EFFECT OF GRAPHENE OXIDE ADDITION IN CEMENT MORTAR

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DECLARATION

I, Ashis Majee, Master of Engineering in Civil Engineering (Structure Engineering), Jadavpur University, Faculty of Engineering and Technology, hereby declare that the work being presented in the thesis work entitled, **"EFFECT OF GRAPHENE OXIDE ADDITION IN CEMENT MORTAR"**, is an authentic record of work that has been carried out in the Department of Civil Engineering, Jadavpur University, Kolkata under **Dr. SAROJ MANDAL** and **Mr. SANTOSH KUMAR DAS** Professor, Department of Civil Engineering, Jadavpur University. The work contained in this thesis has not yet been submitted in parts or full to any other university or institute or professional body for award of any degree or diploma or any fellowship.

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ABSTRACT

Graphene oxide (GO) is a recently invented 2D nanoplane fiber. It is typically produced via chemical oxidation and exfoliation of graphite. It contains active functional groups on its nanoplane surface. At present, the addition of GO in cement based composites becomes an important area of research to improve the overall properties of such cement composite. A limited number of research papers are available on the effect of GO addition in cement based composite. However, a comprehensive study is needed on this topic to utilize its beneficial effects.

Based on this background, an experimental investigation on the effect of GO in cement-mortar has been made. The study includes the effect of addition of GO in different percentage of cement on the workability and the mechanical properties of cement mortar. The effect of GO addition has been made both for OPC and PPC separately with cement sand ratio of 1:2 and 1:3. The microstructure properties of such GO based cement composite have been also studied. The microstructural analysis of cement mortar (with/without GO) have been analyzed through Mercury Intrusion Porosimetry (MIP) for pore size distribution and X-ray Diffraction (XRD) techniques.

It is noted that the cement mortar with GO has a better mechanical properties than the control mortar (without GO). The compressive strength and flexural strength of cement mortar increases up to a certain percentage of GO addition and further addition of GO the strength decreases. In general, the workability of cement mortar decreases due to addition of GO in plain cement mortar. Graphene oxide in the form of nanosheet acts as nano reinforcement in cement mortar. Microstructural analysis of the GO-cement mortar shows much denser structure and better crystallized of hydration product. For OPC the optimal GO addition is 0.05% and 0.04% for cement sand ratio of 1:2 and 1:3 respectively. However, the optimal GO addition in PPC is 0.04% and 0.05% for cement sand ratio of 1:2 and 1:3 respectively.

CONTENTS

ABSTRACT....vi
LIST OF FIGURES....ix
LIST OF TABLES....xi
CHAPTER-1 INTRODUCTION....1
1.1 INTRODUCTION....2
1.1.1 Graphene Oxide (GO)....3
1.1.2 Production of Graphene Oxide....4
1.2 RESEARCH OBJECTIVES....5
CHAPTER-2 REVIEW OF LITERATURE....6
2.1 REVIEW OF LITERATURE....7
2.2 SCOPE OF RESEARCH....19
CHAPTER-3 EXPERIMENTAL INVESTIGATION....20
3.1 MATERIALS....21
3.1.1 Cement....21
3.1.2 Fine Aggregate....21
3.1.3 Graphene Oxide (GO)....22
3.2 MIXTURE PROPORTION....22
3.3 SPECIMENS....23
3.3.1 Specimen Preparation....24
3.4 EXPERIMENTAL PROGRAMME....26
3.4.1 Flow Test....26
3.4.2 Compressive Strength Test....27
3.4.3 Flexural Strength Test....27
3.4.4 Ultrasonic Pulse Velocity (UPV) Test....28
3.4.5 Sorptivity Test....28
3.4.6 XRD analysis....29
3.4.7 MIP analysis....29
CHAPTER-4 RESULTS AND DISCUSSION....30
4.1 RESULT AND DISCUSSION....31

CHAPTER-5 CONCLUSIONS AND SUGGESTION FOR FUTURE RESEARCH 49
5.1 CONCLUSIONS 50
5.2 SUGGESTION FOR FUTURE RESEARCH 51

LIST OF FIGURES

		Page no
Fig 1.1	Lerf and Klinowski model (1998) of GO	3
Fig 1.2	Szabo model (2006) of GO	3
Fig 1.3	Modified Hummer's method for GO production	4
Fig 1.4	Ultrasonication method for GO production	5
Fig 2.1 (a)	SEM image of plain paste showing a straight-through type crack (arrow)	7
Fig 2.1 (b)	SEM image of GO–cement composite showing a number of fine cracks (arrows) with few branches	7
Fig 2.2 (a)	Effect of age on compressive strength	8
Fig 2.2 (b)	Standard deviation of compressive strength results measured at various ages	8
Fig 2.3	Compressive strength for different carbonation	19
Fig 3.1	Flow chart of sample preparation	25
Fig 3.2	Mini Slump-Cone with flow table	26
Fig 3.3	Flow measurement	26
Fig 3.4	Compressive Strength Test by Compression Testing Machine	27
Fig 3.5	Center Point Load Test	27
Fig 3.6	Sorptivity Test	29
Fig 4.1	Flow table test results GO-OPC mortar with cement sand ratio 1:2	33
Fig 4.2	Flow table test results GO-OPC mortar with cement sand ratio 1:3	33
Fig 4.3	Flow table test results of GO-PPC mortar with cement sand ratio 1:2	33
Fig 4.4	Flow table test results of GO-PPC mortar with cement sand ratio 1:3	33
Fig 4.5	Compressive strength of GO based mortar using OPC with cement sand ratio 1:2	34
Fig 4.6	Compressive strength of GO based mortar using OPC with cement sand ratio 1:3	35
Fig 4.7	Compressive strength of GO based mortar using PPC with cement sand ratio 1:2	36
Fig 4.8	Compressive strength of GO based mortar using PPC with cement sand ratio 1:3	37
Fig 4.9	Flexural strength of GO based mortar using OPC with cement sand ratio 1:2	38
Fig 4.10	Flexural strength of GO based mortar using OPC with cement sand ratio 1:3	39
Fig 4.11	Flexural strength of PPC and GO-PPC mortar with cement and sand ratio 1:2	40
Fig 4.12	Flexural strength of PPC and GO-PPC mortar with cement and sand ratio 1:3	41
Fig 4.13	Sorptivity test result of GO based mortar using OPC with cement and sand ratio 1:2	43

Fig 4.14 Sorptivity test result of GO based mortar using OPC with cement and sand ratio 1:3 44
Fig 4.15 Sorptivity test result of GO based mortar using PPC with cement and sand ratio 1:2 44
Fig 4.16 Sorptivity test result of GO based mortar using PPC with cement and sand ratio 1:3 45
Fig 4.17 XRD analysis result of OPC and GO-OPC (0.05% GO) mortar with cement sand ratio 1:2 46
Fig 4.18 XRD analysis result of PPC and GO-PPC (0.05% GO) mortar with cement sand ratio 1:2 47
Fig 4.19 MIP result of OPC and GO-OPC (0.05% GO) mortar with cement sand ratio 1:2 48

LIST OF TABLES

		Page no
Table 3.1	Chemical composition of Ordinary Portland Cement.	21
Table 3.2	Chemical composition of Portland Pozzalona Cement	21
Table 3.3	Technical Parameters of Graphene Oxide	22
Table 3.4	Mix proportion for different tests of cement mortar with GO and without GO	23
Table 4.1	Flow table test results for OPC mortar with GO	32
Table 4.2	Flow table test results for OPC mortar with GO	32
Table 4. 3	Flow table test results for PPC mortar with GO	32
Table 4.4	Flow table test results for PPC mortar with GO	32
Table 4.5	Compressive strength of GO based mortar using OPC with cement sand ratio 1:2	34
Table 4.6	Compressive strength of GO based mortar using OPC with cement sand ratio 1:3	35
Table 4.7	Compressive strength of GO based mortar using PPC with cement sand ratio 1:2	36
Table 4.8	Compressive strength of GO based mortar using PPC with cement sand ratio 1:3	37
Table 4.9	Flexural strength of GO based mortar using OPC with cement sand ratio 1:2	38
Table 4.10	Flexural strength of GO based mortar using OPC with cement sand ratio 1:3	38
Table 4.11	Flexural strength of GO based mortar using PPC with cement sand ratio 1:2	40
Table 4.12	Flexural strength of GO based mortar using PPC with cement sand ratio 1:3	40
Table 4.13	UPV test result of OPC mortar with GO and cement sand ratio 1:2	41
Table 4.14	UPV test result of OPC mortar with GO and cement sand ratio 1:3	42
Table 4.15	UPV test result of PPC mortar with GO and cement sand ratio 1:2	42
Table 4.16	UPV test result of PPC mortar with GO and cement sand ratio 1:3	42

CHAPTER-1

INTRODUCTION

1.1 INTRODUCTION

The incorporation of reinforcing materials into mortar and concrete has become a common practice to improve its mechanical performance. Microfibers such as steel, glass, polymers, and carbon have been extensively used for developing fiber-reinforced composites over the past several decades. Although microfibers enhance the ductility and toughness of the concrete matrix, their influences on compressive strength and durability are considered to be limited. The functionalization of carbon and polymer fibers enables them to form covalent bonds with the cement matrix; however, their small specific surface area limits their contribution to the interfacial strength (Wichmann et al. 2005). As such, nanomaterials can provide a better solution than traditional fibers through reinforcing at the nanoscale and allocating a much higher specific surface area for cement matrix interaction. Some nanomaterials even exhibit pozzolanic characteristics by consuming calcium hydroxide to produce calcium silicate hydrate, i.e., C–S–H (Chuah et al. 2014). These characteristics can improve the interfacial structure and internal matrix properties. One such promising nanofibrous material is graphene oxide (GO). It is typically produced from the chemical oxidation and exfoliation of graphite. GO forms a hexagonal 2D sheet layers having several nanometers thick and several hundred nanometers long. They are long-plane nanofibers containing ranges of reactive oxygen functional groups, which can actively influence microstructure formation of cementitious materials during hydration process.

GO offers several smart properties that can potentially enhance the performance of cement-based materials. Compared to other nanomaterials suitable for cement incorporation, GO has a large specific surface area that contains highly reactive hydroxyl, epoxide, carboxyl, and carbonyl functional groups (Lambert et al. 2009). Although the functionalization of graphene into GO degrades the mechanical properties, GO sheets exhibit a mean elastic modulus of 32 GPa and a tensile strength of 130 GPa, (Lee et al. 2008) which are superior to the elastic modulus and tensile strength of cement.

In the present study, the major focus related to investigations of GO addition in cement-mortar is to study the overall behavior in terms of workability, compressive strength, flexural strength, hydration rate, sorptivity, reinforcing ability and pore size distribution. The micro structure analysis has been also made in this study. The GO addition in cement mortar varies from 0.03% to 0.06% by weight of cement in addition mode. The study has been made for OPC and PPC based mortar separately for cement sand ratio of 1:2 and 1:3.

1.1.1 Graphene Oxide (GO)

On a simple level, graphene oxide can be consider as a single-layer of graphene decorated with different oxygen functionalities mostly in the form of hydroxyl and epoxy groups on the basal plane, with smaller amounts of carboxyl, carbonyl, phenol groups at the sheet edges. GO is mainly produced through chemical oxidation of graphite, with subsequent dispersion and exfoliation in water or in suitable organic solvents.

Due to nonstoichiometric composition of GO, its amorphous characters and the inhomogeneous distribution of oxygen groups, the precise atomic structure of GO is still uncertain. Despite this, several structural models has been proposed over the years. The most well-known model now a days, is the one proposed by Lerf and Klinowski [1], based on nuclear magnetic resonance (NMR) studies. He considers a random distribution of flat aromatic regions with not oxidized benzene rings and wrinkled regions of alicyclic six-membered rings bearing hydroxyl groups and ether groups (Fig 1.1). In 1998, Lerf and Klinowski [2] revisited their previous model, adding carboxyl groups only on the edges of the GO sheets.

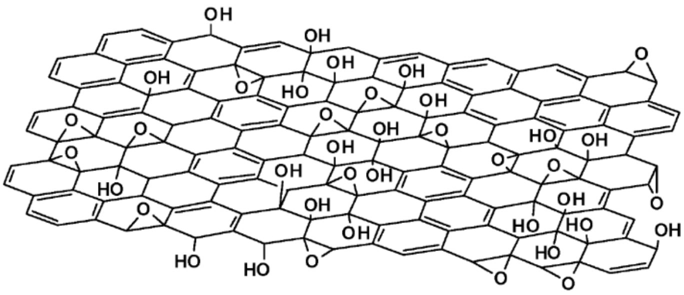


Fig 1.1 Lerf and Klinowski model (1998) of GO

Szabo et al [4] recently proposed a new structural model that involves a carbon network consisting of two kinds of regions: trans-linked cyclohexane chairs and ribbons of flat hexagons with C=C bonds as well as functional groups like hydroxyl, ether, carbonyl and phenolic groups (Fig 1.2).

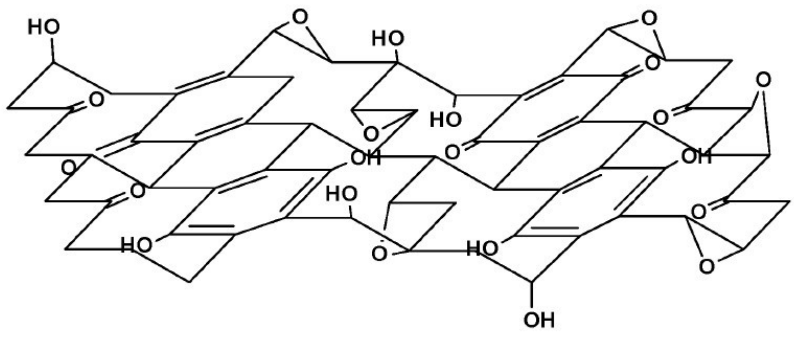


Fig 1.2 Szabo model (2006) of GO

1.1.2 Production of Graphene Oxide

Two suitable industrial scale-up routes for GO production with minimal environmental impacts have been developed: the chemical reduction route (CRR) and the ultrasonication route (USR). In chemical reduction route GO, is commonly synthesized from the oxidation of natural graphite via the modified Hummers method (Park & Ruoff 2009). Briefly, $KMnO_4$, graphite flake, concentrated H_2SO_4, and orthophosphoric acid are mixed and then stirred for 24 h at 50 °C. The resultant mixture is added to H_2O_2 (30%) and centrifuged. The separated product is finally washed with water, HCl, and ethanol at pH 7, and then maintained at 70 °C for 12 h as shown in Fig-1.3.

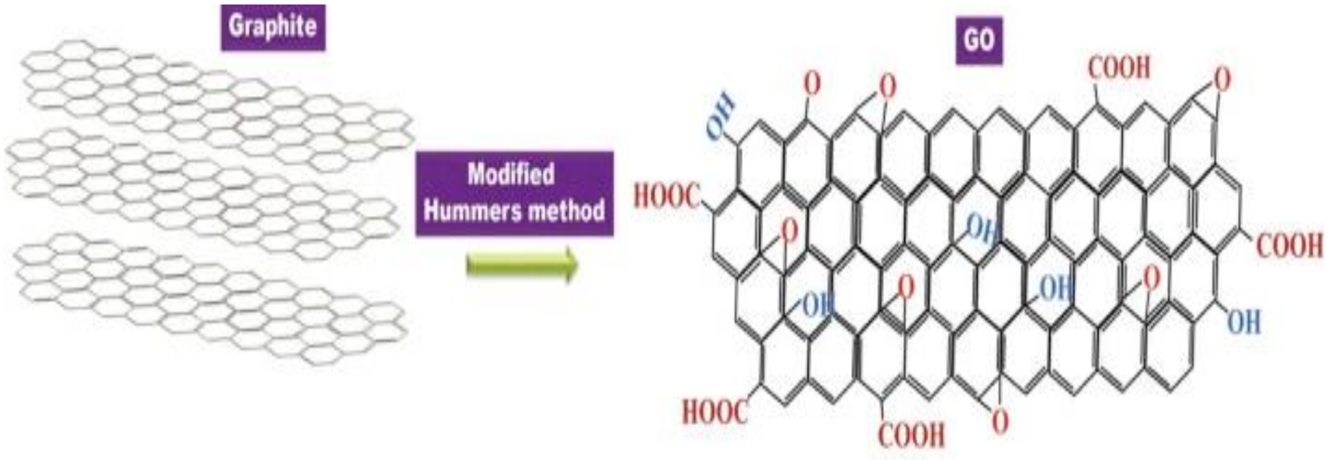


Fig 1.3 Modified Hummer's method for GO production

In addition to the production of GO via the chemical reduction method, it can also be produced through ultrasonication method. Ultrasonic exfoliation is the most widely used delamination technique for the production of high quality graphene oxide (Refer Fig-1.4). In ultrasonic exfoliation method, graphite oxide powder is mixed in aqueous KOH with the pH value 10. For the exfoliation and subsequent dispersion, the probe-type ultrasonicator UP200St (200W) is used. Afterwards, K+ ions are attached onto the graphene basal plane to induce an ageing process. The aging is achieved under rotary evaporation (2 h). In order to remove excessive K+ ions, the powder is washed and centrifuged various times. The obtained mixture is centrifuged and freeze-dried, so that a dispersible graphene oxide powder precipitates.

Preparation of a conductive GO paste: The graphene oxide powder can be dispersed in dimethylformamide (DMF) under sonication in order to produce a conductive paste (Han et al.2014).

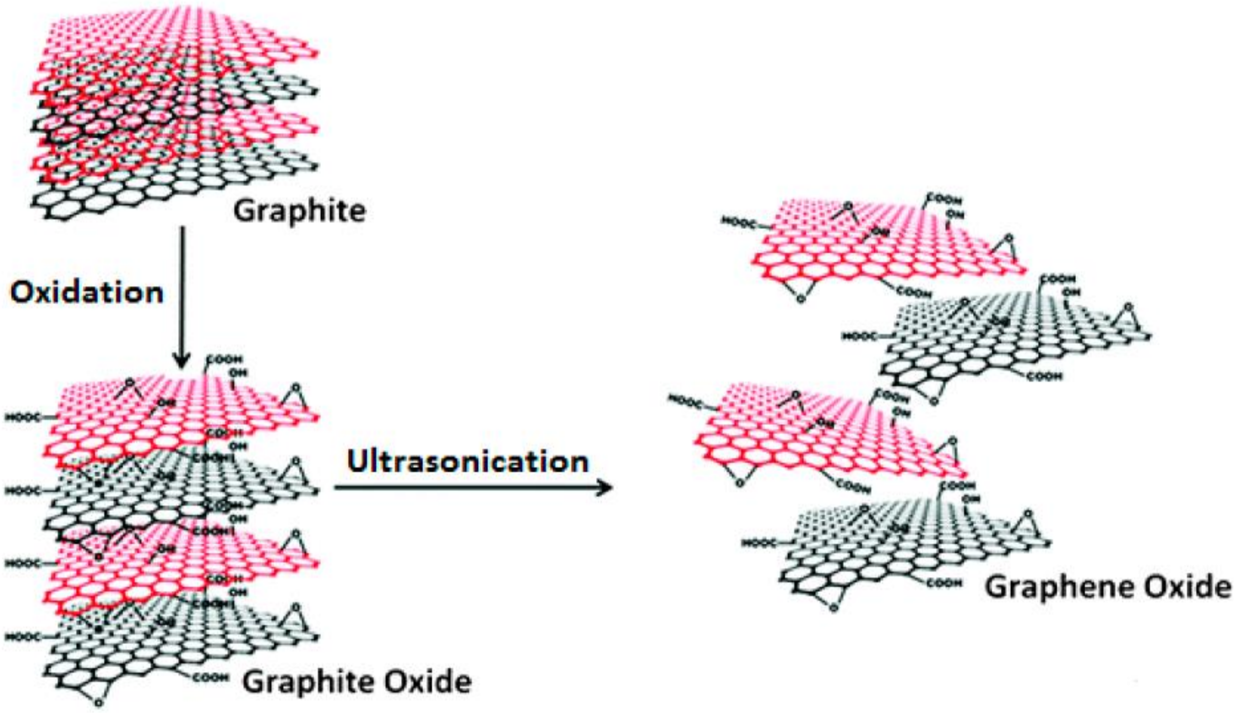


Fig 1.4 Ultrasonication method for GO production

1.2 RESEARCH OBJECTIVES

The objective of this present investigation is as follows

(a) To study the effects of different percentage of graphene oxide addition in cement mortar.

(b) To study the changes in consistency of cement mortar in terms of workability for various percentage of GO addition.

(c) To study the hydration rate and microstructure of the cement mortar due to the addition of GO.

(d) To study the durability of cement mortar for the addition of GO.

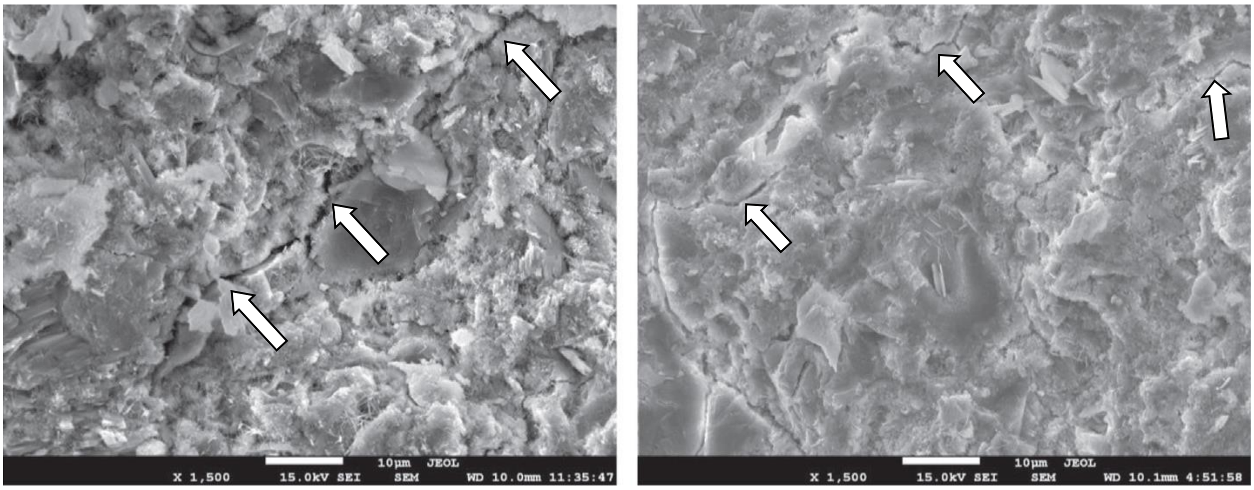
(e) To study the effect of GO in OPC and PPC based mortar.

CHAPTER-2

REVIEW OF LITERATURE

2.1 REVIEW OF LITERATURE

Zhu Pan (2015) et al used GO to enhance the mechanical properties of ordinary Portland cement paste. The introduction of 0.05 wt% GO can increase the GO-cement composite compressive strength by 15-33% and the flexural strength by 41-59%, respectively. Scanning electron microscope imaging of the GO-cement composite shows the high crack tortuosity, indicating that the two-dimensional GO sheet may form a barrier to crack propagation [Refer Fig 2.1 (a) and Fig 2.1 (b)]. Consequently, the GO-cement composite shows a broader stress-strain curve within the post-peak zone, leading to a less sudden failure. The addition of GO also increases the surface area of the GO-cement composite. This is attributed to increasing the production of calcium silicate hydrate. The results obtained in this investigation suggest that GO has potential for being used as nano-reinforcements in cement-based composite materials.



(a) (b)

Fig 2.1 (a) SEM image of plain paste showing a straight-through type crack (arrow) (b) SEM image of GO–cement composite showing a number of fine cracks (arrows) with few branches.

Kai Gong and Zhu Pan (2015) et al investigated on Reinforcing Effects of Graphene Oxide on Portland Cement Paste. They have got from their experimental study that by adding GO nanosheet 0.03% by weight increased the compressive strength and tensile strength of cement composite more than 40% due to reduction of pore structure cement paste. And they have found that mixing of GO nanosheet increased the degree of hydration and reduced the workability of the cement paste.

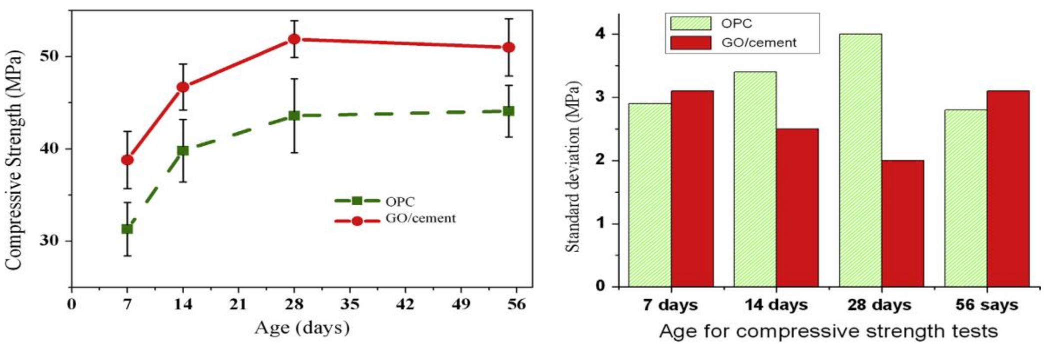
They have used GO nanosheet, OPC and tap water and w/c ratio 0.5 for their experimental investigation. For workability test they have used the mini-slump test and for compressive and tensile strength test they prepared the cylindrical specimen and for measurement of degree of hydration they tested the non-evaporable test.

After the investigation they have reached to the conclusion that the addition of GO nanosheet reduced the workability of OPC paste and the use of GO increases the non-evaporable water content and calcium hydroxide content in OPC paste at different test ages. The results indicate that the degree of hydration of OPC paste is enhanced by GO. On the other hand addition of GO enhances the strength of OPC paste. The 28-day compressive strength and tensile strength are increased by over 40% with 0.03% by weight GO.

Zhu Pan, Li He, Ling Qiu, Asghar Habibnejad Korayem, Gang Li, Jun Wu Zhu, Frank Collins, Dan Li, Wen Hui Duan, Ming Chien Wang (2015) investigated to improve the mechanical properties and microstructure of ordinary Portland cement paste using graphene oxide. They have found that the introduction of 0.05 wt% GO can increase the GO–cement composite compressive strength by 15–33% and the flexural strength by 41–59%, respectively.

Scanning electron microscope imaging of the GO–cement composite shows the high crack tortuosity, indicating that the two-dimensional GO sheet may form a barrier to crack propagation. Consequently, the GO–cement composite shows a broader stress–strain curve within the post-peak zone, leading to a less sudden failure.

They concluded that the incorporation of GO in cement paste significantly increases the volume of gel pores in the composite. The addition of GO also increases the surface area of the GO–cement composite. This is attributed to increasing the production of calcium silicate hydrate. From there results obtained in the investigation they suggested that GO have potential for being used as nano-reinforcements in cement-based composite materials.



(a) (b)

Fig 2.2 (a) Effect of age on compressive strength; (b) standard deviation of compressive strength results measured at various ages.

M. Devasena and J. Karthikeyan (2015) have carried out an investigation on strength properties of graphene oxide (GO) concrete. This study showed the results of an experimental investigation of graphene oxide on physical properties of concrete. This experimental investigation found out that the optimum quantity of graphene oxide required to achieve maximum compressive, tensile and flexural strength of concrete. Graphene oxide was added to the concrete in three mix proportions. Graphene oxide contents were varied by 0.05%, 0.1%, and 0.2% by weight of cement. All the specimens were cured for the period of 7, 14 & 28 days before crushing. Tests were performed at the age of 7, 14 & 28 days. Test results indicated that the inclusion of graphene oxide in concrete enhanced the compressive, split tensile and flexural strength.

They have used for their experimental study, Ordinary Portland cement, 53 Grade (conforming to IS: 12269 – 1987), locally available river sand and locally available crushed blue granite stones nominal size 12.5mm (confined Grading zone II of IS: 383-1970).

Chemical prepared from graphite powder and other chemicals to enhance the strength parameters of the concrete and w/c 0.5. And mix design done for M25 grade concrete. They had made Concrete cubes of size 150mm x 150mm x 150mm and cylinders of size 150mm diameter and 300mm height were casted for the above proportions of concrete to test the compressive strength, the split tensile strength and flexural strength.

After that experimental study they have reached to the conclusion that addition of graphene oxide leads to an increase in compressive strength, tensile strength and flexural strength. 0.1% of GO is needed to improve flexural strength of a PPC matrix about 4% and compressive strength about 11%. The addition of GO improves the degree of hydration of the cement paste and increases the density of the cement matrix, creating a more durable product.

Qin Wang, Jian Wang, Chun-xiang Lu, Bo-wei Liu, Kun Zhang, Chong-zhi Li (2015) investigated the effect of adding graphene oxide (GO) to cement on its microstructure and mechanical strength. A paste of cement (16.5 wt% of water) and GO (0.05 wt %) was prepared together with an identical mixture to which sand (3x the weight of the cement) had been added to form a mortar. The fluidity, viscosity and setting time of the mortar and the morphology, pore structure and compressive and flexural strengths of both the hardened cement paste and mortar, were investigated using SEM, nitrogen adsorption, and fluidity, viscosity, mechanical and hydration tests. The influence of the GO addition on the hydration heat of the cement was also tested. As per the result obtained from their investigation that the addition of GO increases the viscosity, decreases the fluidity and shortens the setting time of the mortar. It also reduces the heat of hydration of the cement. The compressive and flexural strengths of the hardened cement paste at different times are increased by the addition of GO. The flexural strength was greater by 86.1, 68.5 and 90.5% after 3, 7 and 28 days, respectively, and the corresponding compressive strength increases were 52.4, 46.5 and 40.4%.For the hardened mortar, the corresponding increases are 69.4, 106.4 and 70.5% for flexural strength and 43.2, 33 and 24.4% for compressive strength. The addition of GO promotes hydration, decreases pore volume, accelerates crystallite formation and causes the crystallites to align, which increases the tightness of both the hardened cement paste and mortar.

Baig Abdullah Al Muhit et al (2015) in their investigation had incorporated graphene oxide (GO) mechanically with 0.1% and 0.05% dosages and compared with normal cement paste. They tested the compressive strength test for graphene oxide cement composite (GOCC) on 3, 7, 14 and 28days. It was observed that GOCC 0.05 showed highest compressive strength in all curing ages. They have found from X-ray diffraction (XRD) analysis that smaller crystallite sizes of calcium silicate hydrate (C-S-H) and portlandite were responsible for the faster and numerous heterogeneous nucleation and higher compressive strength. They used Ordinary Portland Cement (OPC) as primary binding materials with w/c ratio of 0.50, two dosages of 0.01% and 0.05% by weight of cement are mixed with cement and water. And they also prepared a control specimen with cement and water with same w/c ratio and cast 33mm 3 cubes by both specimens for compressive strength test.

They prepared GO by modified Hummers method from natural graphite powder purchased from Dixon Graphite (Microfine). And they made a solution by 10mg of GO and 10ml of deionized water and used cup-horn ultrasonication to generate a homogeneous brown solution.

They got the compressive strength of GOCC0.05 specimens up to 22.7MPa at 28days compared to 18.2MPa and 17.6MPa for GOCC0.01 and control specimen, respectively, showed 29% and 22% increase in strength over the control specimen and GOCC0.01, respectively, and after 3days, GOCC0.05 and GOCC0.01 specimens showed 24% and 16% increment, respectively in strength compared to the cement paste specimens. Although they have reached this conclusion that GOCC0.01 exhibited higher strength compared to the control specimen, as hydration continues, the strength gain reduced and after 28day, GOCC0.01 showed only 3.4% increased. On the other hand, the compressive strength of GOCC0.05 continued to increase until 28days.

El zbieta Horszczaruk (2015) et al presented how 3 wt% of graphene oxide incorporated into the cement can affect the microstructure and physical–mechanical properties of the cement composite. Therefore, here we present study on early age mechanical response of the cement mortar modified with graphene oxide using atomic force microscopy (AFM). The kinetics of the hydration process was investigated by Infrared, Raman, X-ray diffraction (XRD) techniques. The morphology of the nano composite was revealed by the scanning electron microscopy (SEM).

Graphene oxide (GO) was synthesized by oxidation of natural graphite flakes (Aesar, 325 mesh) according to the modified Hummers method. Briefly, to admixture of $KMnO_4$ and graphite concentrated sulfuric acid and ortho-phosphoricacid were poured. It was stirred for 24 h and heated to 50^{o}C. The resulting mixture was added to H_2O_2 (30%) and centrifuged. The separated solid product was washed with water, HCl and ethanol and kept for 12 h at 70^{o}C.

Two types of the samples have been prepared: the reference sample (OPC, water and sand) and the sample of OPC containing 3 wt% of the graphene oxide (GO3). The nano composite was prepared according to the following procedure: first, grapheme oxide was suspended in distilled water and sonicated for 3 h until the homogeneous solution was obtained. Next, OPC was added to the mixture. In both samples water to cement ratio was kept the same (W/C = 0.6). At the end, the sand has been added. High resolution transmission electron microscopy (HRTEM, FEI Tecnai F30) and atomic force microscopy (AFM –Nanoscope V Multi Mode 8, Bruker) were used to study the morphology of GO. The presence of the functional groups have been clearly proved in XPS analysis. X-ray diffraction technique (X,Pert Philips Diffractometer) was employed to reveal the phase composition of the nanocomposite and the reference sample. Raman spectra were acquired on the in via Raman Microscope (Renishaw) at an excitation wavelength of 785 nm. IR absorption spectra were collected on the Nicolet 6700 FT–IR Spectrometer. FT-IR, Raman and XRD were collected after 10 min 165 min, 24 h and 7 days.

It is shown that graphene oxide additive in the amount of 3 wt% in cement results in significant enhancement of Young’s modulus. Furthermore, the kinetics of the hydration process is not strongly affected by the incorporation of graphene oxide into the cement. The morphology of the modified sample is almost the same as the reference material meaning the homogeneous distribution of the GO flakes in a cement matrix.

This route can pave a novel way to prepare cement paste with strongly enhanced mechanical properties. The results derived from this research and from other studies in the literature leave no doubt of the benefits received by inserting graphene oxide in cementitious products. Besides the increments obtained on mechanical properties of the composites, the experimental results indicate that the studied graphene oxide interact well with the hydration products of Portland cement and have the potential of being used in concrete.

Yu Shang, Dong Zhang , Chao Yang, Yanyun Liu, Yong Liu (2015) investigated the effects of graphene oxide (GO), silica fume (SF) and graphene oxide encapsulated silica fume (GOSF)on the rheological properties of cement pastes were investigated. It was found that the addition of GO into the cement caused a noticeable reduction in fluidity and increased rheological parameters. However, GOSF pastes had better fluidity and lower rheological parameters at a same dosage of SF, indicating that the addition of GO lowers the rheological parameters. A possible mechanism was proposed to explain the different effects of GO on cement pastes. The research provides a pathway to utilizing GO in cement based materials.

Ordinary Portland cement type 42.5R was main materials used in this research SF was used as the cementitious material. The SF particles were dispersed into ethanol solution via sonication. After30 min, 3-Aminopropyltrimethoxysilane (APS) was poured into the above solution and reaction for 24 h to obtain APS-modified silica fume. Then the suspension was washed with ethanol and deionized water. In order to evaluate the influence of GO sheets on the fluidity of the cement pastes, mini-slump test was carried out. After mixing, mixtures were poured into a mini-core (top diameter 36 mm, bottom diameter 60 mm, and height 60 mm) immediately. The rheological measurements of the pastes were performed by means of a NXS-11 rotary viscometer. The method was based on measuring the shear stress(s) along complete cycles containing the ascending and descending shear rates;15 rotating speeds are available. The initial and final speeds were 5.6 and360 rpm, respectively. In order to examine the influence of GO, SF, GOSF on the mechanical properties of cement pastes, compression tests were conducted on the specimens (2 cm × 2 cm× 2 cm). The specimens were tested at the age 28 days. The loading rate was set to 10 mm/min. At least three samples were repeated for each test.

They found that the fluidity of cement pastes is reduced with the increase of the dosage of GO, indicating that GO additives reduce the fluidity of cement paste. While the yield stress and plastic viscosity is increased with the increase of GO addition. The addition of SF or GOSF to cement paste leads to lower fluidity. However, the fluidity of GOSF pastes is higher than that of SF pastes, indicating that the addition of GO increase the fluidity of cement pastes. It is observed that GOSF pastes have a lower yield stress value and plastic viscosity at a same dosage of SF, indicating that GO lowers the yield stress value and plastic viscosity of cement pastes. The effects of GO and GO sheets encapsulating SF on the fluidity and rheological properties of cement pastes are different. This may because the synergetic effect of the shape effect of SF and the surface activity of GO.

Xiangyu Li, Asghar Habibnejad Korayem, Chenyang Li (2016) studied the dispersion of graphene oxide (GO) in simulated pore solution and cement paste. It was found that severe GO aggregation occurred in presence of divalent calcium ions in both pore solution and cement paste. However, the GO aggregates were not stable under shear mixing. After vigorous mixing, the massive GO aggregates split into medium-sized particles, ranging from few to several 100 lm. To improve the GO dispersion in cement paste, silica fume was used to mechanically separate individual GO nanosheets. The dispersion was then investigated using microstructure analysis and mechanical properties. The results showed that, with the addition of silica fume, the dispersion of GO nanosheets was greatly improved.

It was found that GO nanosheets aggregated in simulated pore solution if no prevention was employed. The aggregates formed a paper-like structure by cross-linking of calcium ions in the pore solution. Without prevention, GO aggregates were also found in cement paste. The size of GO aggregates could be reduced by shear mixing. If a sufficient amount of silica fume was used, GO dispersion in the cement paste was highly improved, with the silica fume mechanically separating the GO sheets and preventing aggregation. However, excess silica fume had a negative effect on the compressive strength of the GO reinforced cement paste by preventing interactions between the GO and cement hydration products. The results of this study may be used to establish guidelines for mix design and practical applications of GO reinforced cement composites which exhibit both improved GO dispersion and enhanced mechanical properties.

Baomin Wang, Ruishuang Jiang, and Zhenlin Wu (2016) investigated on the mechanical properties and microstructure of Grapahene Nanoplatelate-Cement composite. They showed that graphene nanoplatelets (GNPs) were dispersed uniformly in aqueous solution using methylcellulose (MC) as a dispersing agent via ultrasonic processing. Homogenous GNP suspensions were incorporated into the cement matrix to investigate the effect of GNPs on the mechanical behavior of cement paste. The optimum concentration ratio of GNPs to MC was confirmed as 1:7 by ultraviolet visible spectroscopy (UV-Vis), and the optical microscope and transmission electron microscopy (TEM) images displayed remarkable dispersing performance. The GNP–cement composite exhibited better mechanical properties with the help of surface-modified GNPs. The flexural strength of cement paste increased up to 15%–24% with 0.05 wt. % GNPs (by weight of cement). Meanwhile, the compressive strength of the GNP–cement composite increased up to 3%–8%. The X-ray diffraction (XRD) and thermal analysis (TG/DTG) demonstrated that the GNPs could accelerate the degree of hydration and increase the amount of hydration products, especially at an early age. Meanwhile, the lower porosity and finer pore size distribution of GNP–cement composite were detected by mercury intrusion porosimmetry (MIP). In addition, scanning electron microscope (SEM) analysis showed the introduction of GNPs could impede the development of cracks and preserve the completeness of the matrix through the plicate morphology and tortuous behavior of GNPs.

They used Ordinary Portland Cement according to Chines Standard and w/c ratio 0.35. They added 5 gm GNPs into 502mL aqueous solution with different MC concentration (ranging from 0.2 g/L to 1.0 g/L), and the suspensions were mechanically stirred for 10 min and treated for 20 min using a sonicator (operating frequency 40 KHz, power 180 W, bottom area 450 cm2).The treated GNP suspensions were tested to determine the best dispersion condition with regards to MC concentration. The amount of GNPs was fixed at 0.05 wt % by weight of cement. The uniform GNP suspension with weighted defoamer and dry cement were placed in an agitator kettle and the composites were mixed using a multispeed planetary mixer at low speed for 2 min and at high speed for 4 min. The mixture was then cast into 40 mm× 40 mm × 160 mm size steel molds and was vibrated for 1 min on an electric vibrator. After 24 h, all samples were demoulded and cured in 20℃ water. These samples were tested for flexural strength and compressive strength at 3 days, 7 days, 14 days and 28 days.

After that experimental investigation they reached that conclusion that the GNPs were treated using MC as a dispersant to improve their dispersibility in aqueous solution. The optimum MC to GNPs ratio of 7:1 by concentration was confirmed using UV-Vis absorbency, and the dispersing performance of GNPs was characterized by optical microscope and TEM. Consequently, the uniform GNP suspensions were used to reinforce the cement matrix as mixing water. The effect of GNPs on the mechanical properties and microstructure of cement based materials were investigated with the help of XRD patterns, thermal analysis, MIP, and SEM analysis. The incorporation of 0.05% GNPs by weight of cement can enhance the flexural strength by15%–24% and the compressive strength by 3%–8%. The XRD pattern and thermal analysis demonstrate that the degree of cement hydration has been promoted by GNPs, especially at an early age. Moreover, a more compact microstructure and finer pore size were detected in the GNP–cement composite using MIP and SEM analysis. However, more research is needed to improve the dispersibility of GNPs in cement matrix and the interfacial interaction between GNPs and the hydration products of cement. This study can provide a suitable method to investigate the two-dimensional nanomaterial cement composites.

Valles Romero Jose Antonio, Emilio Raymundo (2016) et al investigated on the fields of nanomaterials, which represent a valuable opportunity for developing compounds nanomaterials, such as graphene oxide. Five types of sample were prepared for the experimental investigation for both compression and flexural test. Concrete samples were prepared with graphene oxide in percentage of 0% and 25 to 6% by weight and graphene oxide formulate in a liquid base, hence it kept colour of concrete intact and has no interference in the construction process.

After the investigation, they reached to the conclusion, that nanoparticles of graphene oxide improves the mechanical properties of the concrete, both compressive and flexural strength, compressive strength increased with increasing graphene oxide content, up to maximum 5% at 28 days compared to the control sample (fc=350kg/cm2=34.32Mpa). And the flexural strength increased as the content of graphene oxide, peaking 4% after which decreased with further increases in the content of graphene oxide, the maximum increase was 62.2% relative to the sample control at 28 days, it was determined that the flexural strength and compressive strength influence the size of nanoparticles.

Shenghua Lv, Jia Zhang , Linlin Zhu, and Chunmao Jail (2016), had done experimental investigation on structural defects including cracks, hole, and disordered morphology which significantly affects their strength and durability. They had used graphene oxide nanosheets for doping, and they were able to control entire cement matrix to form an ordered microstructure consisting of polyhedron-like crystals and exhibit flower-like patterns. The cracks and holes in the cement matrix just about vanished. The compressive and flexural strengths as well as the parameters for the durability assessment of the corresponding cement composites improved compared with the control samples.

They had used the Polycarboxylate super plasticizers (PCs, with a content of 40% and a water-reducing rate of 32%), Portland cement (P.O. 42.5) and standard sand. The main chemicals used were concentrated sulfuric acid (H_2SO_4, 98%), potassium permanganate ($KMnO_4$), sodium nitrate ($NaNO_3$), and hydrogen peroxide (H_2O_2, 30%). All chemicals used are of reagent purity and not any treatment. And they had prepared GON/cement composite by uniformly mixing water, PCs, and GONs first, and then cement and sand via stirring. The weight ratio of the cement/water/PCs/GONs was 450:1350:160:0.9:0.09. The sample sizes were 40 mm×40 mm×160 mm and 100 mm×100 mm×400 mm, respectively. The specimens were removed from the mould after 24 h and cured in standard conditions until testing.

They had measured the chemical groups in the GONs by Fourier-transform infrared spectroscopy (FTIR; EQUINOX-55, Bruker, Ettlingen, Germany) and X-ray photoelectron spectroscopy (XPS;XSAM 800, Kratos, Manchester, UK). The microstructure and the size distribution of GONs were examined by atomic force microscopy (AFM; SPI3800N/SPA400, Seiko, Osaka, Japan) and a laser particle analyzer (NANO-ZS90, Zetasizer, Worcestershire, UK). X-ray diffraction (XRD; D/max2200PC, Rigaku, Osaka, Japan) was used to examine the crystalline. The microstructures of GON/cement composites were determined with a scanning electron microscope (SEM; S-4800, Hitachi, Tokyo, Japan).The elemental compositions were determined with an energy-dispersive X-ray spectrometer (EDS)(EDAX, Cassatt, SC, USA), which was coupled with the S-4800 SEM. The compressive strength was tested with a concrete compressive strength tester (JES-300, Wuxi, China) at a pressure increase rate of 1 MPa/s. The flexural strength of the GON/cement composites was determined using a concrete three-point flexural strength tester (DKZ-500, Wuxi, China) at a pressure increase rate of 0.25 MPa/s. The water penetration, the freeze thawing, and the carbonation experiment were carried out by GB/T5082-2009 (National Standard of China).

After that experimental study they have reached to the conclusion that GONs can be used to control the formation of Portland cement hydration products into polyhedron-like crystals and an aggregate-forming ordered microstructure with flower-like patterns. The research results indicate that polyhedron products can transform into flower-like patterns and further form ordered microstructures with defect-free structures. And these cement hydration products are easier to grow cracks and holes of the cement matrix. These results had major practical applications for the production of cement composites with high strength, high toughness, and long durability.

Xiangyu Li, Yan Ming Liu, Wen Gui Li, Chen Yang Li, Jay G. Sanjayan, Wen Hui Duan, Zongjin Li(2017) studied about the effects of graphene oxide (GO) agglomerates on the workability, hydration, microstructure, and compressive strength of cement paste. They have found that workability of cement paste reduces because of the presence of GO agglomerates, which entrap a large amount of water. They have used for their experimental investigation GO nanosheet, OPC and tap water, and w/c ratio 0.4.For workability test they have used the mini-slump test and for compressive and tensile strength test they prepared the cylindrical specimen and for measurement of degree of hydration they tested the non-evaporable test.

After the investigation they have reached to the following conclusions.

1) Incorporation of GO reduces the workability of cement paste, due to the formation of GO agglomerates by chemical cross-linking of GO nanosheets by calcium cations. Free water is reduced by these GO agglomerates which have high water entrapment capacity.

2) The hydration of cement paste is accelerated due to the seeding effect of GO agglomerates which provide nucleation sites because of their small particle size and large surface area.

3) The incorporation of GO refines the pore structure of cement paste due to the filling of large pores by GO agglomerates.

4) At 28 days, the incorporation of 0.04% by weight GO produced a 14% improvement in the compressive strength of cement paste. A threshold for GO content was found to be around 0.03% by weight of cement. When the GO content is below the threshold, the influence of incorporated GO is not significant. When the GO content exceeds the threshold, the incorporation of GO increases the compressive strength.

Qin Wang et al (2017) investigated to improve the rheological properties of cement paste with fly ash when GO is present whereby they have shown that fly ash can offset the reduction of fluidity by GO. The effect of fly ash was studied with two dosages of GO 0.01 wt% and 0.03 wt%. The yield stress and plastic velocity decreased with increase in fly ash. At 0.01 wt% of GO and 20 wt% fly ash, the yield stress of the paste decreased 85.81% and the plastic viscosity decreased 29.53% in comparison to the control sample i.e. with no fly ash or GO. At 0.03 wt% of GO and 20 wt% of fly ash, the yield stress of the paste is 50.33% lower and the plastic viscosity decreased slightly by 5.58%.The hysteresis area of the composite paste also decreased with the increase of fly ash. Meanwhile, the results indicated a good correlation between the fluidity and the plastic viscosity. The "ball" effect, grain size gradation and less water demand of fly ash can play an important role in improving the fluidity of the GO-cement systems. Moreover, GO can offset the delay in early-stage strength gain of fly ash-cement systems. When the dosage of fly ash is less than 15%, the compressive and flexural strength of fly ash-GO-cement composites are all higher than the control sample at 3,7 and 28 days.

Liulei Lu and Dong Ouyang (2017), have carried out an experimental study on effect of graphene oxide nanosheets concrete (GONC) additives on the properties of cement mortar and ultra-high strength concrete (UHSC). And they have got results that GONC were easy to prepare and graphene oxide nanosheet affected the mechanical properties of cement composites and fluidity of cement composite decreased with the increasing GONC content. Results indicated that using 0.01% by weight of cement GONCs caused a 7.82% increase in compressive strength after 28 days of curing. Moreover, adding GONCs improved the flexural strength and deformation ability, with the increase in flexural strength compared to that of compressive strength. They have got the morphology of the hardened cement paste and UHSC sample by field-emission scanning electron microscopy (FE-SEM). FE-SEM observations showed that the GONCs were well dispersed in the matrix and the bonding of the GONCs and the surrounding cement matrix was strong. Furthermore, FE-SEM observation indicated that the GONCs probably affected the shape of the cement hydration products.

They had used the Ordinary Portland cement type II 42.5R (C), silica fume (SF), and ground granulated blast-furnace slag (GGBS) in all mixtures. A polycarboxylate-based super plasticizer (PCs) was used in concrete mixtures for workability purposes. The fine aggregate (FA) used in this study was natural river sand with a fineness modulus of 2.79. The coarse aggregate (CA) was crushed granite with a maximum size of 20 mm. They were synthesized by a modified Hummers method and was well dispersed in water. The average size and thickness of GONCs are 100–1000 nm and ~0.7 nm and w/c 0.5.

Three mixtures of cement paste containing GONCs were prepared for FE-SEM observation. The GONCs were added in the amount of 0%, 0.05%, and 0.25% by weight of cement. Six mixtures of cement mortar were prepared by 450 g cement, 1350 g ISO standard sand, 225 g water, and a certain amount of GONCs. GONCs were added at levels of 0.00%, 0.01%, 0.03%, 0.05%, 0.08%, and 0.10% by weight of cement in the mortar mixtures. And three mixtures of the ultra-high strength concrete (UHSC) were prepared by mixing cementitious materials (cm), aggregates, GONC dispersions, water, and PCs and w/c ration 0.2. GONCs were added at levels of 0.00%, 0.01%, and 0.03% by weight of cement.

A. Mohammed; N. T. K. Al-Saadi; and R. Al-Mahaidi, M.ASCEA (2017) nanomaterial represented by graphene oxide and a series of cementitious materials was used in this work. Fresh properties such as setting time and flow ability were tested to ensure suitability for practical applications. Mechanical properties were tested in terms of compressive, tensile, and pull-off strengths. The experimental results proved the adequate performance of the innovative cement-based adhesive in terms of fresh properties and mechanical strength. The results show that the adhesive mixture has a pot life up to 120 min with a flow of 7.5%, and remarkably high 13.8 MPa tensile, 101 MPa compressive, and 1.2 MPa pull-off strengths.

Xiangy Li and Yan Ming Liu et al (2017) showed the effects of graphene oxide (GO) agglomerates on the workability, hydration, microstructure, and compressive strength of cement paste. The workability of cement paste was reduced because of the presence of GO agglomerates, which entrap a large amount of water. And they found the mini-slump diameter was reduced by 21% with the incorporation of 0.03% by weight of GO in cement paste. Also they found that the incorporation of GO, have much greater impact on micro pores than on large and small mesopores, at 28days, the incorporation of 0.04% by weight GO showed a 14% improvement in the compressive strength of cement paste, however the incorporation of GO below 0.03%, had no positive effects on compressive strength.

They had used Ordinary Portland Cement (OPC) confirmed by ASTM C150 and GO nanosheets. They had made a solution, where GO nanosheets were dispersed in water at the concentration of 4mg/ml. And the mean size of GO nanosheets was around 1µm. And they had prepared the cement paste with GO by a high-speed shear mixer.

They had tested four samples for determining the workability of cement paste. The GO content were 0%, 0.02%, 0.03% and 0.04% and w/c was 0.4 for every sample. And they had used TAM Air to examine the hydration heat development of cement paste by incorporating GO nanosheets and around 5g of cement paste. They had begun measurements every 3min after the mixing.

They had also prepared cement paste for microstructure characterization, namely mercury intrusion porosimetry (MIP) and SEM. After 28days of compressive strength tests, the sample were broken into 3-6 mm particles and then soaked in acetone to stop the hydration. Before MIP testing the samples were oven dried at 105ºC for 3 hours after which they had prepared the cylindrical specimens (23.5×47mm) for compressive strength test.

They had concluded that incorporating GO reduces the workability of cement paste, due to the formation of GO agglomerates by chemical cross linking of GO nano-sheets by calcium cations as a result free water was reduced by these GO agglomerates which have high water entrapment capacity. Secondly the hydration of cement paste was accelerated due to the seeding effect of GO agglomerates which provide nucleation sites because of their small particle size and large surface area.

A. Mohammed, J.G. Sanjayan, A. Nazari, N.T.K. Al-Saadi (2018) have carried out an experimental study in order to enhance the resistance of cement matrix against carbon dioxide attack using graphene oxide. They added Graphene oxide to cement mix sufficient amount of water to produce cementitious matrix. Visual colour change, porosity and compressive strength tests were performed to investigate

effect of the carbonation on cementitious materials. They have got the result that shows very low carbonation depth in the graphene oxide cement mix (Refer Fig 2.3). After 18 months, the plain cement mixture was nearly fully carbonated compared to only about 4 mm for the GO mix. This significant result can be attributed to the influence of interlocking of GO to different anionic and cationic ions.
They concluded that one important role of GO is enhancement the microstructure and reduction of porosity which results in slowing carbonation rate by restriction movement of CO_2 molecules into cementitious material. Another factor is the interlocking of calcium and carbonate ions which is found to be very effective in hindering the harmful reaction of carbonation.

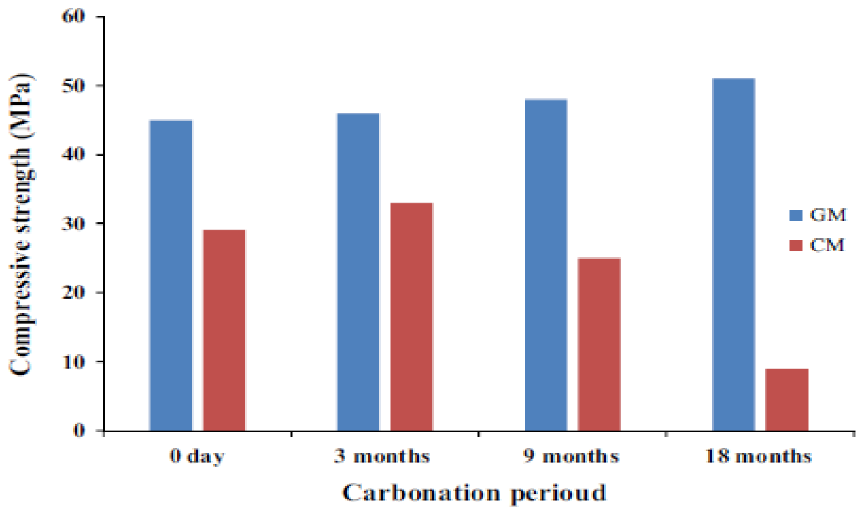


Fig 2.3 Compressive strength for different carbonation

2.2 SCOPE OF RESEARCH

Based on the review of literature on the application of Graphene Oxide in cement mortar, it has been found that most of the study on GO addition have been made on ordinary portland cement. No study have been made on cement mortar with PPC. In all cases they added GO in cement mortar after ultrasonication. So, the scope of the research is to study the effects of various percentage of GO in cement mortar using PPC and OPC also. A comparison on the properties of cement mortar with different percentage of GO is made for OPC and PPC. The study has been made to determine the optimal GO addition depending on mixture proportion and type of cement.

CHAPTER-3

EXPERIMENTAL INVESTIGATION

EXPERIMENTAL INVESTIGATION

This chapter presents the details of experimental investigation on the effect of addition of graphene oxide in cement mortar. The experimental studies were conducted mainly at the Concrete Technology and Structural Engineering Laboratory of Civil Engineering Department, Jadavpur University, Kolkata, India. The details of all materials which are used in this study are presented in section 3.1. The next sections include the detail of experimental programme (3.2) to study the addition of graphene oxide in cement mortar in different percentage (by weight) of cement.

3.1 MATERIALS

3.1.1 Cement

Two types of cement used for the experimental study are (a) Ordinary Portland Cement (OPC) having 28 days compressive strength of 50.12 MPa and (b) Portland Pozzolana Cement (PPC) having 28 days compressive strength of 34.08 MPa. The chemical analysis report of OPC and PPC are given in table 3.1 and 3.2.

Table 3.1- Chemical composition of Ordinary Portland Cement.

	CaO	SiO_2	Al_2O_3	Fe_2O_3	K_2O	MgO	Na_2O	SO_3	LOI
% (Weight)	63.9	19.9	4.7	3.4	0.5	1.3	0.2	2.6	3.0

Table 3.2- Chemical composition of Portland Pozzalona Cement.

	CaO	SiO_2	Al_2O_3	Fe_2O_3	K_2O	MgO	Na_2O	SO_3	LOI
% (weight)	42.6	30.4	8.7	5.3	0.7	1.6	0.3	2.5	3.3

3.1.2 Fine Aggregate

Fine aggregate used for the study was available river sand having specific gravity 2.66 and fineness modulus 2.99.

3.1.3 Graphene Oxide (GO)

Graphene Oxide is a recently invented carbon based 2D nanoplane material. GO act as a nano-reinforcement in cement based composites. The Graphene Oxide was collected from **Ad-Nano Technologies Private Limited.** The mean size of GO nanosheets is around 5 to 10 μm having surface area 450m^2 /gm. In the GO-water solution, GO nanosheets were dispersed in water at the concentration of 5 mg/ml. The major functional groups on the surface of GO are –OH and –COOH. The technical parameters of GO is provided in table 3.3 as per Ad-Nano Technologies Private Limited.

Table 3.3 Technical Parameters of Graphene Oxide

Graphene Oxide	Description
Purity	>99%
Numbers of Layers	1-3 layers
Average Thickness(z)	0.8-1.6 nm
Average Lateral Dimension(X&Y)	5-10 μm
Surface Area	450m^2/g
Carbon %	66%
Oxygen %	32%
Others %	1%

3.2 MIXTURE PROPORTION

The mixture proportion of cement mortar (with/without GO) has been fixed with trials in the laboratory. The low percentage of GO addition has considered based on previous literature. The GO has been added both in OPC and PPC based mortar separately. Total twenty mixes have been prepared based on different cement sand ratio, types of cement and different percentage of GO addition (0.00% 0.03%, 0.04%, 0.05% and 0.06%). The mix proportions of different mixtures are shown in table 3.4.

Table 3.4 - Mix proportion for different tests of cement mortar with GO and without GO.

Mix No	Cement	Type of Mortar	Cement Sand Ratio	Water Cement Ratio	GO % by weight of cement
A0 (Control)	OPC	OPC Mortar	1:2	0.45	0
A3		GO-OPC Mortar			0.03
A4					0.04
A5					0.05
A6					0.06
B0 (Control)	OPC	OPC Mortar	1:3	0.45	0
B3		GO-OPC Mortar			0.03
B4					0.04
B5					0.05
B6					0.06
C0 (Control)	PPC	PPC Mortar	1:2	0.45	0
C3		GO-PPC Mortar			0.03
C4					0.04
C5					0.05
C6					0.06
D0 (Control)	PPC	PPC Mortar	1:3	0.45	0
D3		GO-PPC Mortar			0.03
D4					0.04
D5					0.05
D6					0.06

3.3 SPECIMENS

The standard mortar cube specimen size of 70.6mm x 70.6mm x70.6mm has been prepared for compressive strength test, Ultrasonic Pulse Velocity (UPV) test and sorptivity test. For each mix, 12 cube specimens were prepared for compressive strength and sorptivity test at 3, 7 and 28 days. Before the determination of compressive strength on mortar cubes, Ultrasonic Pulse Velocity were performed. Similarly for each mix, 3 prism specimens were prepared for flexural strength at 28 days.

3.3.1 Specimen Preparation

To prepare GO based cement mortar, at first a certain percentage of water from total water are mixed with GO in a ratio 200:1 by weight. As an example, for a particular cement mortar mix of water cement ratio 0.45, cement sand ratio 1:3 and GO addition in the mix 0.06% by weight of cement (for 3 numbers 70.6mm x 70.6mm x 70.6mm cube size). The amount of cement required is 550 gm. So, the amount of GO required = 0.06*550/100 = 0.33 gm. Water required = 0.45*550 = 247.5 gm. As per water GO ratio 200:1, for 0.33gm GO the amount of water required = 0.33*200 = 66 gm. The remaining water = 247.5-66 = 181.5 gm. Now this 0.33 gm GO and 66gm water are mixed together and then the resulting mixture is ultra-sonicated using a UP100H ultrasonic processor for 45 minutes to one hour to ensure uniform suspension. Then the remaining water (181.5gm) is added to the sonicated mixture. On the other hand, cement and sand are mixed together in the required proportion.

The cement sand mixture and liquid (GO-water suspension) are placed in a plastic bowl and they are mixed together with a trowel. The time of mixing was in between 3 to 4 minutes to obtain uniform colour of mixture.

In assembling the moulds ready for use, cover the joints between the halves of the mould with a thin film of petroleum jelly and apply a similar coating of petroleum jelly between the contact surface of the bottom of the mould and base plate in order to ensure that no water escapes during compaction.

Each mould is filled by three layers of cement mortar of equal thickness. Each layer is compacted by a tamping rod with 25 blows to eliminate entrapped air and honey combing. After compacting the third layer cut the excess mortar and smoothen the top face with a trowel.

The test specimens were stored in the laboratory in moist air of at least 90 percent relative humidity and at a temperature of 27 +2^{0}C for 24 +1/2 hours from the addition of water to the dry ingredients.

After demoulding, the cube specimens were submerged in clean fresh water for water curing and keep there until taken out just prior to test. The sample preparation methods for all samples are similar.

The flow chart of cement mortar preparation for different tests of GO based cement mortar are shown in Fig 3.1.

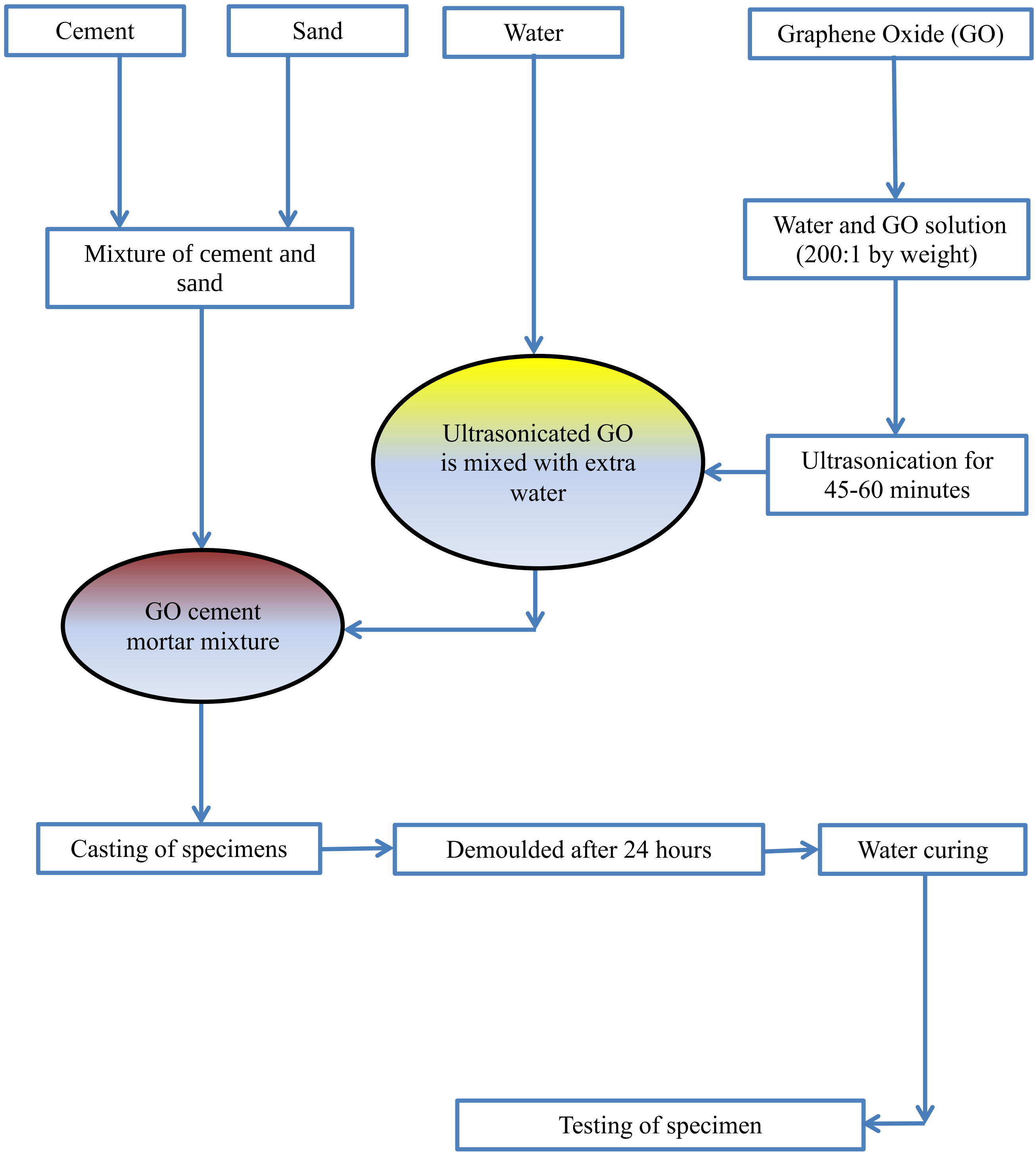


Fig 3.1 Flow chart of sample preparation

3.4 EXPERIMENTAL PROGRAMME

The following tests were conducted mainly at the Concrete Technology and Structural Engineering Laboratory of Civil Engineering Department, Jadavpur University, Kolkata, India are as follows.

1) Flow test on fresh mortar, 2) Compressive strength test, 3) Flexural strength test, 4) Ultrasonic pulse velocity (UPV) test, 5) Sorptivity test, 6) X-ray Diffraction (XRD) analysis and 7) Mercury Intrusion Porosimetry test.

Details of the testing procedure are presented in the next section.

3.4.1 Flow Test

Flow table test was conducted as per IS 4031(part-7) to assess the workability of fresh cement mortar with different percentage of GO addition. A miniature slump cone was employed with top diameter 70 mm, bottom diameter 100 mm, height 50 mm (Refer Fig 3.2 and 3.3). The slump-cone was placed on the center of flow table and filled with fresh mortar. The fresh mortar was tamped down with a spatula to ensure compaction. The slump cone was removed vertically to ensure no lateral disturbance. Immediately the flow table was dropped from a height of 12.5 mm by 25 times in 15 s and then base diameter of mortar mass was measured. The flow is the resulting increase in average base diameter of the mortar mass (measured on at least four diameters at approximately equally spaced intervals) expressed as a percentage of the original base diameter.

Fig 3.2 Mini Slump-Cone with flow table

Fig 3.3 Flow measurement

3.4.2 Compressive Strength Test

To examine the influence of addition of GO on the mechanical properties of cement mortar, compressive strength tests were conducted using standard mortar cube specimen size of 70.6mm x 70.6mm x70.6mm (Refer Fig-3.4). The cube specimens were tested at 3days, 7days and 28 days after casting and water curing to determine the compressive strength of both types of cement mortar (with GO / without GO). The maximum load was determined by compressive strength testing machine. Compressive strength is calculated by dividing the maximum load by the original cross sectional area of the specimen in a compression test.

3.4.3 Flexural Strength Test

The flexural strength test was conducted following ASTM C293 on 50mm x 50mm x 200mm size beams by center point load test (Refer Fig-3.5). The displacement control rate was 1.25 mm/min and the capacity of the flexural testing machine was 10KN.

The flexural strength was calculated using the following formula:

$f = 3WL/2bd^2$…………. (1)

Where f is the flexural strength (MPa),

W is the failure load (Newton),

L is the span between two supporting point (150mm),

b and d are the width and depth of the specimens (mm), respectively.

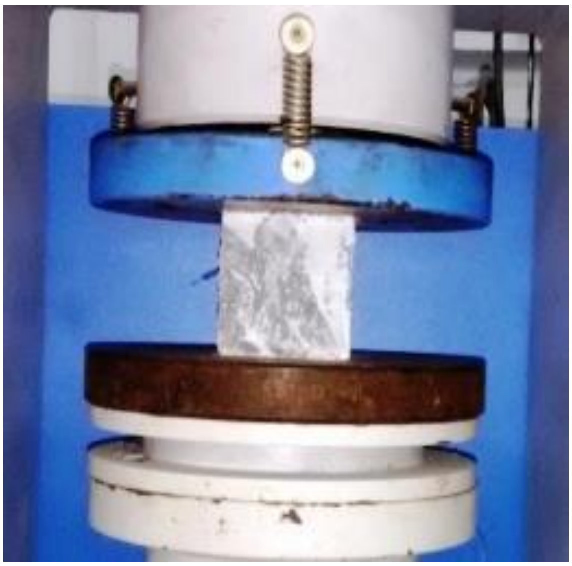


Fig 3.4 Compressive Strength Test by Compression Testing Machine

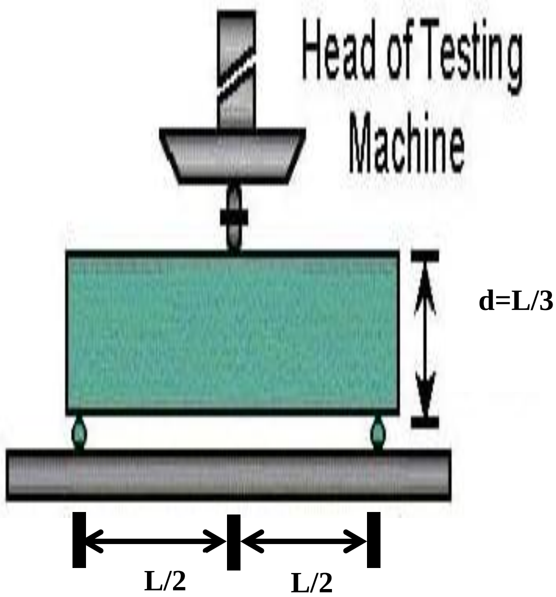


Fig 3.5 Center Point Load Test

3.4.4 Ultrasonic Pulse Velocity (UPV) Test

The ultrasonic pulse velocity test has been conducted as per IS 13311 (Part 1):1992 by using 70.6mm x 70.6mm x 70.6mm size mortar cube to measure the followings : a) The homogeneity of the mortar b) The presence of cracks, voids and other imperfections c) The quality of one element of mortar in relation to another. In this test method, the ultrasonic pulse is produced by the transducer which is held in contact with one surface of the mortar cube under test. After traversing a known path length Q in the cube specimen, the pulse of vibrations is converted into an electrical signal by the second transducer held in contact with the other surface of the cube and an electronic timing circuit enables the transit time (T) of the pulse to be measured. The pulse velocity (V) is given by: $V = L/T$. Once the ultrasonic pulse impinges on the surface of the mortar cube, the maximum energy is propagated at right angles to the face of the transmitting transducer and best results are, therefore, obtained when the receiving transducer is placed on the opposite face of the mortar cube (direct transmission or cross probing).The test was conducted before the compressive strength test.

3.4.5 Sorptivity Test

Sorptivity test was performed as per ASTM C1585-04. The purpose was to determine the rate of absorption of water by unsaturated mortar. Sorptivity is a function of the increased mass of a specimen resulting from absorption of water through one surface is exposed to water with respect to time. The sorptivity can be determined by the measurement of the capillary rise absorption rate on reasonably homogeneous material. Tap water was used as a test fluid. The cement mortar cube specimens size 70.6mm x 70.6mm x 70.6mm after casting and demoulding were immersed in water for 28 days curing. Then the specimens were dried in oven at a temperature of 50 ± 2°C and relative humidity of 80% for 3 days. Then the specimens were cooled. In order to ensure unidirectional flow through the specimen without any influence of wicking action the specimens were sealed on all sides with non-absorbent coating other than the exposure face. The samples were weighed and their weights were recorded to the nearest 0.01gm. A support consisting of iron net was placed in the bottom of container to ensure that exposure to water was even across the exposed surface. The specimen was placed at top of this support and the container was gently filled with tap water until it reached a level approximately 1 to 3 mm above the level of the exposed surface (Refer Fig 3.6). The weight of the specimens were recorded at intervals of 60 seconds, 5, 10, 20, 30, and 60 minutes, 2, 3, 4, 5, and 6 hours and 1, 2, 3, 4, 5, 6, 7 and 8 days. During weighing surface water on the specimen was wiped off with a dampened tissue and each weighting operation was completed within 30 seconds.

Sorptivity (S) is a material property which characterizes the tendency of a porous material to absorb and transmit water by capillarity. The cumulative water absorption (per unit area of the inflow surface) increases as the square root of elapsed time (t)

I $=S\sqrt{t}$ Therefore S$=I/\sqrt{t}$

Where S = sorptivity in mm $/\sqrt{}$sec.

t = elapsed time in seconds.

I = ΔW/Ad = Absorption in mm.

ΔW = change in weight = W2-W1

W1 = oven dry weight of specimen in grams.

W2 = weight of specimen after different time intervals like 60 seconds, 5, 10, 20, 30 and 60 minutes etc.in grams.

A= surface area of the specimen through which water penetrated in mm^2.

d= density of water in gm/mm^3

Fig 3.6 Sorptivity Test

3.4.6 XRD analysis

Samples from cement mortar (with/without GO) specimens for XRD scanning were collected from the central part of broken specimens after compressive strength tests and then crushed to a fine powder. The powder was sieved using 90-μm mesh. The powder samples were sealed in plastic bags and stored for the XRD scan. The scan angle was from 10° to 80°

3.4.7 MIP analysis

This test method is used to determine the volume and pore size distribution in control cement mortar (without GO) and GO based cement mortar. Quantachrome make Poremaster 60 was used to conduct the test by applying different levels of pressure to a sample immersed in mercury. After 28 days compression tests, the cement mortar specimens were broken into 3 to 6-mm particles size for MIP test.

CHAPTER-4

RESULTS AND DISCUSSION

4.1 RESULT AND DISCUSSION

In this chapter, all the experiment results on the effect of graphene oxide addition on the overall behavior of cement mortar are presented. The graphene oxide is used in cement mortar by 0.03, 0.04, 0.05 and 0.06% by weight of cement.

Table 4.1 and 4.2 show the results of flow table tests of mortar with OPC with different percentage of GO for cement sand ratio of 1:2 and 1:3 respectively. The results are also given in Fig 4.1 and Fig 4.2. Similarly, Table 4.3 and Table 4.4 show the flow table test results for mortar with PPC and different percentage of GO addition for cement sand ratio of 1:2 and 1:3 respectively. The results are also presented in Fig 4.3 and Fig 4.4. The results indicate that the workability cement mortar in terms of final flow diameter decreases with respect to control sample (without GO) with the addition of different percentage of GO. It is noted that when the percentage of GO addition was 0.03% by weight of cement, the final flow diameter decreased suddenly with respect to control cement mortar (without GO) for both OPC and PPC based mortar. With the further increase of GO content up to 0.06%, the final flow diameter is not reduced significantly and the behaviour is not uniform. Fig 4.1 and 4.3 shows that the final flow diameter increases when the GO content 0.04% and 0.05% for cement sand ratio 1:2 with both OPC and PPC, although both flow values are less than control mortar (without GO). Similar results were reported by Shang et al. [15] and Xiangyu Li et al. [17]: the incorporation of GO into cement paste caused a noticeable reduction in fluidity and an increase in viscosity. The possible reasons of flow reduction that the GO has a large surface area, allowing it to absorb free water on its surface, leading to the decrease in fluidity [17, 18]. Another reason of flow reduction may be due to the formation of graphene oxide agglomerates/ettringite which has large water entrapment capacity as observed from XRD analysis. Similar observations were also obtained from previous literatures [14, 20]. Again, further increase of GO content in cement mortar, the final flow diameter decreases. The maximum reduction of flow was 22.54% for GO-OPC mortar and 18.57% for GO-PPC mortar due to the addition of 0.03% GO having cement and sand ratio 1:2. Similar flow reduction was observed by 16.54% for GO-OPC mortar and 8.98% for GO-PPC mortar with the incorporation of 0.06% GO having cement and sand ratio 1:3.

The results of compressive strength of cement mortar (cement sand ratio 1:2) using OPC with different percentage of GO addition are presented in Table 4.5 and Fig 4.5. As usual, the compressive strength of each mortar mix (with/without GO) increases with the increase in curing age. It is noted that the compressive strength of GO based cement mortar (cement sand ratio 1:2) increases with the increases of GO addition up to 0.05% at all ages.

Table 4.1 Flow table test results for OPC mortar with GO

Mix with Cement : Sand = 1:2	GO (%)	Initial Flow Diameter (mm)	Final Flow Diameter (mm)	Change of Flow Diameter (%)
A0 (Control)	0.00	100	142	….
A3	0.03	100	110	-22.54
A4	0.04	100	112	-21.13
A5	0.05	100	120	-15.49
A6	0.06	100	115	-19.01

Table 4.2 Flow table test results for OPC mortar with GO

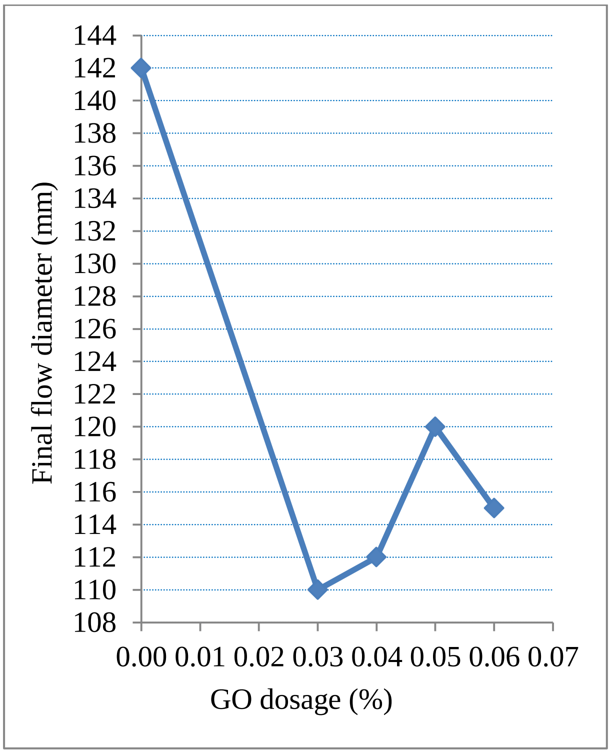
Mix with Cement : Sand = 1:3	GO (%)	Initial Flow Diameter (mm)	Final Flow Diameter (mm)	Change of Flow Diameter (%)
B0 (Control)	0.00	100	130	….
B3	0.03	100	115	-11.54
B4	0.04	100	111	-14.62
B5	0.05	100	109.5	-15.77
B6	0.06	100	108.5	-16.54

Table 4.3 Flow table test results for PPC mortar with GO

Mix with Cement : Sand = 1:2	GO (%)	Initial Flow Diameter (mm)	Final Flow Diameter (mm)	Change of Flow Diameter (%)
C0 (Control)	0.00	100	140	….
C3	0.03	100	114	-18.57
C4	0.04	100	118	-15.71
C5	0.05	100	130	-7.14
C6	0.06	100	119	-15.00

Table 4.4 Flow table test results for PPC mortar with GO

Mix with Cement : Sand = 1:3	GO (%)	Initial Flow Diameter (mm)	Final Flow Diameter (mm)	Change of Flow Diameter (%)
D0 (Control)	0.00	100	128	….
D3	0.03	100	123	-3.91
D4	0.04	100	120	-6.25
D5	0.05	100	118	-7.81
D6	0.06	100	116.5	-8.98



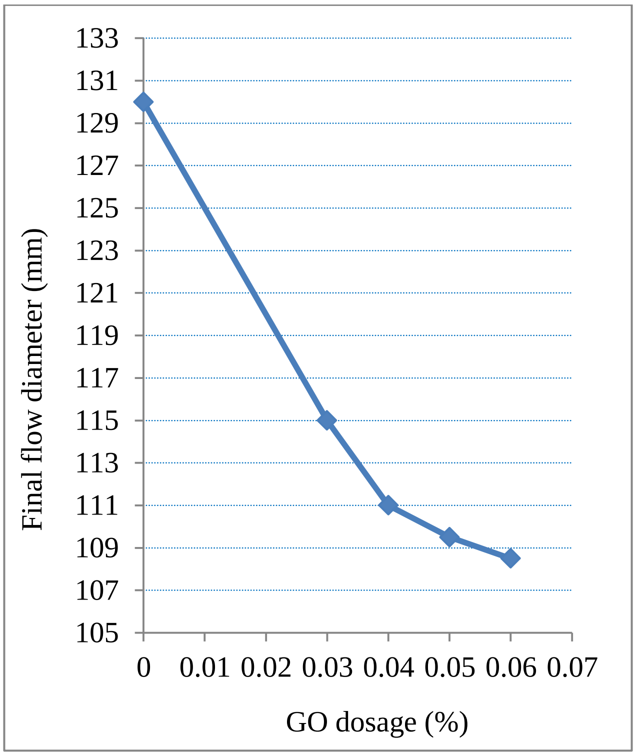
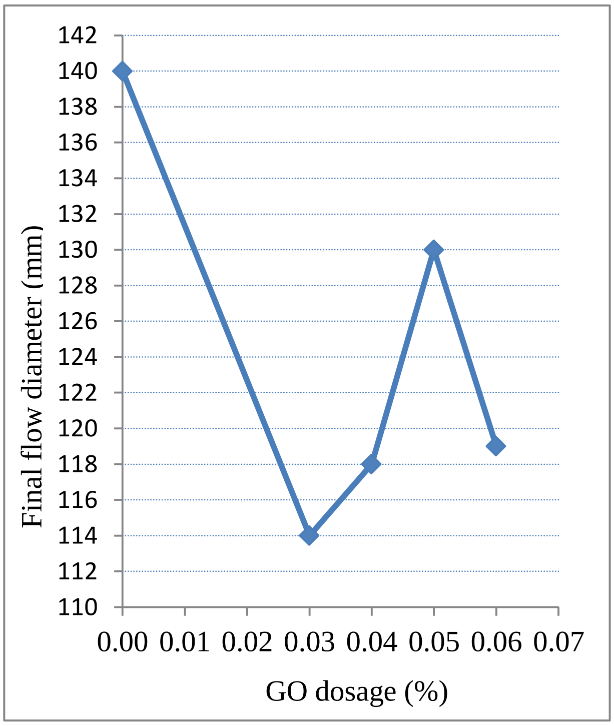


Fig 4.1 Flow table test results GO-OPC mortar with cement sand ratio 1:2

Fig 4.2 Flow table test results GO-OPC mortar with cement sand ratio 1:3



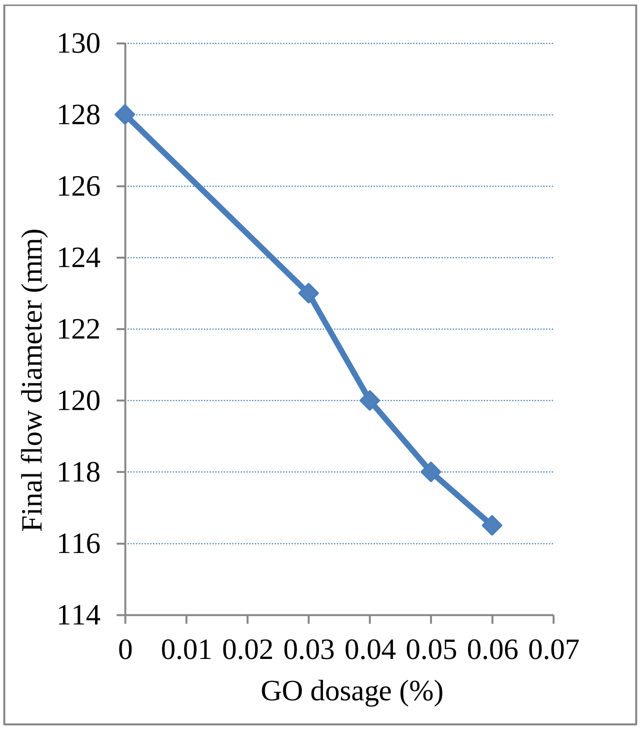


Fig 4.3 Flow table test results of GO-PPC mortar with cement sand ratio 1:2

Fig 4.4 Flow table test results of GO-PPC mortar with cement sand ratio 1:3

With further addition of GO (0.06%) the strength reduces drastically below the control mix (without GO) at 3, 7 and 28 days. However, the reduction in compressive strength is comparatively less at 3 days compared to that of 7 and 28 days. It is also noted that the strength improvement due to GO addition (up to 0.05%) is more at 3 days compared to 7 and 28 days. The improvement in compressive strength with GO addition is mainly due to the formation of more C-S-H gel than control mix (without GO). Similar observations were also reported in previous literatures [20, 21]. The XRD analysis of cement mortar with 0.05% GO and control mortar (without GO) indicates the development of more C-S-H gel in GO based cement mortar than control mortar (Refer Fig 4.17 and Fig 4.18). Further the pore structure in cement mortar is modified by GO agglomerates. However, more studies are needed to investigate the reduction in strength due to higher percentage of GO addition.

Table 4.5: Compressive strength of GO based mortar using OPC with cement sand ratio 1:2

Mix No	GO (%)	3 Days		7 Days		28 Days	
		Compressive Strength (MPa)	Strength change (%)	Compressive Strength (MPa)	Strength change (%)	Compressive Strength (MPa)	Strength change (%)
A0 (Control)	0	19.04	….	34.95	….	50.12	….
A3	0.03	21.23	11.5	38.85	11.16	54.58	8.9
A4	0.04	23.71	24.53	41.18	17.83	56.83	13.39
A5	0.05	28.21	48.16	43.94	25.72	60.72	21.15
A6	0.06	19.43	2.05	27.15	-22.32	47.83	-4.57

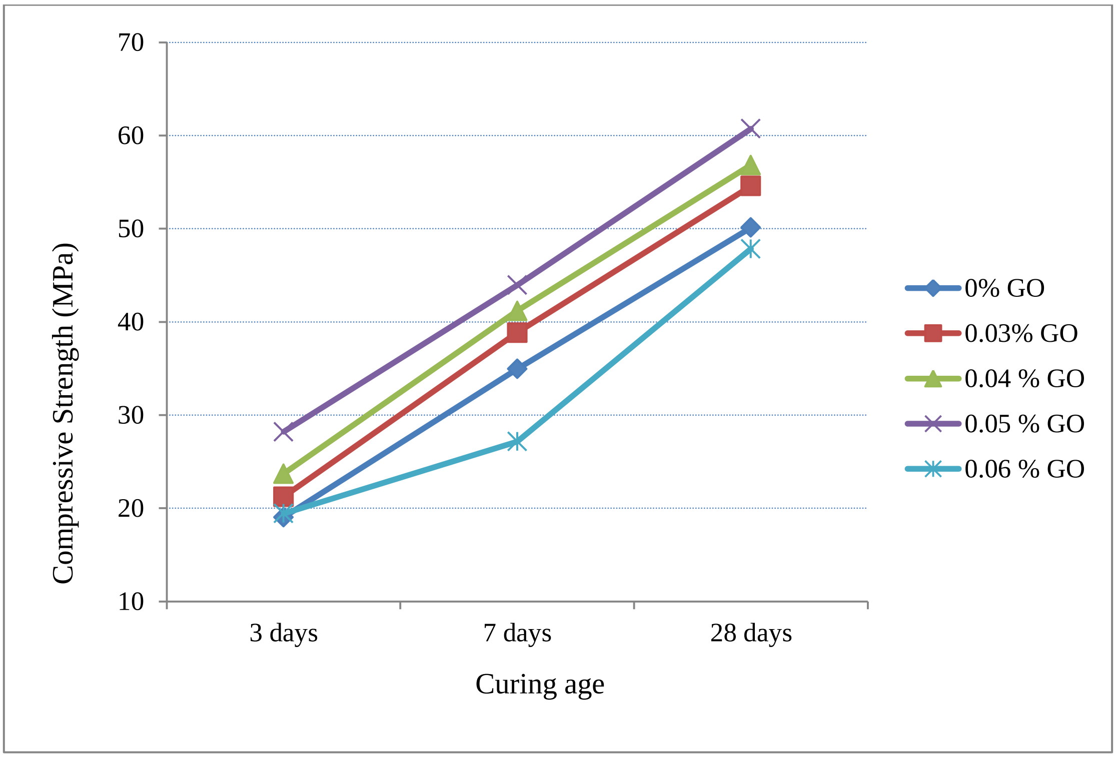


Fig 4.5 Compressive strength of GO based mortar using OPC with cement sand ratio 1:2

Table 4.6 and Fig 4.6 shows the compressive strength of cement mortar (cement sand ratio 1:3) using OPC with different percentage of GO addition at 3, 7 and 28 days respectively. The strength behaviour of mortar with GO addition for cement sand ratio of 1:3 is almost similar to that of cement sand ratio 1:2. However, the maximum compressive strength of GO based cement mortar for cement sand ratio of 1:3 was obtained at GO addition of 0.04% compared to 0.05% GO addition for mortar cement sand ratio of 1:2. Even the strength improvement with respect to control mix (without GO) at optimal GO addition (0.04%) in cement sand ratio of 1:3 is less compared to that of optimal GO addition (0.05%) in cement sand ratio of 1:2 at all ages. Therefore, it may be concluded that the mix proportion has an important role in determining optimal GO addition in terms of strength.

Table 4.6: Compressive strength of GO based mortar using OPC with cement sand ratio 1:3

Mix No	GO (%)	3 Days		7 Days		28 Days	
		Compressive Strength (MPa)	**Strength change (%)**	**Compressive Strength (MPa)**	**Strength change (%)**	**Compressive Strength (MPa)**	**Strength change (%)**
B0 (Control)	0	12.78	….	18.43	….	28.6	….
B3	0.03	14.8	15.81	20.24	9.82	30.05	5.07
B4	0.04	17.09	33.74	23.34	26.64	32.48	13.57
B5	0.05	15.94	24.73	20.97	13.78	31.5	10.14
B6	0.06	13.4	4.85	19.17	4.02	26.75	-6.47

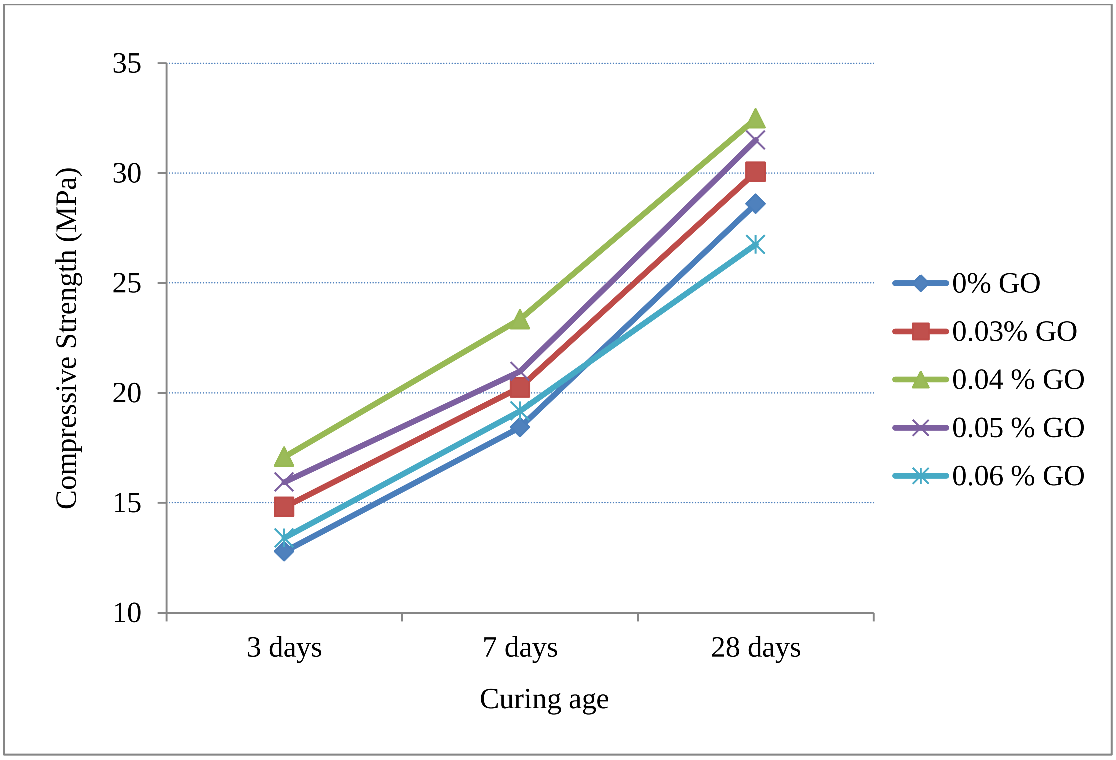


Fig 4.6 Compressive strength of GO based mortar using OPC with cement sand ratio 1:3

The results of cement mortar using PPC instead of OPC with different percentage of GO addition are shown in Table 4.7 & Fig 4.7 for cement sand ratio of 1:2 and in Table 4.8 & Fig 4.8 for cement sand ratio of 1:3. The overall strength behaviour of PPC based cement mortar with GO addition is almost similar to that of OPC based cement mortar. However, the optimum percentage of GO addition in PPC based mortar is 0.04% in case of cement sand ratio of 1:2 and 0.05% in case of cement sand ratio of 1:3. This optimal percentage of GO addition in PPC is opposite to that of OPC based mortar. The strength improvement at optimal GO addition is also more at 3 and 7 days compared to 28 days.

Table 4.7 Compressive strength of GO based mortar using PPC with cement sand ratio 1:2

Mix No	**GO (%)**	**3 Days**		**7 Days**		**28 Days**	
		Compressive Strength (MPa)	**Strength change (%)**	**Compressive Strength (MPa)**	**Strength change (%)**	**Compressive Strength (MPa)**	**Strength change (%)**
C0 (Control)	0	17.82	….	24.44	….	34.08	….
C3	0.03	18.71	5	25.32	3.6	36.11	5.96
C4	0.04	22.52	26.37	27.79	13.71	38.21	12.12
C5	0.05	20.08	12.68	27.68	13.26	37.81	10.94
C6	0.06	18.74	5.16	22.46	-8.1	31.18	-8.51

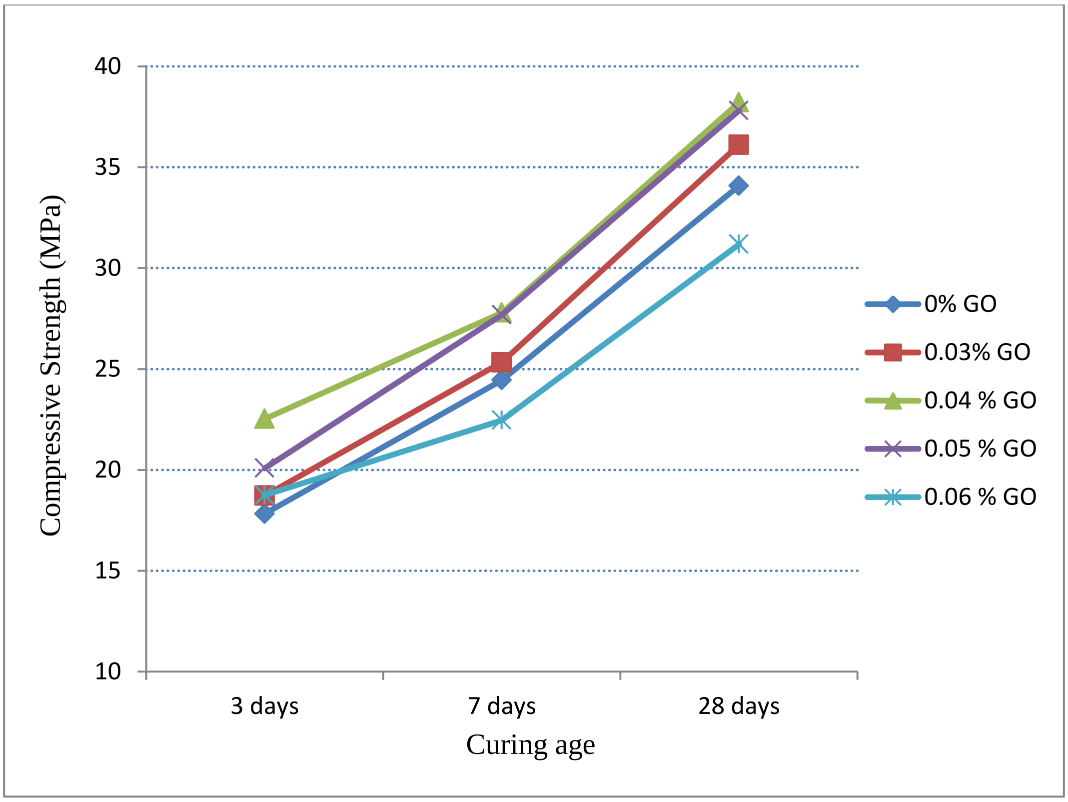


Fig 4.7 Compressive strength of GO based mortar using PPC with cement sand ratio 1:2

Table 4.8: Compressive strength of GO based mortar using PPC with cement sand ratio 1:3

Mix No	GO (%)	3 Days		7 Days		28 Days	
		Compressive Strength (MPa)	Strength change (%)	Compressive Strength (MPa)	Strength change (%)	Compressive Strength (MPa)	Strength change (%)
D0 (Control)	0	15.15	….	21.15	….	30.2	….
D3	0.03	16.98	12.09	21.9	3.56	31.98	5.89
D4	0.04	19.67	29.81	24.39	15.32	32.97	9.16
D5	0.05	20.1	32.67	25.38	20.02	36.49	20.84
D6	0.06	18.13	19.64	23.77	12.42	33.11	9.65

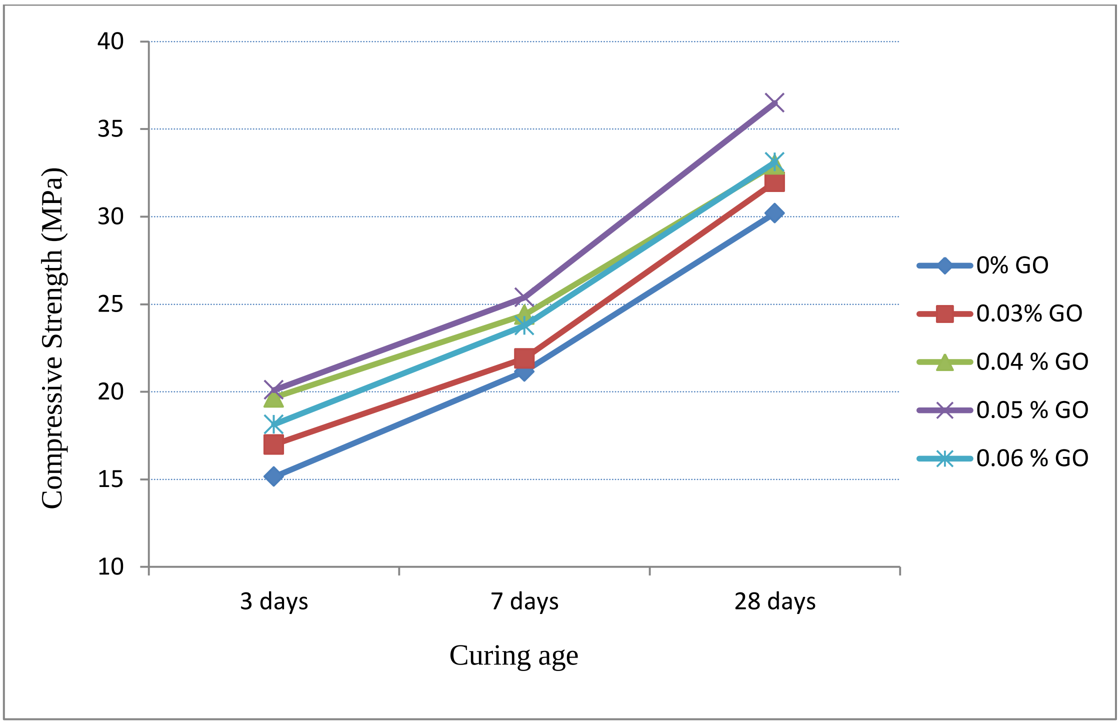


Fig 4.8. Compressive strength of GO based mortar using PPC with cement sand ratio 1:3

The experimental results of the flexural strength of GO based cement mortar using OPC are presented in Table 4.9 & Fig 4.9 for cement sand ratio of 1:2 and in Table 4.10 & Fig 4.10 for cement sand ratio of 1:3. It is noted that the flexural strength of GO based cement mortar increases due to the addition of different percentage of GO (from 0.03% to 0.06% by weight of cement) with respect to control mortar (without GO). The optimum percentage of GO addition to achieve maximum flexural strength of OPC based mortar is 0.05% in case of cement sand ratio of 1:2 and 0.04% in case of cement sand ratio of 1:3.The results are similar to that of compressive strength at 28 days of similar mortar. The maximum improvement in flexural strength was 28.19% for cement sand ratio of 1:2 and 15.22% for cement sand ratio of 1:3 at their corresponding optimal addition of GO.

Table 4.9 Flexural strength of GO based mortar using OPC with cement sand ratio 1:2

Mix No	Graphene Oxide (%)	Flexural Strength (MPa)	
		28 Days Strength	Strength Improvement (%)
A0 (Control)	0.00	4.93	…
A3	0.03	5.56	12.78
A4	0.04	6.03	22.31
A5	0.05	6.32	28.19
A6	0.06	5.61	13.79

Table 4.10 Flexural strength of GO based mortar using OPC with cement sand ratio 1:3

Mix No	Graphene Oxide (%)	Flexural Strength (MPa)	
		28 Days Strength	Strength Improvement (%)
B0 (Control)	0.00	4.29	…
B3	0.03	4.71	9.90
B4	0.04	4.94	15.22
B5	0.05	4.82	12.47
B6	0.06	4.66	8.70

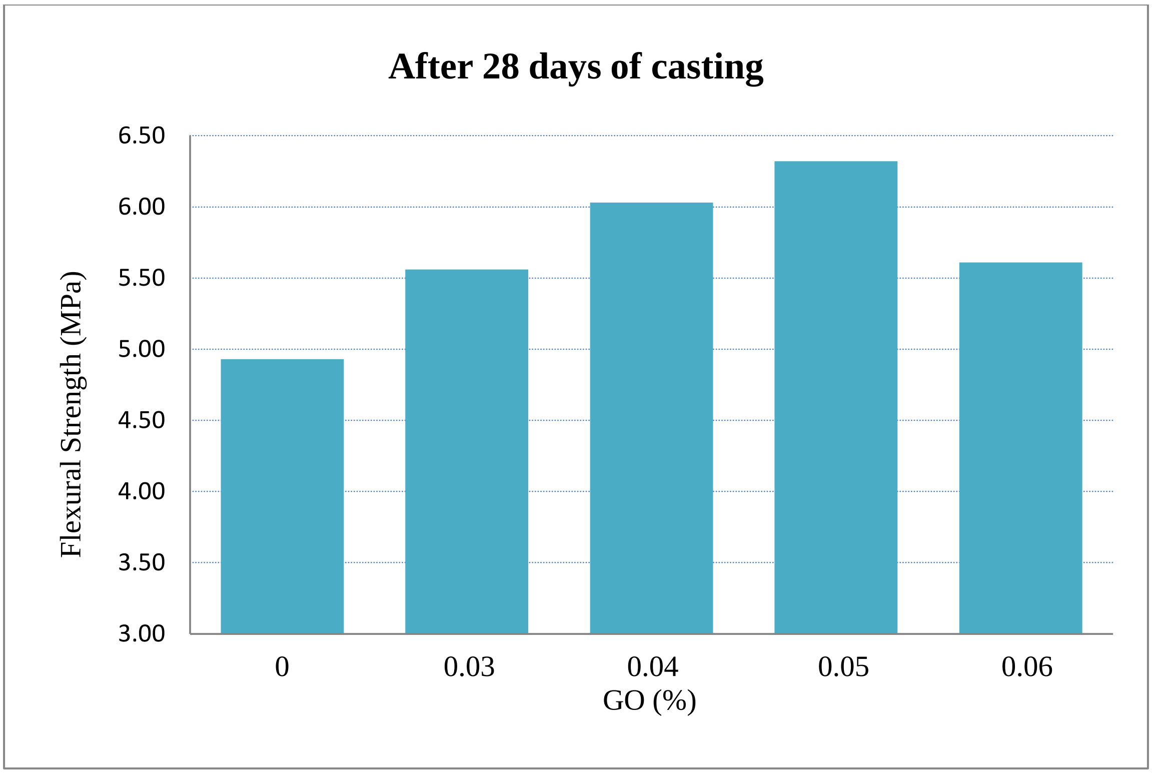


Fig 4.9 Flexural strength of GO based mortar using OPC with cement sand ratio 1:2

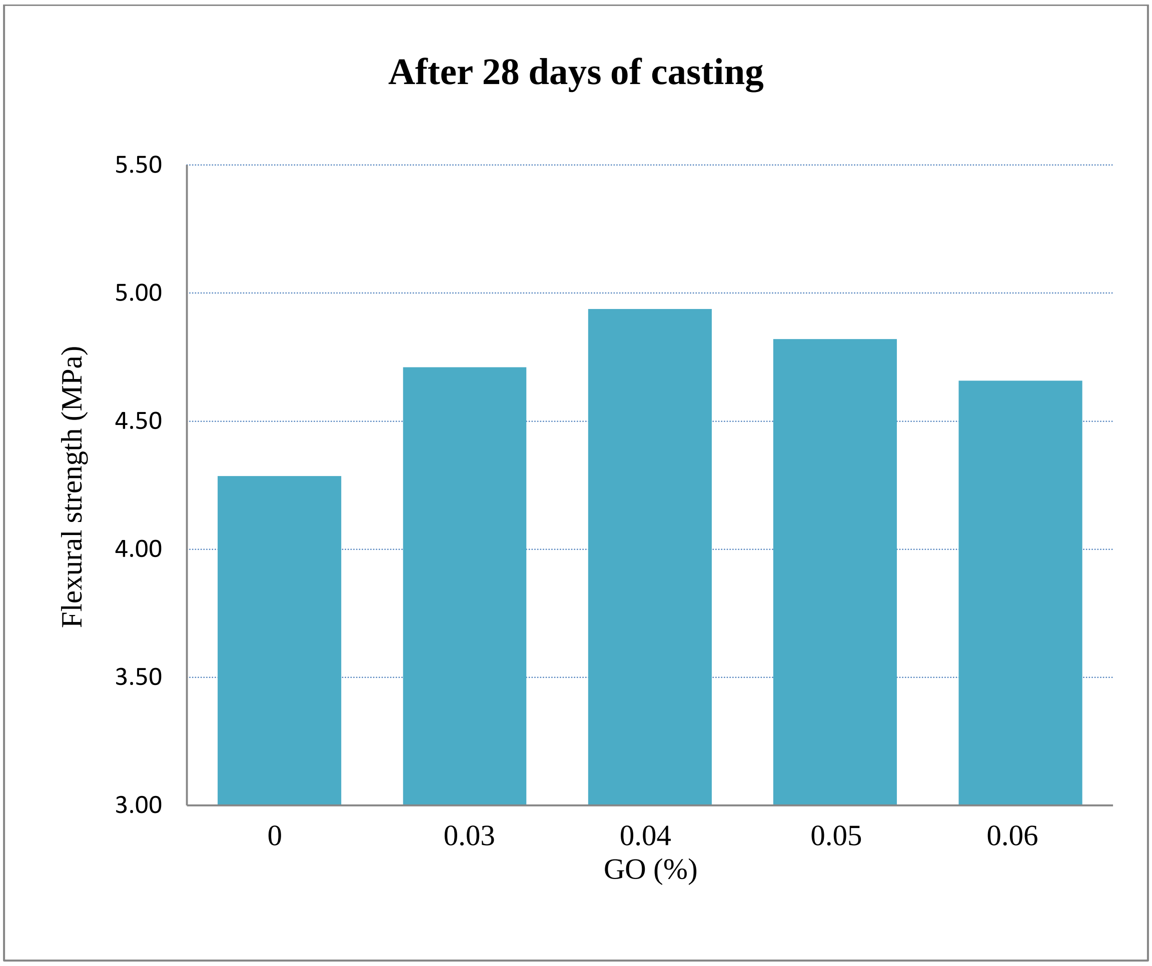


Fig 4.10 Flexural strength of GO based mortar using OPC with cement sand ratio 1:3

The results of flexural strength of GO based cement mortar using PPC instead of OPC with different percentage of GO addition are shown in Table 4.11 & Fig 4.11 in case of cement sand ratio 1:2 and in Table 4.12 & Fig 4.12 in case of cement sand ratio 1:3. The flexural strength improvement pattern of PPC based cement mortar with different percentage of GO addition is nearly similar to that of OPC based cement mortar. However, the optimum percentage of GO addition for maximum flexural strength improvement in PPC based mortar is 0.04% for cement sand ratio of 1:2 and 0.05% for cement sand ratio of 1:3. The maximum improvement in flexural strength was 27.94 % in case of cement sand ratio of 1:2 and 17.05% in case of cement sand ratio of 1:3 corresponding to their optimal GO addition.

As a consequence, the increase in flexural and compressive strengths suggest that the bond developed between GO and cement mortar is effective under loading conditions. Also the addition of GO has a positive impact on the process of hydration, which could directly transform into mechanical properties. In addition, GO, as a nano-scale layer material, can easily fill the pores of the cement mortar, and make the material more solid or denser.

Table 4.11 Flexural strength of GO based mortar using PPC with cement sand ratio 1:2

Mix No	Graphene Oxide (%)	Flexural Strength (MPa)	
		28 Days Strength	Strength Improvement (%)
C0 (Control)	0.00	4.22	…
C3	0.03	5.03	19.12
C4	0.04	5.40	27.94
C5	0.05	5.12	21.32
C6	0.06	4.91	16.18

Table 4.12 Flexural strength of GO based mortar using PPC with cement sand ratio 1:3

Mix No	Graphene Oxide (%)	Flexural Strength (MPa)	
		28 Days Strength	Strength Improvement (%)
D0 (Control)	0.00	4.01	…
D3	0.03	4.13	3.10
D4	0.04	4.60	14.73
D5	0.05	4.69	17.05
D6	0.06	4.22	5.43

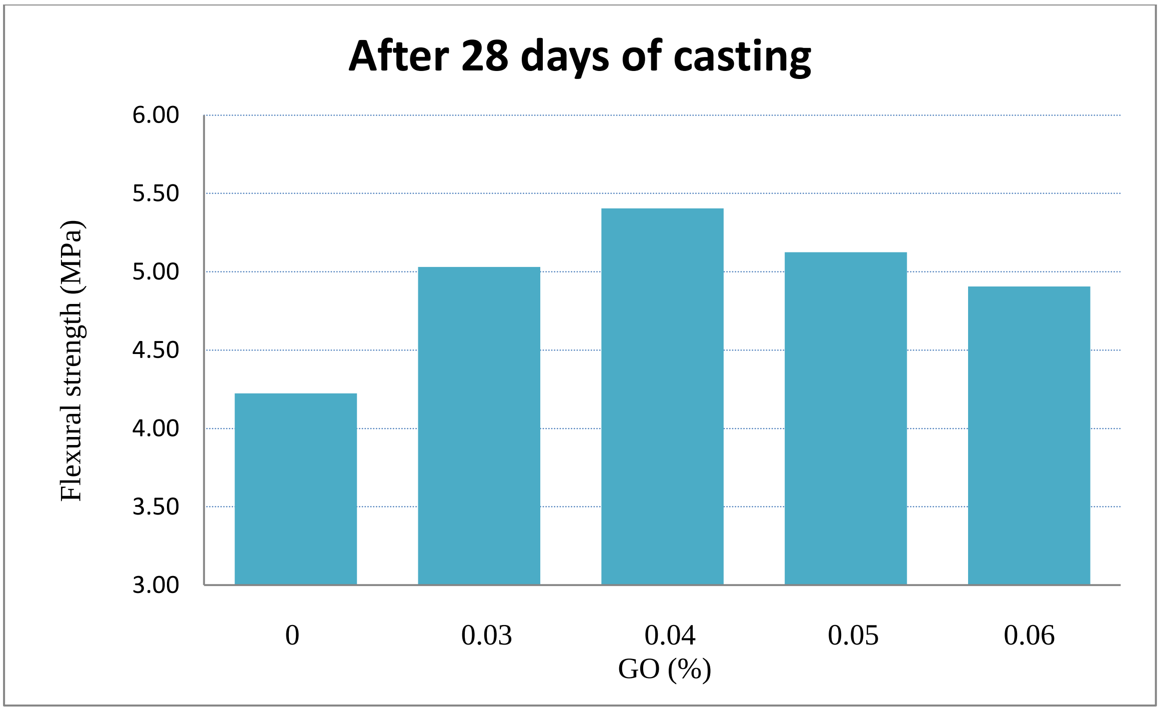


Fig 4.11 Flexural strength of PPC and GO-PPC mortar with cement and sand ratio 1:2

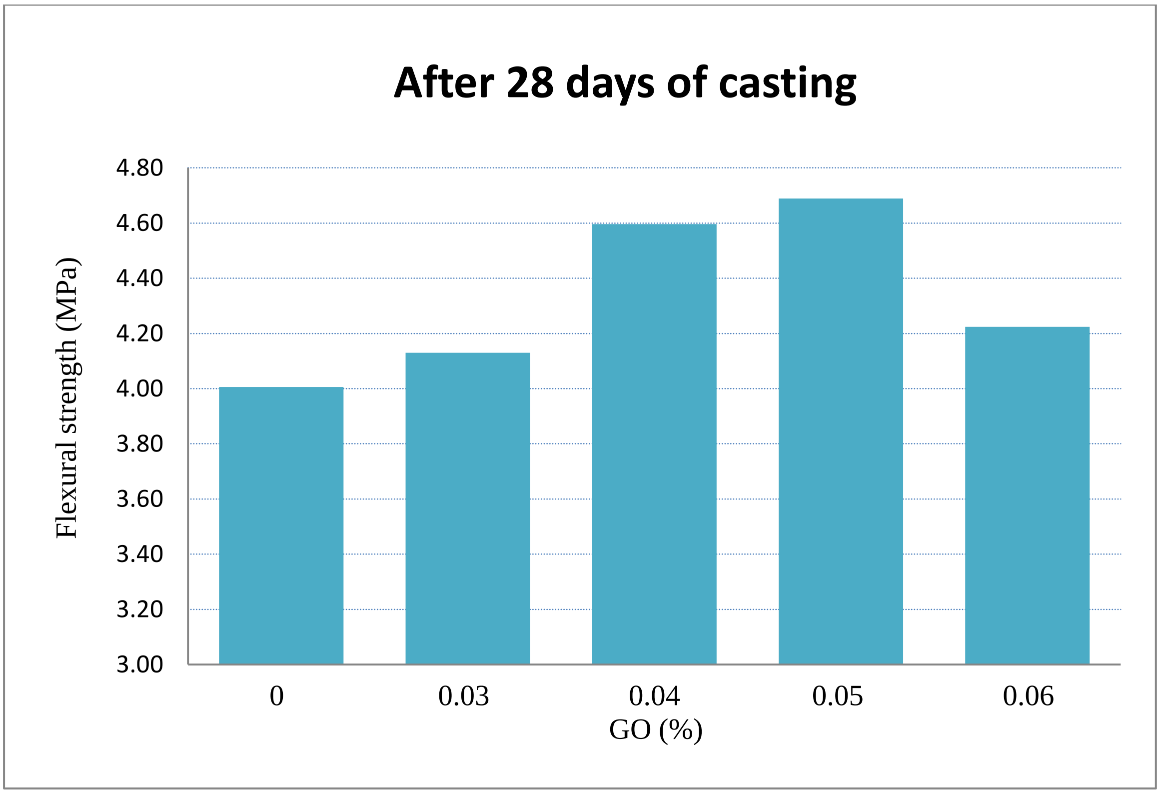


Fig 4.12 Flexural strength of PPC and GO-PPC mortar with cement and sand ratio 1:3

The results of ultrasonic pulse velocity test of GO based cement mortar (cement sand ratio 1:2 and 1:3) using OPC are shown in Table 4.13 and Table 4.14 respectively. The results indicate that the velocity of ultrasonic pulse through GO based cement mortar increases with the increase in the addition of different percentage of GO (0.03%, 0.4%, 0.05% and 0.06%) at 28 days curing. Such increase of pulse velocity indicates that cement mortar becomes stronger, get densified and good bond developed between GO and cement mortar. The pulse velocity is maximum at GO content 0.05% and 0.04% for cement sand ratio of 1:2 and 1:3 respectively. This supports the result of maximum compressive strength and flexural strength at optimum percentage of GO addition.

Table 4.13 UPV test result of OPC mortar with GO and cement sand ratio 1:2

Mix No	**GO (%)**	**Pulse Velocity (km/s)**
A0 (Control)	0	3.774
A3	0.03	3.856
A4	0.04	3.908
A5	0.05	4.067
A6	0.06	3.755

Table 4.14 UPV test result of OPC mortar with GO and cement sand ratio 1:3

Mix No	GO (%)	Pulse Velocity (km/s)
B0 (Control)	0	3.670
B3	0.03	3.688
B4	0.04	3.856
B5	0.05	3.785
B6	0.06	3.562

Similarly, Table 4.15 and 4.16 shows the result of UPV test of GO based cement mortar (cement sand ratio 1:2 and 1:3) with different percentage of GO and using PPC cement. It is noted that the ultrasonic pulse velocity through GO based cement mortar has been increased with respect to control mortar (without GO) due to the addition of different percentage of GO like GO based cement mortar with OPC. The pulse velocity is maximum through cement mortar with GO percentage of 0.04% and 0.05% for cement sand ratio 1:2 and 1:3 respectively.

Table 4.15 UPV test result of PPC mortar with GO and cement sand ratio 1:2

Mix No	GO (%)	Pulse Velocity (km/s)
C0 (Control)	0	4.063
C3	0.03	4.112
C4	0.04	4.219
C5	0.05	4.177
C6	0.06	4.014

Table 4.16 UPV test result of PPC mortar with GO and cement sand ratio 1:3

Mix No	GO (%)	Pulse Velocity (km/s)
D0 (Control)	0	3.341
D3	0.03	3.575
D4	0.04	3.744
D5	0.05	3.841
D6	0.06	3.771

The results of sorptivity test of cement mortar using OPC with cement sand ratio 1:2 and 1:3 for different percentage of GO addition are shown in Fig 4.13 and Fig 4.14. This test measures the rate of water absorption, occurring due to capillary suction along a unidirectional rise, as a function of time. It is noted that the trend of water absorption rate of GO based cement mortar and control cement mortar (without GO) are almost similar. Due to the addition of different percentage of GO (0.03% to 0.05%) in cement mortar, the rate of water absorption of GO based cement mortar decreases with respect to control cement mortar (without GO). When the GO content is 0.06% and cement sand ratio 1:2, the rate of absorption is less than control mortar (without GO) up to 3 days after that rate of absorption is more than control mortar (without GO). For GO addition of GO 0.06% and cement sand ratio 1:3, the rate of absorption of cement mortar is less than control mortar (without GO) up to 8 days. The rate of water absorption is least due to the addition of 0.05% GO (for cement sand ratio 1:2) and 0.04% GO (for cement sand ratio 1:3). So, 0.05% and 0.04% are the optimum percentage of GO addition for cement sand ratio 1:2 and 1:3 respectively. Thus at optimum GO content, cement mortar exhibit maximum strength improvement, less voids, compact microstructure and more durable than control mortar (without GO).

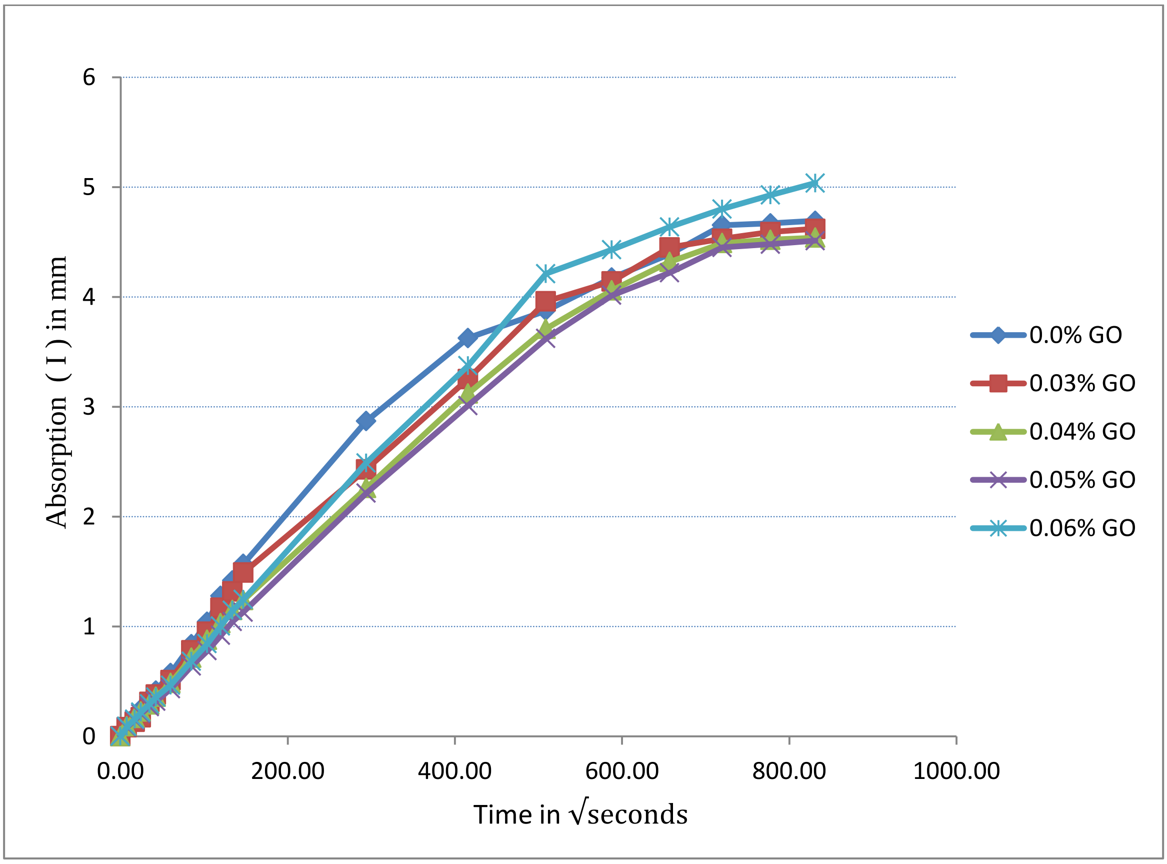


Fig 4.13 Sorptivity test result of GO based mortar using OPC with cement and sand ratio 1:2

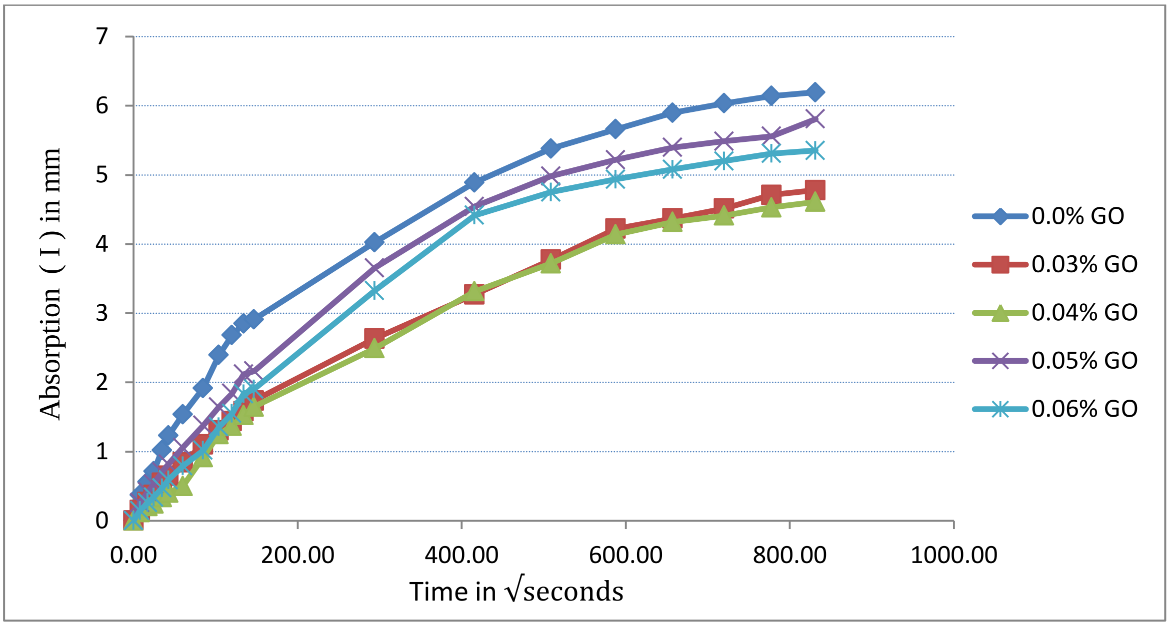


Fig 4.14 Sorptivity test result of GO based mortar using OPC with cement and sand ratio 1:3

Fig 4.15 and 4.16 shows the results of sorptivity test of GO based cement mortar using PPC with cement sand ratio 1:2 and 1:3. It is noted the rate of water absorption of GO based cement mortar decreases with the addition of different percentage (0.03% to 0.06%) of GO in cement mortar with respect to control mortar (without GO). The trend of water absorption rate of GO based OPC mortar and PPC mortar are almost similar. The rate of water absorption decreases due to the improvement of micro structure with less number of capillary pores in case of GO based cement mortar. However, the optimum percentage of GO addition in PPC based mortar is 0.04% in case of cement sand ratio 1:2 and 0.05% in case of cement sand ratio 1:3.

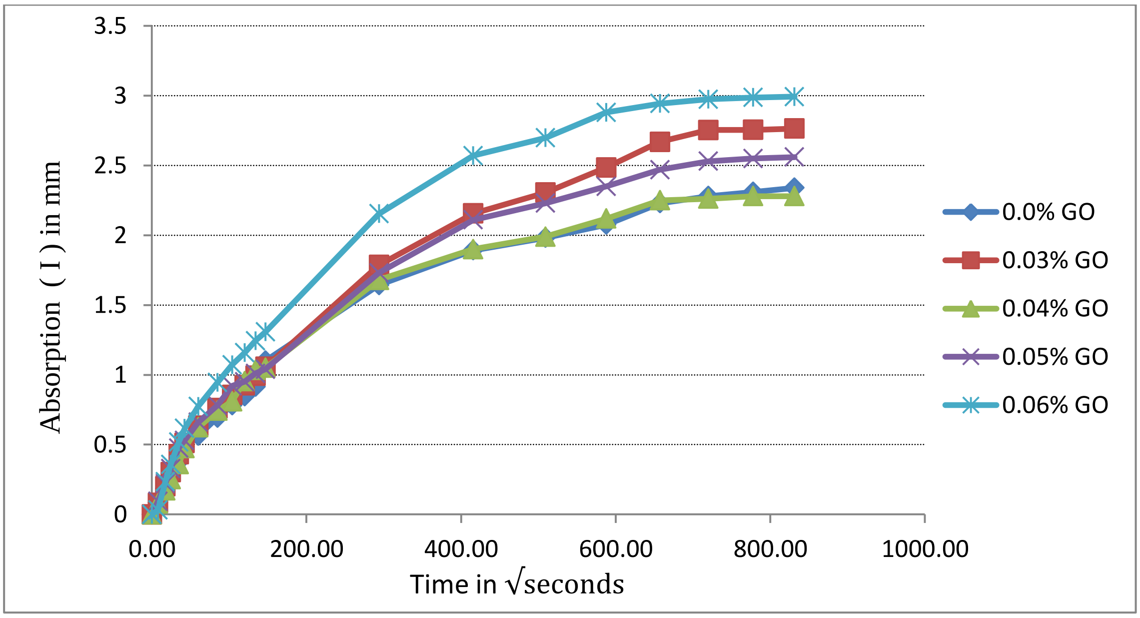


Fig 4.15 Sorptivity test result of GO based mortar using PPC with cement and sand ratio 1:2

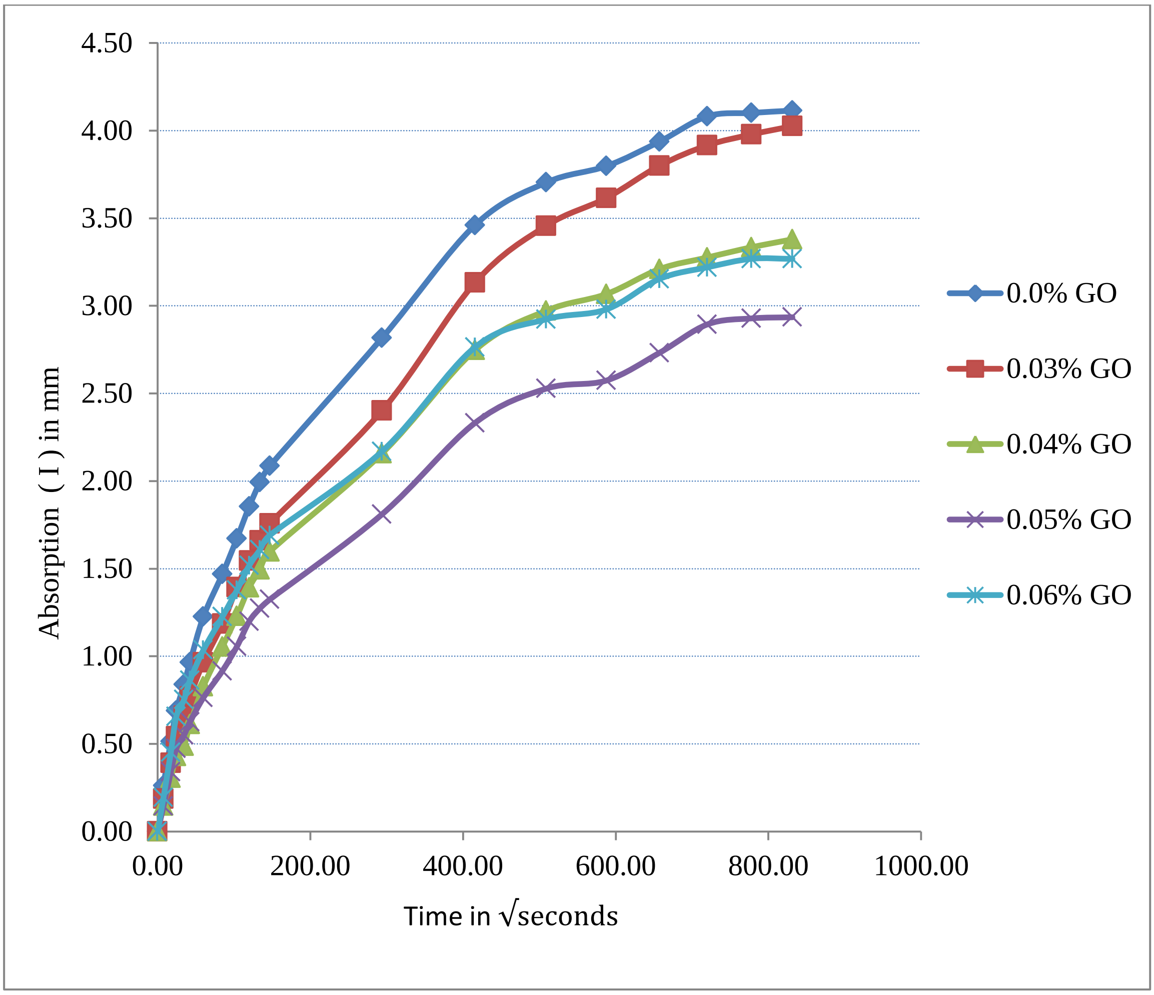


Fig 4.16 Sorptivity test result of GO based mortar using PPC with cement and sand ratio 1:3

The results of XRD analysis for control OPC mortar (without GO) and GO-OPC mortar with 0.05% GO having cement sand ratio 1:2 are shown in Fig-4.17. It is noted that higher and more peaks of C-S-H are observed in GO based OPC mortar than control mortar (without GO). The significant picks are appeared at 2θ = 18.05, 20.9, 26.7, 28.05, 30.75, 34.15, 39.5, 50.15, 54.9, 60, 67.8 and 73.5 degree. These mineralogical results indicate that GO can promote hydration reactions. New compound like Ettringite (CaAlSH), Boggsite, Ankerite, Gismondine and Srebrodolskite are also formed due to addition of GO in control OPC mortar. Due to the formation of Ettringite, Ankerite and Gismondine, the early development of strength of GO-OPC mortar increases compared to control OPC mortar. The Gismondine provides thermal stability and prevent decomposition of cement gel at high temperature.

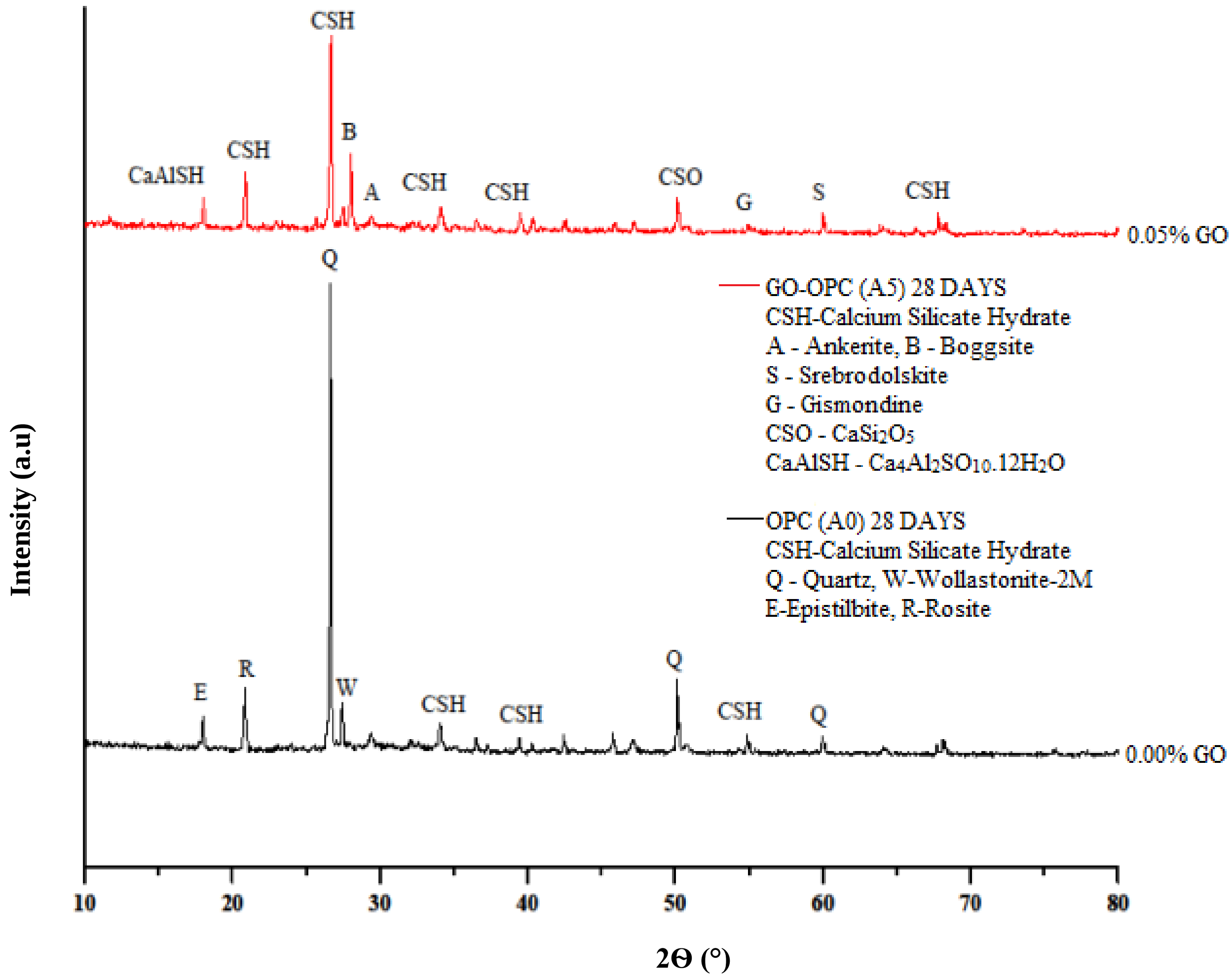


Fig 4.17 XRD analysis result of OPC and GO-OPC (0.05% GO) mortar with cement sand ratio 1:2

The result of XRD analysis for control PPC mortar (without GO) and GO-PPC mortar with 0.05% GO having cement sand ratio 1:2 are shown in Fig 4.18. It is clearly shows that the higher peaks of C-S-H are observed in GO-PPC mortar than control mortar at different values of 2θ. The significant picks are appeared at 2θ =10.5, 18, 20.8, 26.6, 27.9, 50.1, 59.9, 67.7, 68.2, 73.4 and 76 degree. However, the increase of peaks was seen in the by-product of the cement hydration that was created through the reaction of the –COOH functional groups with C_2S, C_3S phase. New compounds mainly Amstallite, Brownmillerite, Ettringite, Boggsite and Gismondine are formed due to addition of GO in PPC mortar. Due to the formation of Brownmillerite, Gismondine and Ettringite the strength of GO-PPC mortar increases compared to control mortar. The Gismondine provides thermal stability and prevent the decomposition of cement gel at high temperature.

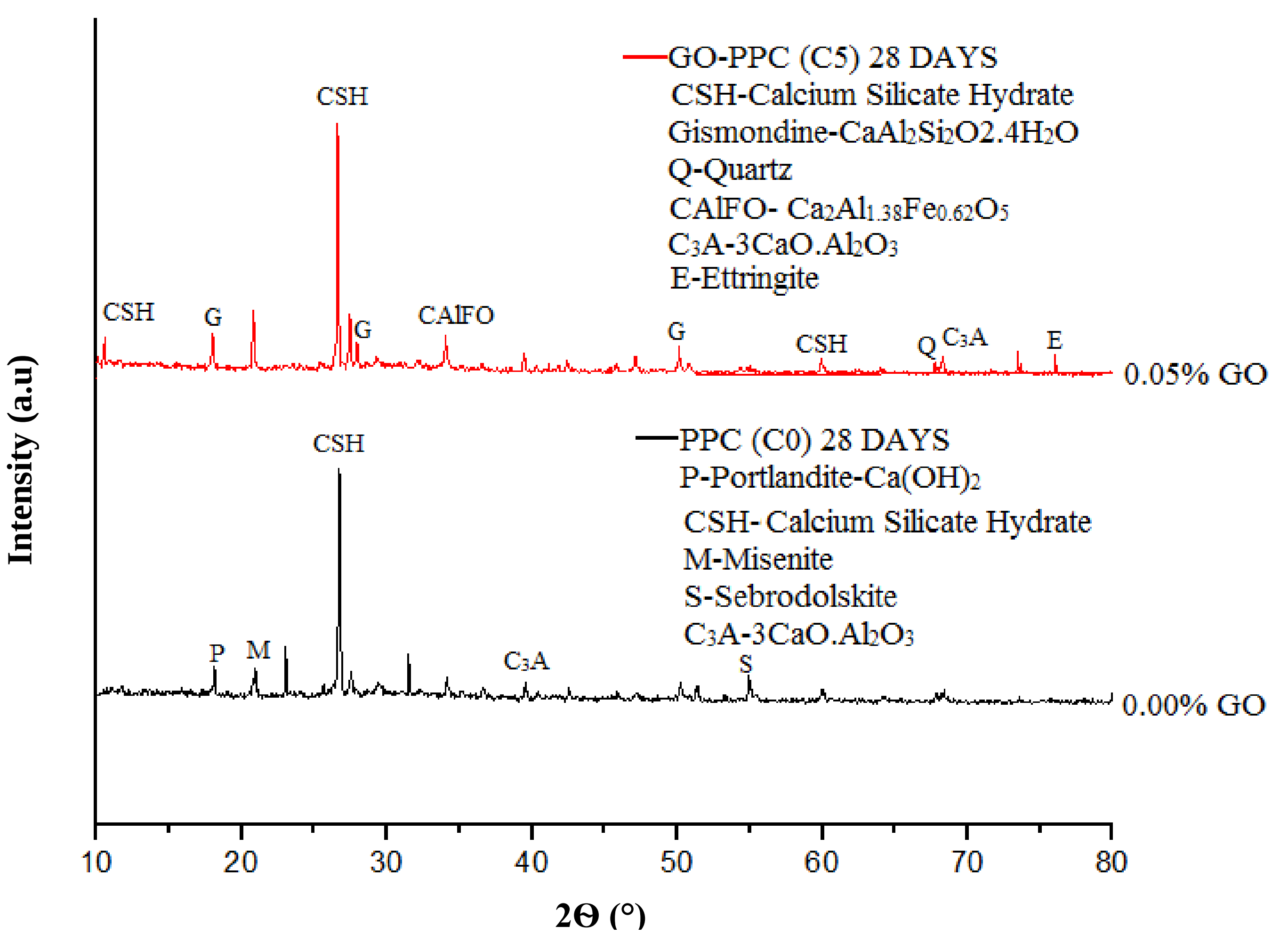


Fig 4.18 XRD analysis result of PPC and GO-PPC (0.05% GO) mortar with cement sand ratio 1:2

The pore size distribution of cement mortar (with/without GO) have been made from 0-50,000 PSI through mercury intrusion porosimetry (MIP) test at 28 days. Fig 4.19 shows the pore size distribution of control OPC mortar (without GO) and GO-OPC mortar with 0.05% GO. It is noted that the pore size distribution of the cement mortar was modified by the addition of GO. It is noted that the volume of mercury intrusion is less in GO-OPC mortar compared to control OPC mortar. A main peak for both curves exist at around 0.00 µm but the peak for GO-OPC mortar is less than control OPC mortar. It can be seen that, after the addition of GO, the GO-OPC mortar have higher proportions of finer pores than the control OPC mortar. It shows clearly that the intruded volume of mercury is more in control mortar than that of GO-OPC mortar for pore diameter from 10µm to 100 µm (Refer-Fig-4.15). Besides reduction of porosity, the addition of GO nanosheets in cement mortar also resulted in refinement of pore structure. The result also supports the strength improvement with the addition of GO in cement mortar.

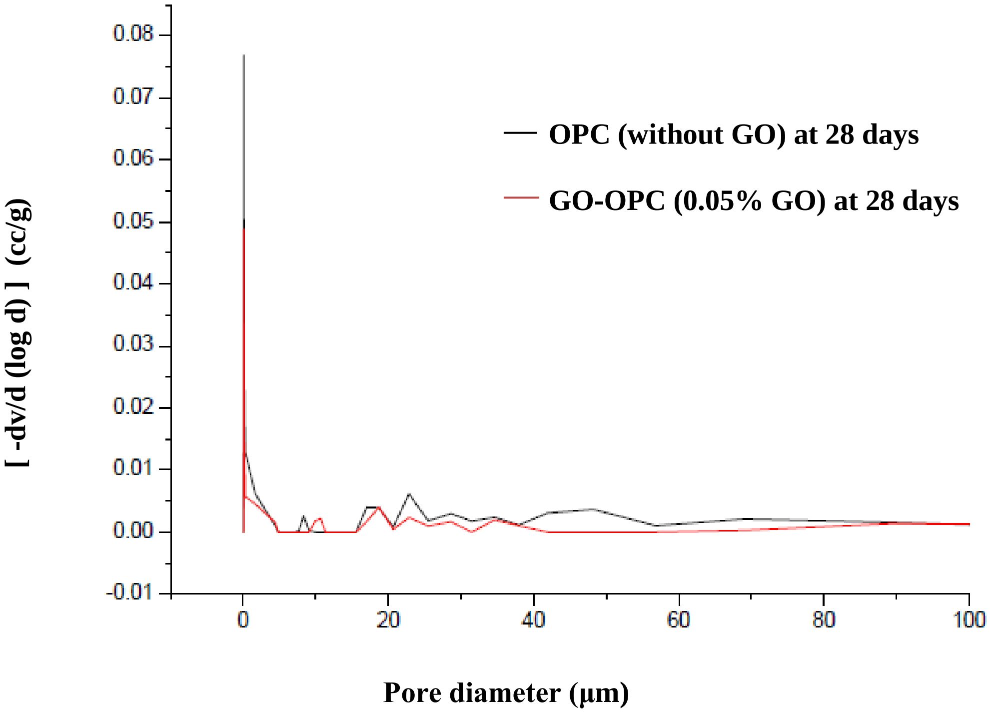


Fig 4.19 MIP result of OPC and GO-OPC (0.05% GO) mortar with cement sand ratio 1:2

CHAPTER-5

CONCLUSIONS AND SUGGESTION FOR FUTURE RESEARCH

5.1 CONCLUSIONS

Based on the experimental investigation on the effect of GO addition in cement mortar with OPC and PPC the following conclusions can be made.

1) The workability of cement mortar for both OPC and PPC is reduced with the addition of GO. This is due to the formation of GO agglomerates/ettringite (as observed from XRD analysis) by chemical cross-linking of GO nanosheets by calcium cations. Free water for mortar is reduced as these GO agglomerates have high water entrapment capacity.
2) The addition of low percentage GO in cement mortar promotes and enhances the compressive strength, flexural strength, cement hydration process and interfacial bond formation through the bridging and nucleation effects.
3) The optimum GO addition in cement mortar with OPC was 0.05%and 0.04% for cement sand ratio of 1:2 and 1:3 respectively. The maximum improvement in compressive strength at 28 days was 21.15% and 13.57 % for cement sand ratio 1:2 and 1:3 respectively. The results of UPV also support the improvement in strength.
4) The optimum GO addition in cement mortar with PPC was 0.04%and 0.05% for cement sand ratio of 1:2 and 1:3 respectively. The maximum improvement in compressive strength at 28 days was 12.12% and 20.84 % for cement sand ratio 1:2 and 1:3 respectively. The results of UPV also support such improvement in strength.
5) The maximum flexural strength was obtained at 0.05% and 0.04% GO addition for OPC based cement mortar with cement sand ratio of 1:2 and 1:3 respectively. However, for PPC based cement mortar, the maximum flexural strength was noted at 0.04% and 0.05% GO addition for cement sand ratio of 1:2 and 1:3 respectively.
6) The sorptivity of the GO based cement mortar is decreases with respect to control mortar (without GO). Hence, the durability of GO based cement mortar is more.
7) The results of XRD analysis indicates that the formation of more C-S-H gel in GO based cement mortar both in OPC and PPC with respect to their corresponding control mortar which is responsible for strength improvement.
8) The MIP results indicate that the addition of graphene oxide improved the pore-diameter distribution and formed a compact microstructure of the cement mortar. The addition of GO refines the pore structure of cement mortar due to the filling of large pores by GO agglomerates.

5.2 SUGGESTION FOR FUTURE RESEARCH

Based on the present experimental investigation the following suggestions for future research can be made.

1) The addition of GO leads to a large reduction in workability of the cement mortar for both cases of OPC and PPC. Further study is needed to improve the workability of fresh GO based cement mortar.
2) The exact relationship between GO and hydration products is still not fully understood. Further in-depth and rigorous investigation should be carried to reveal the true relationship between GO and hydration products.
3) The investigation should be carried out to study the effect of GO addition on concrete also.

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