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Mechanical and durability performance of concrete incorporating graphene oxide

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ABSTRACT

This current investigation was conducted to explore the influence of graphene oxide (GO) on the mechanical and durability properties of normal concrete. A total of five mixes were prepared in addition of laboratory synthesized GO ranging from 0-0.8% with an increment of 0.02% by weight of cement. The synthesized GO was characterized using SEM, FT-IR and XRD techniques. The workability, initial surface absorption and sorptivity values were observed to have reduced 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. The mix with 0.08% GO showed better result compared to rest of the mixes with GO and control mix.

1 Introduction

Nanotechnology has changed the abilities, expectations and vision to control the material world. The advance in nano science may have a great impression in the field of construction materials. The potential of ordinary Portland cement (OPC) which is one of the largest possessions 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 material might definitely result in “smart” properties [1]. 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 [2-3]. The experimental

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results of mechanical strength were reported to 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 [4-5]. Graphene oxide (GO) is a graphene derivative, comprises of mono-layer of sp^2 – hybridized carbon atoms with oxygenated functionalities attached on the edges and basal planes of GO sheets. The intermolecular forces (van der Waals forces) between the GO nanosheets were significantly changed and therefore improve their dispersion in water. GO also exhibits high aspect ratio and large surface area. A high-performance concrete requires a considerably enhanced performances such as high strength, high durability, high chloride ion resistivity, high freeze resistance, high sulphate resistance, low shrinkage, and low carbon footprint, low abrasion, etc. than the conventional/normal concrete [6-9]. Nano reinforcement could be an answer to the expansion of infrastructure, high-rise buildings, bridges, marine or hydraulic works etc., which are generally under the attack of environment through alkalis and salts.

Addition of GO in cement-based composites have an adverse effect on the workability due to its large surface area that tend to absorb more water molecule to get wet and a bulky lateral size with high capacity for water retention created due to cluster of GO nanosheets [10-12]. Regardless of the above mention disadvantage of GO, incorporation of small content of GO of about 1% by weight of cement (bwoc) have improved the compressive strength by 63% [13]. The addition of 0.05% GO (bwoc) was reported to enhanced the compressive strength by 15-33% and flexural strength by 41-58% [6, 14]. Shang et al. have stated that the compressive strength with 0.04% GO inclusion to the cement paste was improved up to 15.1% compared to plain cement paste [5]. The compressive strength and tensile strength were increased by over 40% with 0.03% GO by weight of cement (bwoc) addition to OPC paste of 28 days curing age [15]. Mohammed et al. concluded that the addition of GO led to enhancement of water sorptivity and chloride penetration values whereas it reduces the pore size of the GO reinforced cement paste indicating the improvement of resistance to chemical ingress [9].

Shamsaei et al. prepared a comparison on mechanical performance of GO-reinforced cement-based composites from which an idea can be drawn that the percentage enhancement is dependent on the amount of GO, purity, water binder ratio, the dispersion technique and the matrix (paste and mortar). The enhancement in compressive, tensile and flexural strength were recorded to be 126.6% (0.5% GO content), 78.6% (0.03% GO inclusion) and 203% (0.3% GO addition) respectively [42]. Some researchers have quoted that GO inclusion accelerated the heat of hydration. The GO sheets with oxygenated functionalities make more approachable to the cement particles, thus allowing the nanosheets to act as nuclei for the cement phases boosting the reaction of cement with water [16-17]. Wang et al. have mentioned that the microstructure of the GO reinforced cement matrix have massive crystal structure covering micro pores, implying leaching of calcium hydroxide (CH) during the hydration stages have been improved at 28 days curing age [6].

The GO inclusion cement matrix is in trend due to its exceptional properties such as dispersibility, catalytic behavior, transport properties, electromagnetic shielding etc. [18-19]. Interest in GO has grown worldwide in various fields among carbon-based nanomaterials. As GO is way cheaper compared to extremely expensive multi-walled carbon nanotubes (MWCNTs), single-walled carbon nanotubes (SWCNTs) and carbon nanofibers (CNFs) which is 250, 1280 and 218 times more than price of GO per 100g respectively [20]. Thus, GO became the best candidate for this investigation as the quantity of GO which is to be used in concrete. This paper is to develop a nano-reinforced concrete composite in addition of GO with varying percentage by weight of cement. No study has been reported on GO inclusion in concrete (with natural coarse and fine aggregate) composites with regards to the workability, compressive, tensile strengths, water permeation, sorptivity, quality of the nano-reinforced concrete composites and the cost analysis to get a clear picture whether this investigation will be helpful for practical application in construction industry.

2 Experimental Program

2.1 Materials

OPC 43 grade, meeting the specification as per IS 8112:1989 and ASTM C150 [21-22], fine aggregate used were locally available which lie in Zone II, conforming IS 383: 1970 and ASTM C136 [23-24], and natural coarse aggregate of maximum nominal size 12.5 mm. The fineness modulus, specific gravity and bulk modulus of fine and coarse aggregate were 2.74 and 7.54, 2.67 and 2.64, 1675 kg/m³ and 1690 kg/m³ respectively. The particle size distribution curve for both fine and coarse aggregate has been shown in Fig. 1. Table 1 shows the physical properties of the OPC. And MG SKY 8765 (polycarboxylate ether-based) super plasticizer were procured from local dealer conforming to IS 9103:1999 and ASTM C 494 [25-26].

GO was synthesized using modified Hummer’s method [27-28], graphite powder (10g) and NaNO₃ (5g) were allowed to react with conc. H₂SO₄ (230 mL) at 20°C temperature on heater stirrer for over a period of 30 minutes and then gradually added KMnO₄ (30g). The resulting mixture were heat up to 35°C for another 30 minutes and slowly diluted with 230 ml water. The mixtures were then heated up to 98°C for 15 minutes and 3% hydrogen peroxide (H₂O₂) were added to complete the reaction followed by 400 mL deionized (DI) water and allowed to settle. And finally, washed the dilute solution of GO with 1:10 HCl and 4 L DI water until the pH of the filtrate was up to 7. The filtered paste was then oven dried at 65°C for 24 hours to obtained required GO powder. The chemicals used were procured from LOBA chemicals. And the GO obtained was then characterized structurally using SEM/EDX, XRD and FT-IR techniques to check the composition of the near surface, crystalline structure and to identify the functional groups present.

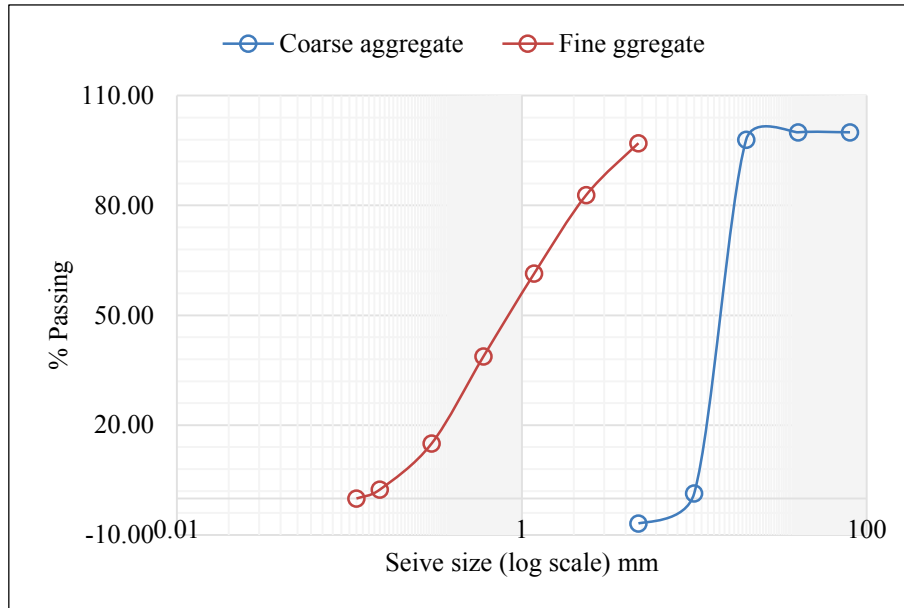


Fig. 1. Particle size distribution of coarse and fine aggregate.

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

2.2 Mix proportions and casting

A total of five mixes were prepared and tests were performed on both fresh concrete and hardened concrete of different curing ages. The binder, fine and coarse aggregate was kept constant at 384 kg/m³, 715 kg/m³ and 1113 kg/m³ respectively (1:1.86:2.89) with w/b ratio 0.50. GO were added with varying percentage content by weight of cement from 0 to 0.08% with an increment of 0.02%. The mix proportions are given in Table 2. The GO was allowed to dispersed in an aqueous solution

containing 0.05% by weight of cement (bwoc) super plasticizer (kept constant for all the mixes) in a sonicator with ultrasonic energy of about 2000J/min at cycles of 20s operated at 50% constant amplitude for 30 mins. The exfoliating was carried out with different loading levels of GO (0.02%, 0.04%, 0.06% and 0.08%). A homogeneous mixture of cement, sand, coarse aggregate and 70% of water was prepared in a power-driven laboratory (220-240 V power input) mixer of 300L capacity at 22-24 rpm for 3 mins. And the remaining 30% water was mixed with the dispersion of GO and added to the mixer then rotated for another 2 mins. The fresh concrete was then poured on the oiled moulds of respective shape and sample sizes. After 24 hours and water cured for 7, 28, 56 and 90 days in a temperature ($27 \pm 2^\circ\text{C}$ and 90% humidity) regulated curing tank till the age of testing.

Table 2. Quantity of materials used in kg/m³

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

2.3 Test Method

2.3.1 Workability

Workability is the ability to be mixed, handled, transported and placed with a minimum loss of homogeneity i.e. segregation or bleeding. Workability of the fresh concrete was performed as per IS: 6461 (Part VII)- 1973 and BS EN 12350 -2 [29-30] and the designed slump value for the control mix i.e. 0% GO content concrete mix was 100 mm. The slump value was evaluated using slump cone test after pouring the fresh concrete in the cone (200 ϕ mm, 100 ϕ mm and 300 mm height) in four layers (1/4 of height of the mould) by tamping each layer 25 times and lifted and the height of coned concrete was measured. The slump value for different mixes were recorded and the influence of GO in the fluidity of the nano-reinforced concrete composites.

2.3.2 Mechanical properties

As per code IS: 516- 1959, ASTM C39/C39M and IS: 5816 -1999 [31-33] were followed to conduct the tests on 100 mm cubes for compressive and splitting tensile strength (diagonally splitting the cube, stress (σ) = 0.5187 Load/Side²) for each curing age 7, 28, 56 and 90 days on 200T capacity Compression Testing Machine (CTM). The load was allowed gradually without shock and increased at the constant rate of 3.5 N/mm²/minute until cube failed.

2.3.3 Ultrasonic Pulse Velocity

The ultrasonic pulse velocity was generated by an electro-acoustical transducer, transmitted on one surface of the concrete cube and receiving (frequency of 54 KHz) the same by a similar transducer in contact with the surface at the other end. A thin layer of coupling gel was applied to facilitate higher signal. The ultrasonic pulse velocity was carried out according to IS 13311(Part 1): 1992 [34]. The principle of evaluating the quality of concrete comparatively higher velocity was obtained on the Pundit Lab when the quality of concrete in terms of density, homogeneity and uniformity is good. In case of poorer quality, lower velocities are obtained. Ranges of velocities of UPV Test are given in IS 13311(Part 1): 1992. The tests were conducted for 28, 56 and 90 days curing period.

2.3.4 Test for water permeation property

2.3.4.1 Initial Surface Absorption

The initial surface absorption test (ISAT) were performed on 150 mm cubes, as per BS 1881: Part 208:1996 [35], this was conducted to test the rate of flow of water into concrete surface per unit area at specified interval. The specimens were oven dried at 105°C until the weight was not more than 0.1%. The setup for the test was made in such a way that a 78mm diameter cap is clamped to the cube surface providing an area of water contact with the surface to be tested of not less than 5000 mm². The water was allowed to flow through the inlet from the reservoir of diameter 100 mm which was fitted with tap to an outlet attached with scale. The water head was maintained between 180 mm to 220 mm for the test. The tap was opened and measurements taken for 1 minute at interval 10 min, 30 min and 60 min from start of the test. The rate of flow was measured in mL/m²s. The tests were performed on 28, 56 and 90 days curing period.

2.3.4.2 Sorptivity

A 100 X 200 mm cylinder was cut into 100 X 50 mm disc and preconditioned by oven drying at 105°C until the difference in weight of the specimen was not more than 0.1%. The test was performed as per ASTM 1585-04: 2009 [36]. The circumferential area was coated with epoxy paint and both the circumferential and top surface were tightly sealed using polyethylene sheets to block evaporation. The weight of the disc was measured at 0, 1, 5, 10, 30, 60 minutes and for every one-hour interval up to 6 hours from the start for initial rate of absorption Trends of plots of cumulative water absorption and square root of time (mL/√S) for all concrete mixes containing graphene oxide (GO) 0%, 0.02%, 0.04%, 0.06% and 0.08%.at 28, 56 and 90 days curing periods were evaluated.

2.3.5 Microstructural observation

The samples of the GO-reinforced concrete mixes using SEM EDX on JEOL JSM-6510LV. The samples were made conductive by gold coating prior to microscopy analysis by sputtering method. To examine the samples at 90 days of curing, the secondary electron detector was used at high vacuum at an accelerating voltage of 15 KV with working distance of 12 mm.

3 Result and discussion

3.1 Structural characterization of synthesized GO

The structural characterization of synthesized powdered GO was approved using field emission scanning electron microscopy (FE-SEM) to attained the surface morphology. Fig. 2 (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 van der Waals force which under the microscope appears to be created.

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 sp² hybridization. The FT-IR spectra as shown in Fig. 2 (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 support 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 K α = 1.540 Å, with fixed divergence slit of 0.2177° having step time of 12.0650 s and step size of 0.0080 was examined at angular range of 2 θ = 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. 2 (c) indicating an interlayer spacing of 0.814 nm and presence of diffraction peak at 2 θ = 10.8° which is assigned to (001) diffraction peak of graphitic oxide.

3.2 Workability

The slump value of the control mix was designed for 109 mm. Fig. 3 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. Comparable results were reported on reduction of fluidity and increase in viscosity with increase in GO content [10-11]. Shang et al. and Gong et al. also concluded that the oxygenated functionalities attached on the GO nanosheets absorbs the water molecule and remains entrapped due to flocculation and agglomeration formation caused by electrostatic interaction between GO and cement particles [5, 15]. 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.

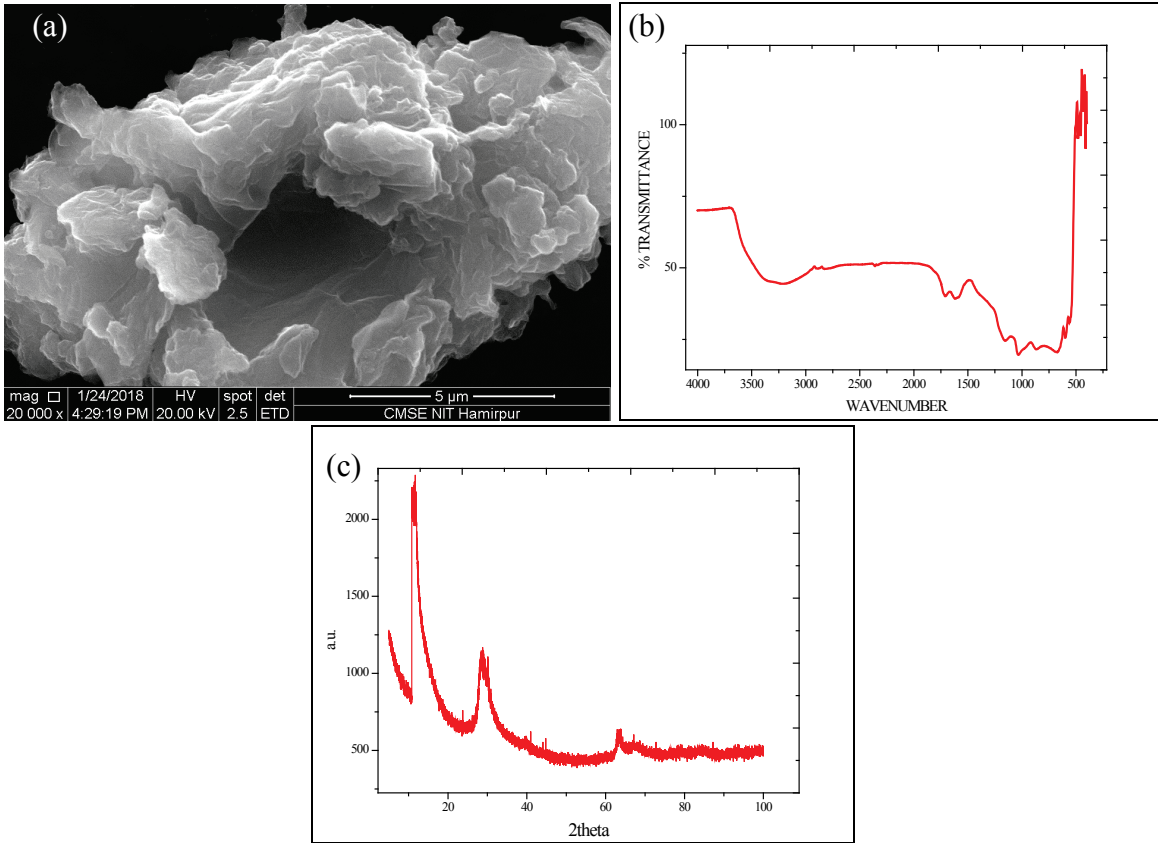


Fig.2. (a) SEM image, (b) FT-IR and (c) XRD of GO

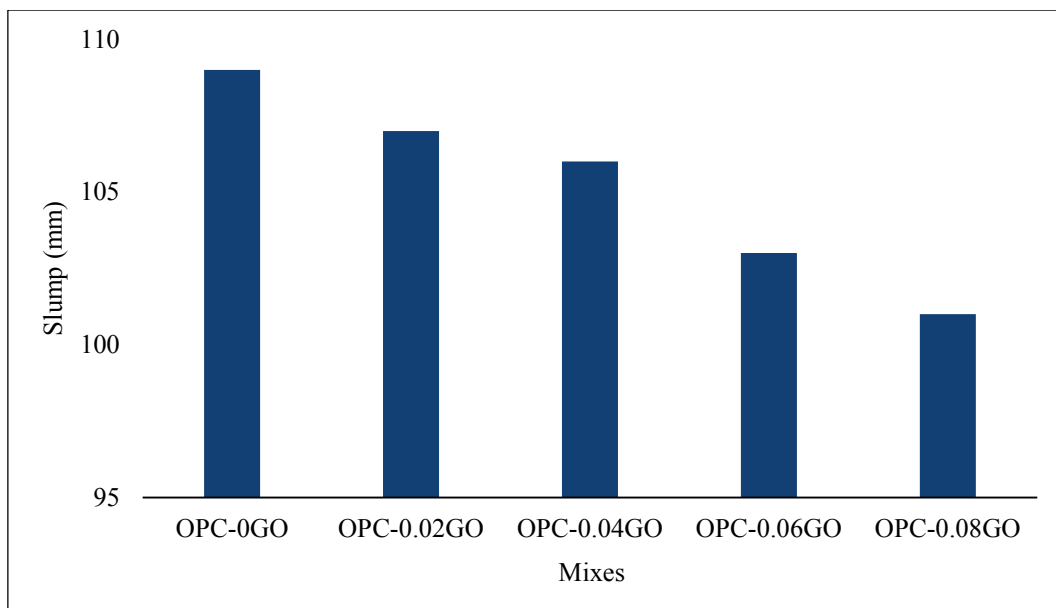


Fig. 3. Slump value of different mixes

3.3 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. 4 (a) & (b) and 5 (a) & (b). 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. Previous studies have mentioned that inclusion of GO nanosheets in cement-based composites has improved the compressive strength at large scale [5, 6, 13, 15, 37,38, 40]. The functional groups on the surface of GO nanosheets acts as active sites that attracts the cement hydrates. The surface area to mass ratio of GO nanosheets assists the nucleation for formation of cement hydrates and develops strong covalent bonds at the interface of cement matrix and dispersed phase (i.e. GO). Thus, a nano-reinforced concrete composite was achieved.

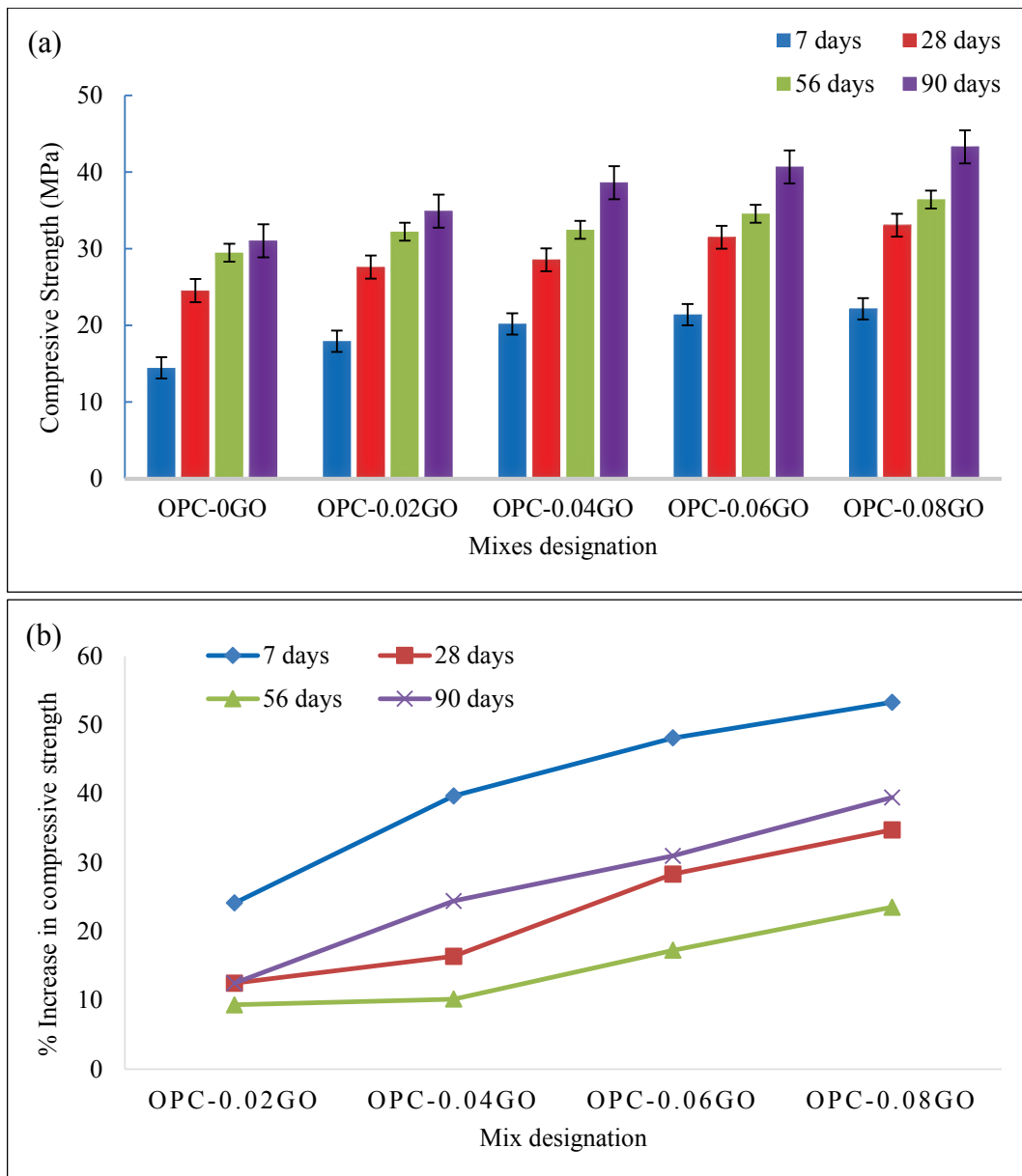


Fig. 4 (a). Comparison of compressive strength, (b) Percentage increase in compressive strength w.r.t control mix

3.4 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. 6. 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.

3.5 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. 7.

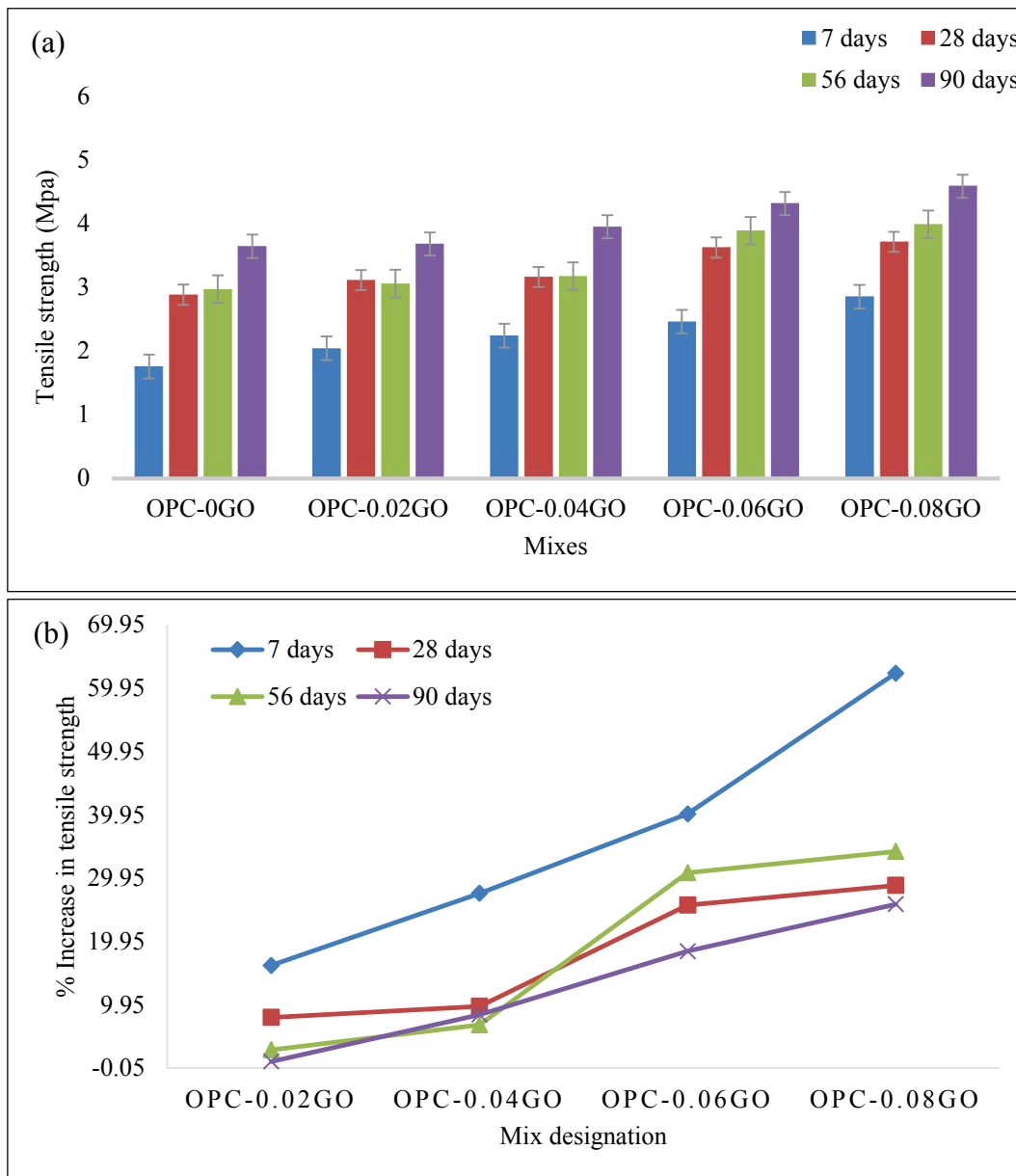


Fig. 5. (a). Comparison of tensile strength, (b) Percentage increase in tensile strength w.r.t control mix

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.

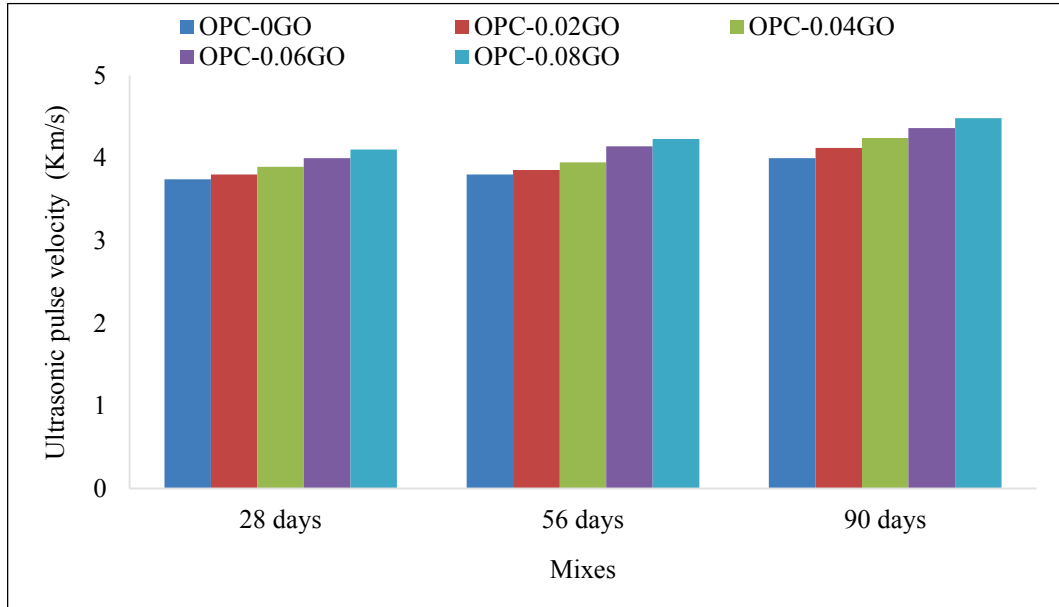


Fig. 6. Ultrasonic pulse velocity of different mixes

3.6 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. 8, 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.

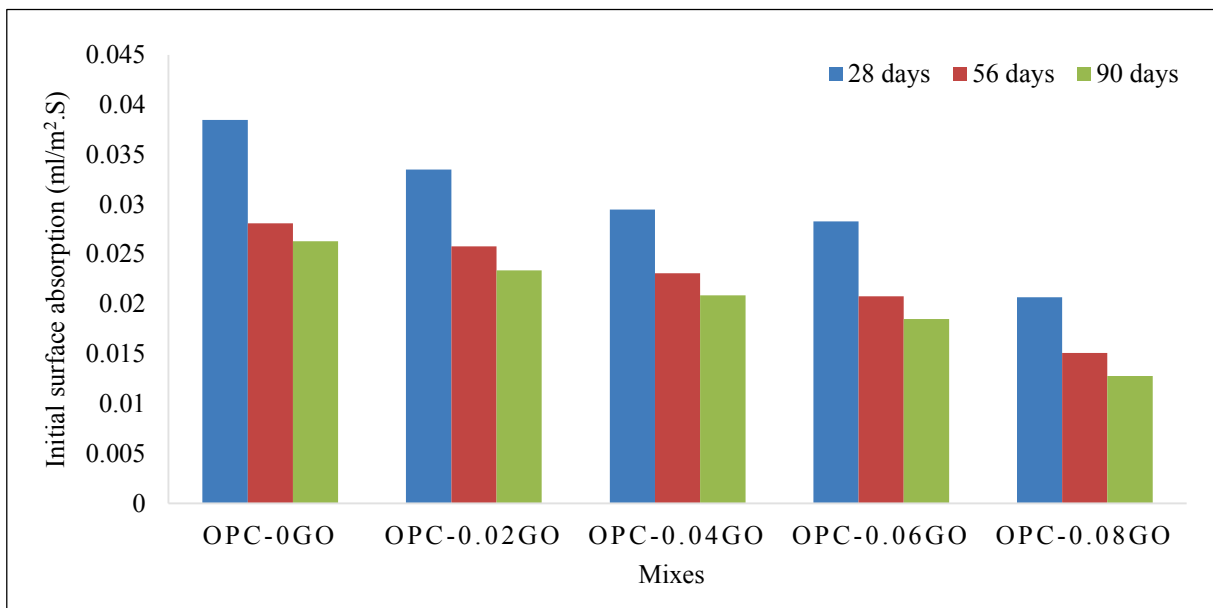


Fig. 7. Initial surface absorption result for all the mixes at 28, 56 and 90 days curing age

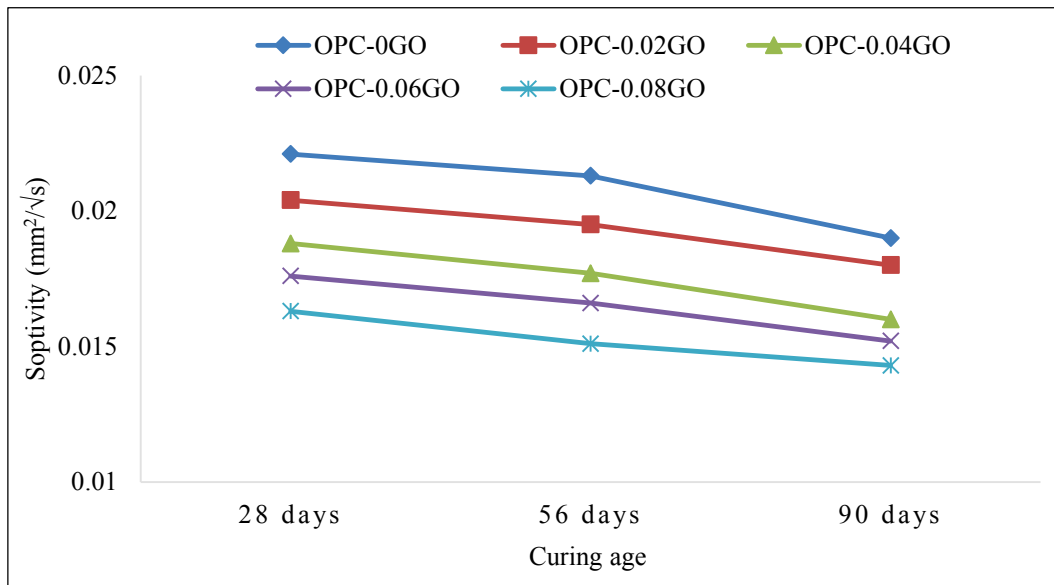


Fig. 8. Initial surface absorption of all the mixes at 28, 56 and 90 days curing age

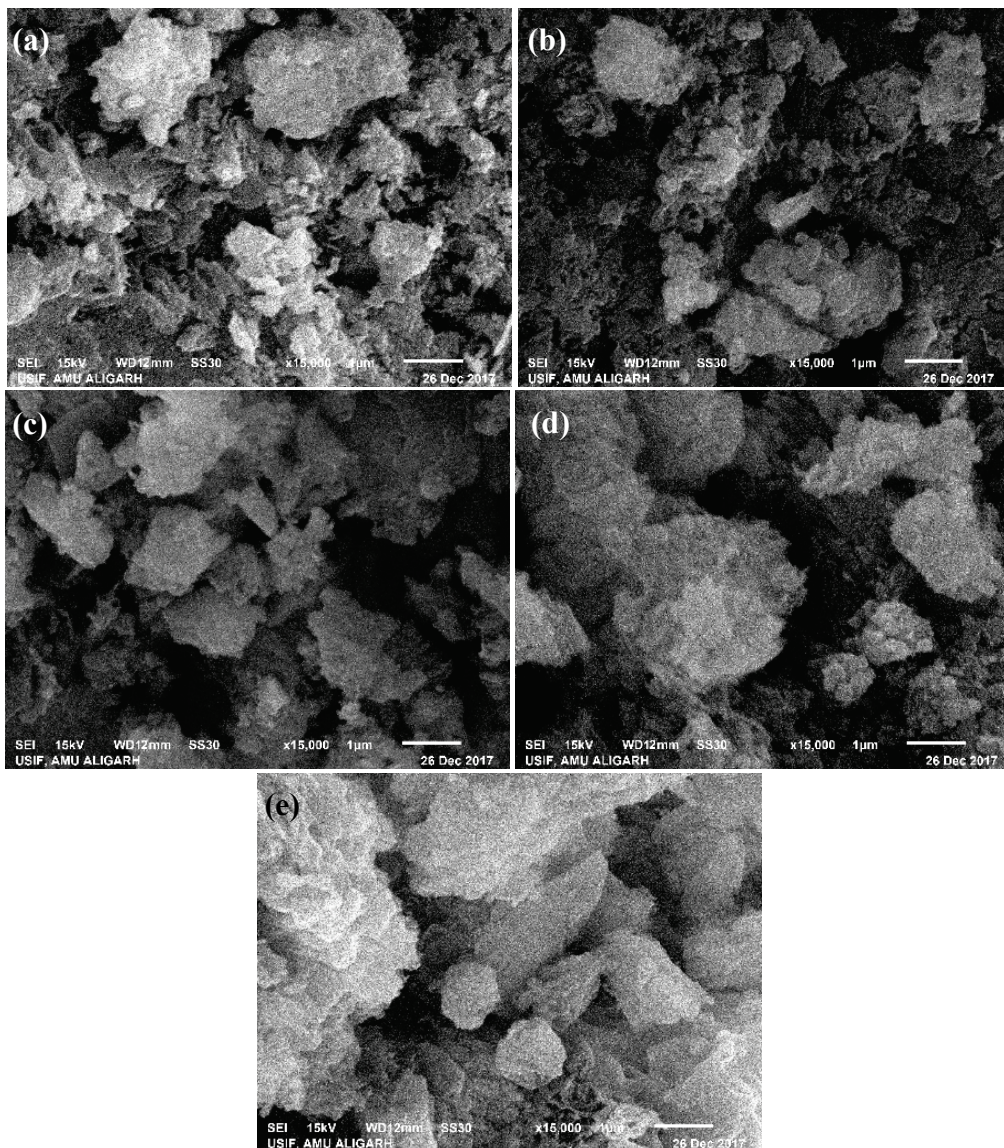


Fig. 9. SEM of mix with (a) GO 0% Control mix, (b) GO 0.02%, (c) GO 0.04%, (d) GO 0.06% and (e) GO 0.08%.

3.7 Microstructural observation

The scanning electron microscopy was carried out to study the comparison in structural morphology of the GO reinforced concrete composites and control mix. Fig. 9. 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

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.

4 Cost Analysis

The cost of casting the mixed designed were analysed and reported in Table 3. The cost of different nano-reinforced concrete composites was evaluated using the commercialized market prices of the materials. The economy index (compressive strength/ cost per m³) was observed to have maximum value at mix M4 (with 0.06% GO inclusion) compare to rest of the mixes. It shows that mix M4 is the optimum mix.

Table 3. Cost Analysis

Material (kg)	Cost (Rs/Kg)	Mix combination				
		OPC-0GO	OPC-0.02GO	OPC-0.04GO	OPC-0.06GO	OPC-0.08GO
OPC	7.48	2872.32	2872.32	2872.32	2872.32	2872.32
FA	1.5	1072.5	1072.5	1072.5	1072.5	1072.5
CA	1	1113	1113	1113	1113	1113
GO	1200	0	92.16	184.32	276.48	368.64
SP	118	22.656	22.656	22.656	22.656	22.656
W	0.05	9.6	9.6	9.6	9.6	9.6
Total (INR)	-	5090.08	5182.24	5274.4	5366.56	5458.72
Compressive Strength (MPa)	-	36.45	46.21	51.17	53.74	54.438
Economy Index (Strength/Cost)	-	0.00716	0.00892	0.0097	0.01001	0.00997

5 Conclusion

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. GO inclusion could accelerated the cement hydration process as it provides nucleation sites to hydrates of cement. But the workability was reduced by 4% due to the agglomeration of GO causing the water molecule to remains entrapped. The water permeation properties tend to decrease with increase in GO content. And the quality of the concrete mixes was improved as the microstructural analysis of the mixes truly indicates that GO acts as filler material mending the microcracks at nano level by aiding in transporting water to the unreacted cement particles. The microstructure of the mixes with GO has shown that inclusion of GO has improved the concrete composites at the micro level as the formation of massive hydrated crystals obtained at 90 days curing age was observed. And from economical index, it can be concluded that mix with 0.06% GO is the optimum mix. Hence, incorporation of GO reinforced the concrete thereby

improving the mechanical and durability properties of the concrete. GO can be a promising nanomaterial and best candidate among the other nanomaterials in the near future for making more durable and smart concrete with more enhanced strength.

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