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ENHANCEMENT OF CONCRETE MICROSTRUCTURE USING GRAPHENE OXIDE AS A CEMENT ADDITIVE: AN EXPERIMENTAL STUDY.

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The current study investigates the graphene oxide's (GO) effect on the mechanical properties of standard concrete. To compare, conventional concrete was designed to attain a compressive strength of 30 MPa, and GO was added to create graphene oxide concrete. The workability and compressive strength of various concrete mixtures were evaluated, and the best mixture, GC2 (0.2% GO), demonstrated a compressive strength of 42 MPa and a flexural strength of 6.0 MPa after 28 days of curing. Additional analysis of GC2 using SEM, EDAX, and XRD revealed a more compact microstructure of hydration products. The results of this study suggest that the mechanical properties of concrete are enhanced by the inclusion of graphene oxide (GO).

Keywords: Concrete, Compressive strength, Flexural strength, Graphene oxide, Microstructure analysis, Mechanical properties enhancement, Workability.

1. Introduction

The widespread use of concrete in the construction sector is due to its favorable properties such as durability, workability, and adequate strength. However, its low tensile strength requires reinforcement with steel or fibers. Despite this, fibers have limited effectiveness in preventing microcrack initiation and have less surface area for the hydration process, leading to concerns about the environmental impact of increased cement production1. To address these concerns and improve concrete's mechanical properties, alternative materials or cement substitutes must be explored. This study focuses on the use of nanomaterials, such as graphene oxide (GO), to further improve the quality of the microstructure of cement. GO is a two-dimensional carbon sheet material with oxygen containing functional groups that, when added to cement, refine the pore structure and form flower-like crystals, improving mechanical properties such as adhesion and filling in the transition zone2. This novel approach using nanotechnology and nanomaterials is expected to bring about significant improvements in concrete properties.

2. Literature review

A study conducted by P. K. Akarsh et al. in 2020 incorporated graphene oxide and silica fumes into high-strength concrete to create a pavement quality mix of M50 grade. Three different kinds of concrete mixture were produced: Silica fume concrete, Graphene oxide concrete, and Graphene oxide and Silica fume Blended concrete. The authors compared the performance of the blended concretes to that of the standard concrete by conducting tests on workability, compressive strength and flexural strength.

They selected the most promising combinations for further investigation based on the trial mixes and test results3.

S.C Devi et al., in the year 2020 attempted to explore the mechanical and durability capabilities of concrete having graphene oxide. They studied five mixes with 0%, 0.02%, 0.04%, 0.06%, and 0.08% of GO by weight of cement. They have reported that the mix having 0.08% GO has a Compressive and tensile strength in relative to the other mixes. Higher GO content is reported to give the reduced sorptivity, and permeability for nano reinforced type concrete mixtures. Later portion of the study included the microstructure examination utilizing SEM/EDX and additionally, the UPV test was conducted to evaluate the quality of the concrete mixture4.

M. Somasri et al., in the year 2021 studied the rheological and mechanical characteristics of self-compacting concrete with GO consolidation. The authors attempted to track down and quantify the expansion in compressive, flexural and split tensile strength and results are 17%, 40%, and 28% respectively for a 0.02%, 0.04%, 0.06%, 0.08% and 0.1 % of GO by weight of cement. The rheological and mechanical characteristics of HSSCC were evaluated for a curing duration of 7, 28, 56 and 90days4.

S. Balaji et al., in the year 2021 conducted a survey on mechanical qualities like flexural, tensile, and compressive strength. The survey was carried out at different ratios of GO blended concrete composites and further the microstructural analysis was carried out through SEM, XRD, mercury intrusion porosimetry, Fourier transform infrared spectroscopy and EDAX. A collection on various techniques to synthesize GO from graphene is presented. The presented survey suggests that using GO as an additional ingredient in concrete helps to improve the characteristics of concrete5.

Su-Jin Leeu et al., in the year 2020 have examined the feasibility of utilizing GO as a supplement to work on the strength of cementitious composites. The authors have tried to evaluate a cementitious composite's compressive strength and pore structure. Wherein 0.025% weight of cement was replaced by GO. For comparison purposes, cementitious composites were substituted with ordinary cement concrete additives like GGBS, silica fume, and flash. Authors reported an improvement in compressive strength by 10.7 -41.5%, relative to plain concrete mixture. The pore structure investigation uncovered that most of the pores were micropores having widths not greater than 2.5nm, adding to the strength parameter of concrete5.

3. Materials and Methods

1. Materials

3.1.1 Cement

According to Indian standard code 12269-2013, In this experiment, 53-grade ordinary Portland cement (OPC) was used. To determine the physical requirements, cement underwent laboratory tests. The test results are shown in Table 1.

Table 1 - Test results of cement

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Type of cement	Property	Results	The limits as per IS 12269-2013	Test methods
53 grade OPC	Standard consistency	29 %	-	IS 4031 (part-4) -1988
	Initial setting time	90 mins	Min. 30 mins	IS 4031 (part-5)-1988
	Final setting time	120 mins	Max. 600 mins	
	Specific gravity	3.14	-	IS 4031 (part-11)-1988

3.1.2 Aggregates:

The well-graded, crushed angular form of coarse aggregates specified in IS 383-2016 is being used, while man-made sand is being utilized as the fine aggregates. The results of the tests conducted on the fine and coarse aggregates can be found in the below tables 2 and 3.

Table - 2 Test results of fine aggregates

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Property	Results	Limits	Test methods
Specific Gravity	2.63	-	IS 2386 (part-3)-1963
Water absorption	1.01 %	Maximum 2 %	IS 2386(part-3)-1963
Fineness Modulus	2.681	2.6 to 2.9	IS 2386(part-1)-1963

Table - 3 Test results of coarse aggregates

Property	Results	Limits	Test Method
Specific gravity	2.72	-	IS 2386 (part-3)-1963
Water absorption	0.55 %	Maximum 0.5 %	IS 2386(part-3)-1963
Fineness Modulus	8.688	> 5	IS 2386(part-1)-1963

3.1.3 Superplasticizer:

Conplast SP430, a chemical that complies with IS 9103-1999, was used. According to the company's recommendations, the dosage varies from 0.6 to 1.5% by weight of cement. It was blended with enough water and added during the concrete mixing process.

3.1.4 Graphene oxide (GO):

"Carborundum Universal Limited", company sponsored graphene oxide. The maximum amount of GO that can be dispersed in water, according to the company's instructions, is 30g/liter. For the current study, five different dosages were selected: 0.1%, 0.2%, 0.3%, 0.4%, 0.5% by the weight of cement. Table 4 lists the characteristics of GO.

Table - 4 Properties of GO

Property	Material Specification
Form	Fluffy powder
Colour	Black
Odour	Odourless
Surface area	200 m^2 /gm
Average thickness	1-4 nm
Average lateral dimension	5-10 micrometer
Bulk density	0.1 *g/cc*
Purity	99%

3.2 Work Methodology:

3.2.1 Adjustment of W/C Ratio and Superplasticizer Dosages:

The mix design was conducted in conforming with IS 10262:20196. To attain the desired strength and workability within the range of 100-125 mm, several trial mixes with varying water-cement ratios (w/c) and superplasticizer dosages were carried out. The workability was assessed using the slump test. After setting the w/c at 0.55, the appropriate grade of concrete was achieved by examining the slump. A dose 0.5% superplasticizer and 0.55 water/cement ratio were established for the conventional concrete.

3.2.2 Preparation of Graphene oxide solution:

To form an aqueous solution, the powder form of GO is utilized. A magnetic stirrer is used to disperse GO in water, which employs a rotating magnet to generate a magnetic field. Stirring activity is induced by dropping a magnetic bead into the water. The device can rotate between 200 and 1800 revolutions per minute. To enhance the adhesion property of graphene oxide, superplasticizer and water were added at the beginning, in a magnetic stirrer. Subsequently, the necessary quantity of GO is added to it. After mixing for approximately 15 minutes at an increasing speed every 5 minutes, the solution is added to the mixture. The volume of this aqueous solution is subtracted from the total volume of water required. When it comes to nanoparticle dispersion, an ultrasonic processor is the most appropriate option3. The preparation of the GO solution using a magnetic stirrer is depicted in Fig.1.

Fig.1 - Preparation of GO solution.

3.3 Mix proportion design details

Two distinct concrete mix types were investigated. The first is plain conventional concrete, while the second is concrete that has been infused with graphene oxide. The experiment's main goal is to evaluate the impact of graphene oxide.

3.3.1 Conventional concrete mix

The mix proportions for conventional concrete were determined using the method recommended by IS 10262-2019 for the mix design of conventional concrete. In this experiment, mix design for M30 grade standard conventional concrete was made with the cement of 333 kg/m3, W/C ratio of 0.55, fine aggregates of 739 kg/m3, coarse aggregates of 1152 kg/m3 with superplasticizer dosage of 1.667 lt /m3 to bring about 100- 125mm slump value.

3.3.2 Graphene oxide concrete mix (GC):

GO is added on to the weight of cement-like 0.1%, 0.2%, 0.3%, 0.4% and 0.5%. The other ingredient quantity remains the same as the conventional concrete mix. Table 5 shows mixed proportion data of GC.

Table 5 - The mix proportions of Graphene oxide concrete (GC) in Kg/m^3.

GO % (by wt. of cement)	Mix ID	Cement	Water	Coarse aggregate	Fine aggregate	Superplasticizer	GO (*gm*)
0%	CC	333	186	1152	739	3.3	0
0.1%	GC1	333	186	1152	739	3.3	333
0.2%	GC2	333	186	1152	739	3.3	666
0.3%	GC3	333	186	1152	739	3.3	999
0.4%	GC4	333	186	1152	739	3.3	1332
0.5%	GC5	333	186	1152	739	3.3	1665

3.4 Blending of materials, casting, and curing.

The designed mix was batched using the appropriate quantities. For conventional concrete, cement, fine aggregate, and coarse aggregate were dry mixed in electrically driven mixer for about 3 minutes, as per the specifications of IS 516:19597. Subsequently, water along with superplasticizer was added and mixed for another 3 minutes until a steady mix was achieved.

To produce graphene oxide concrete (GC), cement, fine aggregates, and coarse aggregates were thoroughly combined in a dry environment condition. The GO solution containing GO and superplasticizer is next poured, followed by water. Up until a consistent form of GC is formed, there is a 3–4minute period of thorough mixing. The water used to create the aqueous GO solution must be subtracted from the total weight of water while preparing GC.

The mixed samples were poured into appropriate moulds. For compaction vibrating table was used, with the moulds being vibrated for 5-10 seconds after each layer was filled, with a minimum of three layers. After providing a smooth finish to the top layer, it was left undisturbed to set. Subsequently, the samples were kept for curing at a steady temperature of 27±3°C for a specific duration.

3.5 Testing of concrete mixes

3.5.1 Fresh properties test

Concrete that is workable has very little particle-to-particle friction or can be compressed tightly enough to overcome the form work's surface or embedded reinforcement's frictional resistance8. Workable concrete can be mixed, placed, compacted, and finished without difficulty. The workability characteristics of the newly made concrete mixtures were examined using the slump cone apparatus and compaction factor apparatus, as per the specifications of IS 1199:20189.

3.5.2 Tests of Hardened Properties:

Tests on compressive strength, split tensile strength, and flexural strength were carried out to assess the qualities of the concrete in its hardened state. And it was conducted in accordance with the relevant Indian standards.

3.5.3 Microstructure analysis:

The heterogeneous microstructure of concrete comprises three components, the cement pastes the pore structure, and the interfacial transition zone between the cement paste and aggregates3. Improving these components enhances the mechanical strength and durability of concrete, as stated in 3. To examine the surface morphology of the concrete, scanning electron microscopy (SEM) is utilized3. Additionally, X-ray diffraction technique, serves as a quick analytical tool to identify the material's crystalline phases10. Energy dispersive analysis by X-ray was performed to investigate elemental composition.

4. Results and discussions

4.1 Workability

Graphene oxide possesses more surface area as it absorbs additional water for making its surface wet1. Due to this fact and to maintain a medium degree of workability, superplasticizer was increased to 1% for graphene oxide mix wherein 0.5% of weight of cement of superplasticizer was sufficient for the conventional mix to achieve a medium degree of workability. The function of a superplasticizer is important in nano-materials-based concrete mixes as it aids in proper dispersion of nanomaterial 11. Table 6 lists the findings of the slump test, whereas Table 7 displays the results of the compaction factor test.

Table 6 - Results of slump test

Type of concrete	Superplasticizer dosage (%)	Slump value (*mm*)	Pattern of slump	Degree of workability
Conventional concrete	0.5 %	100	True slump	Medium
Graphene oxide concrete	1.0 %	89	True slump	Medium

Table 7 - Results of compaction factor test

Type of concrete	Compaction factor	Degree of Workability
Conventional concrete	0.90	Medium
Graphene oxide concrete	0.86	Medium

4.2 Mechanical properties

4.2.1 Compressive strength

Compression test was performed on a compression testing machine for a period of 3, 7, and 28 days of curing. The concrete mixture contained GO with a dosage of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% by weight of cement, and the corresponding results of the compressive strength tests are presented in Table 8. The outcome of the compressive strength test showed that the compressive strength increased up to a certain point with increase in the dosage of Graphene oxide. However, after that point, the compressive strength decreased due to insufficient dispersion3. The mixture containing 0.2% GO displays the maximum compressive strength of 42.228 MPa I.e., 18.15% increase when compared with CC mix of strength 35.738 MPa due to proper dispersion at the completion of 28 days of curing. The GC3, GC4 and GC5 mix shows lesser strength than the GC2 mix and hence 0.2% of GO addition was considered to be the optimum dosage and further, the flexural and split tensile strength tests were performed with 0.2% GO.

Lin et al. (2016)12 reported that GO functions as a catalyst and facilitates the cement hydration process while preserving the oxygenated functional groups on the GO nanosheets 12.
The functional groups act as active sites to attract cement particles 13. GO's high surface area to mass ratio promotes nucleation and facilitates the development of cement hydrates, creating robust covalent bonds at the interface of the cement matrix and GO 14. Consequently, adding GO enhances the strength of concrete composites at the nanoscale, resulting in increased compressive strength compared to the control mix 14.

Table 8 - Compressive strength test results

Mix name	Mix constituent	Compressive strength		
		3days	7 days	28 days
CC	0%	27.63	31.49	35.74
GC1	01% GO	27.37	32.30	38.33
GC2	0.2 % GO	27.67	34.28	42.22
GC3	0.3% GO	27.52	31.71	39.88
GC4	0.4% GO	27.11	29.28	37.50
GC5	0.5% GO	27.18	28.89	36.88

4.2.2 Flexural strength test

From the outcome obtained by the compressive test, it is suggested that a 0.2% dosage of GO should be used for conducting flexural strength tests. The findings from the flexural strength tests showed a 24.8% increase in strength, with a flexural strength of 5.614 MPa for the GC2 mix and 4.496 MPa for the CC mixture after a duration of 28 days of curing. The incorporation of GO inside the cement-based composites has been shown to speed up the hydration rate, enhance the tensile and flexural strength, and improve mechanical interlocking found at the interface between the GO-infused cement matrix 13. For a duration of 3, 7, and 28 days of curing, Table 9 documents flexural strength findings for the CC mix and GC2 mix.

Table 9 - Flexural strength test results

Mix name	Flexural strength		
	3 days	7 days	28 days
CC	3.32	3.90	4.5
GC2	3.86	4.64	5.61

4.2.3 Test for Split Tensile Strength

The outcomes of the split tensile strength test are demonstrated in Table 10, which indicate a 4.6% increase (3.182 MPa) in strength for the GC2 mix compared to the CC mix (3.042 MPa). This improvement in strength is attributed to the densification of concrete as the pores are filled, resulting in improved mechanical properties 15,16. The improvement in both compressive and tensile strength in graphene oxide-infused concrete is attributed to the "bringing action" of GO sheets, which promote the formation of strong hydration products within the concrete 17.

Table 10 - Split tensile strength test results

Mix name	Split tensile strength		
	3 days	7 days	28 days
CC	1.77	2.22	3.04
GC2	1.84	2.38	3.18

4.3 Microstructural analysis

4.3.1 SEM analysis

SEM testing was carried out on the hardened concrete samples after a period of 28 days of curing to examine in detail the surface morphology and microstructure of concrete.

Fig.2 presents SEM photos from CC specimen (a to d) and Fig.3, (a to d) shows SEM photos of hardened concrete from the GC2 specimen. The sample was taken out of the cube's center portion and examined for compression before being subjected to SEM. The CC and GC2 specimen images are displayed at three different scales, namely 20µm, 10µm, 2µm and 5µm.

SEM analysis is a valuable technique that can uncover crucial information about primary hydration products, such as layered calcium hydroxide (Ca(OH)2), needle-like ettringite, and the fibrous calcium silicate hydrates (C-S-H), as well as the inter transition zone which is present between cement hydrates particles and aggregates 3. In Fig (b), fibrous CSH phases at a scale of 2µm and needle-like ettringite at a scale of 5µm are evident. These dense hydration products are visible throughout the cement matrix at a scale of 2 µm, and the calcium silicate hydrates, which are one of the appealing hydration products, is distributed in the form of a dense sponge-like structure, as illustrated in Fig.2 (b).
Fig.3 (a to d) displays SEM pictures of a fractured area of the GC2 sample after 28 days of curing, where needle-shaped ettringite and the widespread dense sponge matrix of C-S-H can be observed mostly at a size of 2µm. Calcium hydroxides are also visible in the form of laminated sheets in Fig.3 (a). Dense hydration products are visible throughout the matrix, as unveiled in Fig.3 (b). However, since the GO content added is only 0.2% by weight of cement, it is challenging to identify the distribution of GO powder in the specimen.
Fig.3 (c) depicts a larger view of the particular section with thick fibrous C-S-H and sharp needle-shaped AFt phases at a scale of 1µm.
The most favorable hydration products like thick fibrous C-S-Hs, progressively spread, merged, and stuck to the GO, helping to strengthen and reduce permeability. Cement particles and GO have a strong bond. Ettringite and C-S-H are typically found in the pores between the zones. Due to the nucleation of hydration products, these pores will accelerate the hydration process

if they are filled with micro and nanomaterial 17, 18.

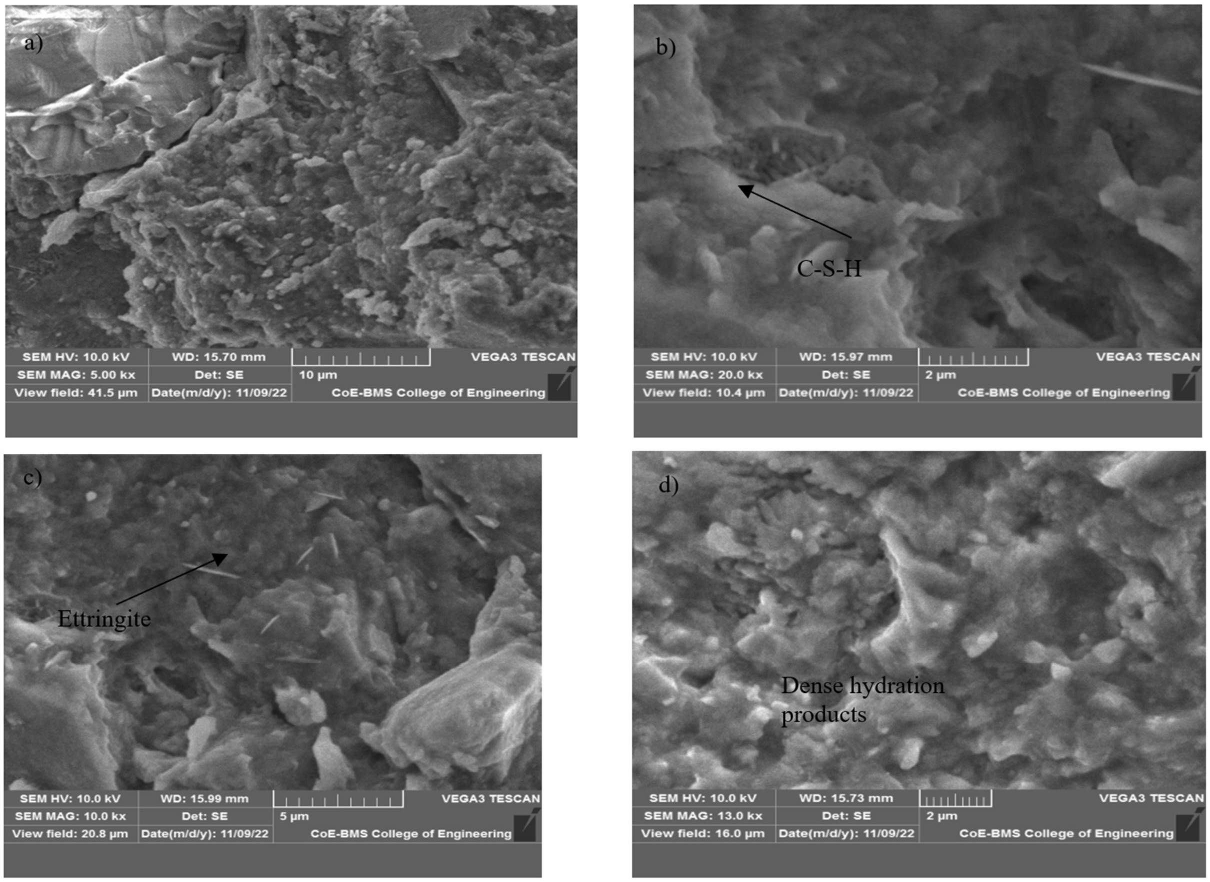
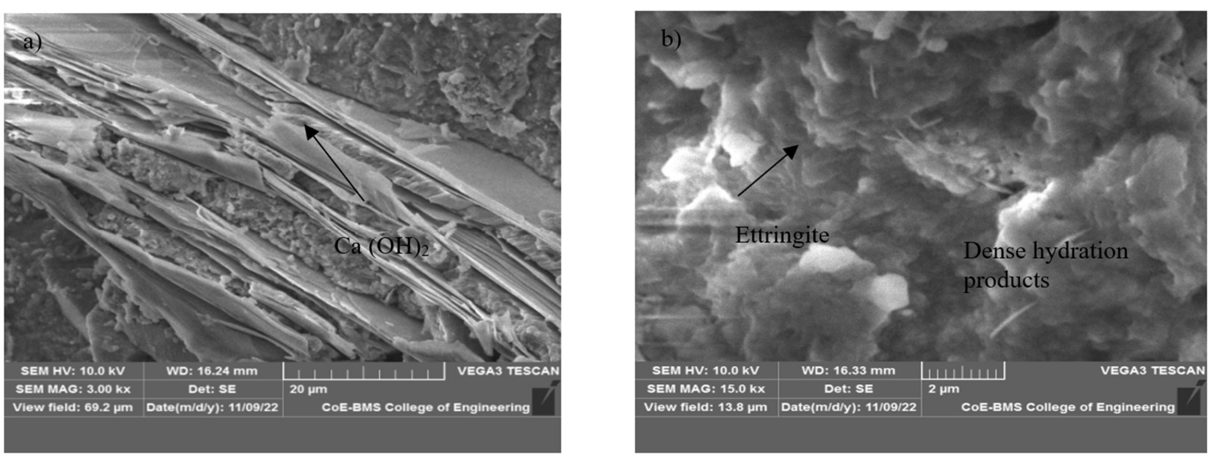


Fig.2 - SEM images of conventional concrete at 28 days.



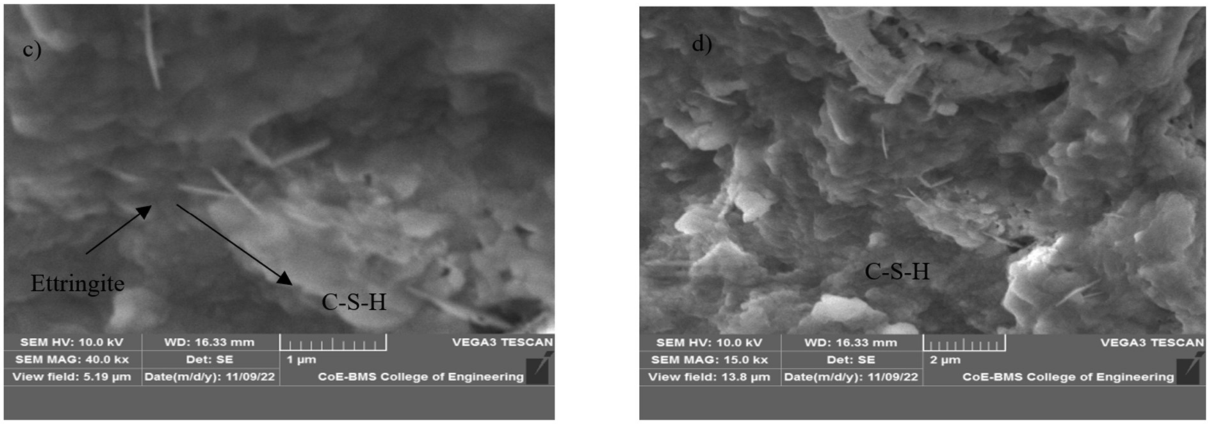


Fig.3 - SEM images of Graphene oxide concrete at 28 days.

4.3.2 XRD analysis

According to 19, XRD measurement provides a diffractogram that displays the present phases, phase concentration, amorphous content, and crystalline size 19. The graph shows the 2θ angle on the x-axis plotted against the diffraction intensity on the y-axis 3. The X-ray diffractogram of powder samples is depicted in Fig.4 for CC mix and Fig.5 for GC2 mix. The intense peak formed at 2θ angle of 20.8714°, 26.5895°, and 50.0657◦ is SiO2 or quartz, which is the main component of concrete aggregate. The calcium carbonate CaCO3 phases are detected at 2θ angle of 24.3288°, 29.3664°. The presence of calcium carbonate may be due to carbonation of calcium-containing hydration products such as CSH, portlandite, and ettringite that combine along with carbon dioxide to generate calcium carbonate 20. The peaks at 26.7538◦ and 28.0979◦ in Fig.4 are observed due to the existence of quartz. The graphene-containing compound peaks were not detected because of the lower dose of GO amount which was used in the concrete mix and also limitations of other equipment 3.

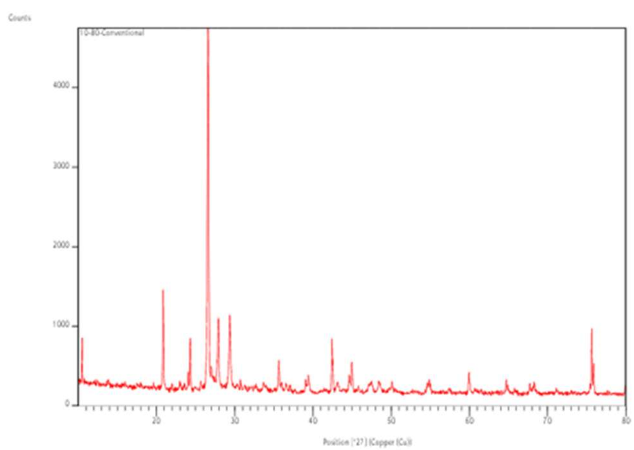


Fig.4 - XRD for conventional concrete specimen.

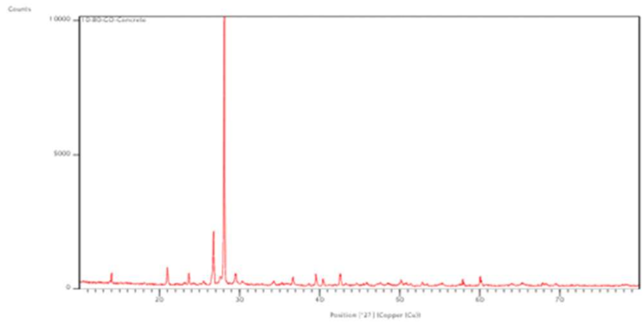


Fig.5 - XRD for Graphene oxide concrete specimen.

4.3.3 Energy Dispersive analysis by X-ray (EDAX)

Specimens of conventional concrete (Fig.6) and GC2 concrete (Fig.7) underwent EDAX testing after a period of 28 days of curing. Test results reveal percentage constituents of cement hydration. The difference in the results between Fig.6 and 7 indicates an increase in calcium element constituents in graphene oxide concrete. This increase in calcium may be attributed to the effect of graphene oxide, which leads to a rise in the formation of C-S-H gel 3.

Table. 11 - eZAF Smart Quant Results of CC mix

Element	Weight %	Atomic %	Net Int.	Error %	K ratio
O	58.22	73.29	257.83	10.01	0.1415
Na	3.43	3.00	20.37	15.82	0.0104
Al	4.56	3.40	62.59	8.32	0.0259
Si	15.44	11.08	250.26	5.60	0.1027
Ca	18.35	9.22	190.32	3.42	0.1610

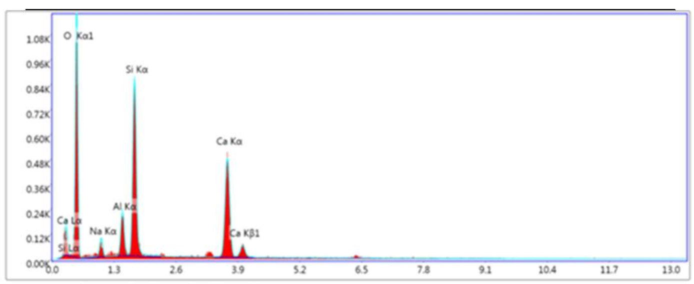


Fig.6 - The percent composition of conventional concrete samples shown in the EDAX graph.

Table. 12 - eZAF Smart Quant Results of GC2 mix

Element	Weight %	Atomic %	Net Int.	Error %	K ratio
O	55.06	73.42	185.59	10.96	0.0932
Al	2.47	1.95	36.76	11.07	0.0140
Si	8.90	6.76	161.75	6.20	0.0610

Ca	33.57	17.87	388.38	2.53	0.3023

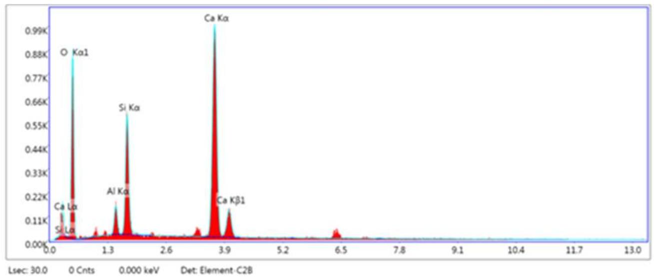


Fig.7 - The percent composition of graphene oxide concrete samples shown in the EDAX graph.

5. Conclusions

1. Workability for conventional concrete shows a significant slump. The high surface area of graphene oxide needs additional amount of water to wet the surface, but the addition of a higher dosage of superplasticizer considerably improved the workability property of graphene oxide concrete.
2. This investigation adopted concrete mix with varying amount of GO-0.1%, 0.2%, 0.3%, 0.4% and 0.5 % by weight of the cement. Among various proportions tested 0.2% of GO was found to be the ideal dosage yielding maximum strength.
3. In addition, the mixes with 0.2% GO prove to have an increase in rate of compressive strength by around 41% with 28 days of curing. Furthermore, the flexural strength increased by around 31% and split tensile strength was found to be increased by 4.60%.
4. The results on mechanical properties were verified by carrying out the analysis on microstructure of concrete. It is seen from SEM analysis that GC2 specimen shows dense hydration products and also the C-S-H gel fills voids, which in turn increases the strength. Further, from XRD analysis peak intensity is observed which may be due to hydration products. e-ZAF quant results of EDAX analysis indicate an increase in hydration products which may be due to the presence of graphene oxide.
5. Also, when superplasticizer is present, the GO is evenly dispersed.
6. With the same cement content better concrete grades can be achieved with GO and hence there is reduction in cement production, and it relatively reduces CO2 emission to some extent.

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