 3 nm layers. Carbon atoms are grey, oxygen atoms are red, and hydrogen atoms are white. network of connections between the levels. Source: Reproduced from with permission. As a result, water molecules gather in between layers, lengthening the gap between GO layers. In contrast to parent graphene, functionalized graphene has less overall surface area, mechanical strength, thermal conductivity, and electrical conductivity. This issue can be solved by further reducing GO to rGO, where the functional groups in the basal planes are eliminated, "flattening" the sheets and producing a material similar to graphene [2]. GO nano-reinforced cementitious composites are a recent field, and without standardization and clear references, Graphite oxide, graphene oxide, and/or reduced graphene oxide have all been referred to as GO or multilayer graphene (MLG) by different researchers. [8]. The many chemical and molecular

properties of GO, such as modest variations in purity, C:O ratio, layer spacing, lateral length of GO layers, etc., must be taken into account for any variant or inquiry. GO and the number of GO layers can have a significant impact on how well it performs as a cementitious compound.

Bentonite:

For many ages, natural pozzolans have been employed in construction. Volcanic ash and heated clay have been used since 2000 BC and possibly earlier in some cultures. The majority of the pozzolan concrete buildings from the Roman, Greek, Indian, and Egyptian civilizations are still standing today, demonstrating the materials' longevity. The most notable technical advantage of adding natural pozzolans to concrete is an increase in the concrete's resistance to various forms of corrosion[9]

The chemical attack on the Mineralogical Society was primarily brought on by reduced permeability caused by a pore refining procedure.

The majority of natural pozzolans used today come from processed resources that are burnt in a kiln before being finely ground into a powder. This preparation technique is well-liked and frequently used in a variety of applications. They consist of metakaolin, calcined clay, and calcined shale.

The main component of bentonite, a naturally occurring pozzolana, is montmorillonite. But bentonite also contains other smectite-related minerals such quartz, feldspar, volcanic glass, organic material, gypsum, and pyrite. According to its chemical makeup, bentonite is a hydrous aluminium silicate that also contains trace amounts of alkali and alkaline earth metals. The Al octahedral sheet and the silica tetrahedral sheet are the two fundamental building components that make up the structure of bentonite[9].

Bentonite, which is derived from volcanic ash, is sold commercially in sodium and calcium forms. When there is water present, sodium bentonite swells and is easily absorbent. Contrarily, calcium bentonite does not expand in this manner when water is introduced. Bentonite is effective in a variety of processes[9]. As an illustration, sodium bentonite is used in drilling mud for oil and gas wells, to seal underground disposal systems and to stop metal contaminants from getting into groundwater.

Use of calcium bentonite to clean the digestive tract is common. Bentonite is also utilized in ceramic structures, cosmetics, cat litter, and adhesives. Additionally, it is used to create end plugs for pyrotechnics and to generate acne-treating face packs. Bentonite is found in the Pakistani province of Pakhtunkhwa[9]. In this research, a bentonite from Jehangira, a town in the Swabi District of Pukhtoonkhwa, was examined.

2.7 Autogenous healing

Cementitious materials that produce cracks can deteriorate more severely. However, cementitious materials can self-heal fissures through the process called autogenous healing. The French Academy of Sciences made the first observation of autogenous healing in 1836[10], which is described as the inherent mending process of fissures caused by different reaction processes in water presence[11]. Closing of fissures caused by various loose particles and water impurities are all factors that affect autogenous healing. Researchers thought that presence of water is very crucial for all components, that help the body mend itself[12]. The hydration of unhydrated cement particles was found to frequently cure cracks in young concrete, whereas CaCO₃ is essential for crack healing in older concrete. A number of steps are involved in producing calcium carbonate[13].

In the first step, atmospheric carbon dioxide disperses in water to produce carbonate and bicarbonate ions[14].



Subsequently, calcium ions are released from the concrete and react with carbonate and bicarbonate ions, giving rise to the formation of calcium carbonate. [14] (2.10 & 2.11).



Specimens are then observe by different test after crack healing to check is healing is restricted to specimens' outer surface of specimen [15].

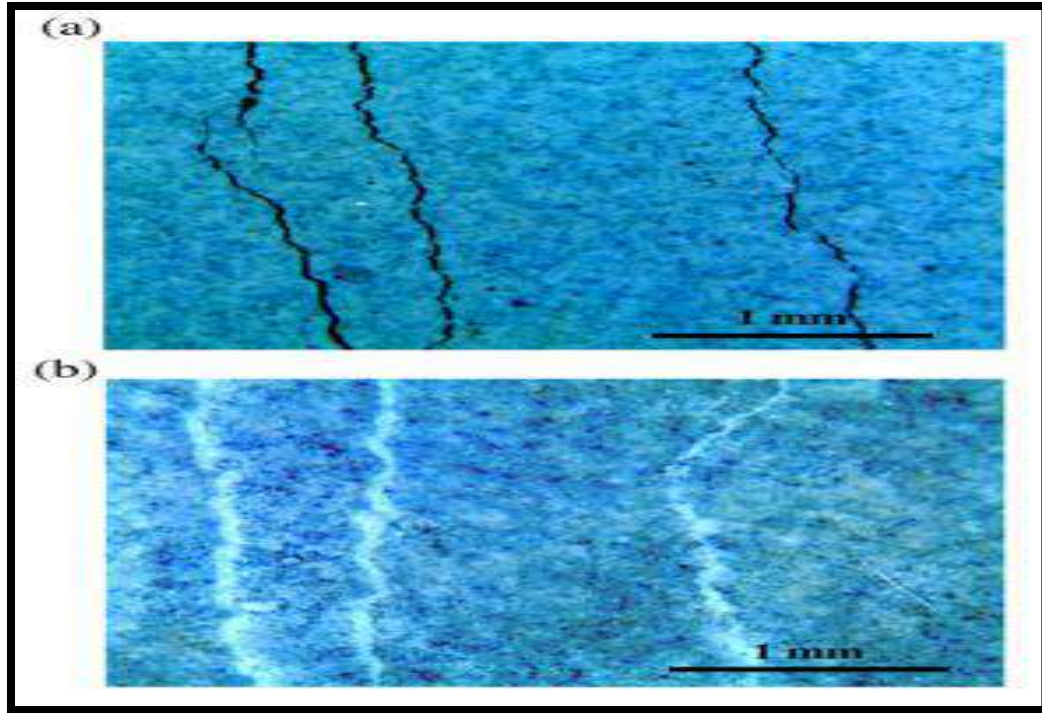


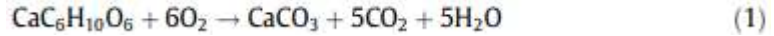
Figure 2.4 Demonstrate Autogenous healing

Figure 2.3 demonstrates a false colour micrograph of a cracked Engineered Cementitious Composite (ECC) specimen. The micrograph in (a) shows the specimen before to healing, whereas that in (b) shows the specimen following healing in CR4 (water).

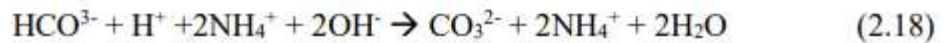
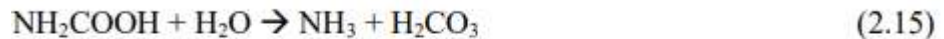
2.8 Bacteria-based self-healing concrete

The saturation of calcium ions, solution pH, the availability of the inorganic carbon that has dissolved in a solution and the existence of locations suitable for nucleation are all ideal factors that affect the precipitation of calcium carbonate in the natural environment[16]. The fourth criterion is given by the bacterial cell itself, while the first three relate to the particular matrix. Bacterial precipitation can be achieved in a variety of methods, includes by the breakdown of calcium molecules by bacteria, such as calcium lactate or urea hydrolysis[6]. Concrete cracks that allow oxygen to enter also allow microorganisms to convert calcium lactate into calcium carbonate and carbon dioxide. Any portlandite particles present will combine with the carbon dioxide to produce further calcium carbonate. which is also useful for healing[16]. Although CaCO_3 crystals are also produced in control samples, the presence of bacteria and calcium lactate might boost CaCO_3 crystal

production. Equation 1 illustrates the chemical process by which bacteria produce calcium carbonate. CaCO₃ crystals develop more frequently and have a comparable structural makeup[1].



However, compared to bacterial specimens, the mechanism of CaCO₃ crystallization in controlled specimens is quite different. The calcium hydroxide carbonation, one of the main cement products produced during hydration, is what causes CaCO₃ to develop in controlled specimens. With the use of the equation shown in [1].It describes the carbonation of calcium hydroxide.



Calcium ions are drawn into negatively charged bacterial cell walls, where they combine with carbonate ions to form calcium carbonate (eqs. 2.19 and 2.20)..[16]



The following elements are added to the cement mortar as part of the (MICP) process for cementitious compounds:

A latent gram-positive alkali-resistant endospore-forming bacteria that becomes active when it comes into touch with water.

A precursor substance[1].

A carrier substance to shield the germs from the mortar's pressure on the outside[1].

2.9 Bacteria

By watching the microbial activity that takes place during the digestion of calcium sources, researchers have been able to follow the creation of calcium carbonate in nature[17]. Concrete can go through the same process as soil thanks to these bacteria. Researchers have been examining the effectiveness of using various bacteria for the precipitation of calcite in concrete 19 specimens over the past few years. gave *Bacillus Sphaericus* to and immobilized it in microcapsules in mortar specimens..[18]. The largest crack that could heal was then measured to be 0.97 mm. A 0.3 mm crack completely healed after. utilized *Bacillus mucilaginous* in a cement paste sample. A 0.81 mm crack was reported to have repaired using *Bacillus subtilis* and graphite nanoplatelets. In cement stone specimens, the number of bacterial spores reduced from after 135 days of curing, according to a study. The decrease in bacterial spores was due to tiny cement stone holes. After 28 days of curing, the majority of pores of cement had diameters between 0.01-0.1 m. nonetheless, bacterial spores have a diameter of between 0.8 and 1 m [1]. This reduced the possibility of germs surviving because of the tremendous pressure applied to the bacterial spores. We expect concrete buildings to normally survive more than 50 years; therefore, self-healing will be successful for a longer period of time. By the use of protective medium, we can increase the self-healing bacterium media's long-term effectiveness.[1].

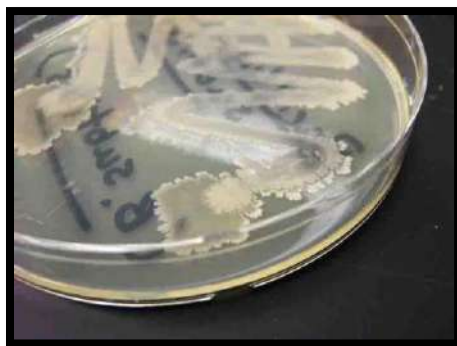


Figure 2.5 Show the culture of Bacteria

2.10 Nutrient substance: -

Nutritional substance are also necessary for the germination of spores and ongoing proliferation of bacterial cells, in addition to bacteria and transport agents. [12]. Numerous healing attempts directly incorporate these nutrients into the concrete mix. Even if these

nutrients are loaded in the carrier compound, due carrier low strength, Nevertheless, during the mixing and laying of concrete[19], they might inadvertently be dispersed in the concrete matrix. The characteristics of concrete can also be impacted by the matrix's inclusion of nutrients. examined the effects of nutrient precursors on the compressive strength of cement mortar, including calcium lactate, calcium formate, calcium nitrate, urea, and yeast extract[20]. They discovered that the addition of calcium formate and calcium lactate increased the compressive strength. The strength of the mortar specimens was unaffected by the addition of urea or calcium nitrate; however, the compressive strength was reduced by the addition of yeast extract. The amount of moisture was decreased since yeast extract contains carbohydrates that serve as retarders..[18] Researchers who studied how dietary precursors affected the hydration of cement found that calcium nitrate accelerated the process and increased the level of hydration.

How well a crack heals can also be influenced by nutrient-derived compounds. tested the precursor solution by immersing the specimens of fractured mortar..[21] investigated the effects of urea, calcium lactate, and calcium gluconate on mortar self-healing. They discovered that calcium lactate and calcium gluconate solutions enhanced self-healing for cracks larger than 0.15 mm, while soaking in urea had no effect..[22].

2.11 Carrier Compounds

There are several techniques to include bacteria into concrete, including directly incorporating bacteria into the concrete, incorporating bacteria in the form of spores because they can endure extreme environmental conditions, and immobilizing dormant bacterial spores in a carrier material and integrating them[23]. As a result of prolonged concrete hydration, Life of a bacterial cell in concrete decreases with time following direct addition as a result of the reduction in pore size, which exerts pressure on bacteria cells and spores when. put bacteria directly to the matrix.[21], they noticed that early-age fissures repaired more quickly than cracks that appeared later in life. Since the bacteria were added directly to the mixture, a poor survival rate was suggested as the cause[21].

The usage of a carrier component for keeping bacteria safe in concrete matrix is equally as important as the type of bacteria. When bacteria are introduced without a carrier component, their capacity to survive over time is significantly reduced[1]. Chemicals can

make it more likely for germs to survive. It is ideal to use a carrier compound that raises both the probability of bacterial survival and concrete's tensile strength, as inadequate tensile strength of concrete is a major contributing factor to the development of cracks in concrete. Chemicals can make it more likely for germs to survive. It is ideal to use a carrier compound that raises both the probability of bacterial survival and concrete's tensile strength, as inadequate tensile strength of concrete is a major contributing factor to the development of cracks in concrete.[1].

Concrete's flexural characteristics are affected by polyurethane (PU) and graphite nanoplatelets (GNP) carrier components. When employed as a carrier component for bacteria in self-healing concrete, light weight aggregates (LWA) offered a better cover to microorganisms but also reduced the concrete's flexural strength and increased its susceptibility to cracking. Bacteria were transported using polyurethane (PU) and silica gel, and it was shown that bacteria immobilized in polyurethane caused superior self-healing. However [16] It was discovered that the flexural strength of lightweight cement-based mortar was negatively impacted by polyurethane foam wastes (PFW). As a result, PU is not ideal for use as a carrier compound, and concrete still requires a carrier compound that increases tensile strength.[1].

2.12 Impact of environmental factor on the effectiveness of bacterial concrete.

Environmental considerations can impact how well bacterial concrete operate its basic function. Since germs require water to survive, the presence of water is crucial for the healing process. Nutrients must first be dissolved in water in order to be accessible across the breach. kept an eye on how the incubation conditions affected the speed at which cracks in bacteria-based concrete healed. Water-cured specimens showed the greatest degree of healing. While samples with less than 95% RH exhibited minimal healing [17]. However, Wang et al. [12] found a distinct set of findings. He discovered that freshwater wet-dry cycles caused specimens to heal the most effectively. We came to the conclusion that wet-dry cycles increased the amount of oxygen available to bacteria and decreased their ability to escape from the matrix along with nutritional components. By submerging concrete specimens made of bacteria in wet soil and freshwater, researchers were able to track how

well they performed. In comparison to specimens in saturated soil, those soaked in waters showed greater healing. However, bacteria-based concrete samples fared better in circumstances with saturated soil than control samples did in freshwater. The bacteria-based cement paste's self-healing mechanism depends on water, making it appropriate for usage in aquatic environments. A significant number of studies on bacteria-based concrete have only been conducted in wet environments. Relatively little study has been done on the creation and application of bacteria-based self-healing concrete in marine environments. The construction of a mortar and assessment of its performance in a submerged zone with low temperatures were the main objectives of Palin's lone inquiry into bacteria-based cementitious materials in a marine environment. An alginate-based hydrogel bead was created as a carrier medium after bacteria were found in a salinity-filled lake in northern Spain. Despite having a negative effect on the durability of concrete, magnesium acetate was utilized as a precursor to nourishment. It was shown that the permeability of 0.4 mm and 0.6 mm fractures was reduced by 95% and 93%, respectively, by adding bacteria-containing beads to the mortar. ²⁴ Although the marine environment's tidal zone has the most degradation, the usage of bacteria-based concrete there has not been studied.

Relevant Work

Numerous bacteria species have been used in concrete crack treatment over the past few years. However, it has been discovered that bacterial addition influences both the concrete's capacity for self-healing and its compressive strength. shows how different microorganisms affect the compressive strength of concrete and cement mortar. At a concentration of 7.6×10^3 cells/cm³, the results showed that using *Bacillus Pasteurii* increased concrete's 28-day compressive strength by 18%. While Ghosh and Mandal's research demonstrates that *Shewanella* results increased 25% compressive strength in 28 days at a concentration of 10^5 cells/cm³. compressive strength increases by 2% when *Escherichia coli* is present. According to Ramachandran and Ramakrishnan, this rise in compressive strength caused by *Shewanella* is bigger than the 18% increase caused by *B. Pasteurii*. *Bacillus pseudofirmus*, which Jonkers and Thiessen utilized, caused a 10% reduction in mortar strength. At the replacement level of 5, *Bacillus sphaericus* decreased

mortar's 28-day compressive strength by 35%. Scientists have been able to see the natural production of calcium carbonate because to the microbial activity that follows from the digestion of calcium sources. [18]. The same process happens in concrete when these bacteria are present. The effectiveness of employing various bacteria for the precipitation of calcite in concrete 19 specimens has been studied by researchers over the past few years. Wang et al. in samples of mortar.[18] *Bacillus Sphaericus* was injected and kept immobilized in microcapsules, and the maximum crack healing was measured at 0.97 mm. claims.[24], Less than $0.5 \times 10^3 \text{ cm}^{-3}$ of bacterial spores remained in cement stone specimens after 135 days of curing, down from $1.8 \times 10^6 \text{ cm}^{-3}$ after nine days. Soens used the microencapsulation technology to provide additional weather protection for microorganisms in concrete. All of the trials stated above used the water permeability test as a barometer for crack healing, and the micro-encapsulation strategy yielded the lowest value of water permeability. However, the polycondensation reaction-based microencapsulation technique is still quite novel and challenging. Therefore, finding a more beneficial and useful carrier technology that can be widely used in concrete practices is important. In addition to making bacteria more likely to survive, carrier chemicals can greatly impact the mechanical properties of concrete. It is preferable to use a carrier material that not only boosts the likelihood of bacterial survival but also strengthens the concrete since poor tensile strength of concrete significantly contributes to the formation of fractures in concrete. Figure 2 depicts the impact of concrete's flexural properties on light weight aggregates (LWA), polyurethane (PU), and graphite nanoplatelets (GNP) carrier compounds. Wictor and Jonker's use of light weight aggregates (LWA) as a carrier component for bacteria in self-healing concrete improved the cover offered to the bacteria while reducing the concrete's flexural strength and fracture susceptibility.[1].polyurethane (PU) and silica gel as carriers for bacteria and their results are bacteria that were immobilized in PU encouraged more self-healing. Gadea and Rodriguez found that polyurethane had a detrimental effect on the flexural strength of cement mortar when they investigated the use of polyurethane foam wastes (PFW) in the production of lightweight cement-based mortar. Because of this, using PU as a carrier compound is not recommended, and concrete still needs a carrier compound that boosts tensile strength. [1]. Sixuan looked into the application of graphite nanoplatelets (GNP) in cement-based mortar.

Chapter 3

3. Research Methodology

3.1 Materials

We use following main materials for our research

- Graphene oxide
- Bacillus Subtilis

3.1.1 Formation of Graphene oxide

- for one gram of graphene oxide. Use 120 mL of concentrated sulfuric acid (95–97%) (H_2SO_4) as shown in figure 3.1 [25].



Figure 3.1 Adding H_2SO_4 in beaker

- Then add 13.4 mL of 85% Concentrated phosphoric acid (H_3PO_4) to H_2SO_4 and allowed it to cool to 20°C as shown in figure 3.2 [25].



Figure 3.2 Adding H_3PO_4 to H_2SO_4

- Then add 1 g of graphite powder and shake mixture at 300 rpm for 10 min to allow intercalation as shown in figure 3.3 [25].



Figure 3.3 Add 1 g graphite

- Then After 10 min slowly added 6 g of solid potassium permanganate (KMnO_4) to the intercalated graphite as shown in figure 3.4 [25]



Figure 3.4 Add KMnO_4 to above solution

- Mix over a period of 5 min. During this period, keep hold ambient temperature of 20°C for 120 h[25].

- Researchers findings were keeping a low temperature generally increases GO performance[25].
- Then add 30% hydrogen peroxide (H_2O_2) dropwise after 120 hours as shown in figure 3.5 [25].

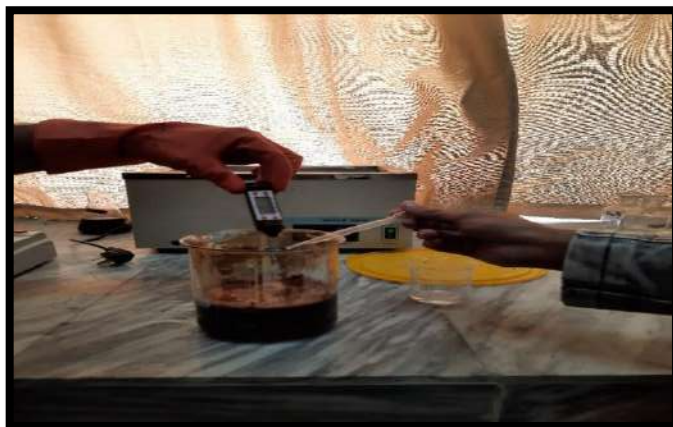


Figure 3.5 Add hydrogen peroxide

Two separate pH readings were recorded, one after 10 s of pH stabilization and another after 20 s to better observe the regenerative capacity of the GO-hydronium layer[25].

Then at last centrifuge and sonicate the above solution as shown in figure 3.6.



Figure 3.6 Sonicate the above solution

3.1.2 Extraction of Bacillus Subtilis

Perform serial dilution: - A step-by-step sequence of dilutions is referred to as a serial dilution when it is used to reduce a material's concentration in a solution to a level that is more useful. After being vigorously mixed for 15 minutes [1], one gramme of soil from the sample was vortexed in 10 ml of distilled water. From 10⁻¹ to 10⁻⁶, each suspension underwent a series of dilutions. [1] as shown in figure 3.7.

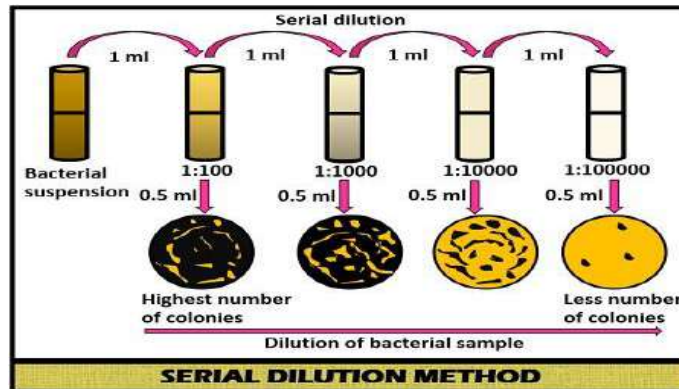


Figure 3.7 Show serial dilution method

Culture media: - Microbial culture media are made by combining nutrients to create an agar or broth that supports the growth and differentiation of microbes. [1].



Figure 3.8 Show culture media

Spread on plate: - Light the glass spreader in the form of a hockey stick over a Bunsen burner. Distribute the sample evenly over the agar's surface using the sterile glass spreader, slowly rotating the Petri dish below. [1] as shown in figure 3.9.

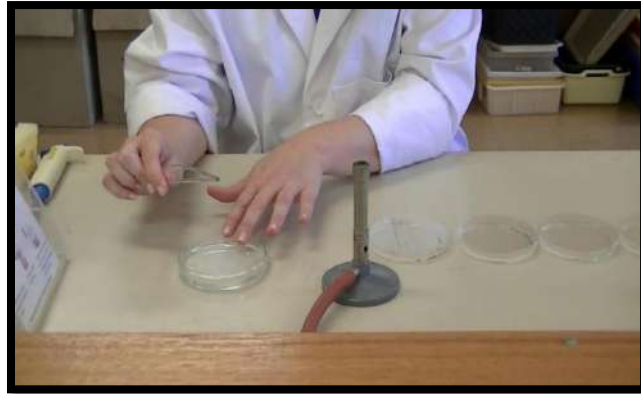


Figure 3.9 Spread bacteria to plate

Incubate the plates: - Every microbiology experiment needs the plates to be incubated in order to promote microbial growth. aerobic conditions and incubation at a temperature below that of the human body [1] as shown in figure 3.10.

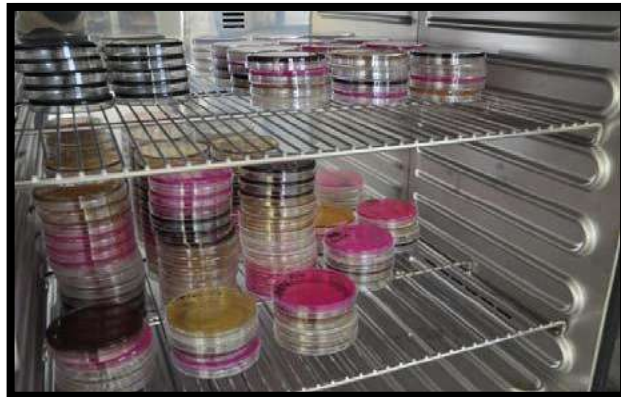


Figure 3.10 Incubation of plates

Observe the Colony: - Bacteria should be observed in a petri dish, and cultures in taped-and-sealed containers should be examined as shown in figure 3.11 [1].



Figure 3.11 Observe the colony

Gram Staining: - Gramme stain is purple in hue. When the stain and bacteria interact, the bacteria in a sample either remain purple or change to pink or red. If the bacteria remain purple, they are Gram-positive. If the bacteria turn pink or red, as in figure, they are Gram-negative. 3.12 [1].

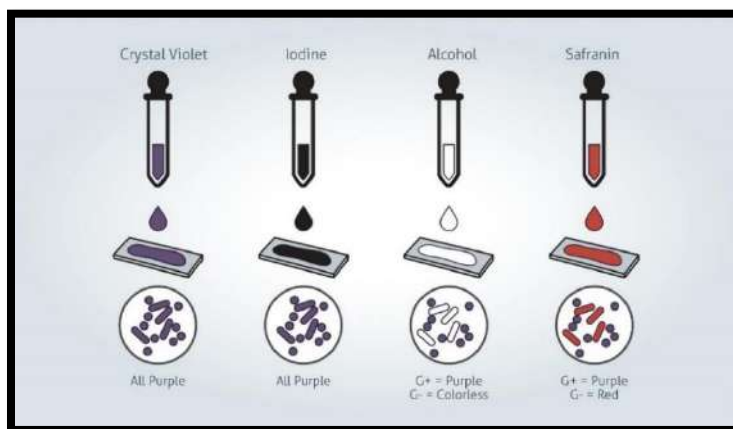


Figure 3.12 show gram staining method

3.2 Mould Selection and Mix Tables.

3.2.1 Mould Selection

For self-healing we use 3-inch diameter and 1-inch-thick moulds

For self-sensing we use 2-inch cube samples as per ASTM (C109/C109M)

3.2.2 Mix Tables

Table: 3.1 Mix for GO Sample

| SR# | Cement | sand | GO%(bwoc) | Water |
|------------|---------------|-------------|------------------|--------------|
| GO 1 | 65 | 180 | 0.04% | 26 |
| GO 2 | 65 | 180 | 0.06% | 26 |
| GO 3 | 65 | 180 | 0.08% | 26 |
| GO 4 | 65 | 180 | 0.10% | 26 |
| GO 5 | 65 | 180 | 0.15% | 26 |

Table: 3.2 Mix for Bentonite samples

| SR# | Cement | sand | Bt%(bwoc) | Water |
|------------|---------------|-------------|------------------|--------------|
| Bt 1 | 65 | 180 | 0.04% | 26 |
| Bt 2 | 65 | 180 | 0.06% | 26 |
| Bt 3 | 65 | 180 | 0.08% | 26 |
| Bt 4 | 65 | 180 | 0.10% | 26 |

Table: 3.3 Mix for simple bacteria sample

| SR# | Cement | sand | Bacteria%(bvow) | Water |
|------------|---------------|-------------|------------------------|--------------|
| BS 1 | 65 | 180 | 6.25% | 26 |
| BS 2 | 65 | 180 | 12.50% | 26 |
| BS 3 | 65 | 180 | 25.00% | 26 |

Table: 3.4 Mix for bacteria with calcium lactate sample

| SR# | Cement | sand | Bacteria%(bvow) | Water | calcium Lactate%(bwoc) |
|------------|---------------|-------------|------------------------|--------------|-----------------------------------|
| BC 1 | 65 | 180 | 6.25% | 26 | 0.5 |
| BC 2 | 65 | 180 | 12.50% | 26 | 0.5 |
| BC 3 | 65 | 180 | 25.00% | 26 | 0.5 |

Table: 3.5 Mix for bacteria with GO sample

| SR# | Cement | sand | Bacteria%(bvow) | Water | GO% |
|------------|---------------|-------------|------------------------|--------------|------------|
| BG 1 | 65 | 180 | 6.25% | 26 | 0.04% |
| BG 2 | 65 | 180 | 12.50% | 26 | 0.04% |
| BG 3 | 65 | 180 | 25.00% | 26 | 0.04% |

For self-sensing mix table: -

- For self-sensing we use 2-inch cube samples as per ASTM (C109/C109M)

Table: 3.6 Mix for self- sensing samples

| SR# | Cement | sand | GO%(bwoc) | Water |
|------------|---------------|-------------|------------------|--------------|
| GO 1 | 85 | 229 | 0.02% | 34 |
| GO 2 | 85 | 229 | 0.03% | 34 |
| GO 3 | 85 | 229 | 0.04% | 34 |
| GO 4 | 85 | 229 | 0.05% | 34 |
| GO 5 | 85 | 229 | 0.06% | 34 |
| GO 6 | 85 | 229 | 0.07% | 34 |

For self-sensing compression mix table: -

Table: 3.7 Mix for self-sensing compression samples

| SR# | No | Cement | sand | GO%(bwoc) | Water(ml) |
|------------|-----------|---------------|-------------|------------------|------------------|
| GO 1 | 6 | 500 | 1375 | 0.02% | 242 |
| GO 2 | 6 | 500 | 1375 | 0.03% | 242 |
| GO 3 | 6 | 500 | 1375 | 0.04% | 242 |
| GO 4 | 6 | 500 | 1375 | 0.05% | 242 |
| GO 5 | 6 | 500 | 1375 | 0.06% | 242 |
| GO 6 | 6 | 500 | 1375 | 0.07% | 242 |
| GO 7 | 6 | 500 | 1375 | 0.08% | 242 |

3.3 Tests

We conduct three main test to analyze our results.

- Optical microscopy test.
- Resistivity test.
- Compression test.

3.3.1 Optical microscopy test.

Objective: To measure the crack healing efficiency of sample.

1: Visual inspection: In visual inspection techniques we use optical microscope of zooming capability up to 1000x to measure the cracks width before and after the damage.



Figure 3.13 Observing samples by Optical microscope

- ▶ This allows for assessments of the closure of cracks the regeneration and overall, the restoration of the structure surface.



Figure 3.14a Crack at day 1

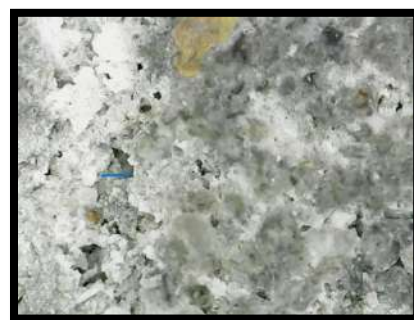


Figure 3.14b Crack after healing

3.3.2 Resistivity Test.

Objective:

To monitor the resistivity of concrete during its setting and curing process to find variation of resistivity values.

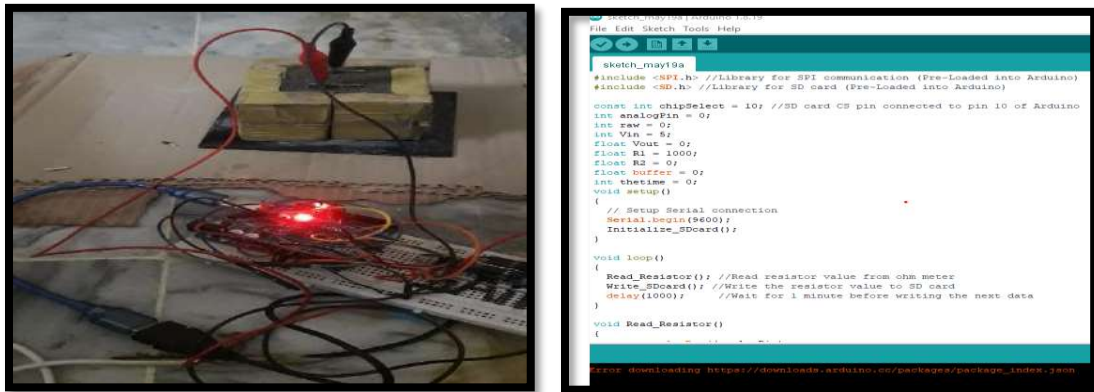


Figure 3.15 Arduino Based resistivity monitoring and its code

Apparatus:-

- Arduino Based Resistivity meter.
- Arduino Software.

Procedure:-

- First we make arduino based multimeter.
- Then we develop arduino code to upload on arduino apparatus.
- Then we upload and run code on the arduino apparatus.
- Then we connect arduino based apparatus to the two electrodes of samples.
- Then we continuously monitor the change in variation of resistivity value for 24 hrs.

Code for Arduino:-

```
#include <SPI.h> //Library for SPI communication (Pre-Loaded into Arduino)

#include <SD.h> //Library for SD card (Pre-Loaded into Arduino)

const int chipSelect = 10; //SD card CS pin connected to pin 10 of Arduino

int analogPin = 0;

int raw = 0;

int Vin = 5;

float Vout = 0;

float R1 = 1000;

float R2 = 0;

float buffer = 0;

int thetime = 0;

void setup ()

{

    // Setup Serial connection

    Serial.begin(9600);

    Initialize_SDcard ();

}

void loop ()

{

    Read_Resistor (); //Read resistor value from ohm meter
```

```
Write_SDcard (); //Write the resistor value to SD card

delay (1000); //Wait for 1 minute before writing the next data

}
```

```
void Read_Resistor ()

{

raw = analogRead(analogPin);

if (raw) {

buffer = raw * Vin;

Vout = (buffer) / 1024.0;

buffer = (Vin / Vout) - 1;

R2 = R1 * buffer;
```

```
Serial.print("t: ");

Serial.print(thetime);

Serial.print(", R2: ");

Serial.println(R2);

thetime = thetime + 1;

}

}
```

```
void Initialize_SDcard ()

{
```

```

// see if the card is present and can be initialized:

if (! SD. begin(chipSelect))

{

  Serial.println("Card failed, or not present");

  // don't do anything more:

  return;

}

// open the file. note that only one file can be open at a time,
// so, you have to close this one before opening another.

File dataFile = SD. open("LoggerCD.txt", FILE_WRITE);

// if the file is available, write to it:

if (dataFile)

{

  dataFile.println("Resistance"); //Write the first row of the text file

  dataFile.close();

}

}

void Write_SDcard ()

{

  // open the file. note that only one file can be open at a time,
  // so, you have to close this one before opening another.

  File dataFile = SD. open ("LoggerCD.txt", FILE_WRITE);

```

```
// if the file is available, write to it:

if (dataFile)

{

dataFile.print("t=");

dataFile.print(thetime);

dataFile.print(", R=");

dataFile.print(R2); //Store resistance value on SD card

dataFile.println(); //End of Row move to next row

dataFile.close(); //Close the file

}

else

Serial.println("SD card writing failed");

}
```

3.3.3 Compression test

Objective: To evaluate the functionality of embedded sensor and ensure the self-sensing capabilities of mortar Under the action of varying load.



Figure 3.16 Compression testing for sensing

Apparatus: -

- Arduino based resistivity meter
- Universal Testing Machine
- Arduino Software

Procedure: -

- First we make arduino based multimeter.
 - Then we develop arduino code to upload on arduino apparatus.
 - Then we upload and run code on the arduino apparatus.
 - Then we connect arduino based apparatus to the two electrodes of samples.
 - Then we put samples in the utm machine and connect the electrodes of sample to the two terminals of the apparatus.
 - Then we monitor the variation of resistivity value with the increase in stress value.
-

Chapter 4

4. Results

4.1 Results of self- healing

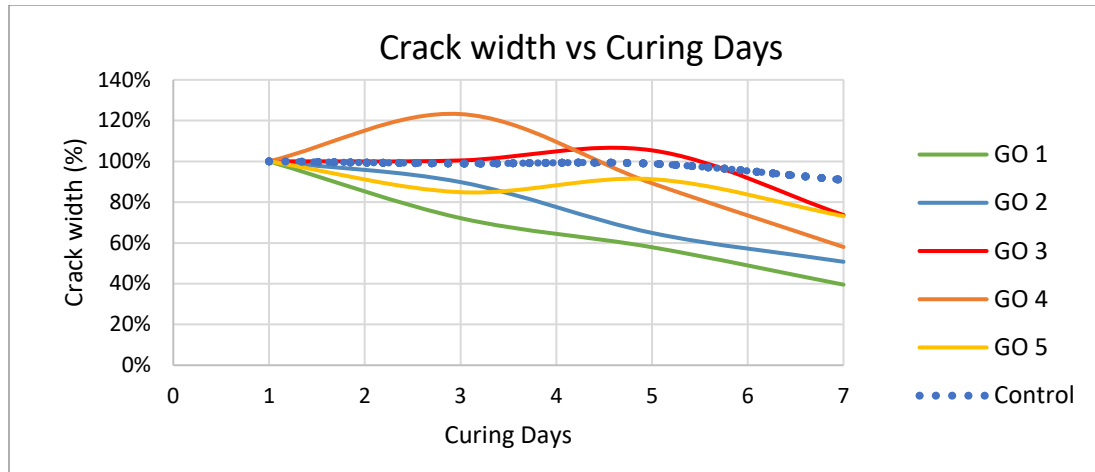
For self-healing we conduct optical microscopy test to find the healing efficiency of the samples. In this we use visual inspection techniques by the use optical microscope of zooming capability up to 1000x to measure the cracks width before and after the damage. The results of optical microscopy test are as follow.

4.1.1 Result of GO Samples

The average Crack healing % of specimens Treated with varying percentages of nano-material (graphene oxide) is given in Table 4.1. After 7 days, all specimens showed a significant improvement in healing efficiency, but sample GO 1 fared better than the other GO samples.

Table 4.1 Results of Self-healing GO samples

| GO (%) | Days | | | |
|--------|-----------------|------|------|-----|
| | 1 | 3 | 5 | 7 |
| | Crack Width (%) | | | |
| GO 1 | 100% | 72% | 58% | 40% |
| GO 2 | 100% | 90% | 65% | 51% |
| GO 3 | 100% | 100% | 105% | 74% |
| Go 4 | 100% | 123% | 89% | 58% |
| Go 5 | 100% | 85% | 91% | 73% |



Graph 4.1 Shows the result of self-healing GO samples

The graph represents the relationship between the crack width (expressed as a percentage) and the number of days for different percentages of "GO." Each GO has its own data series on the graph. The x-axis represents the number of days, ranging from 1 to 7, and the y-axis represents the crack width percentage, ranging from 0% to 100% and beyond. For each GO level, there are four data points on the graph, corresponding to the crack width percentage.

GO 1: At the beginning (1 day), the crack width is 100%. As the days progress, the crack width decreases, reaching 72% at 3 days, 58% at 5 days, and 40% at 7 days.

GO 2: At 1 day, the crack width is 100%. However, the crack width decreases at a slower rate compared to GO 1. At 3 days, it is 90%, at 5 days it is 65%, and at 7 days it is 51%.

GO 3: At 1 day, the crack width is 100%. In this case, the crack width increases from the initial value at 3 days, reaching 105%. At 7 days, it decreases to 74%.

GO 4: Similar to GO 3, the crack width starts at 100% on the first day. It experiences a more rapid increase compared to the previous two cases, reaching 123% at 3 days. However, it decreases to 89% at 5 days and further reduces to 58% at 7 days.

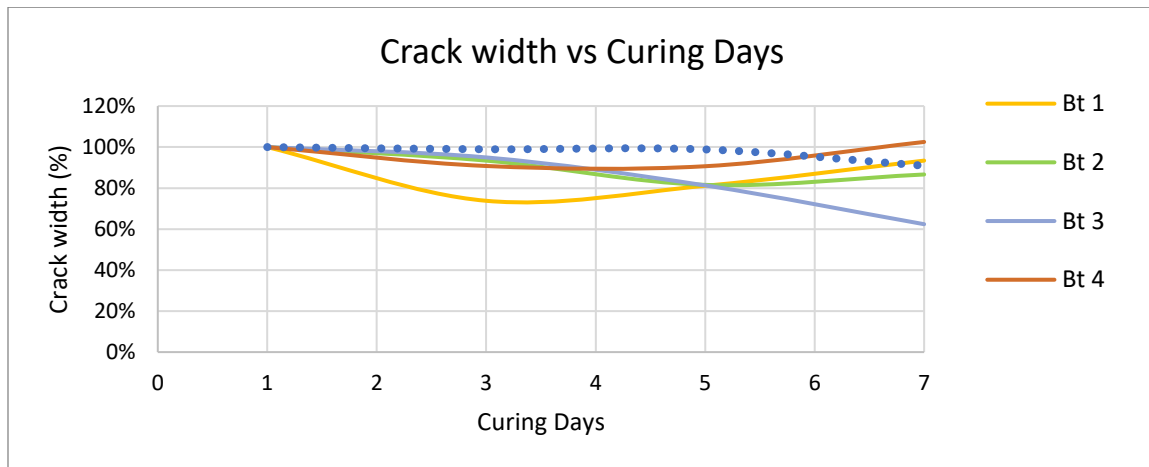
GO 5: The crack width is 100% on the first day. At 3 days, it decreases to 85%. However, it starts increasing again and reaches 91% at 5 days. Finally, it further decreases to 73% at 7 days.

4.1.2 Result of Bt Samples

The average Crack healing % of specimens Treated with varying percentages of bentonite given in Table 4.2. specimens, bc 3 demonstrated a significant healing efficiency after 7 days compared to other bc samples.

Table 4.2 Results of Self-healing Bt samples

| Bt (%) | Days | | | |
|--------|-------------|-----|-----|------|
| | 1 | 3 | 5 | 7 |
| | Crack width | | | |
| Bt 1 | 100% | 74% | 81% | 93% |
| Bt 2 | 100% | 93% | 82% | 87% |
| Bt 3 | 100% | 95% | 81% | 62% |
| Bt 4 | 100% | 91% | 91% | 103% |



Graph 4.2 Shows the result of self-healing Bentonite samples

The graph represents the relationship between the crack width (expressed as a percentage) and the number of days for different levels of a variable labeled "Bt." Each Bt level has its own data series on the graph. The x-axis represents the number of days, ranging from 1 to 7, and the y-axis represents the crack width percentage, ranging from 0% to 100% and

beyond. For each Bt level, there are four data points on the graph, corresponding to the crack width percentage.

Let's analyze the data for each Bt level:

Bt 1: At the beginning (1 day), the crack width is 100%. As the days progress, the crack width decreases, reaching 74% at 3 days, it increases to 81% at 5 days, and 93% at 7 days.

Bt 2: At 1 day, the crack width is again 100%. The crack width decreases slightly at 3 days, reaching 93%. At 5 days, it decreases further to 82%, and at 7 days, it decreases again to 87%.

Bt 3: At 1 day, the crack width is 100%. In this case, the crack width decreases from the initial value at 3 days, reaching 95%. At 5 days, it decreases further to 81%, and at 7 days, it decreases again to 62%.

Bt 4: Similar to Bt 1, the crack width is 100% on the first day. It experiences a slight decrease at 3 days, reaching 91%. However, at 5 days, it remains the same at 91%. Finally, it increases to 103% at 7 days.

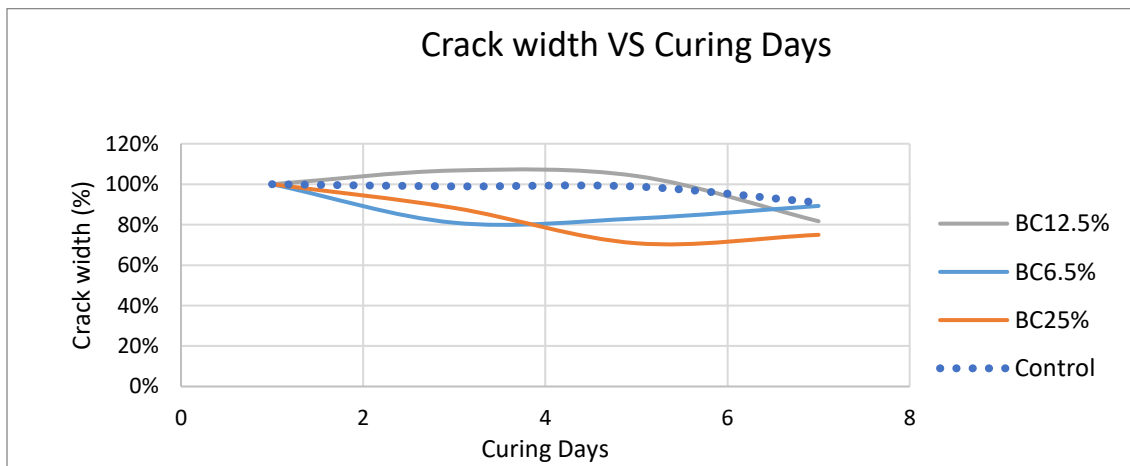
The graph demonstrates how different levels of the Bt affect the crack width over time. It shows the variation in crack width for each Bt level, providing insights into the behavior and trend of crack development under different conditions.

4.1.3 Results of Bacteria with calcium lactate

The average Crack healing % of specimens Treated with varying percentages of bacteria with calcium lactate is given in Table 4.3. specimens, bc 3 demonstrated a significant healing efficiency after 7 days compared to other bc samples.

Table 4.3 Results of self-healing Bacteria with calcium lactate samples

| SR.NO | Days | | | |
|-------|-------------|------|------|-----|
| | 1 | 3 | 5 | 7 |
| | Crack width | | | |
| BC 1 | 100% | 81% | 83% | 79% |
| BC 2 | 100% | 107% | 104% | 82% |
| BC 3 | 100% | 88% | 71% | 75% |



Graph 4.3 Shows the result of bacteria with calcium lactate samples

The graph represents the relationship between the crack width (expressed as a percentage) and the number of days for different levels of a variable labeled "BC." Each BC level has its own data series on the graph. The x-axis represents the number of days, ranging from 1 to 7, and the y-axis represents the crack width percentage, ranging from 0% to 100% and beyond. For each BC level, there are four data points on the graph, corresponding to the crack width percentage.

Let's analyze the data for each BC level:

BC 1: At the beginning (1 day), the crack width is 100%. As the days progress, the crack width decreases slightly, reaching 81% at 3 days, 83% at 5 days, and 79% at 7 days.

BC 2: At 1 day, the crack width is 100%. The crack width increases at 3 days, reaching 107%. However, it starts to decrease afterward, reaching 104% at 5 days and further reducing to 82% at 7 days.

BC 3: At 1 day, the crack width is 100%. In this case, the crack width decreases from the initial value at 3 days, reaching 88%. At 5 days, it further decreases to 71%, and at 7 days, it slightly increases to 75%.

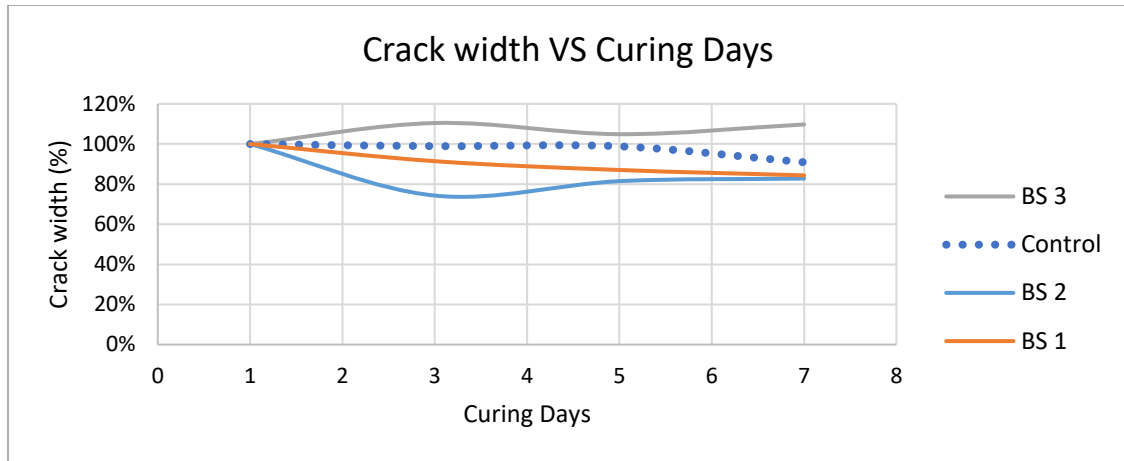
The graph demonstrates how different levels of the BC affect the crack width over time. It shows the variation in crack width for each BC level, providing insights into the behavior and trend of crack development under different conditions.

4.1.4 Results of Simple Bacteria

The average Crack healing % of specimens Treated with varying percentages of Simple bacteria given in Table 4.4. specimens, BS 2 demonstrated a significant healing efficiency after 7 days compared to other bc samples.

Table 4.4 Results of self-healing simple bacteria sample

| SR.NO | Days | | | |
|-------|-------------|------|------|------|
| | 1 | 3 | 5 | 7 |
| | Crack width | | | |
| BS 1 | 100% | 91% | 87% | 84% |
| BS 2 | 100% | 74% | 81% | 83% |
| BS 3 | 100% | 110% | 105% | 110% |



Graph 4.4 Shows the result of self-healing of simple bacteria sample

The graph represents the relationship between the crack width (expressed as a percentage) and the number of days for different levels of a variable labeled "BS." Each BS level has its own data series on the graph. The x-axis represents the number of days, ranging from 1 to 7, and the y-axis represents the crack width percentage, ranging from 0% to 100% and beyond. For each BS level, there are four data points on the graph, corresponding to the crack width percentage.

Let's analyze the data for each BS level:

BS 1: At the beginning (1 day), the crack width is 100%. As the days progress, the crack width decreases, reaching 91% at 3 days, 87% at 5 days, and 84% at 7 days.

BS 2: At 1 day, the crack width is again 100%. The crack width decreases at 3 days, reaching 74%. However, it starts to increase afterward, reaching 81% at 5 days and further increasing to 83% at 7 days.

BS 3: At 1 day, the crack width is still 100%. In this case, the crack width increases from the initial value at 3 days, reaching 110%. At 5 days, it remains at a high level of 105%, and at 7 days, it continues to stay at 110%.

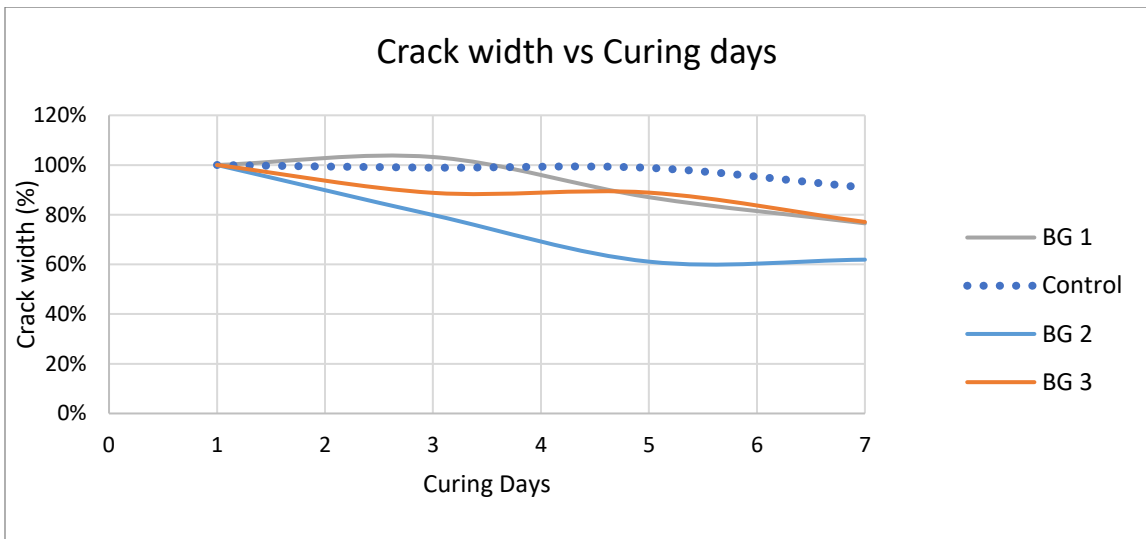
The graph demonstrates how different levels of the variable BS affect the crack width over time. It shows the variation in crack width for each BS level, providing insights into the behavior and trend of crack development under different conditions.

4.1.5 Results of Bacteria with GO

The average Crack healing % of specimens treated with varying percentages of bacteria with GO is given in Table 4.5. specimens, BG 2 demonstrated a healing efficiency 62% after 7 days which is much significant as compared to other BG samples.

Table 4.5 Results of self-healing of Bacteria with GO samples

| Sr. No | Days | | | |
|--------|-------------|------|-----|-----|
| | 1% | 3% | 5% | 7% |
| | Crack width | | | |
| BG 1 | 100% | 103% | 87% | 77% |
| BG 2 | 100% | 80% | 61% | 62% |
| BG 3 | 100% | 89% | 89% | 77% |



Graph 4.5 Shows result of self-healing bacteria with GO samples

The graph represents the relationship between the crack width (expressed as a percentage) and the number of days for different levels of a variable labeled "BG." Each BG level has its own data series on the graph. The x-axis represents the number of days, ranging from 1 to 7, and the y-axis represents the crack width percentage, ranging from 0% to 100% and

beyond. For each BG level, there are four data points on the graph, corresponding to the crack width percentage.

Let's analyze the data for each BG level:

BG 1: At the beginning (1 day), the crack width is 100%. As the days progress, the crack width increases slightly, reaching 103% at 3 days. However, it starts to decrease afterward, reaching 87% at 5 days and further reducing to 77% at 7 days.

BG 2: At 1 day, the crack width is 100%. The crack width decreases at 3 days, reaching 80%. It continues to decrease at 5 days, reaching 61%, and remains relatively stable at 62% at 7 days.

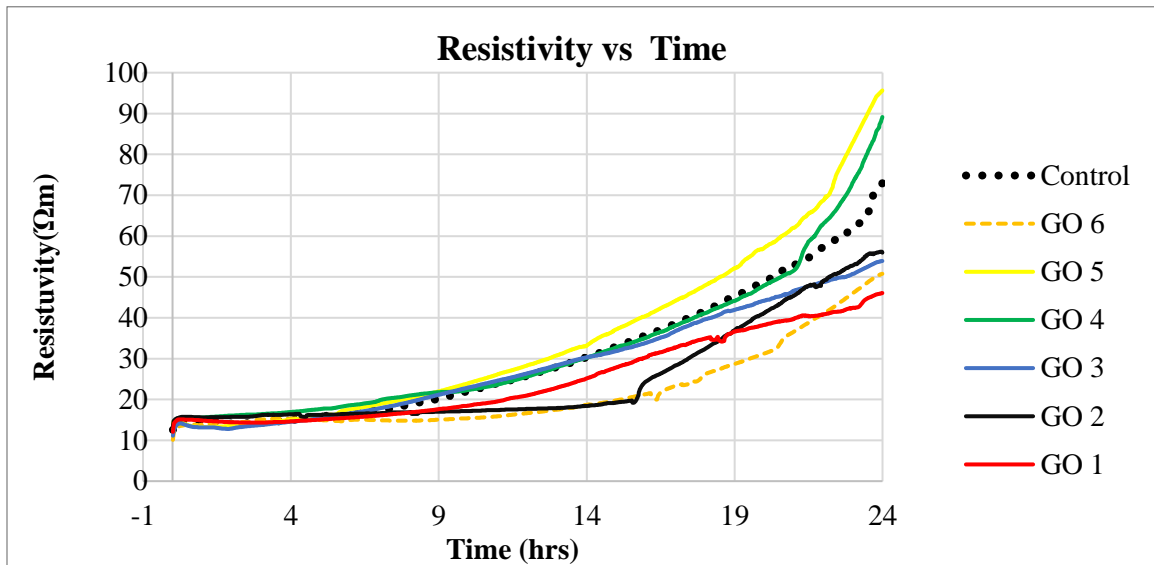
BG 3: At 1 day, the crack width is 100%. In this case, the crack width decreases slightly from the initial value at 3 days, reaching 89%. It remains constant at 89% at 5 days and decreases slightly to 77% at 7 days.

The graph demonstrates how different levels of the variable BG affect the crack width over time. It shows the variation in crack width for each BG level, providing insights into the behavior and trend of crack development under different conditions.

4.2 Self-Sensing Results

For Self-sensing we conduct electric resistivity test for this we check the resistivity of concrete during its setting and curing process to find variation of resistivity values. The test results are given below for different samples Results are shown in graph 4.6.

Results of resistivity test:



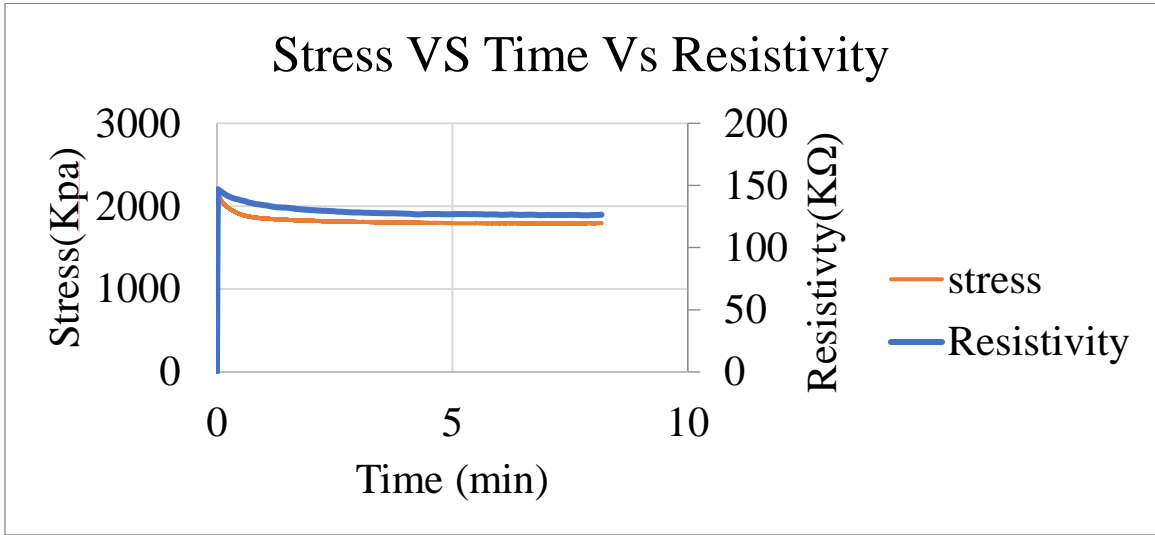
Graph 4.6 Shows the results of self-sensing samples

The graph represents the relationship between the resistivity (expressed as a percentage) and the time for different levels of a variable labeled "GO." Each Go level has its own data series on the graph. The x-axis represents the time in (hrs), ranging, and the y-axis represents the resistivity value. For each GO level, there is a variation in resistivity value at different time interval.

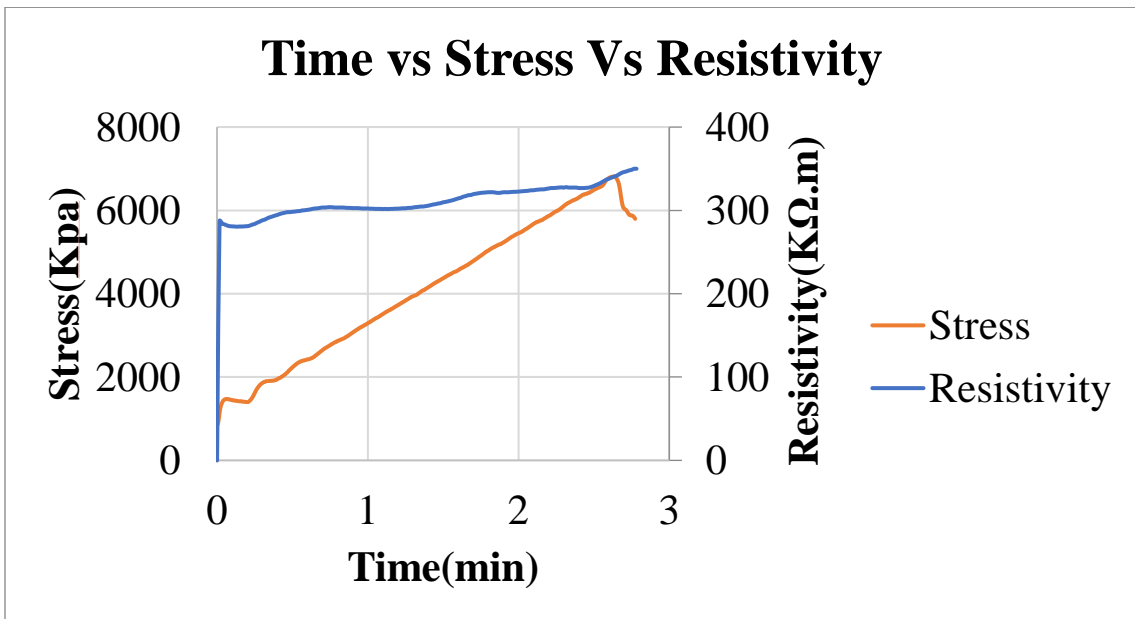
Let's analyze the data for each GO level:

The above graphical line of GO 2 and GO3 show a stable change and at the end of 24 hrs its resistivity value is as compared to control. The line of GO 2 show a stable value at starts its resistivity value drop after 14 hrs and then after that it increase its value again and at end of 24 hrs its resistivity value is as compared to control. The line of GO 4 and GO 5 increase its resistivity value instantly and at the end of 24hrs its resistivity value is high as compared to control. The line of GO 6 increase its resistivity value gradually with time and at end of 24hrs its value is less as compared to other except GO 2 resistivity value.

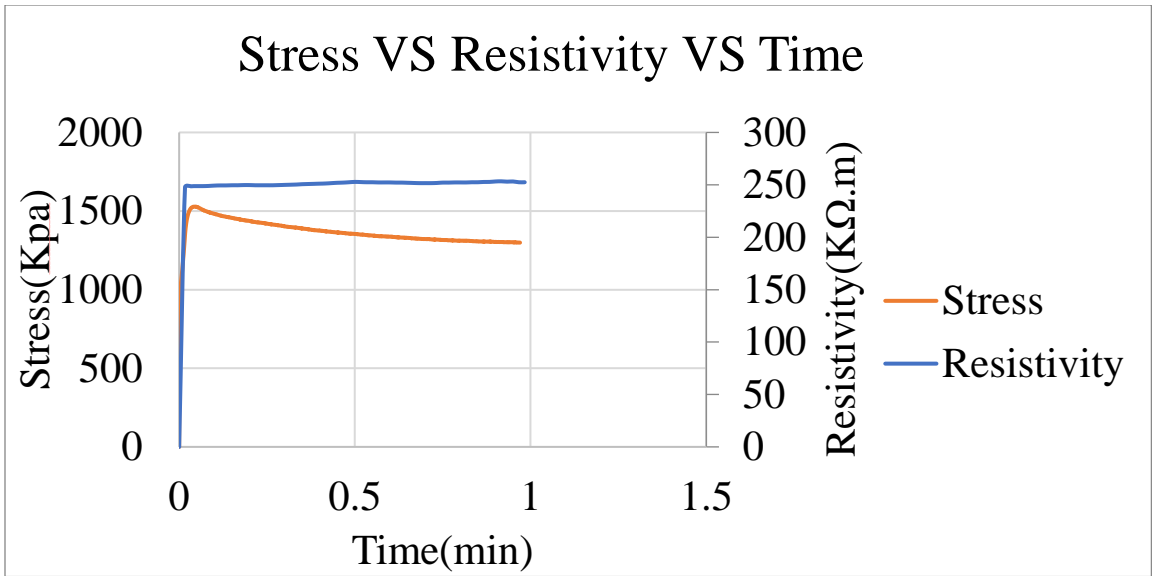
Results of Compression Sensing test:



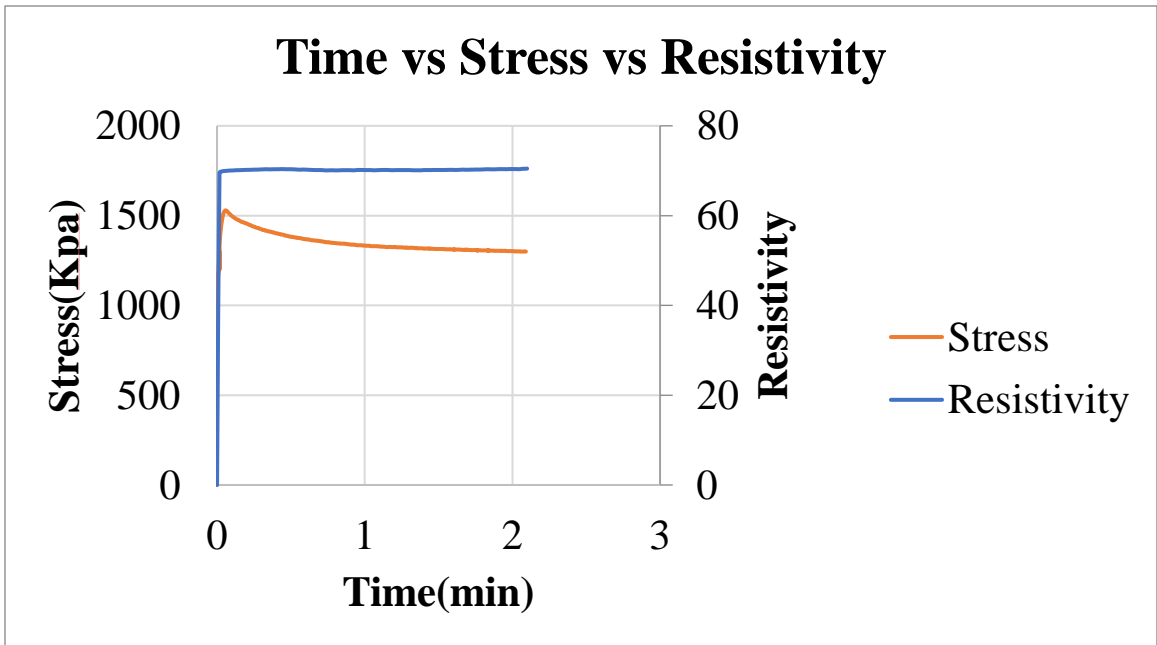
Graph 4.7 Compression Sensing Result of GO 0.02%



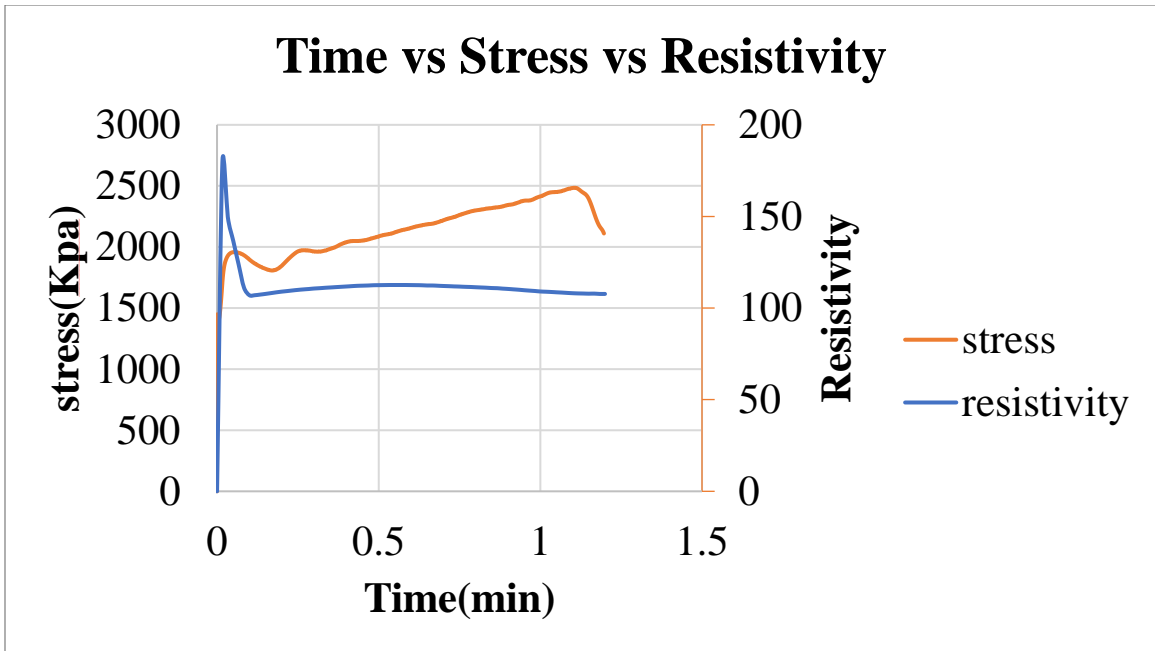
Graph 4.8 Compression Sensing Result of GO 0.03%



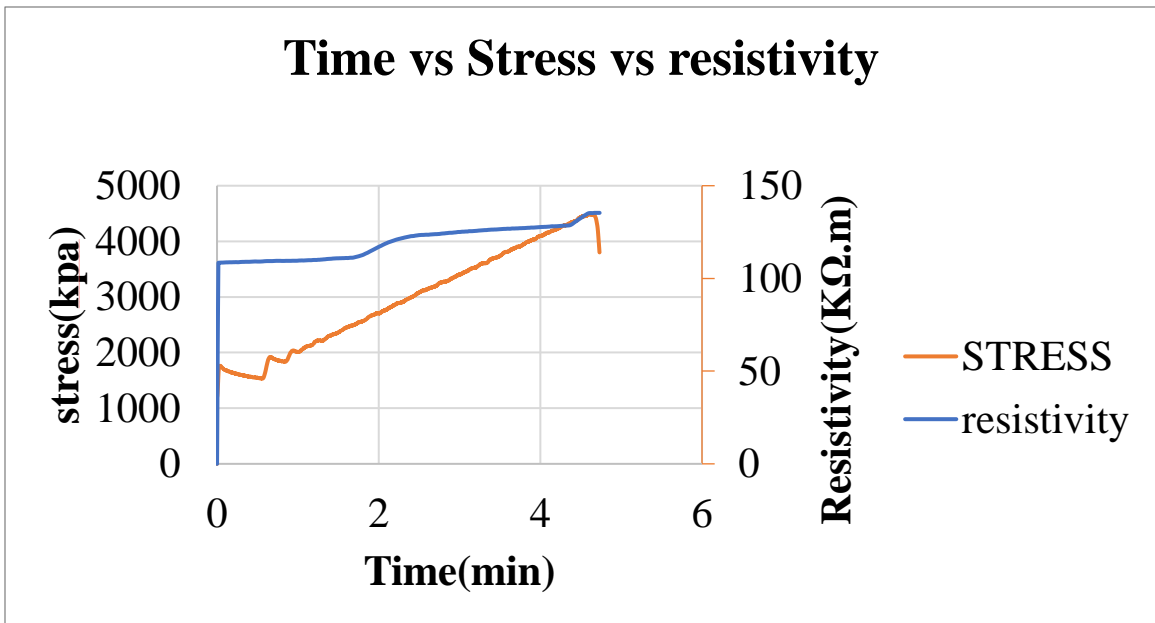
Graph 4.9 Compression Sensing Result of GO 0.04%



Graph 4.10 Compression Sensing Result of GO 0.05%



Graph 4.11 Compression Sensing Result with no GO



Graph 4.12 Compression Sensing Result of GO 0.07%

Chapter 5

5. Conclusion and Recommendation

5.1 Conclusion: -

- 0.04% GO addition by weight of cement fully healed cracks up to 0.50mm in 7 days.
- The nanomaterial exhibited best self-healing properties, significantly higher than bacterial concrete.
- However, GO capabilities as conductive filler is poor, and limited self-sensing abilities were observed.

5.2 Recommendation

- Standard measurement for bacterial concentration should be implemented for accurate comparisons between research literature.
 - A combination of conductive filler and graphene oxide should be tested for self-sensing and self-healing abilities
-

References:

- [1] W. Khaliq and M. B. Ehsan, “Crack healing in concrete using various bio influenced self-healing techniques,” *Constr. Build. Mater.*, vol. 102, pp. 349–357, Jan. 2016, doi: 10.1016/j.conbuildmat.2015.11.006.
- [2] T. M. Sheikh, M. P. Anwar, K. Muthoosamy, J. Jaganathan, A. Chan, and A. A. Mohamed, “The mechanics of carbon-based nanomaterials as cement reinforcement — A critical review,” *Construction and Building Materials*, vol. 303. Elsevier Ltd, Oct. 11, 2021. doi: 10.1016/j.conbuildmat.2021.124441.
- [3] Z. Bekzhanova, S. A. Memon, and J. R. Kim, “Self-sensing cementitious composites: Review and perspective,” *Nanomaterials*, vol. 11, no. 9, 2021, doi: 10.3390/nano11092355.
- [4] P.K. Mehta, “Concrete in the Marine Environment,” *Eng. technology*, 1991.
- [5] M. Santhanam and M. Otieno, “Deterioration of concrete in the marine environment,” *Mar. Concr. Struct.*, vol. 1, pp. 137–149, 2016, doi: 10.1016/b978-0-08-100081-6.00005-2.
- [6] Y. Yang, E. H. Yang, and V. C. Li, “Autogenous healing of engineered cementitious composites at early age,” *Cem. Concr. Res.*, vol. 41, no. 2, pp. 176–183, 2011, doi: 10.1016/j.cemconres.2010.11.002.
- [7] N. Hearn, “Self-sealing, autogenous healing and continued hydration: What is the difference?,” *Mater. Struct. Constr.*, vol. 31, no. 8, pp. 563–567, 1998, doi: 10.1007/bf02481539.
- [8] T. M. Sheikh, M. P. Anwar, K. Muthoosamy, J. Jaganathan, A. Chan,

- and A. A. Mohamed, “Graphene oxide’s regenerative acidity and its effects on the hydration of Type II Portland Cement,” *Constr. Build. Mater.*, vol. 364, Jan. 2023, doi: 10.1016/j.conbuildmat.2022.129933.
- [9] S. Ahmad, S. A. Barbhuiya, A. Elahi, and J. Iqbal, “Effect of Pakistani bentonite on properties of mortar and concrete,” *Clay Miner.*, vol. 46, no. 1, pp. 85–92, 2011, doi: 10.1180/claymin.2011.046.1.85.
- [10] N. De Belie *et al.*, “A Review of Self-Healing Concrete for Damage Management of Structures,” *Advanced Materials Interfaces*, vol. 5, no. 17. Wiley-VCH Verlag, Sep. 07, 2018. doi: 10.1002/admi.201800074.
- [11] K. Vijay, M. Murmu, and S. V. Deo, “Bacteria based self healing concrete – A review,” *Constr. Build. Mater.*, vol. 152, pp. 1008–1014, 2017, doi: 10.1016/j.conbuildmat.2017.07.040.
- [12] K. Vijay, M. Murmu, K. Vijay, and M. Murmu, “Effect of calcium lactate and *Bacillus subtilis* bacteria on properties of concrete and self-healing of cracks,” 2020.
- [13] M. S. S. Zabanoot, “Review of autogenous and autonomous self-healing concrete technologies for marine environments,” *WIT Trans. Built Environ.*, vol. 196, pp. 31–38, 2020, doi: 10.2495/HPSM200041.
- [14] Mohd. Warid Hussin and Muhd Zaimi Abd. Majid and Rosli Mohamad Zin, “A Review of Self-healing Concrete Research Development”.
- [15] A. R. Suleiman and M. L. Nehdi, “Effect of environmental exposure on autogenous self-healing of cracked cement-based materials,” *Cem. Concr. Res.*, vol. 111, pp. 197–208, Sep. 2018, doi:

10.1016/J.CEMCONRES.2018.05.009.

- [16] S. Gupta, S. D. Pang, and H. W. Kua, “Autonomous healing in concrete by bio-based healing agents – A review,” *Construction and Building Materials*, vol. 146. Elsevier Ltd, pp. 419–428, Aug. 15, 2017. doi: 10.1016/j.conbuildmat.2017.04.111.
- [17] M. Seifan, A. K. Samani, and A. Berenjian, “Bioconcrete: next generation of self-healing concrete,” *Applied Microbiology and Biotechnology*, vol. 100, no. 6. Springer Verlag, pp. 2591–2602, Mar. 01, 2016. doi: 10.1007/s00253-016-7316-z.
- [18] J. Y. Wang, H. Soens, W. Verstraete, and N. De Belie, “Self-healing concrete by use of microencapsulated bacterial spores,” *Cem. Concr. Res.*, vol. 56, pp. 139–152, 2014, doi: 10.1016/j.cemconres.2013.11.009.
- [19] I. Sandalci, M. M. Tezer, and Z. Basaran Bundur, “Immobilization of Bacterial Cells on Natural Minerals for Self-Healing Cement-Based Materials,” *Front. Built Environ.*, vol. 7, no. April, pp. 1–15, 2021, doi: 10.3389/fbuil.2021.655935.
- [20] R. Maddalena, H. Taha, and D. Gardner, “Self-healing potential of supplementary cementitious materials in cement mortars: Sorptivity and pore structure,” *Dev. Built Environ.*, vol. 6, no. January, p. 100044, 2021, doi: 10.1016/j.dibe.2021.100044.
- [21] M. Luo, C. X. Qian, and R. Y. Li, “Factors affecting crack repairing capacity of bacteria-based self-healing concrete,” *Constr. Build. Mater.*, vol. 87, pp. 1–7, 2015, doi: 10.1016/j.conbuildmat.2015.03.117.

- [22] J. Ducasse-Lapeyrusse, R. Gagné, C. Lors, and D. Damidot, “Effect of calcium gluconate, calcium lactate, and urea on the kinetics of self-healing in mortars,” *Constr. Build. Mater.*, vol. 157, pp. 489–497, 2017, doi: 10.1016/j.conbuildmat.2017.09.115.
- [23] J. Feng, B. Chen, W. Sun, and Y. Wang, “Microbial induced calcium carbonate precipitation study using *Bacillus subtilis* with application to self-healing concrete preparation and characterization,” *Constr. Build. Mater.*, vol. 280, p. 122460, 2021, doi: 10.1016/j.conbuildmat.2021.122460.
- [24] H. M. Jonkers, A. Thijssen, G. Muyzer, O. Copuroglu, and E. Schlangen, “Application of bacteria as self-healing agent for the development of sustainable concrete,” *Ecol. Eng.*, vol. 36, no. 2, pp. 230–235, 2010, doi: 10.1016/j.ecoleng.2008.12.036.
- [25] B. Thomas *et al.*, “Pr ep rin er r Pr ep ot pe er ed,” vol. 10, no. 4, pp. 212–217, 2019.
-