

AN EXPERIMENTAL STUDY ON GRAPHITE/GRAPHENE NANO PARTICLES IN REINFORCED CONCRETE



Final Year Project UG 2017

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PARTICLES IN REINFORCED CONCRETE”**

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in

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In the name of Allah, the most Beneficent, the most Merciful as well as peace and blessings be upon Prophet Muhammad, His servant and final messenger.

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Last but not the least, we are grateful to the National University of Sciences and Technology (NUST) for providing us an environment to achieve what we have always dreamt of and enabling us to play our part for the betterment of humanity.

DEDICATED

TO

Our supervisor Dr. Musaad Zaheer Nazir Khan

and our parents.

ABSTRACT

In the recent years, due to increase in population, construction demand has increased and the trend is still rising. Nearly 10% of all carbon emissions come from construction industry. Concrete is a fundamental material used in almost every type of construction, but one of its main constituents, “cement” due to its intensive manufacturing process causes increase in temperature. Due to concerns of high rising temperature the research community started searching for novel methods to substitute the cement with “greener” additive whilst at the same time improving the intrinsic properties of concrete. A novel and truly revolutionary method of enhancing the performance of concrete, thus allowing for decreased consumption of raw materials, lies in nano-engineering the cement crystals responsible for the development of all mechanical properties of concrete in this study graphene nano particles were added in the conventional concrete.

Graphene is the most promising nanomaterial for composites' reinforcement to this date, due to it's exceptional strength and toughness and its unique ability to retain original shape after strain, water impermeability properties and thereby enhance the ductility and fracture toughness of the resultant matrix. In this study, 28 days cured GNPs nano-reinforced concrete specimens were tested to check the compressive, split tensile and flexure strength under strain controlled Universal Testing Machine (UTM). Some non destructive tests were also performed to check the quality of GNPs reinforced concrete. Test results reveal that the strength, ductility, and fracture toughness can be improved with the addition of GNPs. A scanning electron microscope (SEM) is used to verify the involved strengthening mechanisms encompassing crack bridging and crack branching effects of inducted GNPs.

In general, all experimental results show a consistent improvement in concrete's performance when enhanced with graphene. The nanomaterial improves the mechanical interlocking of cement crystal, thus strengthening the internal bonds of the composite matrix.

TABLE OF CONTENTS

LIST OF FIGURES	i
LIST OF TABLES	ii
LIST OF ACRONYMS	iii
CHAPTER 1	1
INTRODUCTION	1
1.1 General.....	1
1.2 Graphene Reinforcement	1
1.3 Environmental Impact.....	1
1.4 Goals and Objectives	2
1.6 Thesis Overview	3
CHAPTER 2	4
LITERATURE REVIEW	4
2.1 Graphene.....	4
2.2 Preparation Routes	5
2.2.1 Mechanical Exfoliation.....	5
2.2.2 Chemical Vapor Deposition (CVD).....	5
2.2.3 Liquid Phase Exfoliation.....	6
2.2.4 Electrochemical Exfoliation.....	7
2.2.5 Chemical reduction of graphene oxide	8
2.2.6 Thermal Shock Exfoliation	8
2.2.6 Bottom-up synthesis.....	8
2.3 Analysis.....	9
CHAPTER 3	10
MATERIAL AND EXPERIMENTAL METHODOLOGY	10
3.1 Materials	10
3.1.1 Graphene Nano platelets (GNPs).....	10

3.1.2 Cement	12
3.1.3 Sand.....	13
3.1.3 Coarse Aggregate.....	14
3.2 Experimental Methodology	15
3.2.1 Mix Design.....	15
3.2.2 Mixing Regime	16
3.2.3 Dispersion Checks for GNPs	16
3.2.2 Curing	18
CHAPTER 4	19
EXPERIMENTAL TESTS AND RESULTS	19
4.1 Compressive Test.....	19
4.2 Split Tensile Test	21
4.3 Flexure Test	23
4.4 Elastic Modulus Test.....	24
4.5 Electrical Resistivity Test	26
Sample.....	27
Electrical Resistivity	27
4.6 Pulse Velocity Test	28
4.7 Rebound Hammer Test	29
4.8 Scanning Electron Microscopy of the Concrete	31
CHAPTER 5	33
CONCLUSIONS.....	33
RECOMMENDATIONS	34
CONSTRAINTS	35
ANNEXURE-A	36
ANNEXURE-B.....	37
REFERENCES	38

LIST OF FIGURES

Figure 1: shows a schematic which shows how graphite is separated into graphene sheets.....	5
Figure 2: shows the comparison of methods for graphene production.....	9
Figure 3: shows ultrasonication bath being used for dispersion of GNPs.	10
Figure 4: shows the SEM image of GNP flakes.	11
Figure 5: shows the XRD analysis of GNP flakes.	11
Figure 6: shows Raman shift of GNP flakes.....	12
Figure 7: shows the gradation curve of sand.	13
Figure 8: shows the gradation curve of coarse aggregate.	14
Figure 9: shows settlement of GNPs with time.	17
Figure 10: shows relative absorption value at different concentrations.	17
Figure 11: shows the compressive test results over a period of 28 days	20
Figure 12: shows sample after split tensile test.	21
Figure 13: shows the split tensile test results.....	22
Figure 14: shows flexure testing assembly with beam specimen.	23
Figure 15: shows flexural test strength results at the end of 28 day period.....	24
Figure 16: shows stress-strain curves of various GNP concentrations along with control specimen.	25
Figure 17: shows stress strain curve of optimum and control sample	26
Figure 18: shows electrical resistivity test apparatus and specimen.....	27
Figure 19: shows pulse velocity test apparatus.....	28
Figure 20: shows the standard ranges of pulse velocity test.....	29
Figure 21: shows the rebound hammer.....	30
Figure 22: shows the rebound hammer graph for strength estimation.....	30
Figure 23: shows the SEM image of control sample.	31
Figure 24: shows the SEM image of 0.8 g/L concrete sample.	31

LIST OF TABLES

Table 1: shows the properties of cement.....	12
Table 2: shows the properties of sand.....	13
Table 3: shows the properties of coarse aggregate.	14
Table 4: shows the details of casting.	15
Table 5: shows the detail of concrete formulations.	15
Table 6: shows the compressive strengths of different concrete formulations.....	19
Table 7: shows split tensile strengths of different concrete formulations.	21
Table 8: shows flexural strength of different concrete formulations.	23
Table 9: shows the elastic modulus values of different concrete formulations.	24
Table 10: shows the toughness values of different concrete samples.....	25
Table 11: shows the electrical resistivity values of different concrete samples.	27
Table 12: shows standard resistivity level compared to possible corrosion rate.....	27
Table 13: shows the pulse velocity values of different concrete samples.....	29
Table 14: shows the compressive strength of different concrete samples through rebound hammer test.....	30
Table 15: shows sieve analysis of fine aggregate.	36
Table 16: shows sieve analysis of coarse aggregate 1.	36
Table 17: shows sieve analysis of coarse aggregate 2.	37
Table 18: shows sieve analysis of coarse aggregate 3.	37

LIST OF ACRONYMS

C-S-H	Calcium Silicate Hydrate
CNTs	Carbon Nanotubes
GO	Graphene Oxide
rGO	Reduced Graphene Oxide
CVD	Chemical Vapor Deposition
GNPs	Graphene Nanoplatelets
SEM	Scanning Electron Microscope
OPC	Ordinary Portland Cement
w/c	Water to Cement Ratio
ACI	American Concrete Institute
XRD	X-Ray Diffraction
ASTM	American Society for Testing and Materials

INTRODUCTION

1.1 General

In recent years, due to increase in population, the demand for construction material has increased in construction industry. Due to this much production of concrete the carbon dioxide emission has increased which is impacting the climate negatively. Cement is the cause for 7% of the global CO₂ emissions [1]. Therefore, it is essential to decrease the amount of concrete used per m³ while maintaining and improving its mechanical and other performance properties through various reinforcements.

1.2 Graphene Reinforcement

Graphene is one of the allotropes of carbon and it is an abundant material. It consists of single atomic layer of graphite. It is in form of hexagonal lattice which consists of tightly bonded carbon atoms. Interaction of graphene with concrete takes place at nanoscale. Graphene changes the morphology of hydration crystal and it forms vast elements of CSH groups after interacting with concrete. Due to large surface area and high surface energy of graphene, CSH particles make bonding with graphene which and it acts as nucleation site which promotes the growth of denser CSH gel along the graphene flakes. This process results in increased bonding strength of cement with higher degree of crystallinity in CSH gel as compared to standard concrete, therefore increasing the overall strength of concrete. Moreover, existing research shows that graphene reinforced concrete has high thermal and electrical conductivities, low weight and offers better resistant to water infiltration. It has been found that graphene reinforced also increases post crack behavior.

1.3 Environmental Impact

Cement production accounts major contribution towards global CO₂ production. Introducing graphene in concrete decreases the amount of concrete required to fulfill

the specification of building loadings by 50%. This would result in significant reduction of 446 kg per ton of the carbon emissions due to cement manufacturing. [2]

1.4 Goals and Objectives

Our objective is using Graphene Nano Platelets (GNPs) from a highly scalable and novel method, then to study the effect of Graphene as a reinforcement on the physical, mechanical and microstructural properties of concrete. Our goal is to show how this novel, nanoengineered concrete can benefit from reduced amount of cement while keeping it's mechanical (and other properties) performance to a very high level.

1.5 Problem Statement

Previous studies were focused on incorporation of nanomaterials such as carbon nanotube and graphene oxide in cement based composites which resulted in 50% and 33% improvement in compressive strength for CNT and GO respectively, while industrial thin graphite platelets (100nm thickness) improved thermal conductivity. However, these findings can not be directly applied to concrete as addition of aggregates i.e. sand and coarse aggregate would alter the physio-mechanical behavior of the material.

Up until now the effect of atomically thin materials on nanoengineering of concrete is yet to be discovered because of the conservative nature of the industry. Due to huge gap between academia and industry, the exploitation of nanotechnology in concrete on a commercial scale remains quite limited. This is due to the very expensive nature of nanomaterials and the complicated multiple-step procedure of it's production. However, this ensures more sustainable urbanization which will result in much lower carbon footprint. It also ensures more resilient constructions against natural disasters.

In the following decade, the scientific community developed various alternatives to mechanical separation for synthesizing graphene sheets (layers) such as Chemical Vapor Deposition, Chemical derivation from graphene oxide and liquid exfoliation in water. The method of chemical exfoliation includes hazardous use of reducing agents that are both dangerous to both humans and the environment. Yet there is no greener,

effective and economical study conducted for the production of soluble defect free graphene and thus to make a “Graphene Reinforced Concrete”.

1.6 Thesis Overview

Followed by introduction, a detailed literature review has been provided in Chapter 2 pertaining to structure of graphene followed by a discussion of exfoliation methods.

Chapter 3 explains the experimental methodology, material characterization utilized in the project and testing procedures acquired for this research study comprising of details of experiments performed. Mix design and casting regime have also been discussed in this chapter.

Chapter 4 carried the results of tests and their interpretative explanations for exfoliation of graphene using a method and graphene reinforced concrete.

Chapter 5 discusses the conclusions based on findings of this research and future recommendations.

LITERATURE REVIEW

2.1 Graphene

Graphene is one of the allotropes of carbon and it is an abundant material. It consists of single atomic layer of graphite. It is in form of hexagonal lattice which consists of tightly bonded carbon atoms. Graphene is found in graphite and in 2004, for the first time, Novoselov and Geim introduced the idea behind decomposing bulk graphite into thin graphene layers by using scotch tape exfoliation method, which includes that after multiple repeated steps, one can reduce the structure of graphite to graphene flakes. The schematic in fig. 1 illustrates this method. Graphite comprises of hundreds of layers of carbon atoms arranged in hexagonal, honeycomb lattice, which are vertically stacked. The layers in graphite can be easily separated due to weak Van der Waal forces holding carbon atoms in Z plane. Graphene is single layer of carbon atoms which are held together by strong covalent bonds. Graphene is a strongest material discovered; it has Young's modulus of 1 TPa and it is 100-300 times stronger than steel and has tensile strength of 130 GPa, which are the most important properties for applications with composites.

The two-dimensional structure endows graphene with built-in advantages over more widely used carbon allotropes such as carbon nanotubes (CNTs) and graphite. The two-dimensional structure provides more specific surface area to interact with matrix material, whether the interaction require transfer of electrons or mechanical stresses. Such properties are very engaging for the application of graphene to use it as a reinforcement in matrix.

These characteristics of graphene are due to 2p orbitals which form π state delocalized bands over the sheet of carbon atoms which constitute graphene. Hence, graphene is extremely high stiffness, has very high thermal conductivity, it show zero permeability towards gases, shows zero effective mass, shows high mobility of charge carries. All these extraordinary properties give graphene a big advantage over other materials which are used in making different composites.

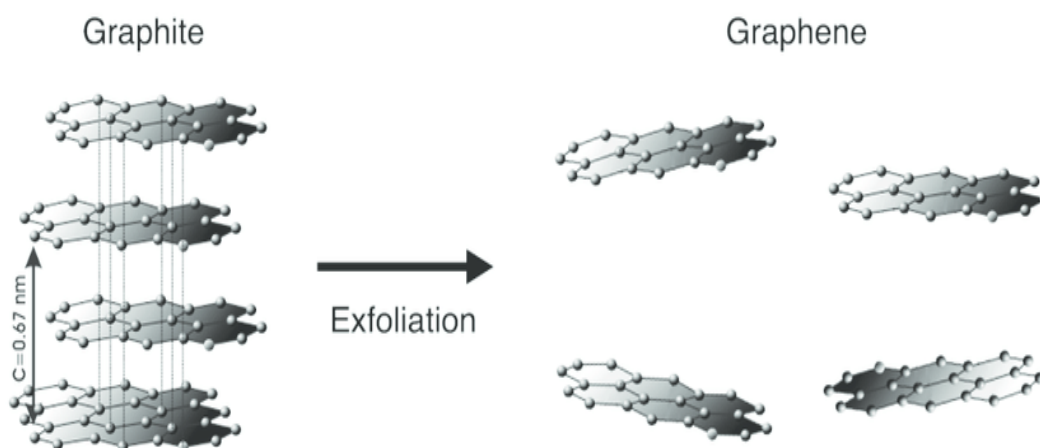


Figure 1: shows a schematic which shows how graphite is separated into graphene sheets.

2.2 Preparation Routes

2.2.1 Mechanical Exfoliation

It is the simplest among the methods of preparation and Geim and Novoselov was awarded the Nobel Prize in 2010 for this method. In this method, repeated tape exfoliation of graphite is done and then it is transferred to a substrate. Methods like optical microscope, scanning tunneling microscopy, atomic force microscopy and Raman spectroscopy, are used to determine the number of layers in graphene flakes. This method makes highest quality crystals, but it can only be used for prototyping and lab scale experiments as large scale production is not possible.

2.2.2 Chemical Vapor Deposition (CVD)

CVD is one of the most useful methods to prepare high structural quality monolayer graphene. A metal is exposed to different hydrocarbon precursors at high temperatures to produce large area samples. Plasma-enhanced CVD, hot/cold wall CVD thermal CVD, and many others are different types of methods available. The exact mechanism of formation of graphene depends upon substrate growth. However, it usually begins with carbon atoms growth which nucleate on the metal after hydrocarbons decomposition and then nuclei grow into larger domains. In addition to gaseous hydrocarbons i.e. ethylene, methane or acetylene, liquid precursors like pentane or

hexane have also been used while the quite diverse set of materials like food, insects and waste can also be used for the CVD production of graphene. Difficulty is faced while transferring graphene from the growth substrate to a substrate of interest because of the chemically inert nature of graphene and it can cause defects and wrinkles in the material, while the stability of grown material can also be affected by thermal fluctuations. Moreover, due to complex nature of the CVD process and the high energy demands for the specific method, the task becomes more difficult.

2.2.3 Liquid Phase Exfoliation

Liquid Phase Exfoliation is another widely used method for the production of graphene. It involves two methods of exfoliation i.e. ultrasonication and high shear blending. It consists of three different steps; 1st dispersion in a solvent or surfactant, 2nd exfoliation and 3rd purification in order to separate the non-exfoliated material from the exfoliated material and if it is to be supplied as powder, complete removal of any solvent traces. By longer sonication period, higher concentrations of graphene can be attained at the expense of energy consumption. After sonication the material obtained consists of thicker flakes. Ultracentrifugation is done to remove these thicker flakes. Higher centrifugation speeds produce thinner flakes with a small lateral size, which are not so useful for composites. Dispersion of graphene can be done in a variety of liquids including aqueous surfactants. In both cases the surfactant bridges the hydrophobic carbon atoms with water molecules, allowing the graphene sheets to oat freely without restacking. The output of the process is estimated from the yield by single-layer graphene percentage. The yield is defined as the ratio of the number of single layer flakes to the total number of graphitic flakes in the dispersion. By altering the sonication time, the starting amount of graphite and the rotational speed of the centrifuge, the yield can be altered. Electrical properties is one of the problems related with the specific process. These properties (electrical) can be similar to electrical properties of graphene oxide as a result of poor transport at contacts between the graphene sheets. In addition to this, the cost is increased due to the use of highly reactive solvents. The process itself is potentially not ecologically-friendly as due to very low solubility of graphene, large amount of solvent is used. Paton et al. showed that high shear forces can be used instead of ultrasonication and thus exfoliated graphene on a 100 liter scale. 10^4 s^{-1} was found to be the critical shear rate for exfoliation of graphene which can be achieved even from

a conventional kitchen blender. The mean number of layers was found to be less than 10 after centrifugation and the typical sizes of the nanosheets in lateral direction were in the range of 300–800 nm. However, it is to be noted that relatively low yield was obtained and exfoliation efficiency can be highly affected by choice of starting material and the rotor optimization. Recently, Dimiev et al. prepared graphene nanoplatelets over the time period of 3-4 hours at room temperature and almost 100% conversion yield from graphite to graphene was obtained. The liquid exfoliation method turns out to be particularly useful for applications in composite materials, due to its scalability (more than 100L/h) (**more than 100L/h**) and yield of high-quality graphene. This method of extraction of graphene in water does not introduce any defects to the sheets, which preserves their intrinsic properties.

2.2.4 Electrochemical Exfoliation

In this method a liquid solution i.e. electrolyte is used and electric current is used to consume electrode which is made up of graphite. In this process oxidation takes place at anode and reduction takes place at cathode of the graphite-based electrode. In order to prepare high quality graphene consisting of few layers for use in optical and energy applications, cathodic reaction methods are comparatively more suitable. But in literature anodic oxidation is used more extensively. The resulting material formed on anode has resemblance with graphene oxide as they have similar oxidation states and it consists of multiple layers of graphene. The advantage of this process lies in its simplicity i.e. it takes place in a single step and also it is less time consuming. The lateral size of flakes is an important criterion to be taken into account. The initial graphite material used, and the conditions of exfoliation affect the lateral size of flakes. This process is eco-friendly due to use of surfactant in aqueous form and electrolyte in liquid form. This method is also used to make a product which resembles graphene oxide when LiClO_4 is used as electrolyte. In this method there is potential of producing graphene at larger scale. However, there are some drawbacks i.e. the electrolytes used can be expensive and graphene produced has crushed morphology which put a limit to its uses.

2.2.5 Chemical reduction of graphene oxide

In this method graphene oxide in the form of single layered sheets are formed when graphene oxide is subjected to exfoliation. Then graphene resembling monolayers are produced after reduction (in-situ) of graphene oxide. The choice of surfactant, reducing agent and solvent is made carefully as these materials effect the process. The material produced is not of good quality because of many defects in its structure such as oxygen and hydrogen contaminating groups are attached and also structure is not uniform as it has deformations and irregularities. So, we cannot be sure if this material should be categorized as graphene or not. Strong reducing agent is required to remove oxygen group from graphene oxide. Also, reduction is incomplete because of which the product formed is termed as reduced graphene oxide.

2.2.6 Thermal Shock Exfoliation

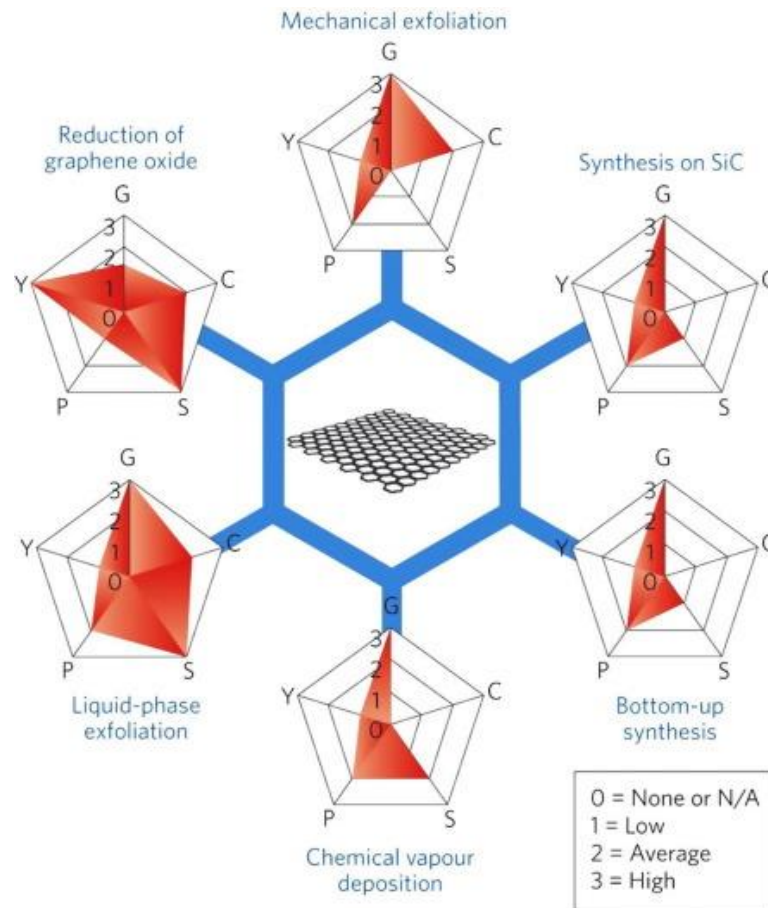
In this method, graphene is synthesized via rapid high temperature heating. In this process, high temperature up to 3000K is applied for milliseconds to just seconds. This is followed by rapid cooling. Thermal shock exfoliation utilizes high temperatures at non-equilibrium and extreme conditions to drive the reactions. The reaction rate is dependent upon the temperature; more high temperature results in high reaction rates. This method produces high rates of graphene manufacturing with little defects.

2.2.6 Bottom-up synthesis

In this process the preparation of graphene molecules starts from small building blocks or elements which are accurate at atomic level. In this process sites of coupling are required on these building blocks which can be energized externally to congregate next structural unit. This process requires high temperature. In this process structural alternatives are formed and after this some of these structural variants or alternatives are discretized to produce the required structure. The main advantage of this process is that it produces high quality graphene. A disadvantage or drawback of this method is restrictions in transferring and handling of the product. Also, the possibility of producing graphene with this method on larger scale is less.

2.3 Analysis

In the following figure all these methods are compared with respect to their yield, quality, scalability, purity and cost of production.



G: graphene quality
 C: cost of production (a low value corresponds to high cost of production)
 S: scalability
 P: purity
 Y: yield of each preparation method

Figure 2: shows the comparison of methods for graphene production.

MATERIAL AND EXPERIMENTAL METHODOLOGY

3.1 Materials

Materials used in manufacturing of graphene reinforced concrete as well as their characterization is mentioned in the upcoming section followed by a detailed experimental methodology.

3.1.1 Graphene Nano platelets (GNPs)

Graphene nano platelets were procured from SCME Nano synthesis lab which was later sonicated in an ultrasonication bath for 2 hours.



Figure 3: shows ultrasonication bath being used for dispersion of GNPs.

Then to investigate the microstructure of GNPs, we performed Scanning Electron Microscopy (SEM) and got the following results as shown in Fig. 4. The image shows that GNPs are 2 dimensional flakes in the given solution and it has size <100 nm.

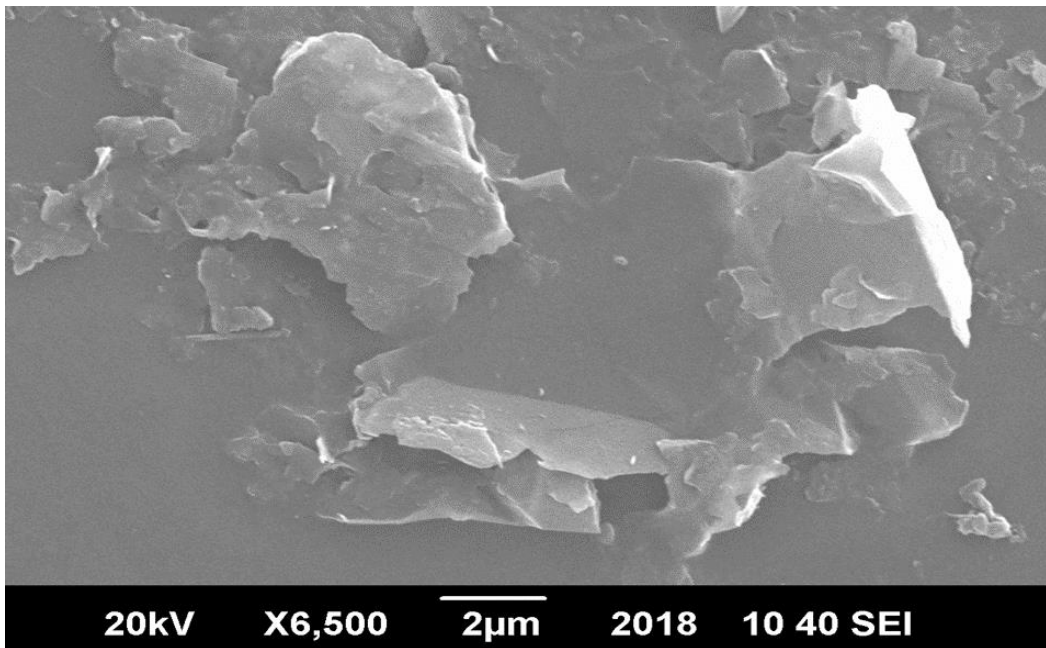


Figure 4: shows the SEM image of GNP flakes.

X-Ray Diffraction (XRD) was performed for the GNPs. The peaks show that it consists of multiple layers of graphene.

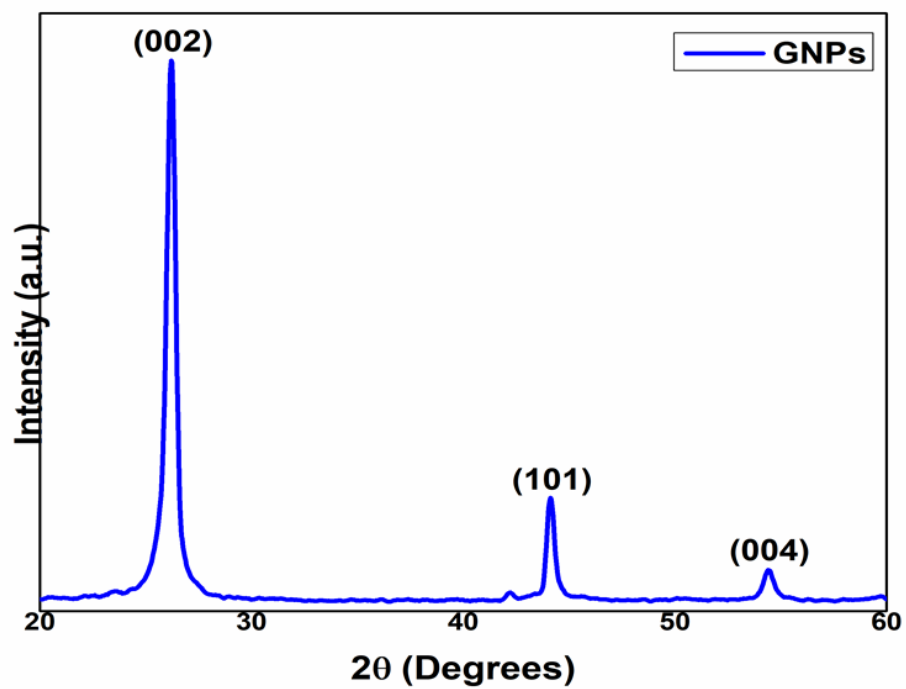


Figure 5: shows the XRD analysis of GNP flakes.

Raman spectroscopy was performed to assess the number of layers in GNPs and level of defects. The results show that thickness of GNP flakes is about 100 nm consisting of multiple layers of graphene and watching well with literature.

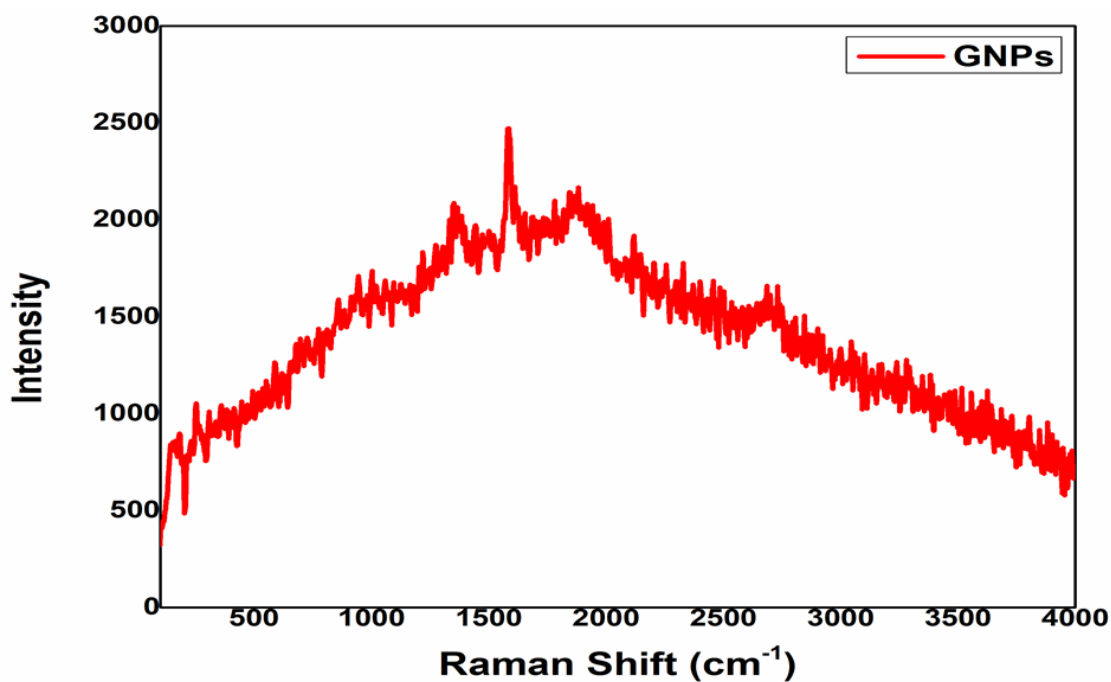


Figure 6: shows Raman shift of GNP flakes.

3.1.2 Cement

Bestway grade cement, free from moisture was used in our research project. The table below shows the properties of cement.

Properties	Values
Initial Setting Time	110 min
Final Setting Time	160 min
Specific Gravity	3.15
Median Size (D50) (μm)	9.5

Table 1: shows the properties of cement.

3.1.3 Sand

Lawrencepur sand was used in our research work having a fineness modulus of 2.71. which lies within limits prescribed by ASTM i.e. 2.2 to 3.2. Fig. 7 shows the gradation curve obtained after performing sieve analysis according to ASTM C33 and tab. 2 shows the properties of sand.

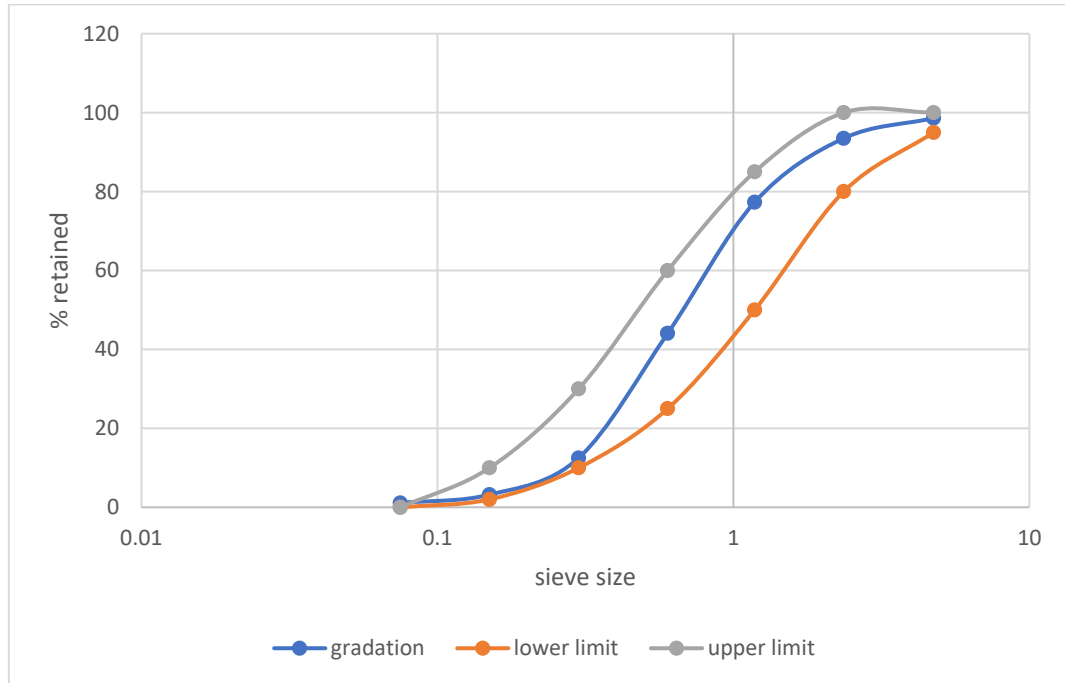


Figure 7: shows the gradation curve of sand.

Properties	Values
Specific Gravity	2.67
Water Absorption	1.2%
Fineness Modulus	2.71

Table 2: shows the properties of sand.

3.1.3 Coarse Aggregate

Margalla crush was obtained from I-12 batching plant. The figure and table below show the gradation curve obtained after performing sieve analysis according to ASTM C33 and properties of coarse aggregate, respectively.

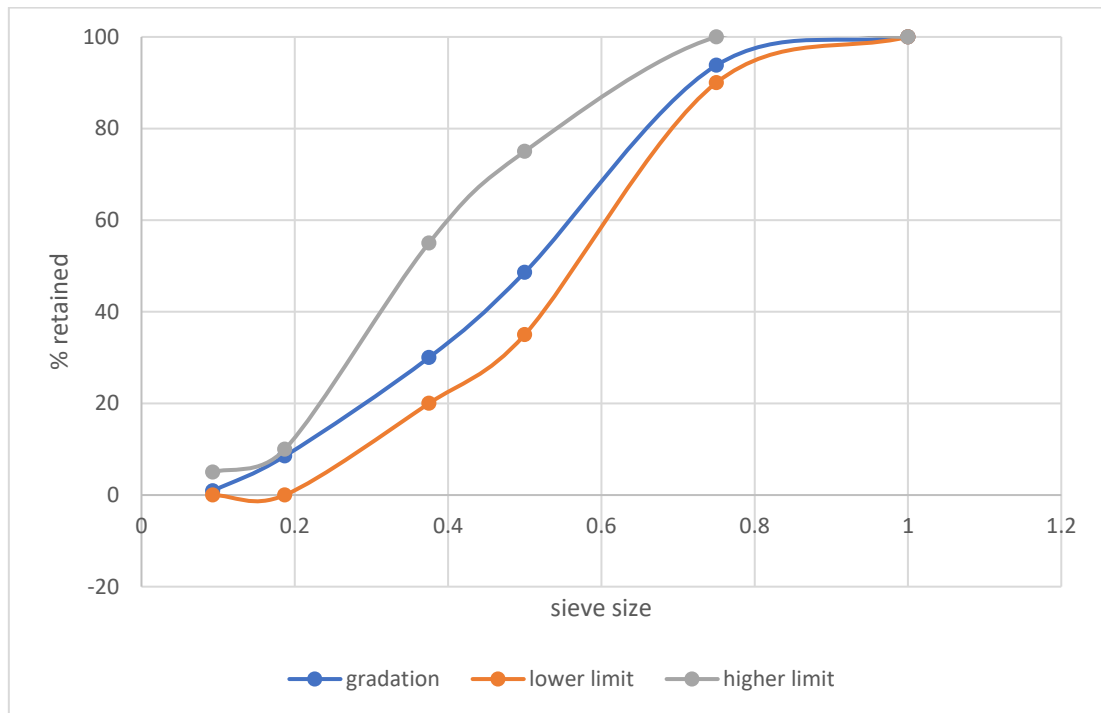


Figure 8: shows the gradation curve of coarse aggregate.

Properties	Values
Specific Gravity	2.59
Water Absorption	0.87%
Surface Moisture	0.1%

Table 3: shows the properties of coarse aggregate.

3.2 Experimental Methodology

In order to investigate the desired results of graphene reinforcement in concrete, we performed a series of tests on 75 concrete samples.

Test	Moulds	Quantity	Formulation	Total
Compression	Cylinder (4"x8")	3	15	45
Split tensile		3	5	15
Elastic Modulus		2	5	10
Flexure	Beam (4"x4"x20")	1	5	5

Table 4: shows the details of casting.

GNPs were added to the concrete mix after sonication for 2 hours using ultrasonication bath to ensure the effective dispersion of GNPs in water. Later UV spectroscopy was done on different formulations to check the dispersion of nano particles in water.

3.2.1 Mix Design

To work out the mix design, literature was consulted, and different concentrations of GNPs were formulated to check the optimum concentration based on hit and trial method. A mix design was worked up for five different formulations as shown in the table below. One of which are the control samples and rest of the formulations had different concentrations of GNPs in concrete mix.

Sample Name	Mix Design	W/C Ratio	GNPs
Control Samples	1 : 1.85 : 2.56	0.47	0%
0.3 g/L			2.41%
0.6 g/L			4.80%
0.8 g/L			6.40%
1 g/L			7.97%

Table 5: shows the detail of concrete formulations.

3.2.2 Mixing Regime

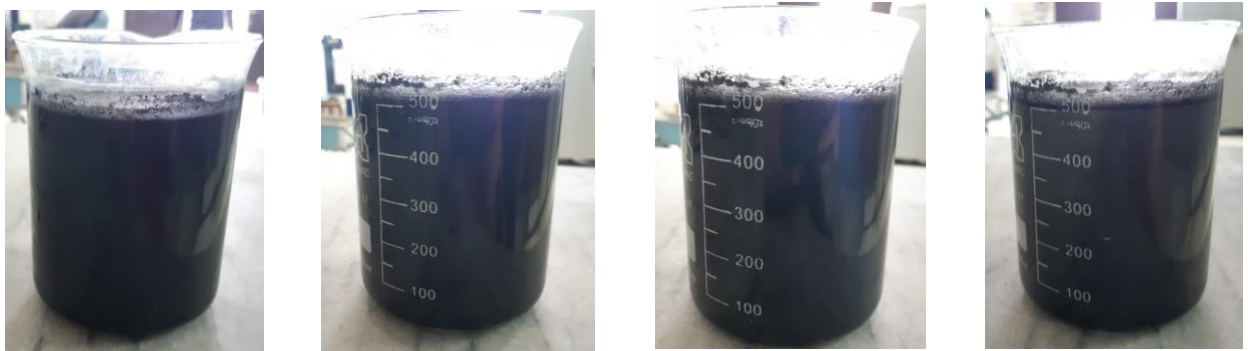
The dispersed GNP solution was prepared using 3 500 ml beaker in an ultrasonication bath for 2 hours. After sonication, the solution was swiftly poured and mixed manually in the remaining water to be used for concrete. Meanwhile, the constituents of the concrete were mixed in a dry state for 45 seconds and then 80% of the GNPs solution was added to the dry mix which was mixed for 1 minute and 30 seconds. Then remaining solution was added and mixer was kept running for further 2 minutes. The concrete mix was poured into the greased moulds, to avoid concrete sticking to it's walls, and vibrated on the vibrating table for even compaction and the surface of the moulds were levelled with a trowel.

3.2.3 Dispersion Checks for GNPs

In order to ensure that GNPs are properly dispersed, a couple of dispersion checks were performed.

3.2.2.1 Visual inspection at 5-minute intervals

Pictures of GNPs suspension after dispersion were taken in order to estimate the time by which GNPs settle down. It took about 40 minutes for the dispersion to become partially clear. After 40 minutes, considerable amount of GNPs were still in dispersed. Mixing of GNPs in concrete was performed within 5 minutes after removing from sonication bath.



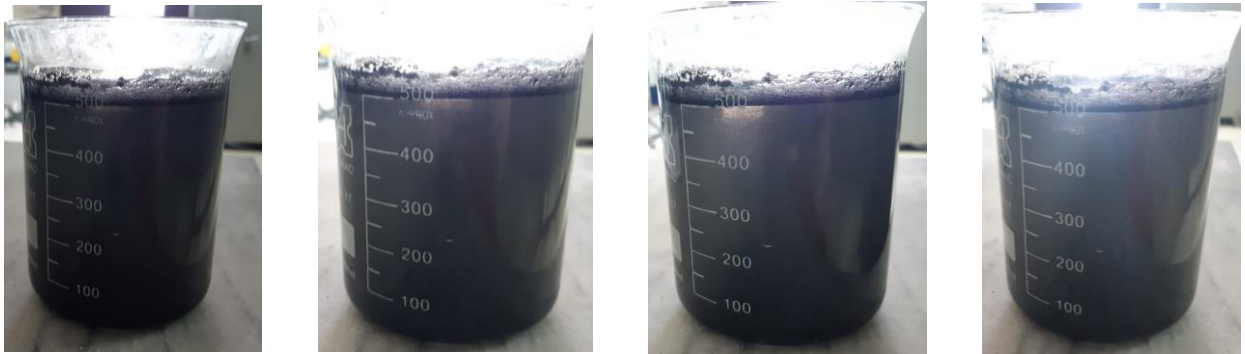


Figure 9: shows settlement of GNPs with time.

3.2.2.2 UV Spectroscopy of the suspensions

UV spectroscopy on GNPs suspensions for all the 4 concentrations was performed. The results show the relative degree of dispersion by varying the concentrations

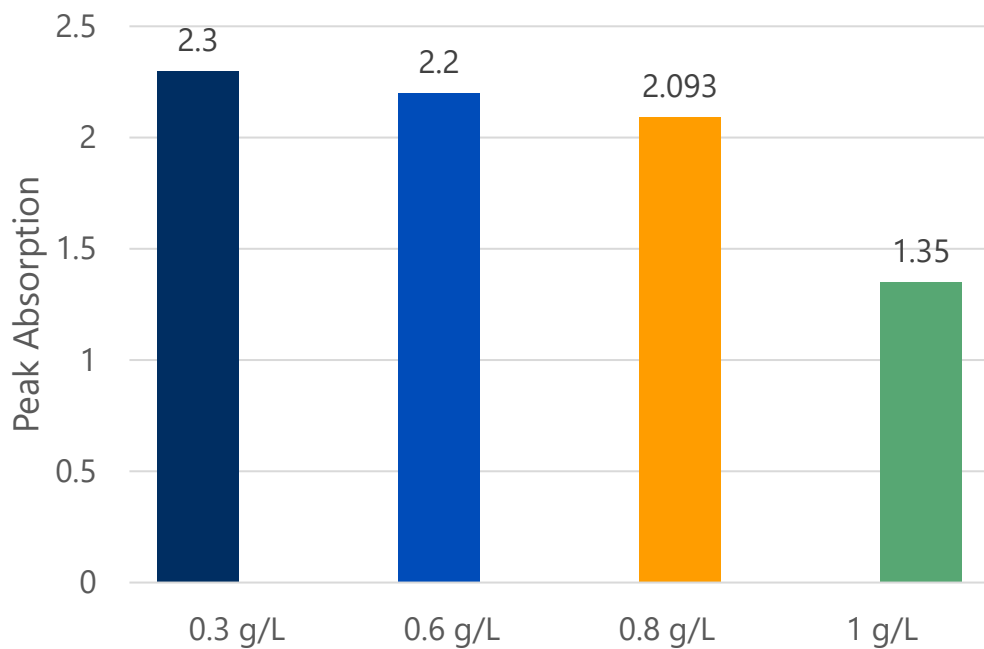


Figure 10: shows relative absorption value at different concentrations.

3.2.2 Curing

24 hours after the specimens were casted, they were placed in the temperature controlled curing tank at 25°C for 28 days for experimental testing. For compression tests, the specimens were cured for 7 and 14 days, respectively.

CHAPTER 4

EXPERIMENTAL TESTS AND RESULTS

4.1 Compressive Test

Axial Compression test was performed on cylinders according to the ASTM C39 standards. Testing was performed at 7, 14 and 28 days curing period. Three 4x8 in. cylinders were used for the data dates and their results were plotted as shown in the following table.

Samples	0.3 g/L	0.6 g/l	0.8 g/l	1 g/l	Control
7 days	2887.77	2790.65	3521.85	3096.56	2712.2
14 days	3885.39	3654.95	4158.77	3894.26	3292.36
28 days	4329.38	4084.15	5032.81	4385.47	3590.9

Table 6: shows the compressive strengths of different concrete formulations.

The experimental results show an increase in compressive strength at the end of 28 day period by 20% in 0.3 g/L concentration samples, 13.7% increase in 0.6 g/L concentration samples, 40.2% increase in 0.8 g/L concentration samples, and 22.1% increase in strength in 1 g/L concentration. A graph plotted shows the peak of strength of 0.8 g/L concentration of Graphene Nano Platelets suggesting the optimum concentration lies in this range.

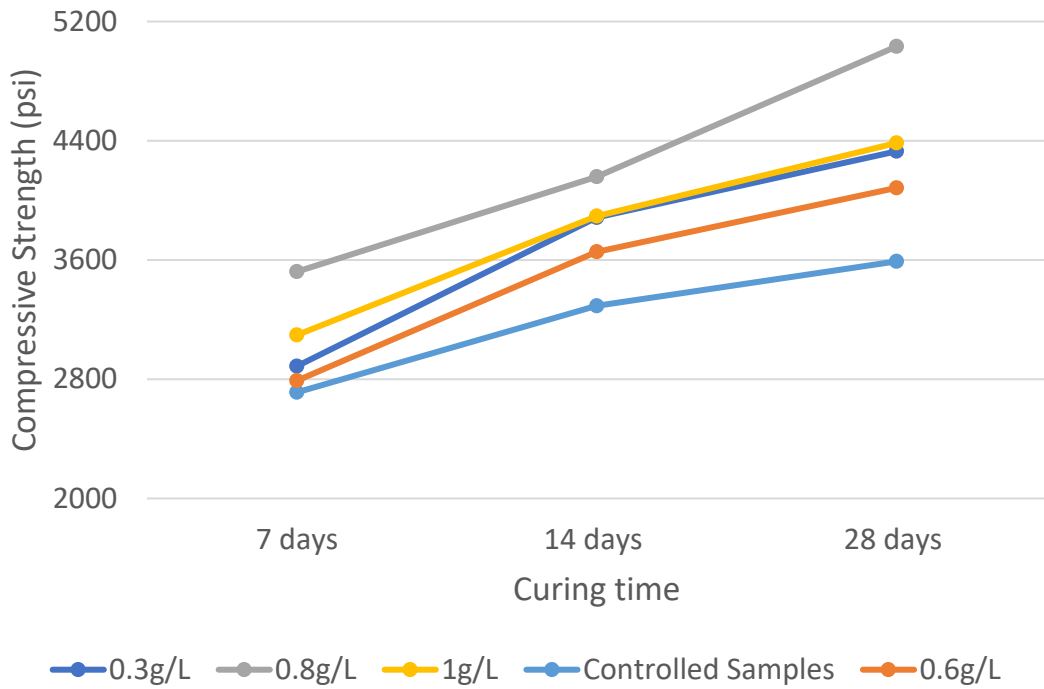


Figure 11: shows the compressive test results over a period of 28 days

It can be noted in the graph that the initial rate of strength gain is comparatively higher in samples of concentrations other than the 0.8 g/L concentration but the ultimate strength of the 0.8 g/L is the highest, which is coherent with the similar observation in plain concrete added with admixture.

4.2 Split Tensile Test

Split tensile test was performed on concrete cylinders according to ASTM C496 standards after 28 days of curing.



Figure 12: shows sample after split tensile test.

Increase in strength was noted in the concrete reinforced with Graphene Nano Platelets. The strength increase in 0.3 g/L concentration was 5%, 19.6% increase in strength of 0.6 g/L concentration, 27.1% increase in strength of 0.8 g/L concentration, and 15% increase in strength of 1g/L concentration of Graphene Nano Platelets reinforced concrete when compared with control samples. 0.8 g/L concentration was noted to have the highest split tensile strength.

Control Sample	0.3 g/L	0.6 g/l	0.8 g/l	1 g/l
419.85 psi	441.20 psi	502.23 psi	533.62 psi	482.97 psi

Table 7: shows split tensile strengths of different concrete formulations.

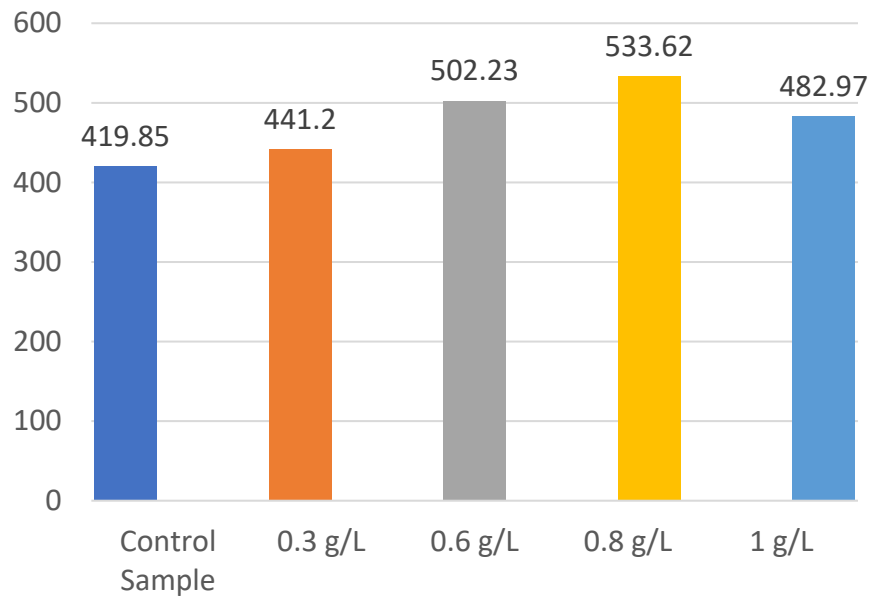


Figure 13: shows the split tensile test results.

Comparing the experimental and theoretical tensile strength of concrete cylinders in figure 18 we note that the experiment strength values are comparable with the values calculated from the formula $7.5\sqrt{fc'}$. This suggests that the formula is applicable to graphene reinforced concrete.

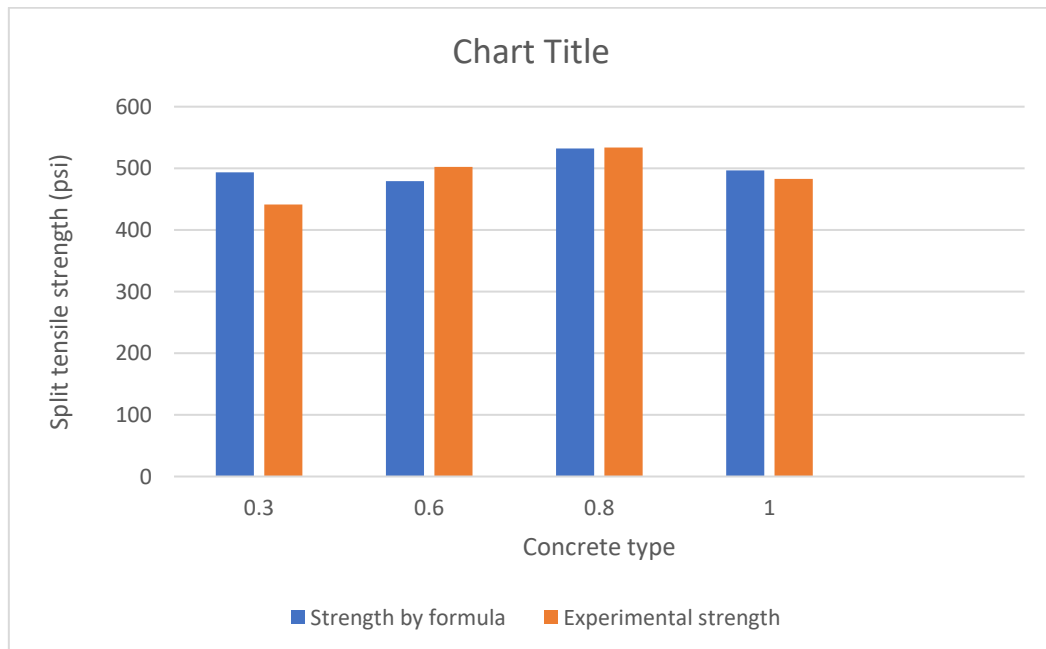


Figure 18: Split Tensile Experimental and formula results compared

4.3 Flexure Test

Flexure test was performed on 100x100x500 mm beam according to ASTM C293 at the end of 28 day period of curing. The prism was mid-point loaded.

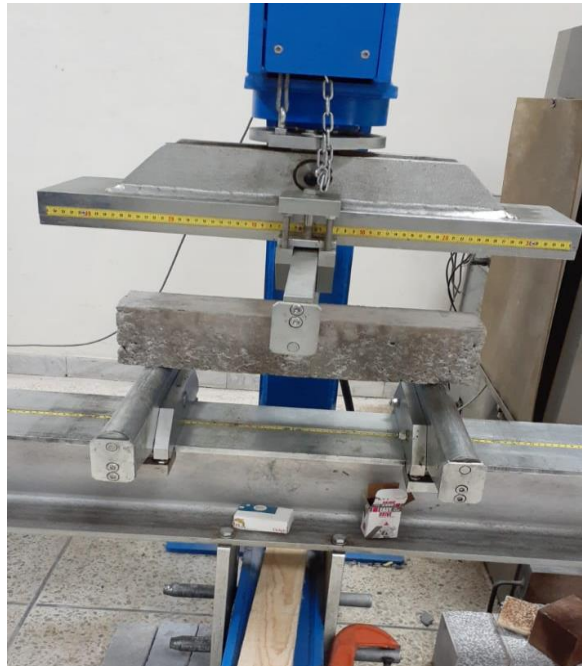


Figure 14: shows flexure testing assembly with beam specimen.

The results noted showed highest strength gain in 0.8 g/L concentration reporting it to be the optimum concentration. The strength gain of 4.4% was observed in 0.3 g/L concentration prism, 21.3% increase in flexural strength was noted in 0.6 g/L concentration, 69.86% increase in strength gain of 0.8 g/L concentration prism, and 39.98% increase in strength of 1 g/L concentration of Graphene Nano Platelets reinforced prism when compared with control group.

Control Sample	0.3 g/L	0.6 g/l	0.8 g/l	1 g/l
3859.45 psi	4029.15 psi	4681.82 psi	6725.4 psi	5402.66 psi

Table 8: shows flexural strength of different concrete formulations.

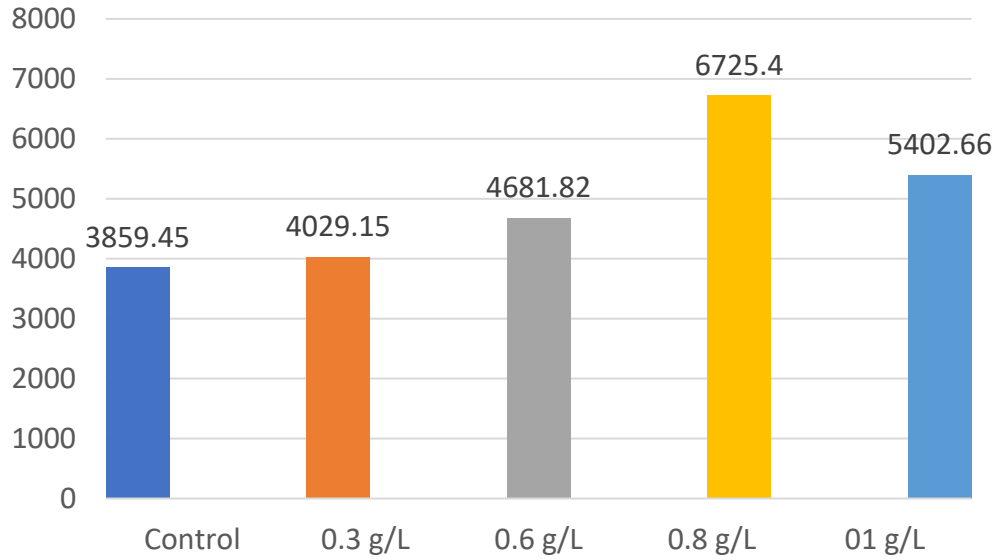


Figure 15: shows flexural test strength results at the end of 28 day period

4.4 Elastic Modulus Test

Elastic Modulus test was done according to the ASTM C469 standards. The highest elastic modulus was achieved by 0.8 g/L concentration. The Tab. 10 and fig. 16 illustrates the results.

Sample	Elastic Modulus (GPa)
0.3 g/L	25.12
0.6 g/L	38
0.8 g/L	47.6
1 g/L	34.8
Control	25.12

Table 9: shows the elastic modulus values of different concrete formulations.

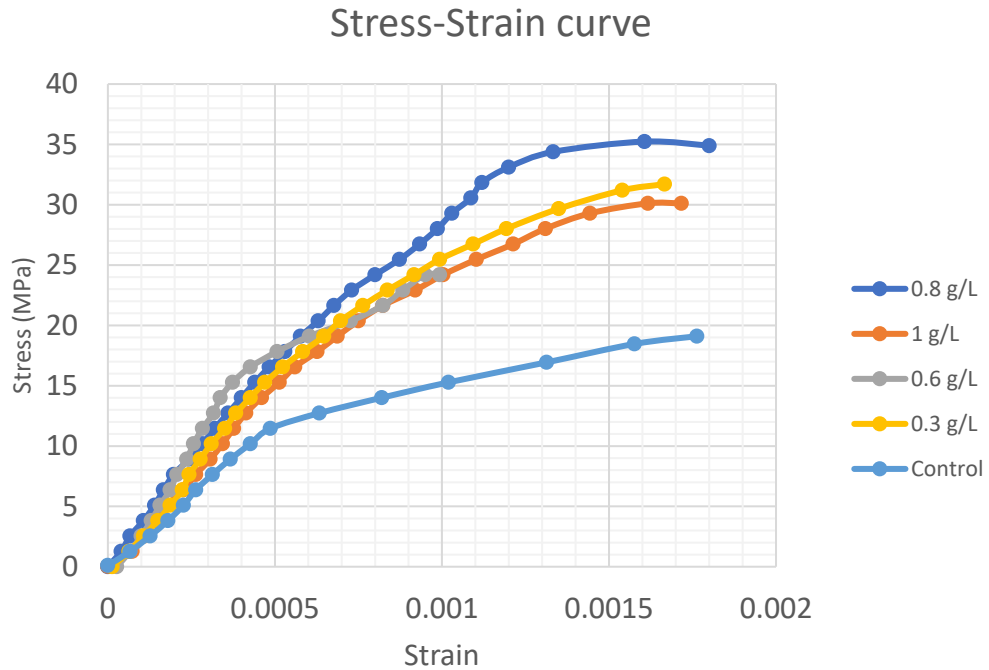


Figure 16: shows stress-strain curves of various GNP concentrations along with control specimen.

The toughness of optimum sample (0.8 g/L concrete) and control sample was calculated by computing the area under the curve of stress strain curves. The optimum sample reported increased toughness as compared to control sample. The Tab. 11 and fig. 17 illustrates the results.

Concrete Type	Area Under The Curve
0.8 g/L	0.04292
Control	0.0292

Table 10: shows the toughness values of different concrete samples.

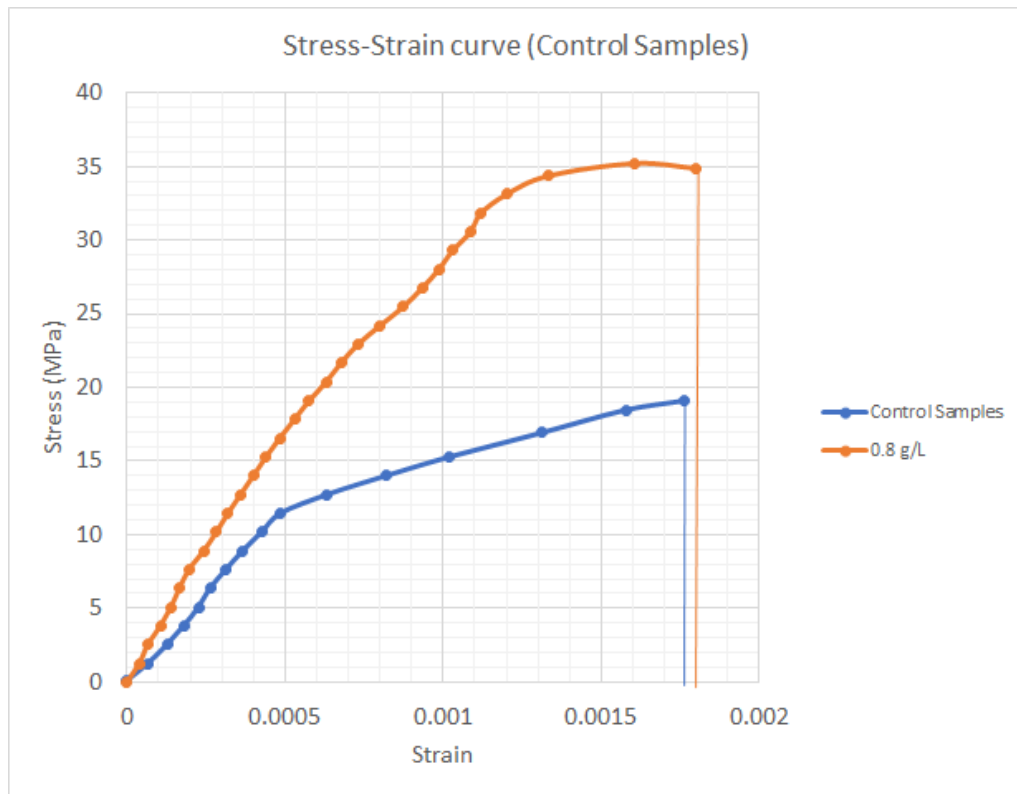


Figure 17: shows stress strain curve of optimum and control sample

4.5 Electrical Resistivity Test

Electrical Resistivity test was done on a cylinder of 0.8 g/L concentration of GNPs according to ASTM's C1876 standard. This test is used to check resistance by concrete against chloride ion penetration. Although chloride penetration doesn't have any effect on concrete, but it results in corrosion of steel. The results show that GNP reinforced concrete shows more resistance against chloride penetration. Electrical resistance offered by concrete is directly proportional to resistance against chloride penetration.



Figure 18: shows electrical resistivity test apparatus and specimen

Table 12 below shows the results of electrical resistivity test:

Sample	Electrical Resistivity
0.8 g/L	10.2 k Ω cm
Control	7.7 k Ω cm

Table 11: shows the electrical resistivity values of different concrete samples.

Resistivity level (k ohm cm)	Possible corrosion rate
<5	Very high
5 to 10	High
10 to 20	Moderate to low
>20	Insignificant

Table 12: shows standard resistivity level compared to possible corrosion rate

The results showed that the resistivity of 0.8 g/L sample lie in moderate to low range of corrosion while the resistivity of control sample lie in the high range of corrosion. The results also show that the concrete with GNPs have less pores as compared to normal concrete making Graphene concrete more durable.

4.6 Pulse Velocity Test

Like the electrical resistivity test, pulse velocity test was done on the same cylinder of 0.8 g/L concentration according to ASTM's C597 standards using Pundit apparatus. This test is used to estimate the quality of concrete by passing ultrasonic pulse through specific length of concrete sample. Time taken by pulse to travel through given length of concrete sample is given by the apparatus the length of sample is divided by time taken to get the velocity.

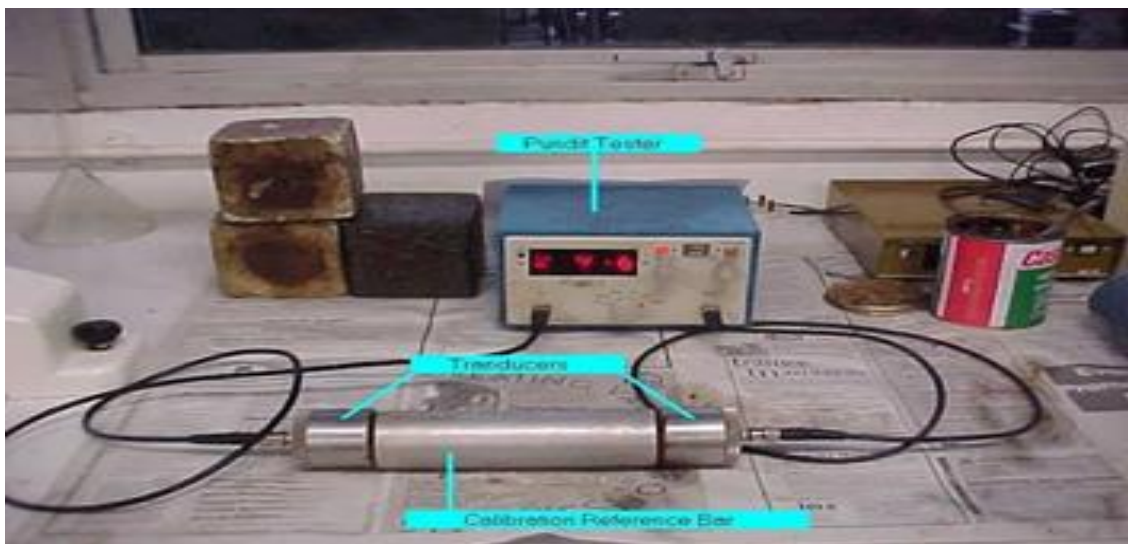


Figure 19: shows pulse velocity test apparatus

The results, comparing with fig. 20, show that optimum sample has higher value of pulse velocity, and it has very good quality while in control sample slight porosity may exist.

SAMPLE	TIME (μ sec)	PULSE VELOCITY
0.8g/L	52	3.9
Control	57	3.57

Table 13: shows the pulse velocity values of different concrete samples

Table: 1 – Concrete Quality based on Ultrasonic Pulse Velocity Test

PULSE VELOCITY	CONCRETE QUALITY
>4.0 km/s	Very good to excellent
3.5 – 4.0 km/s	Good to very good, slight porosity may exist
3.0 – 3.5 km/s	Satisfactory but loss of integrity is suspected
<3.0 km/s	Poor and los of integrity exist.

Figure 20: shows the standard ranges of pulse velocity test

4.7 Rebound Hammer Test

Furthermore, like previous non destructive tests, a rebound hammer was used on the same cylinder to get an empirical estimate of the strength of the concrete according to ASTM C805. The apparatus mainly gives results on basis of surface hardness and penetration resistance.



Figure 21: shows the rebound hammer

The results show that optimum sample has considerably higher strength and surface hardness than control sample.

Sample	Rebound Number	Compressive Strength (psi)
0.8g/L	28	3200
Control	21	2000

Table 14: shows the compressive strength of different concrete samples through rebound hammer test.

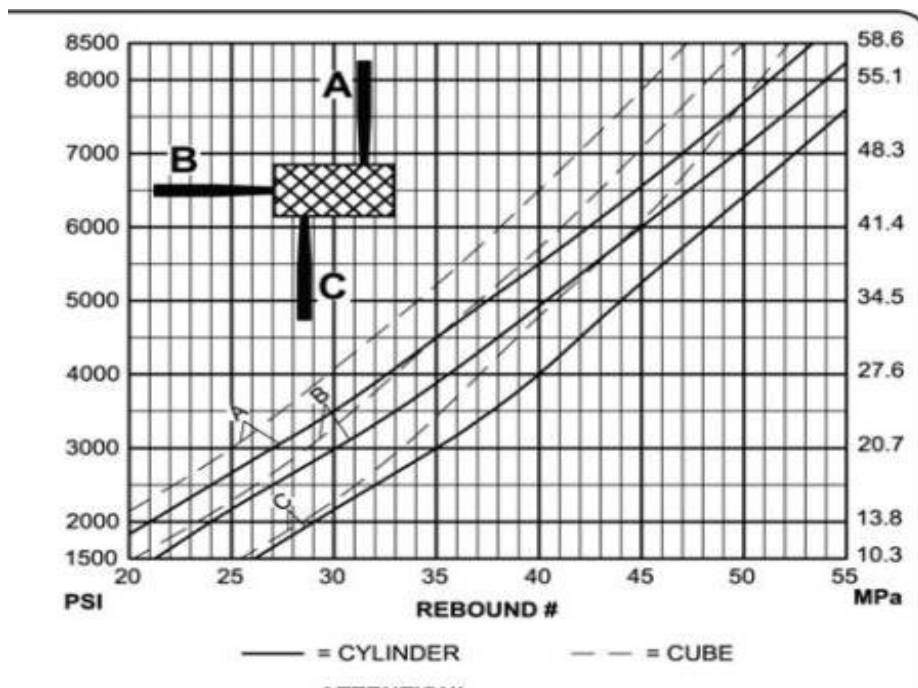


Figure 22: shows the rebound hammer graph for strength estimation

4.8 Scanning Electron Microscopy of the Concrete

To observe changes in microstructure, SEM for optimum and graphene reinforced samples was performed.

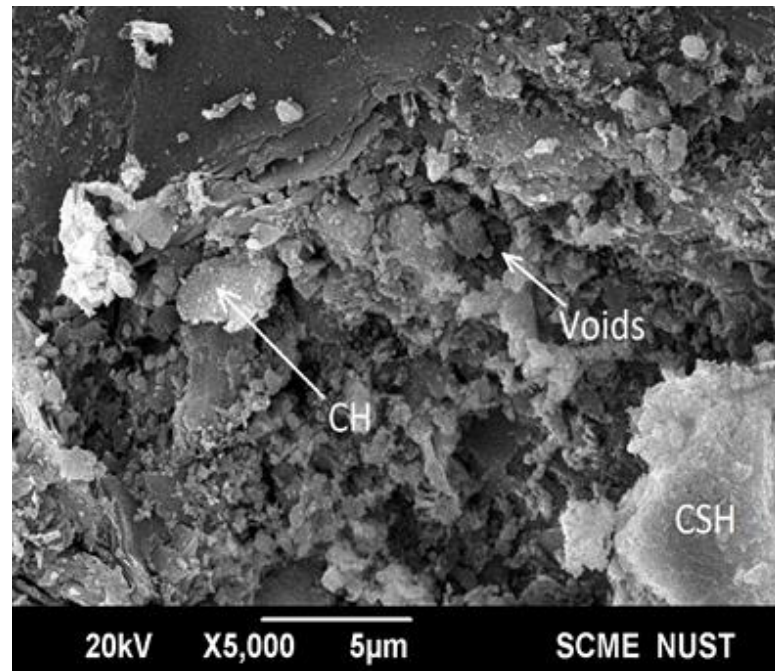


Figure 23: shows the SEM image of control sample.

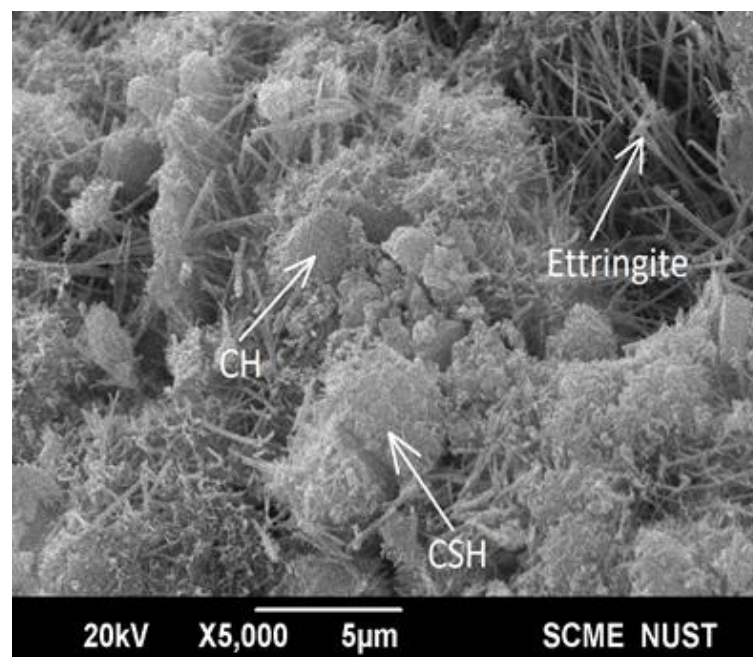


Figure 244: shows the SEM image of 0.8 g/L concrete sample.

In SEM of control sample shown in fig. 23, it can be seen that the degree of crystallinity is poor and hydration products can be hardly identified. Also, it has more voids.

Graphene reinforcement in concrete promotes the growth of hydration products and results in improvement in crystallinity. So, in SEM of graphene reinforced concrete samples the microstructure and degree of crystallinity has improved. It can be clearly identified that the hydration products i.e., CSH gel, calcium hydroxide and ettringite. In fig. 25 which is the optimum sample, the microstructure is dense and has less voids. Hence, addition of GNPs in concrete showed better growth of CSH gel and better structure of hydration products.

CONCLUSIONS

1. Addition of Graphene Nano Platelets sonicated in water and added in the concrete mixer during batching leads to an increase in the quality and durability of concrete proven by the pulse velocity test on 0.8 g/L sample where the speed of pulse velocity is 10% faster than control sample.
2. Up to 40% increase in compressive strength, 27% increase in tensile strength and 47% increase in toughness has been observed.
3. 0.8 g/L concentration of GNPs sourced from SCME nano-synthesis lab showed the highest strength gain in concrete.
4. Electrical resistivity test and pulse velocity test results suggest that there is a reduced chloride penetration rate causing reduction in the rate of steel corrosion and increase in durability of concrete.

RECOMMENDATIONS

1. Further research is needed to analyze GNPs interaction in the microstructure of concrete. Further research needed to analyze the structural performance of Graphene reinforced concrete members.
2. The carbon footprint of the method of Graphene Nano Platelets synthesis as well as the concrete reduced due to increase in strength should be compared for the prospects of Green Concrete.
3. The prospects of industrial scale synthesis of Graphene Nano Platelets at an economical rate must be explored.
4. Interaction of Graphene Nano Platelets with various admixtures is needed.

CONSTRAINTS

1. Novel method of GNPs synthesis not fully understood.
2. Due to a limited quantity of provided graphene tests for tensile strength and flexural strength were performed only at the end of 28 day period unlike compressive strength tests.
3. Lack of molds prevent large scale casting; hence the casting regime must be extended

ANNEXURE-A

Sieve No	Weight Retained (g)	% age weight Retained	Cumulati ve % retained	% Passing	ASTM min. % passing	ASTM max. % passing
#4	14	1.4	1.4	98.6	95	100
#8	51	5.1	6.5	93.5	80	100
#16	162	16.2	22.7	77.3	50	85
#30	332	33.2	55.9	44.1	25	60
#50	316	31.6	87.5	12.5	10	30
#100	93	9.3	96.8	3.2	2	10
#200	21	2.1	98.9	1.1	-	-
Pan	11	1.1	100	0	-	-

Table 15: shows sieve analysis of fine aggregate.

Sieve No.	Weight retained (g)	% weight retained	Cumulative % retained	% passing
1''	0	0	0	100
¾''	273	27.3	27.3	72.7
½''	727	72.7	100	0
3/8''	0	0	100	0
Pan	0	0	100	0
Total	1000	-	-	-

Table 16: shows sieve analysis of coarse aggregate 1.

ANNEXURE-B

Sieve No.	Weight retained (g)	% weight retained	Cumulative % retained	% passing
1''	0	0	0	100
¾''	0	0	0	100
½''	358	35.72854291	35.72854291	64.27145709
3/8''	345	34.43113772	70.15968064	29.84031936
Pan	299	29.84031936	100	0
Total	1002	-	-	-

Table 17: shows sieve analysis of coarse aggregate 2.

Sieve No.	Weight retained (g)	% weight retained	Cumulative % retained	% passing
#4	706	70.6	70.6	29.4
#8	294	29.4	100	0
Pan	0	0	-	-
Total	1000	100	-	-

Table 18: shows sieve analysis of coarse aggregate 3.

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