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EXFOLIATED GRAPHENE NANOPARTICLES FOR HIGH STRENGTH SELF-COMPACTING CONCRETE

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ABSTRACT:
The main idea of this project is about to study the effect of partial replacement of cement with Graphene oxide (GO) nanoparticles for high strength self-compacting concrete. These nanoparticles have the potentiality to recast the elements at Nano scale. By amending cement at Nano scale, we can improve the micro structure of cement.
The reason of this project is to increase the surface roughness of the Graphene oxide by nanoparticles, which intensify the interaction between filler and cement matrix. In this project we investigated various chemical and mechanical properties of GO contain high strength self-compacting concrete. Due to the nanoparticles of Graphene, it will incorporate the property of self-compacting itself in the concrete.
GO is added to cement at 0.02%, 0.04%, 0.06%, 0.08% by powder to cement in the form of a serum to determine the achievement of high strength self-compacting concrete.
Further SEM analysis, XRD analysis, Raman spectra, BET surface area, FTIR are the chemical tests are conducted in order to determine the micro level inherent properties of the Graphene oxide.
Keywords: Graphene oxide, DIMETHYL Form amide, ultra-sonication.

INTRODUCTION:

Concrete is the most used construction material across the world wide. We know now-a-days so many materials are partially replacing in the cement concrete in order to increase the strength of the structures. Graphene is the strongest material in the world, so that if we add the Graphene in the concrete, it has ability to increase strength, reduce clinker factor in cements (reducing carbon footprint), high electrical and thermal conductivity, and it has potential to deliver stronger, less permeable concrete structures enabling a new generation of concrete designs. It also improves the durability, mechanical strength, hardness and flexibility.
Graphene is an allotrope of carbon consisting of a single layer of atoms arranged in a 2-dimensional honey-comb lattice Nano structure containing polar and non-polar functional groups.
Due to the strength of its 0.142 Nm-long carbon bonds, Graphene is the strongest material ever discovered, with an ultimate tensile strength of **130,000,000,000 Pascal's** (or 130 gigapascals), compared to 400,000,000 for A36 structural steel, or 375,700,000 for Agamid (Kevlar).

MATERIALS USED:

1. Graphene
2. DMF
3. Aggregate
4. Water
5. Cement

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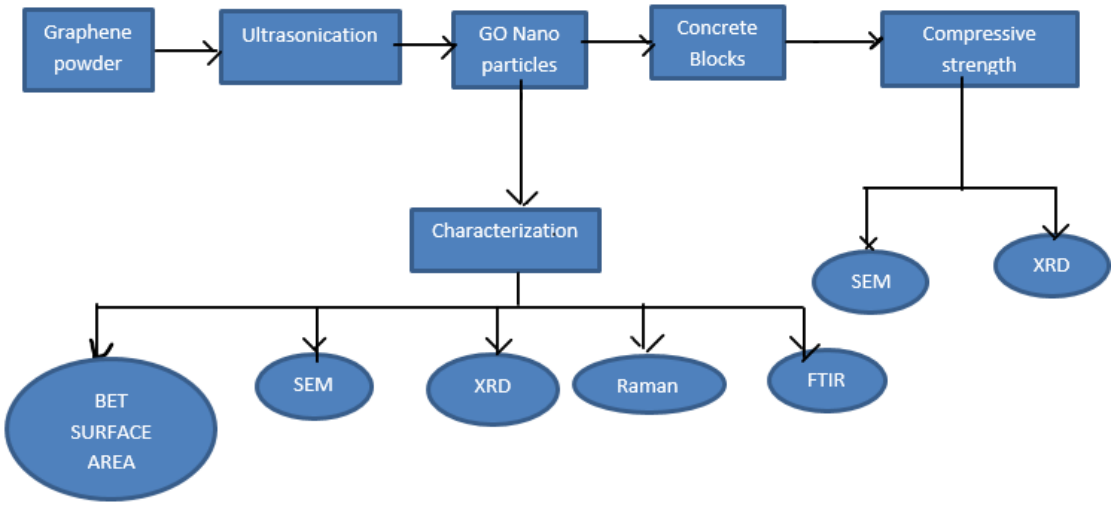
1) Graphene oxide: At first Graphene oxide (GO) was regarded as result of chemical exfoliation and oxidizing of layered crystalline graphite (natural or artificial). Amazingly, at definite conditions of oxidizing graphite carbon atoms conserve integrity 2D structure of layers with oxygen-containing functional groups attached to both sides of carbon plane and to the edges. However quite recent results demonstrated that the same structure could also is obtained using alternative (bottom up) method by hydrothermal treatment of glucose or even by chemical vapor deposition (CVD) technique. Therefore, today it worth giving more general definition on the basis of its structure: single atom carbon layer with both surfaces modified by oxygen containing functional groups. Like any 2D carbon GO can have single layer or multilayer structure. Carbon layers in multilayer GO are separated by functional groups bonded to each layer of carbon atoms. Although GO like Graphene is two-dimensional carbon material, its properties are extremely far from that of Graphene. It does not absorb visible light, have very low electric conductance compared to that of Graphene and demonstrates significantly higher chemical activity.
2) DMF: Dimethylformamide is an organic compound with the formula $(CH_3)_2NC(O)H$. Commonly abbreviated as DMF,this is colorless liquid is miscible with water and the majority of organic liquids. DMF is a common solvent for chemical reactions. Dimethylformamide is odorless, but technical-grade or degraded samples often have a fishy smell due to impurity of dim ethylamine. Dim ethylamine degradation impurities can be removed by sparing samples with an inert gas such as argon or by sonicating the samples under reduced pressure. As its name indicates, it is structurally related to form amide, having two methyl groups in the place of the two hydrogen's. DMF is a polar (hydrophilic) aprotic solvent with a high boiling point. It facilitates reactions that follow polar mechanisms, such as S_N2 reactions.

METHODOLOGY:

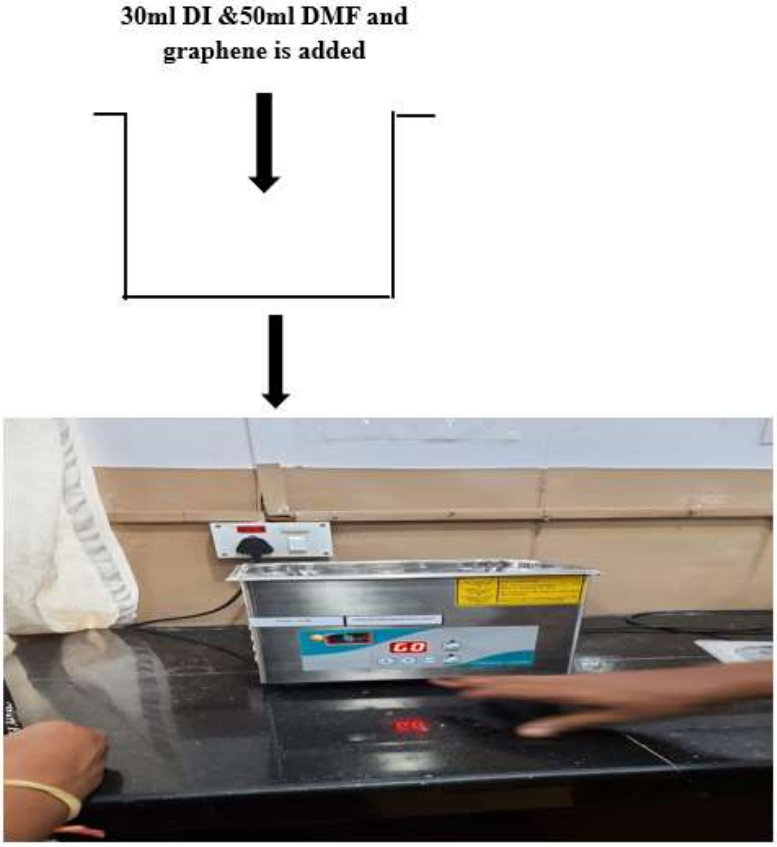


EXPERIMENTAL PROCEDURE:

- Preparation of Graphene serum: Take a beaker and add 30ml of distilled water and 50ml of DMF and the Graphene powder is added as weighted by percent of cement by weight.
  After adding all these materials stir it to get a uniform liquid. The sample is then kept in sonicator for 2hrs. In this process the Graphene particles are exfoliated into the small Nano particles and it incorporate the property of self-compacting and the high strength.

The chemical which is used have the property that will not allow the particles to settle down at the bottom. They will be in dispersion so that while mixing with concrete it will give a uniform strength throughout the structure.

Other than this chemical will settle at one place and will not spread uniformly throughout the structure and there will be a strength deficient at one place and more strength at other place.



Ultra-Sonication

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Chemical Used

Graphene Sample

- **Mix Proportion:** Concrete mix was designed in order to obtain the strength parameters of concrete conventionally of M30.
  - STEP – 1: DETERMINING THE TARGET MEAN STRENGTH
    F'ck = fck + 1.65 x S = 30 + 1.65 x 5.0 = 38.25 N/mm2
    Where,
    F'ck = Target average compressive strength at 28 days
    Fck = Characteristic compressive strength at 28 days
    S = Assumed standard deviation in N/mm2 = 5 (as per table -1 of IS 10262- 2009)

  - STEP – 2: SELECTION OF WATER CEMENT RATIO
    From Table 5 of IS 456, Maximum water-cement ratio = 0.50

  - STEP – 3: SELECTION OF WATER CONTENT
    Maximum water content for 20 mm aggregate = 186 Kg (for 25 to 50 slump)
    We are targeting a slump of 100mm; we need to increase water content by 3% for Every 25mm and above 50mm i.e., increase 6% for100mm slump.
    i.e., Estimated water content for 100 Slump = 186+(6/100) X 186 = 197 liter
    Water content = 197 liters.

  - STEP – 4: CALCULATION OF CEMENT CONTENT:
    Water-Cement Ratio = 0.50
    Water content from Step – 3 i.e., 197 liters
    Cement Content = Water content / "w-c ratio" = (197/0.50) = 394 kgs
    From Table 5 of IS 456,
    Minimum cement Content for moderate exposure condition = 300 kg/m3
    394 kg/m3 > 300 kg/m3, hence, OK.

As per clause 8.2.4.2 of IS: 456
Maximum cement content = 450 kg/m3, hence ok.

- STEP – 5: PROPORTION OF VOLUME OF COARSE AGGREGATE AND FINE AGGREGATE
  From Table 3 of IS 10262- 2009, Volume of coarse aggregate corresponding to 20 mm size and fine aggregate (Zone I) = 0.60
  In the present case water-cement ratio is 0.5. So, there will be no change in coarse aggregate volume i.e., 0.60.

- STEP – 6: ESTIMATION OF CONCRETE MIX CALCULATIONS:
  The mix calculations per unit volume of concrete shall be as follows:
  Volume of concrete = 1 m3
  Volume of cement = (Mass of cement / Specific gravity of cement) x (1/100) = (39/3.15) x (1/1000) = 0.125 m3
  Volume of water = (Mass of water / Specific gravity of water) x (1/1000) = (197/1) x (1/1000) = 0.197 m3
  Total Volume of Aggregates = 1- (b+c) =1- (0.125+0.197) = 0.678 m3
  Mass of coarse aggregates = d X Volume of Coarse Aggregate X Specific Gravity of Coarse Aggregate X 1000 = 0.678 X 0.60 X 2.80 X 1000 = 1139 kgs/m3
  Mass of fine aggregates = d X Volume of Fine Aggregate X Specific Gravity of Coarse Aggregate X 1000 = 0.678 X 0.40 X 2.70 X 1000 = 732 kgs/m3.

- STEP – 7: CONCRETE MIX PROPORTIONS
  Cement = 394 kg/m3
  Water = 197 kg/m3
  Fine aggregates = 732 kg/m3
  Coarse aggregate = 1139 kg/m3
  Water-cement ratio = 0.50

➢ **MIXING:**

1. Initially weigh all the materials according to the ratio and collect all the materials separately.
2. Now, on a clean platform first pour the coarse aggregate and fine aggregate then mix them uniformly until the voids of coarser are filled by the finer.
3. Next pour the cement and mix them thoroughly after mixing add graphene serum and then add the measured water to it and start the hand mixing, due to the small work hand mixing is carried out to be done.
4. Mix all the ingredients in 3 minutes and after mixing them uniformly then apply the grease to the cube mould and then pour the concrete in three layers and compact it with the hand compaction rod of 25 blows for each layer.
5. On the top the excess matrix is removed by trowel and leave the mould filled with concrete for 24 hrs.
6. After the cube is settled remove the cube from the mould and place it in the curing tank for 28 days.

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- **TESTING**

Tests on cement:

1. Specific gravity of cement:

Clean the flask and dry it. Now, weigh the empty flask with stopper which is considered as W_1.
Next, fill the cement half of the flask and weigh it W_2.
Add water to the cement to the top of the flask. Mix well to remove the air bubbles and weigh it, W_3.
Empty the flask. Now, fill the flask with water completely and weigh it with stopper and it is W_4.
Specific gravity of cement $= \frac{w_2 - w_1}{(w_2 - w_1) - (w_3 - w_4)}$

2. Fineness of cement:

Take 100gm of cement into the empty pan. And weighed as W_1.
The cement sample should be free from lumps.
Place the sample of cement into the 90µ IS sieve and closes it with a lid.
Continuously shake the sieve gently in all directions for 15 minutes.
Now, note down the weight of the retained cement sample and weighed as W_2.

% Weight of the residue = $\frac{Weight\ of\ the\ sample}{Total\ weight\ of\ the\ sample}$

3. Standard consistency of cement: The consistency of cement is the minimum water requirement to start the chemical reaction between water and cement.

- Fix and place the Vicat apparatus on a flat surface and make necessary adjustments.
- Now take 400gm of cement specimen and add 28% of water by the weight of cement and mix gently for up to 3 to 5 minutes.
- Then fill the cement paste into the Vicat mould and remove the excess cement paste by the trowel.
- Now place the Vicat mould on the Vicat apparatus and release the plunger gently by contacting the cement paste surface.
- Then release the plunger and allow it to penetrate the cement paste and note down the reading from the gauge scale from the bottom of the Vicat mould.

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- Now again, add water with cement paste at a different water ratio until the reading lies between 5mm and 7mm.
  P = (W/C) x 100
  P – Percentage of water
  W – Weight water added
  C – Weight of cement

Tests on Fine aggregates:

1. Specific gravity of fine aggregate:
   Clean the pycnometer and dry it. Now, empty weight of the pycnometer is determined as W_1.
   Fill the pycnometer with sand of 1000g of clean sample and it is weighed as W_2.
   Now, add water into the pycnometer which is already filled with sand. And weighed it as W_3.
   Now, the sample is discharged and cleaned it. Completely fill with water and weigh it. It is determined as W_4.
   Specific gravity of fine aggregate = $\frac{w_2-w_1}{(w_2-w_1)-(w_3-w_4)}$

2. Bulking of sand:
   Take the sample of sand and fill into the measuring cylinder pf 200ml.
   Don't compact the sand, make the necessary by using the steel rod.
   Place the sample into the container.
   Fill the measuring cylinder with 100ml water.
   And pour the sand into the measuring cylinder. Stir it with the help of steel rod.
   Allow it to settle sometime.
   The sand will be the below 200ml. now, note down the value, X.
   Repeat the same procedure for 2 more samples.
   Bulking of sand = $\frac{200-X}{X} * 100\%$

3. Fineness modulus of fine aggregate:
   Take all the IS sieves and arrange them in descending order.
   Take 1kg of sand into a tray and break the lumps.
   Pour the sample at the top of the sieve and shake the sieves continuously for 10 to 15 minutes.
   Now, take the weight of the retained sample into the IS sieves.

4.Compressive test: After curing period of 28 days is completed take the mould from the curing tank and surface dry the cube.
Now place the cube in compressive testing machine and apply the constant load on the cube then note down the reading where the cube is failed and further calculate the compressive strength of the cube.

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Compressive test machine

Surface drying of cubes

Placing of cube in ucs machine

Crushing of cube

SEM ANALYSIS ON CRUSHED CUBE SAMPLE:

1. SEM TEST: In order to do the Sem analysis first the sample is grinded into small particles and after that the sample thoroughly washed and oven dried at 96 °C. The specimen should pass through the 75-micron sieve. Furthermore, the uncoated sieved samples were then kept securely on top of a glass plate (Canada balsam coated) [1]. In this experiment the size of glass plate used is 25.4 cm×6.35 cm. The samples are then analyzed among LEICA microscope and envisioned using QWIN software. This analytical experiment will give us the surface texture of the samples.

RESULTS:

53 GRADE GRAPHENE CUBES OF M30 (serum)					
RATIO	MOULD PLACED	OPENED	PLACED IN CURING TANK	FOR 28 DAYS	
				OPENED	COMPRESSIVE STRENGTH(N/mm^2)
0.02%	04-11-2022	05-11-2022	05-11-2022	02-12-2022	13.15
0.04%	04-11-2022	05-11-2022	05-11-2022	02-12-2022	11.60
0.06%	04-11-2022	05-11-2022	05-11-2022	02-12-2022	24.48

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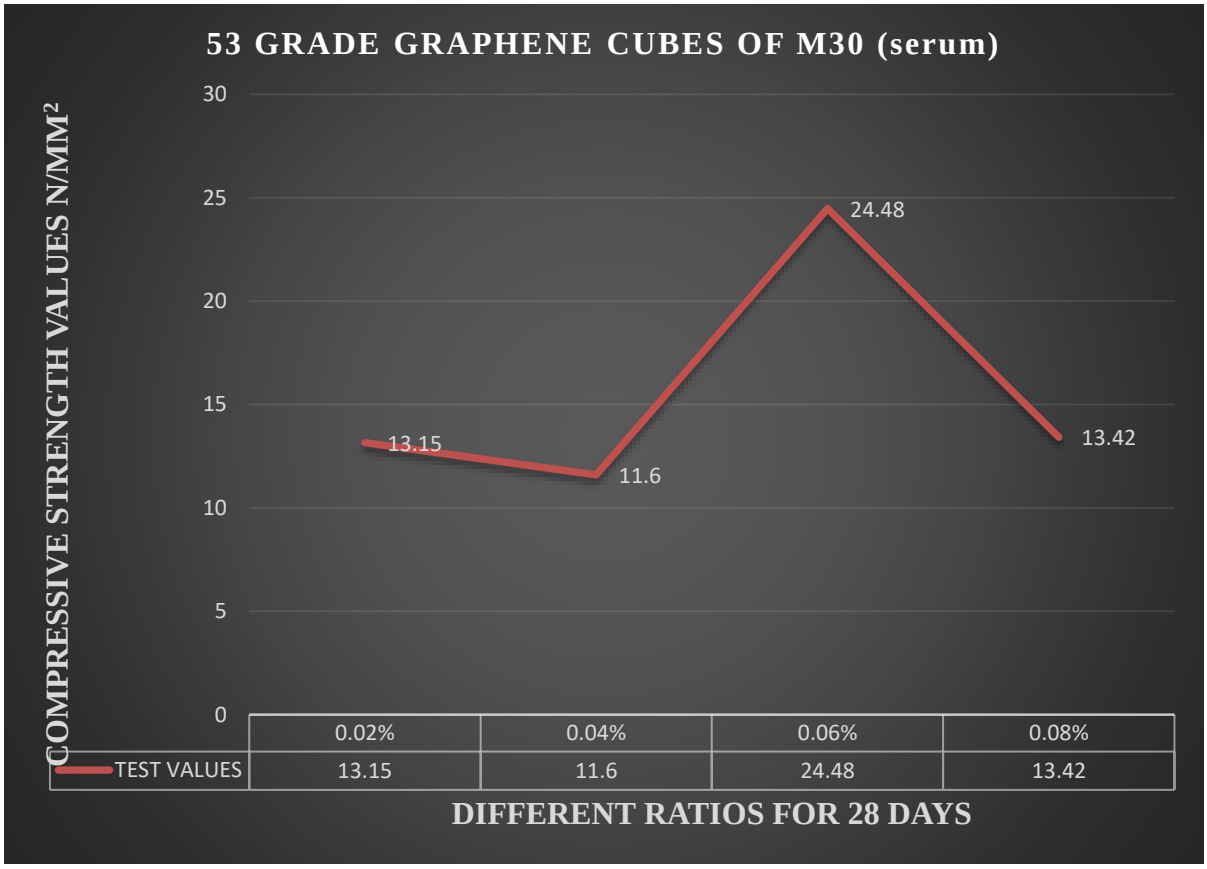
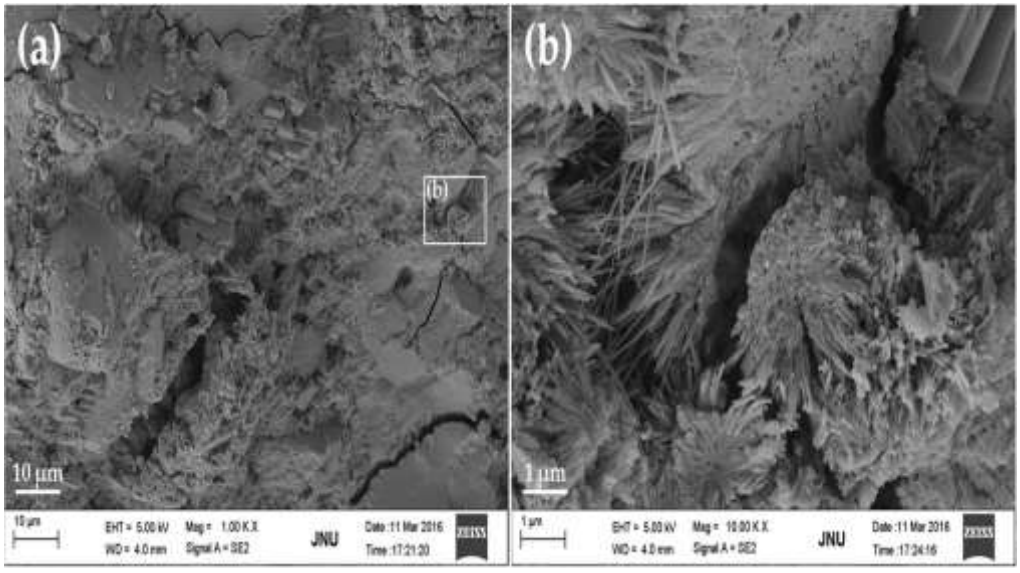
0.08%	04-11-2022	05-11-2022	05-11-2022	02-12-2022	13.42



GRAPH1

SEM TEST VALUES



CONCLUSION:

On the basis of the results observed in this study, the following conclusions can be drawn:
As the percentage of GO increases up to 0.075%, the mechanical and the durability properties improve significantly. This is possibly due to GO's acceleration of hydration on concrete. But due to some modifications in mixing and proportions we got highest value at 0.06% and after that the value got decreased. The primary goal of this research is to determine that by partial replacement of graphene can increase the compressive strength. Being graphene more cost but we use a very little quantity so that it is reasonable and gives better results. Generally, now a days all over the world buildings are constructed using cement but the top most hardest material is graphene. So, if we partially replace the cement with graphene oxide it can give more strength comparing to before and there are more other properties like it is ecofriendly so that it can reduce the carbon footprint. As the graphene oxide is exfoliated into nano particles, they will have more properties and enable them to compact by themselves. The chemical di methyl formamide is used in this study because other than dmf if we use any other while mixing all the graphene nano particles settle at only one place and there will be more strength at one place and less strength at other place. So that in order to overcome this effect dmf chemical is used which does not allow to settle at one place it will have the property of being in suspension.

FURTHER STUDY AND REASEARCH:

As we didn't do the proportions and mixing properly there is a further scope to continue this project again and to do all remaining different types of tests. And further more in order to know the micro level analysis of graphene oxide some of the chemical tests are to be conducted like SEM analysis, XRD analysis, RAMAN SPECTRA, FTIR tests.

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