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Analyse the Environmental Combine Effect of Graphene Oxide and Rice Husk Ash in Concrete Properties

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Abstract

Rice husk ash is environmentally hazardous if not dispose properly. Rice Husk Ash with graphene oxide as 50% cement replacement in concrete were used and analysed for their fresh and hardened properties. Also, this samples were mould as reinforced concrete beam specimens and tested for their flexural behaviour. Eleven cube samples were made with 4.9% RHA-0.1%GO, 9.8%RHA-0.2%GO, 14.7%RHA-0.3%GO, 19.6%RHA-0.4%GO,24.55%RHA-0.5%GO, 29.4%RHA-0.6%GO, 34.3%RHA-0.7%GO, 39.2%RHA-0.8%GO, 44.1%RHA-0.9%GO and 49%RHA-1%GO. Control concrete mix with 100% OPC were used to compare the results after 7,14 and 28 days for compressive strength, modulus of elasticity, flexural strength etc. Fresh concrete properties such as slump, bulk density and entrained air content were tested for eleven concrete mixes and SEM images for materials and cured concrete were presented in this paper. Seven concrete mixes were used for beam specimens that is up to 30% RHA-GO replacement and tested for deflection, load carrying capacity and crack pattern. As a result 25 to 30% RHA-GO mix can be used as cement replacement in concrete.

Keywords: graphene oxide, rice husk ash, concrete, cement, heat of hydration, cracks, environmental pollution

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INTRODUCTION

"Graphene-based greener, stronger concrete developed" which was an article in 'The Hindu' on April, 2018. This article clearly shows that graphene in concrete is an emerging trend. The trend of using graphene for various purposes in construction industry is because graphene proved the strongest material till now. Graphene is a hexagonal structure made of carbon atoms. Graphene is made of single layer, but it forms most of the other carbon allotrope. There are various forms of graphene such as graphite, graphene solution, reduced graphene, graphene oxide, graphene nanotubes etc. This graphene increases the material strength enormously in whatever form it added. Also, only a small dosage of graphene is more than enough to increase strength and other properties. Shenhua et al. investigated cement composite with graphene oxide nanosheets. They proved that with 0.03% of GO in cement composite increases its flexural strength, tensile strength, compressive strength and its toughness (Lv et al. 2013).

Graphene in the form of graphene oxide solution was used in this paper. Extraction of graphene oxide from graphene can be done mechanically or chemically. In this paper graphene oxide solution was extracted using Modified Hummer's method and the process of synthesis was taken from the paper written by Narasimha Rao et al. (Narasimharao et al. 2016). The raw material used for the synthesis of graphene oxide solution was flake graphite which is a naturally occurring material.

There were so many researches ongoing to use graphene as cement replacement in concrete (Valles Romero et al. 2016) and various admixtures were also added with graphene to find its changing characteristics. The combination tested in this paper were graphene oxide and rice husk ash as 50% cement replacement.

Rice husk are the outer protective covering of grain and it is an unused material. Particularly in India, large amount of rice husk is produced which are disposed at no cost and creates environmental pollution. Burning of rice husk to form rice husk ash is very crucial because it

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Table 1. Physical properties of fine aggregate and coarse aggregate

Sl. No.	Properties	Fine aggregate	Coarse aggregate
1	Size	5 mm	20 mm
2	Specific gravity	2.59	2.65
3	Water absorption	1.2 %	0.56 %
4	Fineness module	2.5	5.85
5	Bulk density (kg/m^3)	1510	1645

affects the properties of concrete (Xiong et al. 2009). Ru-Shan Bie et al. investigated the rice husk ash burned at various temperatures and its effects in properties of concrete (Bie et al. 2015). These rice husk must be burned at 600^0C at muffle furnace to obtain the rice husk ash which can be used in concrete.Using of rice husk ash in concrete also provides a solution for waste disposal and pollution control to some extent.A.N. Swaminathan presented a paper that explains using RHA alone in concrete increases its strength properties (Swaminathan 2013). Graphene oxide and rice husk ash independently increases concrete strength when used separately (Zareei et al. 2017) so to obtain its full potential, it was used in combinations in this paper.

In this paper, combinations experimented were0,5,10,15,20,25,30,35,40,45,50 % rice husk ash and graphene oxide as cement replacement in concrete. A proper proportion of cement, water and aggregates forms the concrete. M43 grade Ordinary Portland Cement (OPC) with tap water and fine aggregate of size 5mm and coarse aggregate of size 20mm was used for concrete mix. Water cement ratio was fixed at 0.42. 150mm x 150mm x 150mm cube moulds were used to form cube specimens and tested for their fresh and hardened properties**.** Beam specimens of size 150 mm x 150 mm x 1700 mm were made with 7 mix samples which is up to 30% RHA-GO replacement were tested for load carrying capacity, deflection and its crack pattern. Flexural behavior was studied by so many researches and that were taken as reference for testing beams in this paper (Imjai et al. 2016, Tejaswi et al. 2015, Zheng et al. 2016).

EXPERIMENTAL WORK

Fine Aggregate

Fine aggregate of good quality confirming to zone II from **Table 4** of IS383-1970 (IS: 383 1970) has been used. Sand in river made of fine particles of minerals and rocks is fine aggregate. This was used in concrete to resist compressive stress and to fill the voids in the coarse aggregate.

Coarse Aggregate

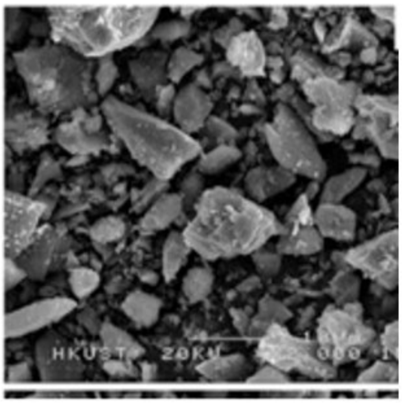
Maximum size of 20mm crushed granite aggregate from local crusher has been used. Coarse aggregate were the particles retained from 4.75mm sieve. Hardness, abrasion resistance and elastic modulus were influenced by adding coarse aggregate to make concrete durable, strong and cheaper.

Cement

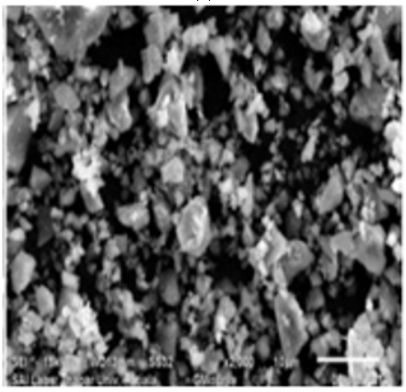
Ordinary Portland cement of40 grade from Ultra Tech cement Ltd., Tamil Nadu was used in this experiment. Cement is the bonding agent of concrete which binds all the elements of concrete together and provides strength to the concrete.

Rice Husk Ash

Rice husk are the protective covering of rice grains and these husks were burned at high temperature to form rice husk ash. Rice husk generated in a large amount and is less expensive. 100 kg of rice produce 20 kg of rice husk. Silica concentration of rice husk ash is more than 80-85%. RHA concrete is highly pozzolanic and reduces the permeability of concrete.



(a)



(b)

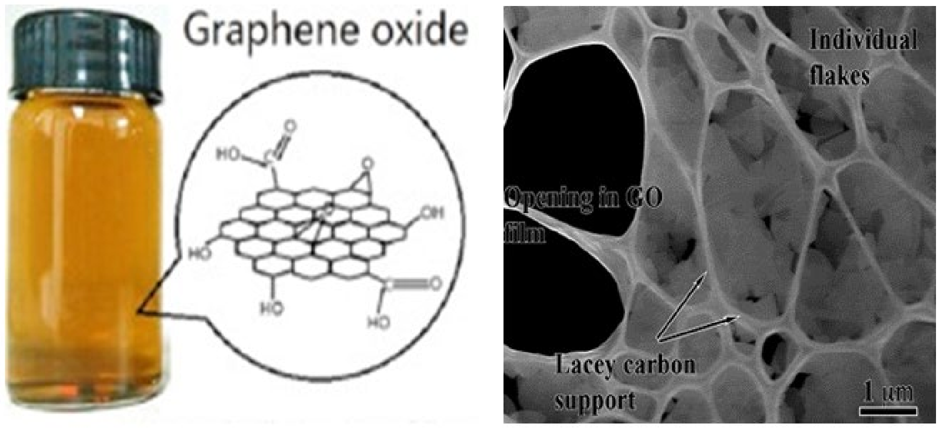
Fig. 1. SEM images of (a) cement (b) RHA

Table 2. Physical properties and chemical composition of cement and mineral admixtures

Physical property	**Cement** [12]	**Rice husk ash**
Specific gravity	3.16	2.06
Fineness (kg/m^2)	335.9	-
Bulk density	-	-
Initial setting time	50 min	-
Final setting time	365 min	-
Chemical Constituents	**(%)**	**(%)**
SiO_2	19.71	90.99
Al_2O_3	5.20	2.90
Fe_2O_3	3.73	0.30
CaO	62.91	0.89
MgO	2.54	0.96
LOI	0.96	4
SO_2	2.72	0.2
K_2O	0.90	0.56
Na_2O_3	0.29	0.10

(a) (b)

Fig. 2. (a) natural graphite flakes (b) SEM image of graphite flakes



(a) (b)

Fig. 3. (a) Prepared GO solution (b) SEM images of GO solution

Graphene Oxide

Hexagonal structure made of single layer of carbon atoms is graphene. The strongest material ever tested in the world and the basic building block of other important allotropes of carbon such as diamond, graphite etc. is graphene. In this paper flake graphite was used to synthesis graphene oxide solution. Flake graphite mostly occur in metamorphic rocks which was obtained from Tamil Nadu Minerals Limited. The process of synthesis of graphene oxide solution from natural graphite flakes using Modified Hummer's method is explained in flow chart shown in **Fig. 4**.

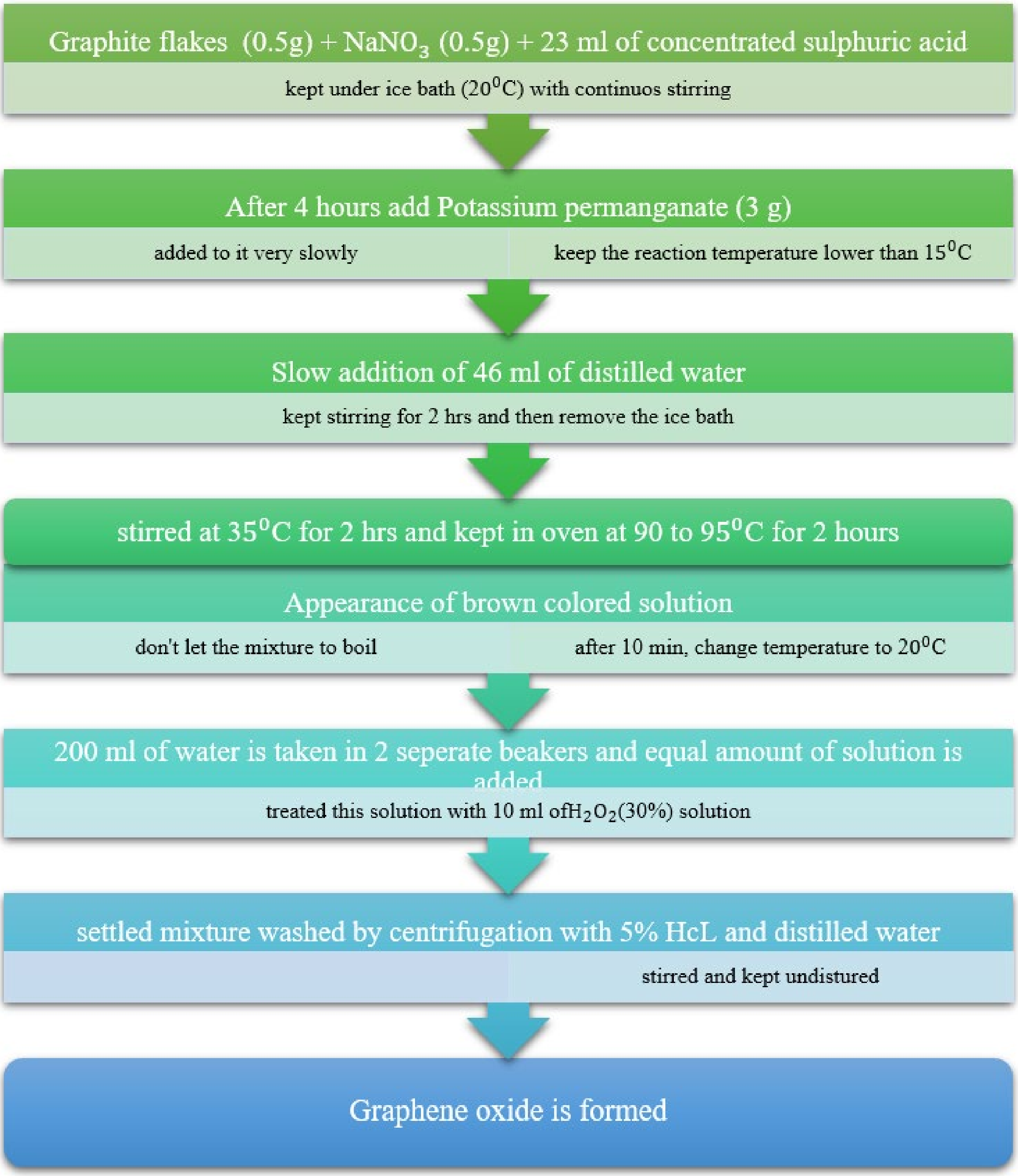


Fig. 3. Preparation of diluted GO solution using Modified Hummer's method

Table 3. Physical properties of graphene oxide

Physical property	Characteristics value
Physical state and appearance	liquid
Odor	Odorless
taste	tasteless
Molecular weight	12.01 g/mole
color	Yellow or black

Mix Proportions

As per IS: 10262-2009 (IS: 10262 2009), concrete mix were made in the ratio of 1:2.46:1.36.

Table 4. Mix proportions for RHA-GO mix samples

No.	Cement		Admixtures				Fine aggregate	Coarse aggregate	Water	Water cement ratio
			Rice husk ash		Graphene oxide					
	%	Kg/m³	%	Kg/m³	%	Kg/m³	Kg/m³	Kg/m³		
1	**100**	450	**0**	0	**0**	0	1109	612	189	0.42
2	**95**	427.5	**4.9**	22.05	**0.1**	0.45	1109	612	189	0.42
3	**90**	405	**9.8**	44.1	**0.2**	0.9	1109	612	189	0.42
4	**85**	382.5	**14.7**	66.15	**0.3**	1.35	1109	612	189	0.42
5	**80**	360	**19.6**	88.2	**0.4**	1.8	1109	612	189	0.42
6	**75**	337.5	**24.5**	110.25	**0.5**	2.25	1109	612	189	0.42
7	**70**	315	**29.4**	132.3	**0.6**	2.7	1109	612	189	0.42
8	**65**	292.5	**34.3**	154.35	**0.7**	3.15	1109	612	189	0.42
9	**60**	270	**39.2**	176.4	**0.8**	3.6	1109	612	189	0.42
10	**55**	247.5	**44.1**	198.45	**0.9**	4.05	1109	612	189	0.42
11	**50**	225	**49**	220.5	**1.0**	4.5	1109	612	189	0.42

Table 5. Results for fresh concrete properties

No.	Cement	Admixtures		Bulk density	Slump	Air content
		Rice husk ash	Graphene oxide			
	%	%	%	kg/m3	mm	%
M1	**100**	**0**	**0**	2250	100	5.1
M2	**95**	**4.9**	**0.1**	2198	105	5.7
M3	**90**	**9.8**	**0.2**	2167	115	5.6
M4	**85**	**14.7**	**0.3**	2109	120	5.5
M5	**80**	**19.6**	**0.4**	2056	125	5.3
M6	**75**	**24.5**	**0.5**	2032	130	5.2
M7	**70**	**29.4**	**0.6**	2014	135	5.0
M8	**65**	**34.3**	**0.7**	2008	140	4.9
M9	**60**	**39.2**	**0.8**	1979	145	4.7
M10	**55**	**44.1**	**0.9**	1953	150	4.6
M11	**50**	**49**	**1.0**	1925	155	4.5

The concrete was mixed in the laboratory temperature of 20^0C. In dry condition both fine and coarse aggregate were placed, and normal tap water was weighed and added to the mixture. All concrete mixtures were prepared using mixer. To obtain a good concrete mix, the following procedure was adopted.

Add quarter of water to fine aggregate helped to absorb water by fine aggregate. Later 60 seconds of mixing, coarse aggregate were put in and furthermore quarter of water was added to it. After this process cement and other supplementary cementitious material was added and the left-out water as per the mix proportion too added to the mixture and mix properly until a uniform mixture was obtained and well compacted. After compaction, casting was performed.

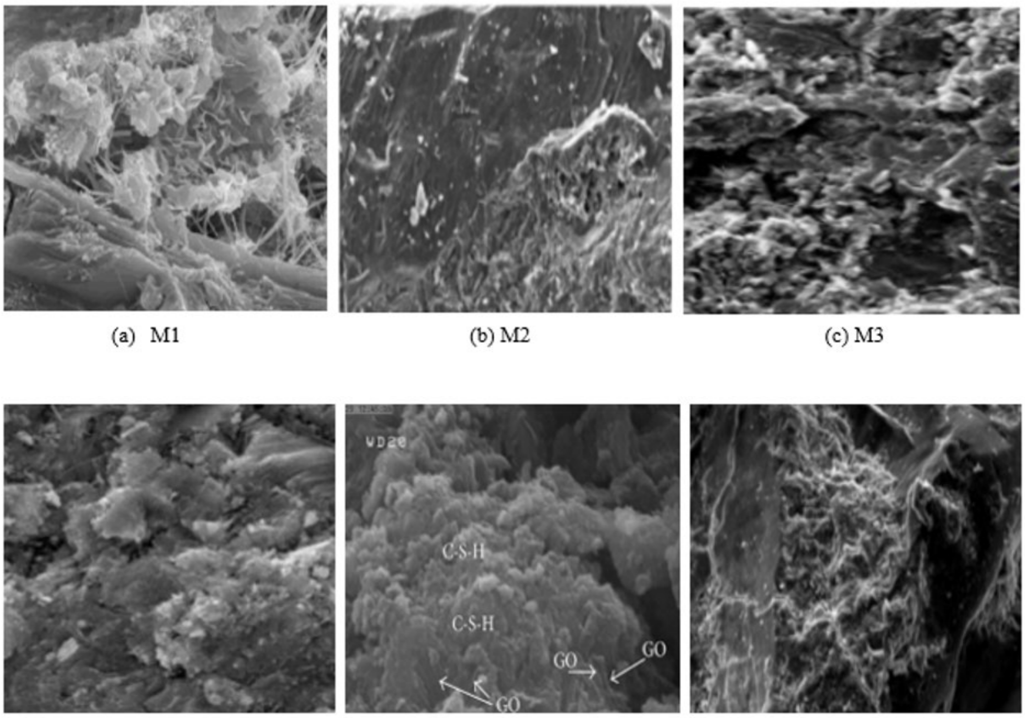
The cube of size 150 mm x 150mm x 150 mm mould with lubricated inner surface were placed on the floor. The floor must be levelled with concrete. To maintain the effective depth cover block of required thickness were to be placed below the bottom of the case. The first layer of concrete was placed in the mold and compacted with tamping rod and second layer was added to the mold. Concrete cube mold was now ready to be kept undisturbed for 24 hours. After 24 hours, demolded and place inside the curing tank. Following 7, 14 and 28 days, the specimens were removed from water and sponged off the water in the surface of the cube and made dry.

It was very crucial to set temperature and humidity favorable for hardening of concrete and this process is called curing. Curing conditions made huge difference in degree of hydration heat and resultant microstructure of hydrated cement which largely affects the mechanical properties of concrete. The concrete specimens were cured for up to minimum 3 days and maximum 28 days.

RESULTS AND DISCUSSIONS

Test for Fresh Properties of Concrete

According to IS456:2000 (IS: 456 2000), degree of workability is measured by slump in mm. from the above results the RHA-GO mix of M1 to M10 shows the high workability concrete as the value falls between 100 – 150.Bulk density was reduced from M1 to M7. As the result these concrete mixes with low bulk density can be used as a light weight concrete in construction. For 20 mm aggregate, entrained air content for fresh concrete during construction must be 5±1. All the mixes produced almost equal air content which is suitable for construction. M7 mix of RHA-GO has 30% replacement of cement shows the bulk density which can be used as normal weight concrete.



(a) M1 (b) M2 (c) M3

(d) M4 (e) M5 (f) M6

Fig. 5. SEM images of concrete cube specimens after 28 days

Table 6. Test results of concrete specimens

No.	Cement	Admixtures		Compressive strength		
		Rice husk ash	**Graphene oxide**	**7 days**	**14 days**	**28 days**
	%	%	%	N/mm^2	N/mm^2	N/mm^2
M1	**100**	**0**	**0**	25.13	27.64	40.19
M2	**95**	**4.9**	**0.1**	28.64	29.35	41.58
M3	**90**	**9.8**	**0.2**	29.60	29.95	41.93
M4	**85**	**14.7**	**0.3**	30.56	30.99	42.84
M5	**80**	**19.6**	**0.4**	32.87	33.45	43.69
M6	**75**	**24.5**	**0.5**	31.65	32.68	43.95
M7	**70**	**29.4**	**0.6**	30.58	31.75	42.56
M8	**65**	**34.3**	**0.7**	29.26	30.62	41.34
M9	**60**	**39.2**	**0.8**	28.47	29.31	41.80
M10	**55**	**44.1**	**0.9**	27.31	26.32	40.56
M11	**50**	**49**	**1.0**	24.48	25.59	39.28

Heat of Hydration

Heat of hydration is the process of generation of heat when cement and water is mixed. It is the product of exothermic reaction between cement and water. OPC is hydraulic cement consists of four kinds of minerals such as alite (C3S), belite (C2S), aluminate (C3A) and aluminoferrite (C4AF). Above four compounds form the 90% of Portland cement which defines the hydraulic characteristics of cement. The other admixtures such as silica fume, fly ash, rice husk ash and graphene oxide have the similar compounds. After the process of hydration, Calcium-Silicate-Hydrate (C-S-H gel) and calcium hydroxide were formed. Aluminate creates the final product ettringite. Formation of C-S-H gel directly relates to the strength and durability of concrete.

$$2C_2S + 9H \rightarrow C_3S_2H_8(C-S-H) + CH \quad (1)$$

$$2C_3S + 11H \rightarrow C_3S_2H_8(C-S-H) + 3CH \quad (2)$$

The SEM image of sample M1, M2, M3, M4, M5 and M6 at 28 days is shown in **Fig. 5**. C-S-H gel, CH crystals and calcium-sulpho-aluminate (ettringite) were observable from the images. The SEM image of M5 (20% RHA-GO) concrete shows the lower traces of CH crystals. This makes the concept clear that M5 concrete is more durable. When compared to M1 concrete, M5 and M6 as 20 and 25 % RHA-GO mix shows smooth mixing of concrete paste. This was evident via formation of C-S-H and CH in the picture.

Compressive Strength

M6 – 25% RHA-GO concrete sample tends to increase compressive strength around 20% higher than

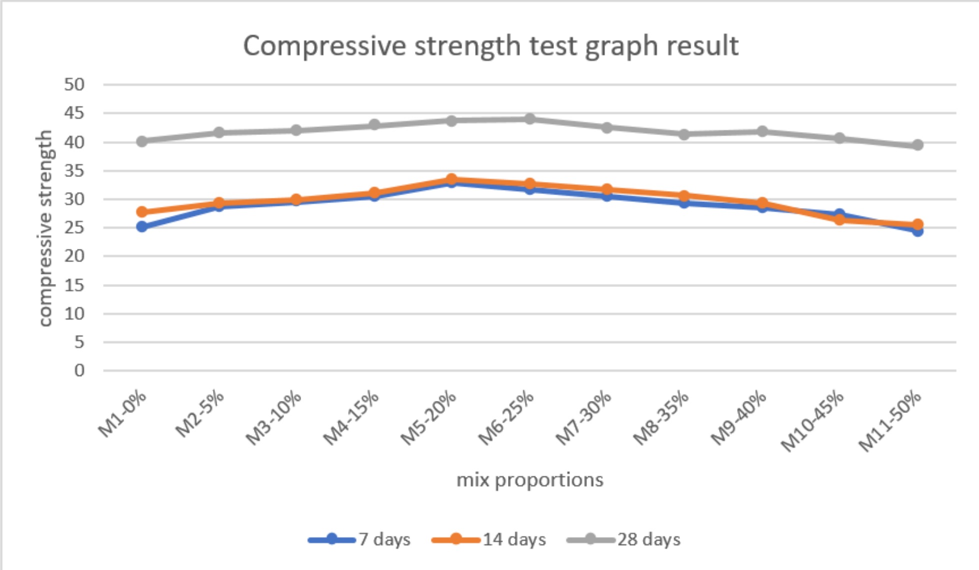


Fig. 6. Compressive strength test graph result

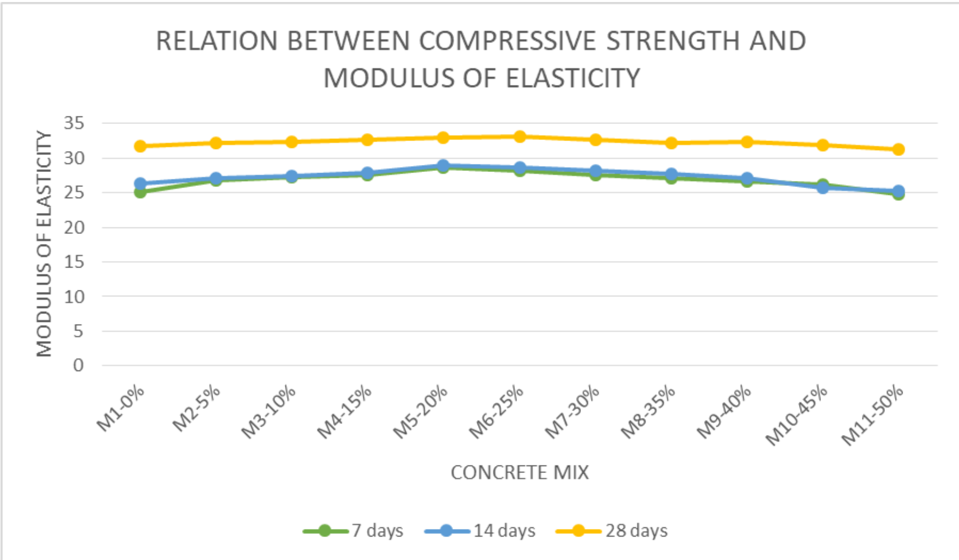


Fig. 7. Compressive strength test graph result

control concrete mix. Above 25% RHA-GO in concrete, there was a decrease in its compressive strength. For M7 concrete there was clear decrease of about 2.5% of compressive strength from control concrete mix. From the above comparison M6 RHA-GO mix performs better than other concrete mixes. Also, other RHA-GO mix below 25% tends to increase its compressive strength, but it is lower as compared to other rice husk ash-graphene oxide mixture.

Modulus of Elasticity

Modulus of elasticity fluctuates due to number of curing days, curing temperatures and humidity, yielding properties of aggregate, duration of concrete, mix proportions and the cement selected which regulates the efficacy of concrete. Modulus of elasticity is given by the formula

$$E_c = 5000\sqrt{f_{ck}} \tag{3}$$

where, E_c – Elastic modulus, N/mm^2

f_{ck}– characteristic compressive strength of concrete, N/mm^2

Fig. 7 clearly presents the M6 concrete mix with graphene oxide and rice husk ash shows the high modulus of elasticity at 28 days than control concrete mix.

Table 7. Test results for cube specimens

No.	Cement	Admixtures		Flexural strength		
		Rice husk ash	Graphene oxide	7 days	14 days	28 days
	%	%	%	N/mm^2	N/mm^2	N/mm^2
M1	**100**	**0**	**0**	6.45	7.14	8.54
M2	**95**	**4.9**	**0.1**	6.77	7.28	8.69
M3	**90**	**9.8**	**0.2**	6.58	7.39	8.72
M4	**85**	**14.7**	**0.3**	6.69	7.42	8.93
M5	**80**	**19.6**	**0.4**	6.83	7.45	9.28
M6	**75**	**24.5**	**0.5**	7.03	7.53	9.48
M7	**70**	**29.4**	**0.6**	7.15	7.67	9.67
M8	**65**	**34.3**	**0.7**	6.91	7.64	9.05
M9	**60**	**39.2**	**0.8**	6.64	7.56	8.76
M10	**55**	**44.1**	**0.9**	6.25	7.38	8.35
M11	**50**	**49**	**1.0**	6.13	6.91	8.16

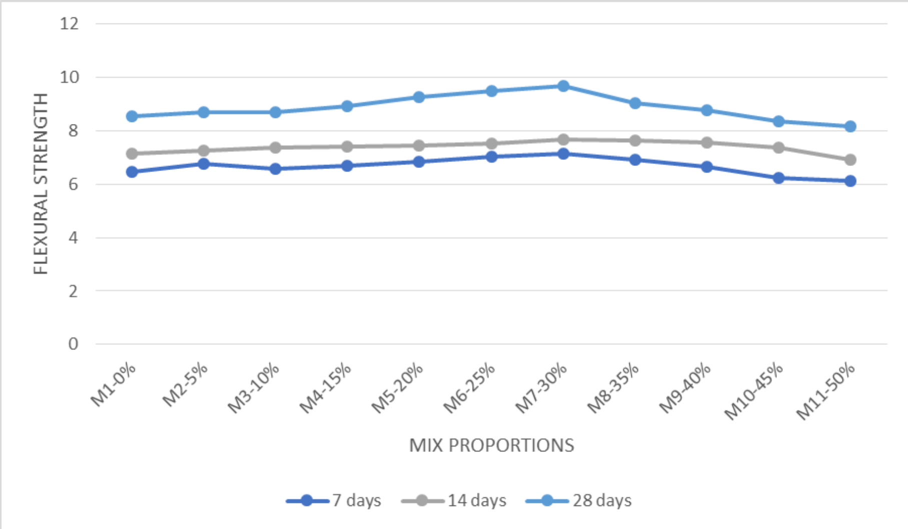


Fig. 8. Flexural strength graph result

Table 8. Test results for cube specimens

No.	Cement	Admixtures		Split tensile strength		
		Rice husk ash	Graphene oxide	7 days	14 days	28 days
	%	%	%	N/mm^2	N/mm^2	N/mm^2
M1	**100**	**0**	**0**	3.13	4.23	5.69
M2	**95**	**4.9**	**0.1**	3.34	4.38	5.78
M3	**90**	**9.8**	**0.2**	3.58	4.65	5.91
M4	**85**	**14.7**	**0.3**	3.82	4.73	6.05
M5	**80**	**19.6**	**0.4**	4.09	5.01	6.37
M6	**75**	**24.5**	**0.5**	4.58	5.59	6.64
M7	**70**	**29.4**	**0.6**	4.37	5.75	6.89
M8	**65**	**34.3**	**0.7**	3.98	5.68	6.57
M9	**60**	**39.2**	**0.8**	3.57	5.44	6.25
M10	**55**	**44.1**	**0.9**	3.31	4.87	5.78
M11	**50**	**49**	**1.0**	2.98	4.01	5.23

Flexural Strength

Flexural strength was a form of tensile strength and M7 mix RHA-GO tends to increase its flexural strength by around 10% from M1 control concrete. Around 10% increase of flexural strength from the RHA-GO in concrete was the highest increment as compared to other percentages of RHA-GO concrete. Above 30% addition of RHA-GO in concrete leads to a gradual decrease of its strength and it decreases to lower than control concrete mix. So, it was concluded that up to 30% RHA-GO can be added to concrete as cement replacement.

Split Tensile Strength

M7 RHA-GO mix tends to increase its split tensile strength by around 30% from control concrete mix at 14 and 28 days. For 7 days of curing M6 RHA-GO concrete mix shows better strength than M7. From the

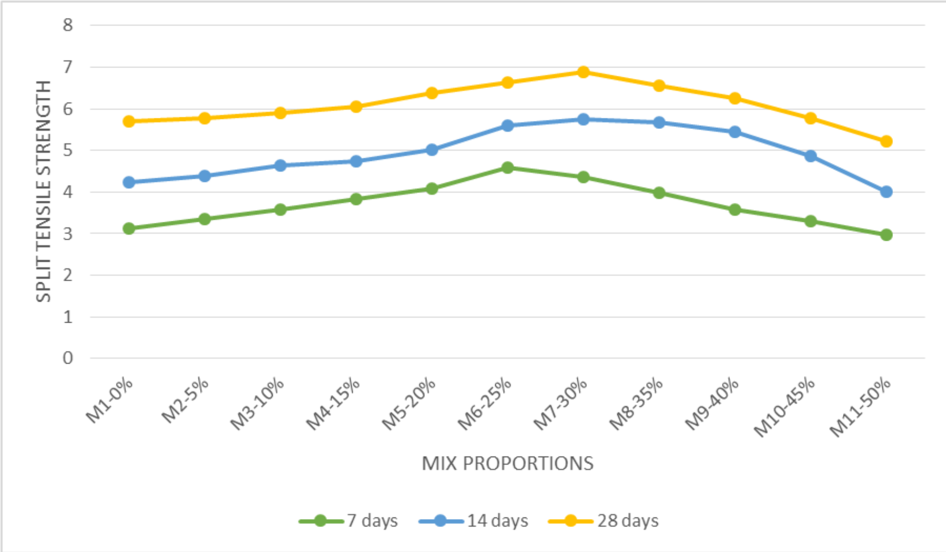


Fig. 9. Split tensile strength graph result

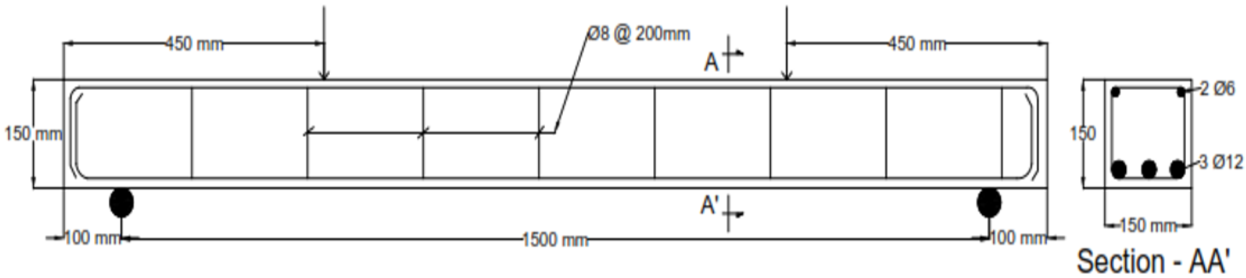


Fig. 10. Reinforcement detail same for all beams

overall results it was observed that 30% RHA-GO concrete mix was better to use as cement replacement in concrete.

Testing of RHA-GO Concrete Beams

Geometry of beamand test set up

The flexural test was conducted for 7reinforced concrete beam specimens made of size 150 mm x 150 mm x 1700 mm. Concrete mix for beam specimens were the same proportions tested before but only up to 30% RHA-GO replacement were tested in beams. The concrete mix proportions were M1-0%, M2-5%, M3-10%, M4-15%, M5-20%, M6-25%, M7-30% RHA-GO mix as cement replacement in reinforced concrete. The seven concrete beams were provided with same reinforcement as 3 bars of 12 mm diameter at the bottom, 2 bars of 6 mm diameter at the top and 8 mm diameter bars at 200 mm spacing as stirrups. The beam was simply supported with an effective span length of 1500 mm.

The test method adopted is as per IS-516, 1959 (IS: 516 1959) two points loading methods. Axial weight was loaded to the specimen through two rollers in the absence of any torsional stresses. The maximum load at which the beam cracks that is how much bending moment can a beam stands is known as modulus of rupture or flexural strength.The crack pattern of beam specimens was observed at regular intervals for the loads applied. The loads were increased gradually by adding 10 kN for each interval.

Load vs deflection behavior

The load Vs deflection behavior for seven concrete beams is shown in the figure below. It was clear that M2 and M7 concrete shown minimum deflection than M1 concrete which means it takes maximum load than control concrete beam.

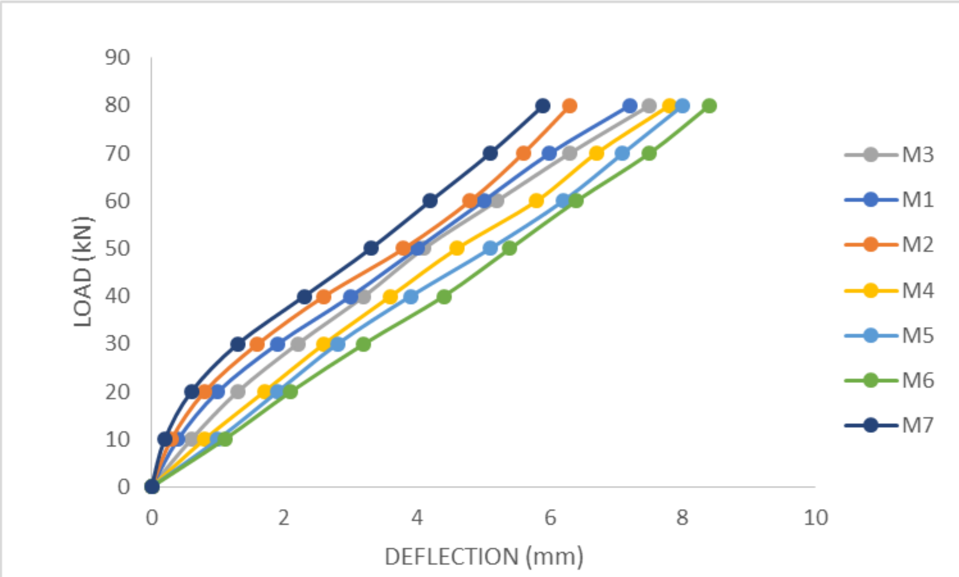
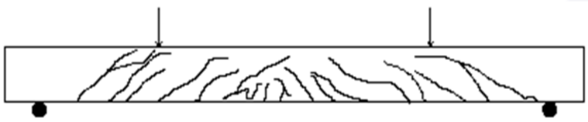


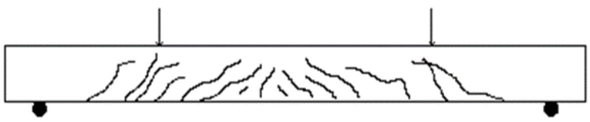
Fig. 11. Load vs deflection behavior

Crack pattern

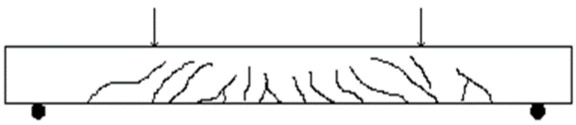
The crack pattern for 7 beams is given below in the figure. From the figure the obtained results were shown that RHA-GO mix can be used in concrete. The results were the crack behavior of M7 concrete beam shows there was less spacing of cracks, minimum number of cracks and minimum crack width than M1 control concrete mix. M2, M3 and M5 concrete beams shows the crack behavior almost like M1 as maximum crack width and less spacing of cracks. This was due to lesser modulus of elasticity.



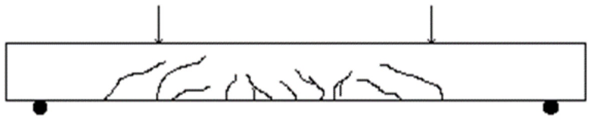
Maximum crack width = 50 mm
Number of cracks = 19
M1 concrete mix – 100% cement



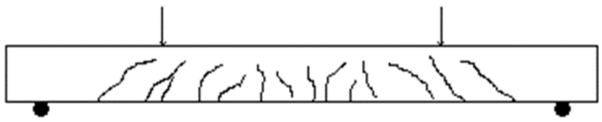
Max. crack width = 42 mm
Number of cracks = 15
M2 concrete mix – 5% RHA-GO



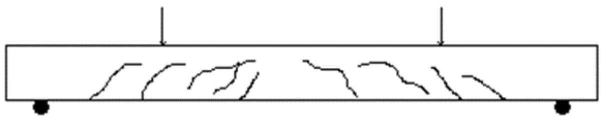
Max. crack width = 38 mm
Number of cracks = 13
M3 concrete mix – 10% RHA-GO



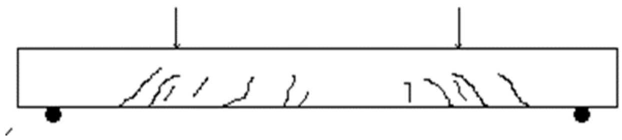
Max. crack width = 30 mm
Number of cracks = 11
M4 concrete mix – 15% RHA-GO



Max. crack width = 32 mm
Number of cracks = 13
M5 concrete mix – 20% RHA-GO



Max. crack width = 25 mm
Number of cracks = 9
M6 concrete mix – 25% RHA-GO



Max. crack width = 20 mm
Number of cracks = 10
M7 concrete mix – 30% RHA-GO

CONCLUSION

It concluded that using of Fly ash and Rice husk ash helps in reducing the environmental pollution during the disposal of excess Fly ash and Rice husk ash. From the above results graphene oxide and rice husk ash in concrete increases the strength and is suitable for use. Graphene oxide increases the workability of concrete which was reduced by rice husk ash in concrete mix. The workability of concrete was analyzed by slump. Almost all concrete mixes show the slump value which gives good workability. Air content for all RHA-GO mix was around 5±1 as per IS456-2000 and its bulk density was low as compared to other mixtures. The results derived from our experimental tests showed that compressive strength increases around 20% in the percentage of 29.4% rice husk ash and 0.6% graphene oxide (M7) of cement in concrete. Modulus of elasticity is related to compressive strength also presented in the graph which shows 24.5% rice husk ash and 0.5% graphene oxide (M6) mixture shows high modulus of elasticity at 28 days than control mix. This helps to reduce the number cracks during flexural testing. The maximum flexural strength was obtained with 29.4% rice husk ash and 0.6% graphene oxide. 29.4% RHA and 0.6% GO shows the maximum split tensile strength. From beam testing, M6, M7 concrete mix shows low deflection hence it proves the high load carrying capacity. The schematic diagram of crack pattern shows that M7 concrete gives a smaller number of cracks and minimum crack width. Graphene oxide increases the durability of concrete by improving the formation of C-S-H gel. Pore size can be reduced by graphene oxide thereby reducing the permeability and hence reduce the formation of microcracks and make it more durable structure. Graphene oxide also act as a protective shield to environmental deterioration attack and hence improves durability. The above results show that graphene oxide-based concrete has been the strongest among other mixtures. It also provides corrosion resistance and durability. Natural graphite flakes were the inexpensive source from which graphene oxide can be obtained. The use of secondary materials like rice husk ash which are industrial byproducts reduce the waste disposal crisis and environmental hazards. The conclusion will be around 25 to 30% RHA-GO as cement replacement is more suitable in concrete and it can be used as an admixture for concrete.

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