INFLUENCE OF GRAPHENE OXIDE ON THE MICROSTRUCTURE AND DURABILITY PROPERTIES OF CONCRETE

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ABSTRACT. This study was executed to explore the effect of graphene oxide (GO) on the durability properties of normal concrete. A total of five mixes were prepared in addition of GO (0-0.8%) synthesized in the laboratory. The synthesized GO have been characterized using SEM, FT-IR and XRD techniques. The workability, initial surface absorption and sorptivity reduce with increase in GO content in the cement matrix. Whereas, the ultrasonic pulse velocity (UPV), compressive and tensile strength were observed to enhance with percentage increment of GO in the concrete compare to control mix. The microstructural analysis was performed using SEM/EDX at 90 days curing age and the image of the mix with 0.08% GO has massive hydrated crystals, filling the void/cracks created during hydration of cement. Thus, the nano-reinforced concrete has constructive microstructure with better durability properties compared to normal concrete process.

Keywords: Graphene oxide (GO), Durability, Microstructure, Sorptivity, Initial surface absorption.

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INTRODUCTION

Our abilities, expectations and vision to control the material world has changed by nanotechnology. The advance in nano-science can also have a great impression on the field of construction materials. The potential of ordinary Portland cement which is one of the largest commodities consumed by mankind is not completely explored. A new generation of concrete which is stronger and more durable with desired stress strain behaviour can be achieved through a better understanding and engineering of complex structure of cement-based composites at nano-level and possibly the whole range of newly introduced will definitely result in “smart” properties. Inclusion of nanomaterials such as carbon nanotubes (CNTs), carbon nanofibers (CNFs) nano silica, graphene based derived, graphene oxide (GO) etc. in cement-based composites and the study of its influence in the cement matrix was carried out by many researchers. The experimental results of mechanical strength were increased up to certain level and decreased with varying water binder ratio and percentage content of nanomaterials at early stages of hydration as well as the durability of the cement-based composites.

Graphene is known for its excellent toughness, thermal, optical and electrical properties [1-2]. As a graphene derivative, graphene oxide (GO) comprises a mono-layer of sp^2 – hybridized carbon atoms the oxygenated functional groups attached on the edges and basal planes of GO sheets, significantly to change the intermolecular forces (Vander Waals forces) between the GO nanosheets and therefore improve their dispersion in water. GO also exhibits high value of tensile strength, aspect ratio and large surface area. High performance concrete requires the concrete with considerably enhanced performances such as high strength, high durability, high chloride ion resistivity, high freeze resistance, high sulphate resistance, low shrinkage, low abrasion, and low carbon footprint etc [3-6] than the conventional concrete. Nano reinforcement could be an answer to the development of infrastructure, high-rise buildings, subsea tunnel and cross-sea bridge, hydraulic or marine works etc., which are usually under the environment attacked easily through salts and alkalis.

Matrix material that exhibits improved compressive strength which is formed by graphene oxide. They have developed a cementitious matrix with enhanced strength and durability through the incorporation of graphene oxide with different percentage [7-10]. Other studies concluded that it acts as reinforcements to hold crack formation and propagation of micro cracks which are caused by aggressive chemical ingress from the environment, resulting a more durable and strengthened cement-based composites [10-15].Although, inclusion of GO in cement matrix reduces fluidity thus increasing the viscosity of the cement-based composites [16-19]. The current study will give an overview on the GO incorporation in concrete composites and its impact on the microstructural and durability properties, as many of the research was carried on cement past and mortar and not on actual concrete.

EXPERIMENTAL PROGRAM

An experimental program was to investigate the strength, water permeation, sorptivity homogeneity and microstructural properties of concrete composite in addition of synthesized graphene oxide in laboratory. The basic properties of concrete constituent materials, concrete mix details along with method of casting and curing, workability of concrete, details of tests performed on hardened concrete are as follows.

Materials

In this work Ordinary Portland Cement of grade 43 (OPC-43) was used for preparing the mixes. Physical Properties of the cement as determined from various tests confirming to Indian Standard IS: 1489-199 (I) are given in Table 1 and the tests were carried out as per IS: 4031-1988. Locally available fine aggregate (zone II) and crushed stones were used of size of maximum nominal size of 12.5 mm. The physical properties and fineness modulus of the aggregate were determined as per standard procedures in IS 383 (1970), shown in Table 2.
Master Gelenium SKY 8765, a poly carboxylate ether based super plasticizer was used for all the mixes. Graphite powder, sodium nitrate ($NaNO_3$), sulphuric acid (H_2SO_4), potassium permanganate ($KMnO_4$), hydrogen peroxide (H_2O_2) and hydrochloric acid (HCl) were obtained from nearest chemical distributor.

Table 1 Physical properties of Cement

Fineness (m^2/kg)	300
Standard consistency (%)	32
Initial Setting time (minutes)	62
Final Setting time(minutes)	270
Specific gravity	3.15
Soundness by Le-Chat Expansion(mm)	1
Compressive Strength (MPa)	
3 Days	24.6
7 Days	34.3
28 Days	45.2

Table 2 Physical properties of Aggregate

	Fine aggregate	Coarse aggregate
Fineness Modulus	2.74	7.54
Specific gravity	2.67	2.64
Bulk Density (kg/m^3)	1675	1690

Synthesis of GO using modified Hummers method

5g of graphite flakes and 2.5g of $NaNO_3$ was added to conical flask (200ml capacity) containing 115ml conc. H_2SO_4 and was allowed to mix with the help of magnetic stirrer for over a period of 45mins at 20℃ temperature. Then 15g $KMnO_4$ was added to the mixture slowly and gradually as the reaction is exothermic, the flask was placed on ice bath and temperatures was check regularly so that it does not exceed 35℃, over a period of 30 mins, then carefully dilute with 230 ml water. The mixtures are heated up to 98℃ for over a period of 15 mins. The resulting solution was allowed to cool down and 400 ml deionised (DI) water was added. To complete the reaction 12 ml H_2O_2 (10% conc.) was added to the solution and kept for 24 hours. The final chemically exfoliated GO sheets were further diluted until the pH was up to 7 and wash with 1:10 HCL solution and by DI water. The resultant product was then filtered, dried and crushed into powder form and was sent to laboratory for structural characterization using SEM, XRD and FT-IR.

Casting and curing

The concrete mix contains 384 kg/m^3 OPC, 715 kg/m^3 fine aggregate, 1113 kg/m^3 coarse aggregate of mix proportion 1:1.86:2.89, with water cement ratio 0.5 and 0.05% super plasticizer was kept constant for all the mixes. 70% of water was introduced to a dry mix of cement, fine and coarse aggregate (electrically operated mixer). The remaining 30% water was mixed with super plasticizer sonicated with GO, then added to the mix and the mixer was run for another 3 mins. The fresh concrete mix was then casted by pouring on properly oiled and tightened cubes of 100 mm and 150 mm size and cylinders of 100 mm x 200 mm. A total of five mixes, control mix with 0%GO and with GO inclusion with 0.02%, 0.04%, 0.06% and 0.08% by weight of cement were casted and water cured at room temperature until the day of testing at 7, 28, 56 and 90 days. The mix designations with mixes are given in Table 3.

Workability test on fresh concrete

To assess the workability- ease of concrete to handle and transport without segregation or bleeding was attained using the slump cone method, the frustum of cone of top dia. 100 was, bottom dia. 200 mm and height of 300 mm. The fresh concrete was poured in three layer each compacted by tapping with rod (25 blows distributed for three layer), then the frustum was lifted allowing the concrete to flow (settle). The depth of concrete was measure from the top of the cone to evaluate the slump value and the slump for the control mix was designed as per IS: 6461 (Part VII) - 1973. The slump values of all mixes were recorded.

Table 3 Quantity of materials used in Kg/m^3

Mix designation	Cement	GO (%)	Fine Aggregate	Coarse Aggregate	Water	Super Plasticizer (%)
OPC-0GO	384	0	715	1113	192~	0.05
OPC-0.02GO	384	0.02	715	1113	192~	0.05
OPC-0.04GO	384	0.04	715	1113	192~	0.05
OPC-0.06GO	384	0.06	715	1113	192~	0.05
OPC-0.08GO	384	0.08	715	1113	192~	0.05

Mechanical strength test

As per code IS: 516- 1959, the test was conducted on cubes of size100 mm at curing age 7, 28, 56 and 90 days. The specimens were tested on 200 tonnes capacity compression testing machine (CTM). The load was allowed gradually increased to avoid shock and was increased at the constant rate of 3.5 N/mm^2/min till the cube failed. Compressive strength was taken the mean of three cubes as a representative value in MPa for each mix of concrete. The compressive strength was calculated by dividing the maximum compressive load to cross sectional area of that cube.

As per code IS 5816: 1999, the test was conducted on cylinders of size 100 mm X 200 mm. The specimens were tested for curing age of 7, 28, 56 and 90 days under same CTM at same rate as mention above.

Ultrasonic pulse velocity test (UPV)

The ultrasonic pulse velocity method was conducted to assess the homogeneity (quality) of the concrete and to check the presence of cracks, voids and other imperfections. The ultrasonic pulse velocity is generated by an electro-acoustical transducer, held in contact with one surface of the concrete cube and receiving the same by a similar transducer in contact with the surface at the other end, carried out as per IS 13311(Part 1)1992. The test was performed on the surface of 100 mm cubes at 28, 56 and 90 days of curing for all the mixes.

Initial surface absorption test

The initial surface absorption test was conducted as per BS 1881: Part 208:1996, this test assesses the rate of flow of water into concrete surface of 150 mm size cube per unit area at specified interval at 10, 30 and 60 mins at curing age of 28, 56 and 90 days. The samples were conditioned by placing in oven at 105˚C until constant mass or mass difference not more than 0.1% weight change over any 24 hours drying period achieved as per code. The specimens were then kept in desiccators cabinet to cool down and to prevent from moisture till the testing day. A 78mm diameter cap was clamped to the cube surface. Cap is providing a minimum area of water contact with the surface to be tested of 5000 mm^2. There are two pipes exit from this cap. One is called as inlet and other is outlet. The reservoir with 100 mm diameter was connected to inlet pipe which is fitted with tap and outlet connected to scale. Head of water was maintained between 180 mm to 220 mm just before and after starting the test. The cap was fitted on the cube with the help of grease (to avoid leakage) and tightened with nut and clamp. The tap was opened and the timer was set to start. Measurements of 1 min was taken at 10 min, 30 min and 60 min interval from start of the test. During 10 min interval, the mean time to collect 10mm of water was taken which was used in an equation given in the code for the value of t, time in sec to find the radius of bore of capillary tube

Capillary suction test

This test method for sorptivity was performed to assess the penetration of water through capillary pores from on face of saturated concrete and was performed per codeASTM C 1585-04, 2009.The standard test specimen was 100 ± 6 mm diameter disc, with length of 50 ± 3 mm. Three specimens were obtained after cutting of cylinder of 100 mm X 200 mm at 28, 56 and 90 days of curing. Procedure of conditioning was similar to ISAT. The enhancement in the mass of a specimen resulting from absorption of water as a function of time when only one surface of the specimen when exposed to water was measured for 1, 5, 10, 20, 30 and 60 mins. The exposed surface of the specimen was immersed in water and water ingress of unsaturated concrete dominated by capillary suction during the initial contact with water. The initial rate of water absorption (mm/√s) is defined as the slope the line plotted against the square root of time (√s). The test was repeated for all the specimens of different mixes at 28, 56 and 90 days.

Microstructural observation

Field emission scanning electron microscopy (FE-SEM) of GO was performed on powdered on BRUKER QUANTA FEG 450. And the samples of the GO-reinforced concrete mixes using SEM on JEOL JSM-6510LV. The samples were made conductive by gold coating before the microscopy analysis was performed. To examine the samples at 90 days of curing, the secondary electron detector was used at high vacuum, accelerating voltage of 15 KV with working distance of 12 mm.

RESULTS AND DICUSSIONS

Structural characterization of GO synthesized

The structural characterization of synthesized powdered GO was approved using field emission scanning electron microscopy (FE-SEM) to attained the surface morphology. Fig. 1(a) represents the SEM image of GO showing wrinkly surface or cringed sheets which directs the attachment of oxidative functional groups. The large surface area of GO nanosheets interlocked with vanderwaals force which under the microscope appears to be creased.

Fourier transmission infrared (FT-IR) spectra shows the existence of oxygen containing functional groups attached on the surface of GO sheets i.e. the interruption of oxygen containing functional groups on the sp^2 hybridization. The FT-IR spectra as shown in Fig. 1(b), Hydroxyl (-OH) and carbonyl groups (C=O) were observed at peak 3217$cm^{-1}$and 1711 cm^{-1}, alkenic bonds (-C=C- stretch) was seen at 1619 cm^{-1}. At peak 870 cm^{-1} C-O stretch in epoxides was visible which was further supported by absorption peak at 1032 cm^{-1}, thus concluding the presence of oxygenated functionalities on the GO nanosheets.

The diffraction pattern for GO powder using PANalytical EMPYREAN diffractometer with Cu Kα = 1.540 Å, with fixed divergence slit of 0.2177° having step time of 12.0650 sand step size of 0.0080 was examined at angular range of 2θ = 5° to 100° and ran at 40 mA and 45 kV to differentiates the interplanar spacing and the crystalline nature. The XRD spectra of the GO is shown in Fig. 1(c) indicating an interlayer spacing of 0.814 nm and presence of diffraction peak at 2θ = 10.8° which is assigned to (001) diffraction peak of graphitic oxide.

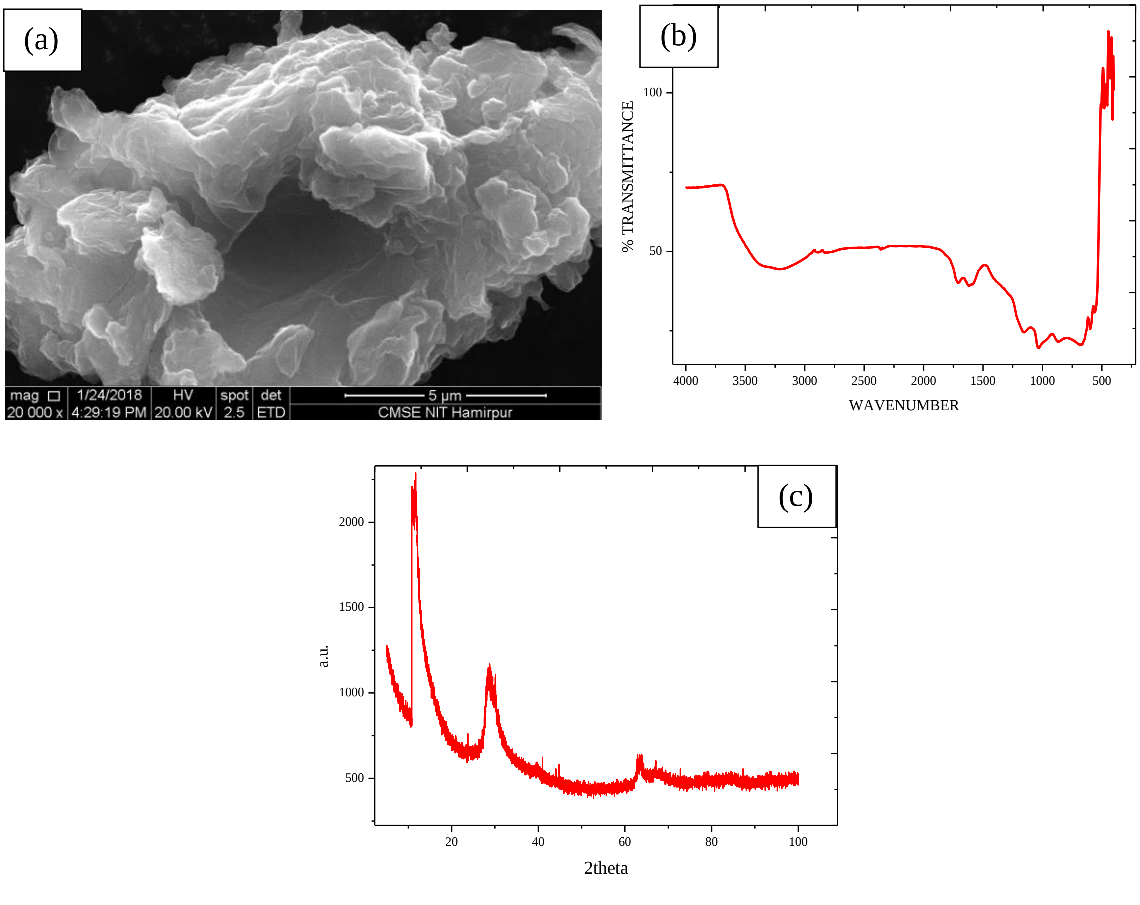

Fig.1 (a) SEM image, (b) FT-IR and (c) XRD of GO

Workability

The slump value of the control mix was designed for 109 mm. Fig. 2 shows the comparison of slump values of the control mix and the mixes made with GO inclusion. It can be clearly visible that the mix concrete with GO 0.08% has the minimum slump value implying that with increase in GO content, the fluidity decreases. Pan et al. have mentioned that workability decreases with percentage increase of GO in the cement-based matrix. GO has high specific surface area with hydrophilic functionalities which absorbs water molecule during mixing of the concrete, thus reducing the fluidity. Hence the GO content in the concrete affects the workability by lowering the fluidity.

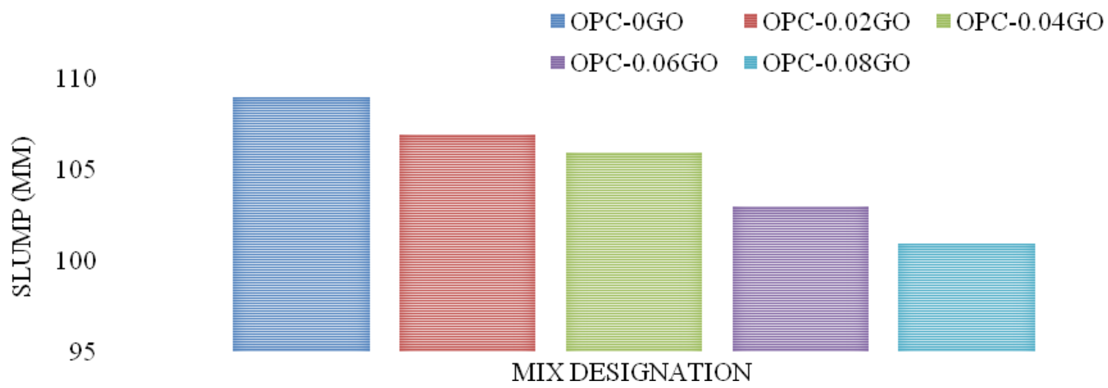

Fig. 2 Slump value of different mixes

Mechanical properties

The compressive and tensile strength of all the concrete mixes were recorded for 7, 28, 56 and 90 days curing age. Both the strength properties were observed to be improved with GO incorporation in the cement matrix compared to control mix as shown in Fig. 3 & 4. The percentage increase in compressive strength was higher at 7 days than rest of the curing age, which means that GO acts as a catalyst in accelerating the heat of hydration of cement in concrete. Farther, the increase in percentage on 90 days was observed to be higher compared to 28 and 56 days, indicating the chances of unreacted cement particles getting hydrated at later age. This means that GO not only has catalytic behaviour, it also has transport properties of reached out to the unreacted particle by delivering water which are absorbed easily by the hydrophilic oxygenated functional groups. Similarly, the percentage increase in tensile strength was higher at 7 days followed by 28, 56 and 90 days. Likewise, with increase in GO content the strength enhancement increases drastically 9-53 % and 10-62% for compressive and tensile strength respectively.

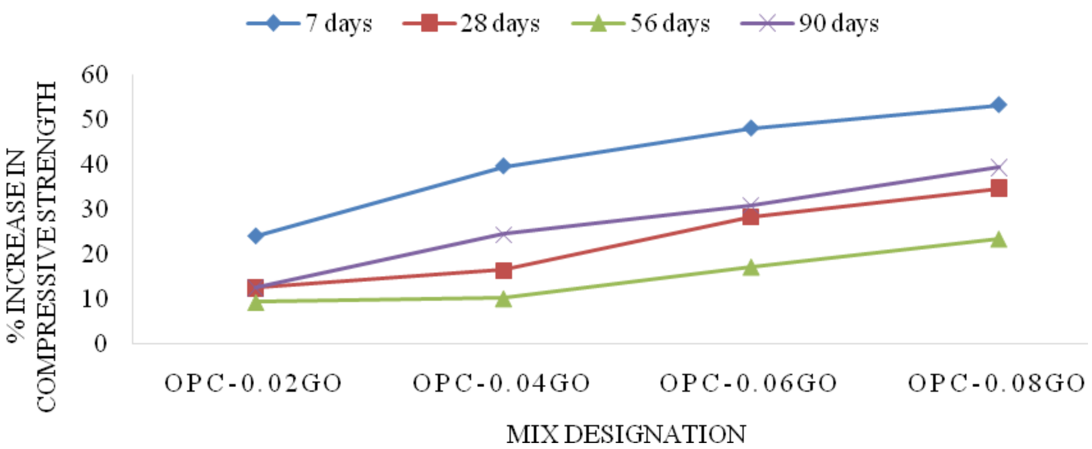

Fig. 3 Percentage increment in compressive strength with respect to control mix

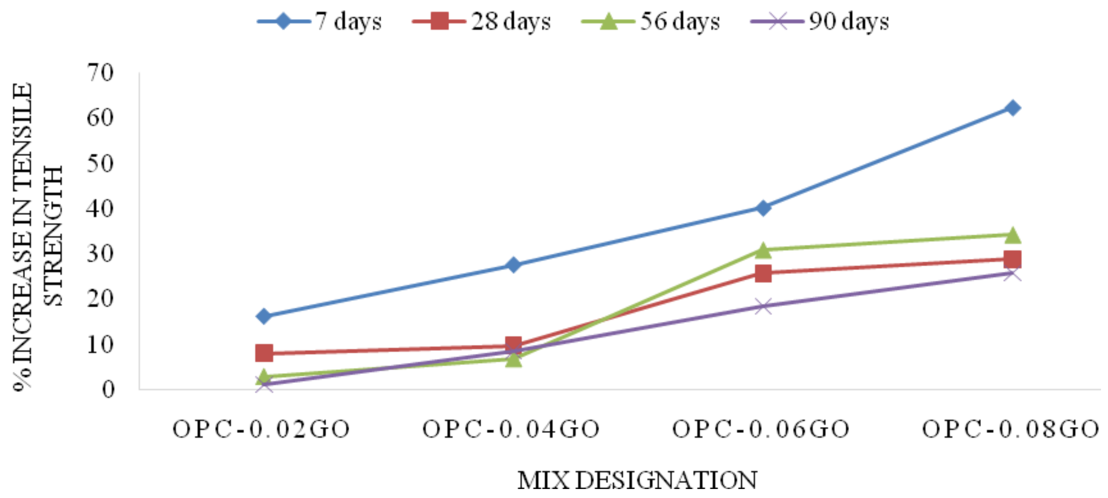

Fig. 4 Percentage increment in tensile strength with respect to control mix

Ultrasonic pulse velocity

The homogeneity of the concrete was determined by conducting a non-destructive UPV test on the hardened concrete of all mixes at 28, 56 and 90 days curing. The quality of the all the concrete mixes in comparison to the control mix are showed in Fig. 5. The pulse velocity above 4.5 km/s is considered to be excellent followed by the good quality concrete, 3.5-4.5 km/s, medium of 3 – 3.5 km/s and poor quality for below 3 km/s. It was observed that all the mixes were good quality concrete mixes but a significantly improvement in the average velocity was observed with increase in percentage content of GO in the concrete composites compared to the conventional mix. And at 90 days the enhancement in strength from the core showing its improvement in the homogeneity of the whole concrete was visible from the result, with incorporation of GO into the cement matrix.

Initial surface absorption

The initial surface absorption test (ISAT) was conducted at 28, 56 and 90 days of curing. The absorption rate was attained by evaluating the uniaxial water penetration on to the surface of the concrete. The comparison of results is shown in Fig. 6. It was noted that the absorption rate was higher for all the mixes at 28 days compared to the 56 and 90 days of curing. Addition of GO has undoubtedly reduced the absorption rate implying less permeable for the water to flow through the pores. With GO percentage content increase in the concrete mix, the density of the cement-concrete matrix has been improved thus, filled the pores/cracks at nano-level leaving no connectivity of the pore to pass the water molecule.

Sorptivity

This method was used to administrate the rate of absorption of water by assessing the increase in mass caused by the water absorption through capillary pores on face of saturated concrete with respect to time. It was recorded that the sorptivity (mm/√s) for all the mixes at 28, 56 and 90 days curing as shown in Fig. 7, mix with GO 0.08% has the least absorption rate compared to the rest of the mixes with GO and control mix. It simply confirms the above statement that the capillary pores were filled by GO (nano-filler). The sorptivity decrease with passing curing days i.e. 90 days has less absorption rate than 56 and 28 days.

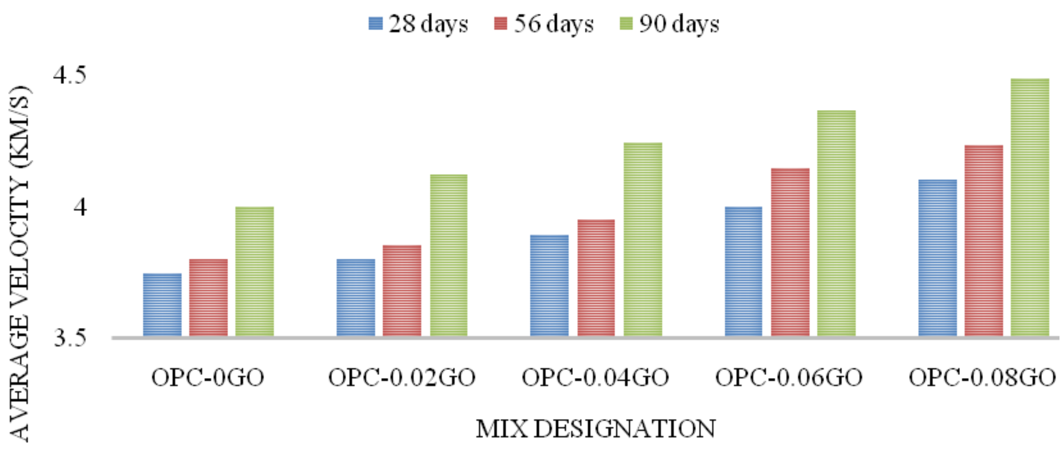

Fig. 5 Ultrasonic pulse velocity of different mixes

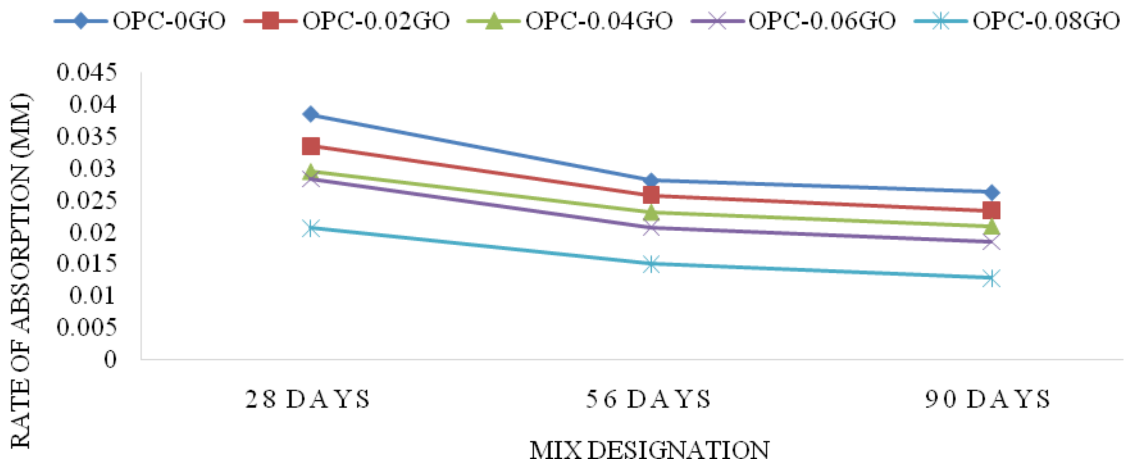

Fig. 6 Initial surface absorption result for all the mixes

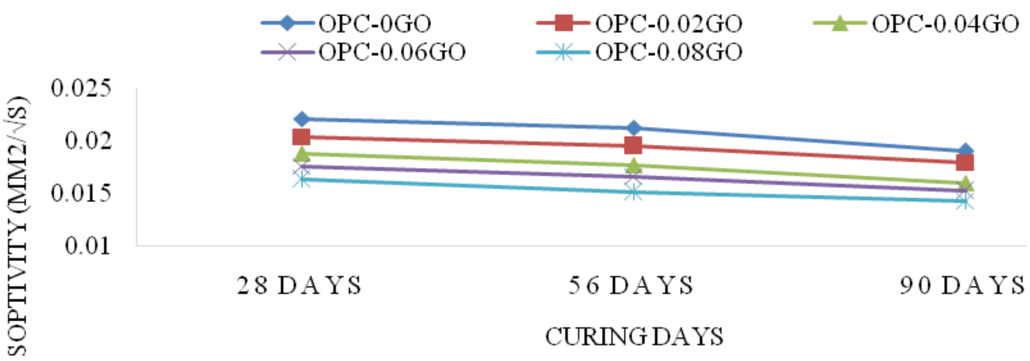

Fig. 7 Initial surface absorption of all the mixes

Microstructural analysis

The scanning electron microscopy was carried out to study the comparison in structural morphology of the GO reinforced concrete composites and control mix. Fig.8**.** Represents the

SEM images of all the five mixes with different percentage of GO of 90 days old concrete. Presence of ettringites, pointy crystal and pores was visible with hydrated crystals.

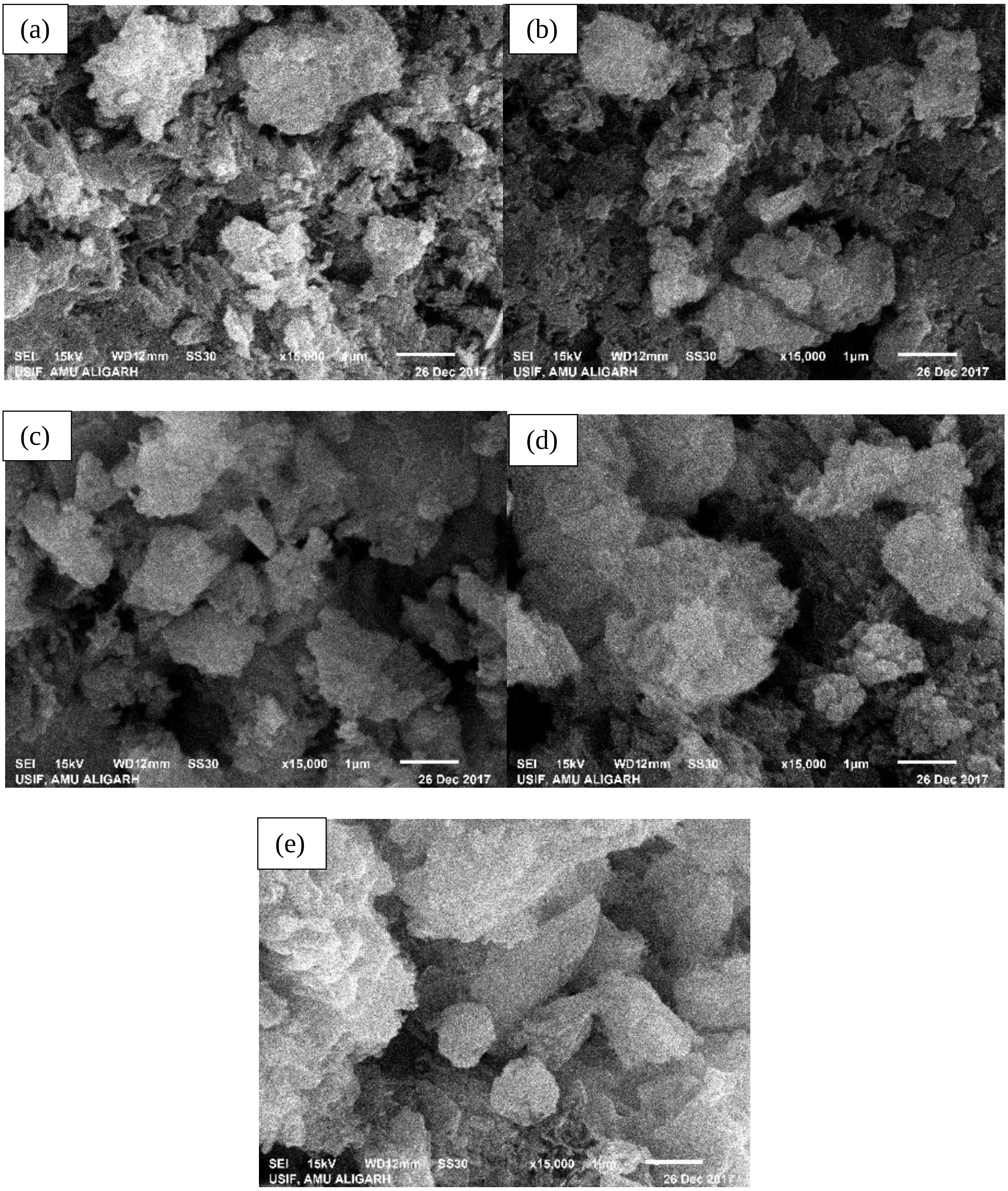

Fig. 8 SEM of mix with (a) GO 0% Control mix, (b) GO 0.02%, (c) GO 0.04%, (d) GO 0.06%, (e) GO 0.08%

are visible on the SEM micrograph of control mix, but massive hydrated crystals, calcium silicate hydrates (C-S-H) was observed in mixes prepared with GO. With percentage content of GO increases the size of the hydrated crystals increases indicating the pores being filled by GO nanosheets. In Fig. 8 (e) smooth GO sheets embedded between hydrated crystals leaving no rooms for pore capillaries which made the concrete less permeable thus, enhancing the durability and strength of the concrete.

CONCLUDING REMARKS

The current investigation suggested that inclusion of GO with even 0.02% by weight of cement has enhanced the strength by 12- 24% and durability properties but somewhat reduces the workability by 4% and water permeation properties tends to decrease with increase in GO content. The quality of the concrete mixes was improved as the microstructural analysis of the mixes truly indicates that GO acts as filler material at nano-scale by aiding in transporting water to the unreacted cement particles, hence, it can be concluded that the size of the hydrated crystals increases at 90 days curing age. So, if the future construction wants a durable with more enhanced strength, then inclusion of GO in a very small amount can be an option.

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