International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 05, May-2016

Experimental Investigation on Influence of Carbon Nanotubes & Halloysite Nanoclay on Strength of Cement Mortar

Ghazala Anjum
Student, Department of Civil Engineering,
Ghousia College of Engineering, Ramanagaram,
Karnataka (Affiliated to VTU, Belgaum)

Dr. N. S. Kumar
Professor and Director (R&D- Civil Engg.,)
Ghousia College of Engineering, Ramanagaram,
Karnataka (Affiliated to VTU, Belgaum)

***Abstract*:- Cementitious composites have high compressive strength and modulus of elasticity, but relatively low tensile strength, toughness and ductility. In order to compensate for that, additional reinforcing agents are used to hold the cement matrix in a much stronger way as compared to conventional calcium silicate hydrate gel.[1] Hence, in such a scenario CNT's have been regarded as ideal reinforcing agents because of their incredible strength and binding properties which increase the compressive and flexural strength to a much greater level. CNT's are graphite sheets with carbon atoms bonded in a hexagonal pattern. However, they are not considered safe for humans as well as the environment because of their toxicology potencies and with the advancement of research in the Nano composite field, another material has emerged as a safe option. Halloysites are naturally occurring eco-friendly nanotubes with low cost and harmless to humans. Because of HNC's higher aspect ratio and easy dispersability in polymer matrix and more importantly its abundant availability makes it the next ideal material for nano cement paste.[6] In this project, an effort is made to study the effect of Carbon Nanotubes; Single walled and Multi walled and Halloysite nanoclay (0.5%, 0.75%, and 1%) on compressive and split tensile strength of cement mortar in comparison with plain cement paste. This study aims to determine the optimum dosage of nano material require to obtain higher compressive and split tensile strength and also to determine Stiffness, Strain energy, Young's modulus, and Modulus of toughness for the Nano composites. To study the SEM (scanning electron microscope) images are taken during mixing, before testing and after testing and Energy-dispersive X-ray spectroscopy (EDX) Analysis.**

Keywords: Carbon Nanotubes, SWCNT, MWCNT, Compression, Split tensile, SEM, EDX

1. INTRODUCTION

Portland cement is the most widely used construction material. Along with fine aggregates and coarse aggregates, it forms the main ingredient of Portland cement concrete (PCC). The mechanism of PCC of providing strength to the concrete has been the most adopted field of research ever since it was discovered as a potential material of construction. Over the course of years, the research has led to the basic conclusion that complex chemical compounds eventually formed due to the hydration of cement, provides the strength to PCC by binding the aggregates together.[1] The prefix 'Nano' in the word Nano-technology means a billionth (1×10^{-9}). Nano-technology deals with various structures of matter having dimensions of the order of a billionth of a meter. Though the word 'Nano-technology' is relatively new, the existence of functional devices and structures have existed on Earth as long as life itself. The recent developments in the field of Nano-technology and Nano-science have opened up new careers and opportunities in science and material engineering. The new materials that are being developed and used have advanced chemical and physical properties with unique characteristics. The properties of these materials depend not only on the size and morphology but also on their composition and chemical bonding. Carbon Nanotubes (CNT) are defined as tubular structures made of one or more layers of graphite sheets. Graphite is a flat sheet formed by layers of carbon atoms bonded in a hexagonal pattern. CNT's are basically classified into single-walled CNT's (SWCNT) and Multi-walled CNT's (MWCNT). SWCNT's have a diameter of 1 to 3 nm, while MWCNT's have a diameter somewhere between 10 to 100 nm. The length of both the CNT's ranges from 0.5 µm to 50 µm.[1] However, the demand for CNT's exceed its supply exponentially. The methods adopted to manufacture the Nanotubes provide small yields skyrocketing its prices to $1500 per gram. CNT's are not only costly but also by nature are potentially toxic because of which, in the recent years, these as been a growing concern about their effects on environment as well as human health. Alternate Nano materials are being searched for to compensate the use of CNT's. Hence, another Nano material comes into picture. Halloysite Nano clays are naturally formed in the earth, hence they are ecofriendly and also cost less than Carbon Nanotubes. Halloysite Nano clay (HNC) is an Aluminosilicate clay ($Al_2Si_2O_5(OH)_4.H_2O$) with Nano tubular and hollow micro structure. Its theoretical composition is of 13.96% H_2O, 39.5% Al_2O_3 and 46.54% SiO_2. They are mined from the natural deposits in China, New Zealand, Brazil, France and USA. HNT's are closely similar to Kaolinite and are used in the manufacture of ceramic white-ware. HNT's have an outer diameter of 10-15nm and an inner diameter of 5-10 nm with 2-40 mm in length.[6] The distinctive chemical and physical properties of Nano materials result in the improvement of overall material performance.

International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 05, May-2016

1.1 ADVANTAGES OF CNT'S

2. They are excellent replacements for metallic wires by virtue of being light weight and small.
3. Not only can they be manufactured with very small amount of material also the resources required for their production are available in plenty.
4. CNT's perform equally well both under extreme heat and extreme cold as they have high resistance to temperature changes.
5. Since CNT's have been the most sought out research topic under Nano-materials, most of the perks have been worked out ever since its invention.
6. There is a significant boost in the economy as investor have been piling into these R&D companies for further progress of this new technology.

Fig 1: Single Walled Carbon Nanotubes

Fig 2: Multi Walled Carbon Nanotubes

1.2 ADVANTAGES OF HNC

1. They are highly dispersive with fine particle size and high surface area.
2. They are available in many implementable forms such as powders, creams, gels etc.
3. They have superior loading rate compared to other materials and also possess fast adsorption.
4. They are non-swelling compounds with high aspect ratio and high porosity.
5. They have increased efficacy.


Fig 3: Halloysite Nanoclay

2. EXPERIMENTATION

In this project, experiment was carried out with specimens of constant diameter of 20mm and constant length of 40mm for both compression and split tensile strength. OPC 53 grade cement and sand passing through 2.3mm IS sieve was used to prepare the specimens. CNT's and HNC was added into the specimen in dosages of 0.5 wt%, 0.75 wt% and 1 wt% of the cement. W/C ratio was arbitrarily fixed at 0.45. The moulds were then placed for air drying for 24 hours. The specimens were removed from the moulds and placed into curing boxes. Tests were conducted after 7, 14 and 28 days of curing for both compression and splitting tensile. For compression test, the specimen was placed upon a steel disk to elevate the level of placement and aligned with the load cell. EMPEROR™ software from Mecmesin was used to provide commands to the machine for conduction of the test. In the software, a graph of load v/s deflection is plotted as the test is being conducted. Calculations of maximum load, maximum displacement, average value, sum etc can be done in the software itself. Once the test is completed, the software automatically displays the value of maximum load at which the specimen fails.

Fig 4: MultiTest 25-i Nano Universal Testing Machine (Funded by VGST, Bangalore)

International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 05, May-2016

2.1 FEATURES

- Maximum Load – 25,000N
- Depending upon the load cell it can perform both compression and tension testing.
- Head height – 1140mm
- Load resolution – 1:6500
- Maximum cross head movement – 950mm
- Weight of machine – 140kg
- Clearance between columns – 400mm
- Load scale accuracy
  - From 2 to 2500N – 0.1% of full scale
  - From 5000 to 25,000N – 0.2% of full scale
- Speed range
  - Up to 10kN: 1-1000mm/min
  - Above 10kN: 1-500mm/min
- It can hoist up to 10 different load cell capacities from 50N to 25kN
-

3. MATERIAL CHARACTERISATION

3.1 SCANNING ELECTRON MICROSCOPY

Fig 5: SEM imaging

The samples required for imaging were extracted from the cement mortar specimens. Samples extracted were less than a pinch in quantity which is more than enough for SEM imaging. These samples were mounted on aluminum stub over a vacuum compatible carbon tape. The samples were then subjected to gold coating to prevent charging. A thin layer of 10nm gold coat was applied on the sample. When the samples are bombarded with the electron peaks in the machinery, if there is no reception involved there is no image obtained. Hence in order to provide a proper potential for the electron beams gold coating is done.

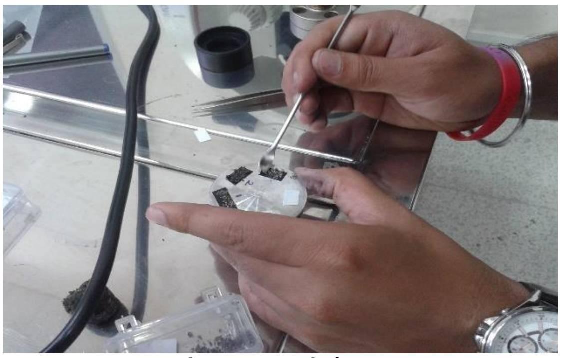


Fig 6: Sample preparation before SEM testing

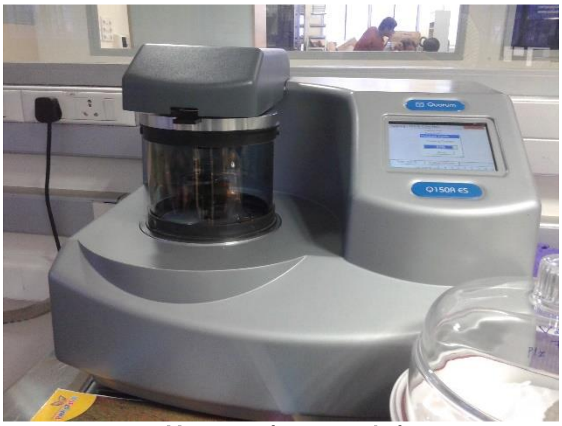


Fig 7: Gold coating of specimens before SEM

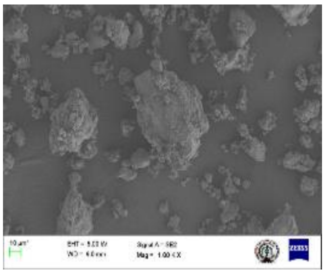
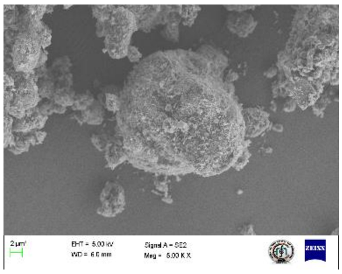
3.2 Energy Dispersive X-Ray Spectroscopy (EDX) Analysis

EDX is a used for the elemental analysis or chemical characterization of a sample. It is generally conducted along with SEM. When electron beams are absorbed by the samples in SEM, they emit X-rays which act as a source of emission for the spectroscopy. These x-rays emitted by the sample are characteristic to the electron hole pair formation at the atomic level of the sample used which in-turn is specific to the composition of the sample. Hence analysis of these emitted x-rays are used to study the chemical composition of the specimen.

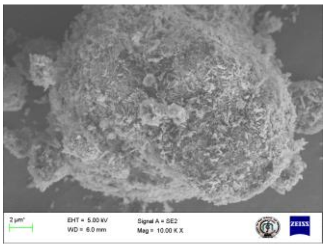
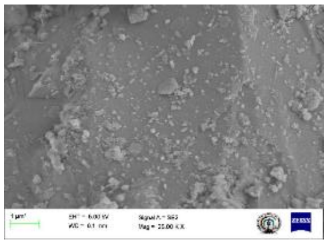
4. RESULTS AND DISCUSSIONS

4.1 SEM AND EDX

a) Sample 1: Cement: Sand (1:2) [before testing]

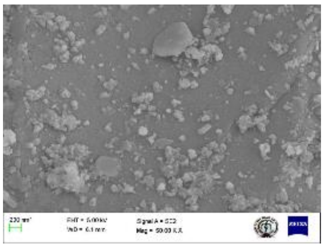



a) b)

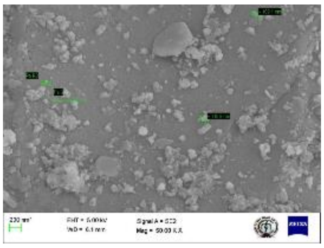



c) d)

International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 05, May-2016

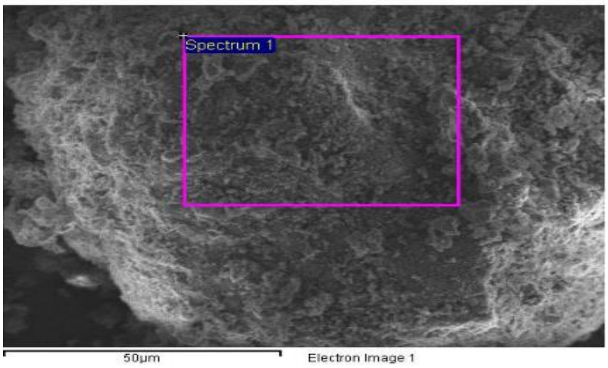


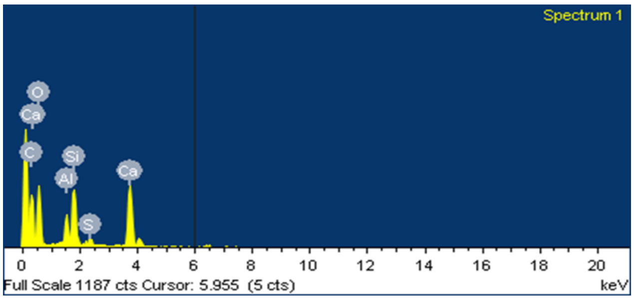
e)



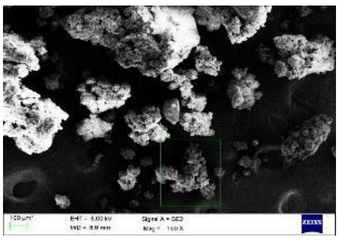
f)

- Sample 1: EDX analysis (before testing)

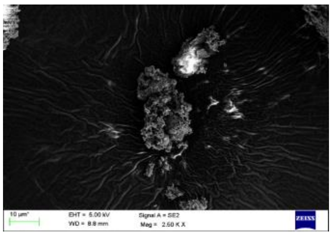




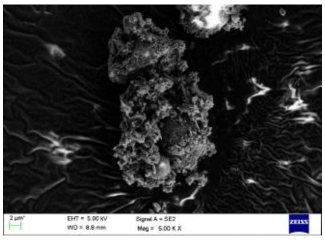
b) Sample 1: Cement: Sand (1:2) [after testing]



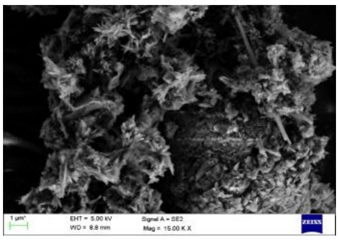
a)



b)

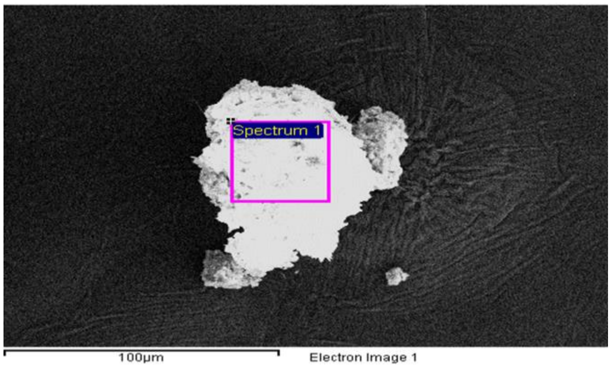


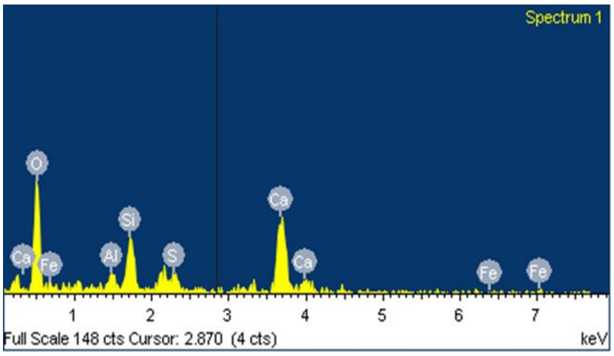
c)



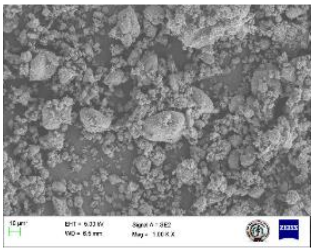
d)

- Sample 1: EDX Analysis (after testing)

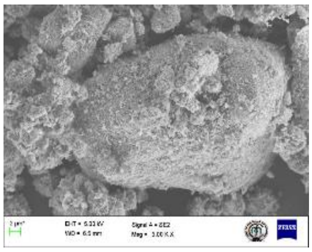




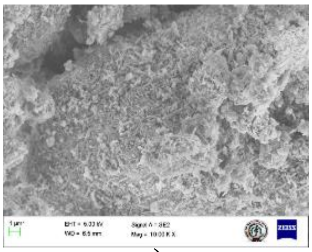
c) Sample 2: CM+HNC (before testing)



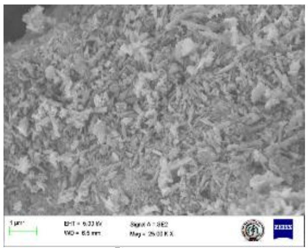
a)



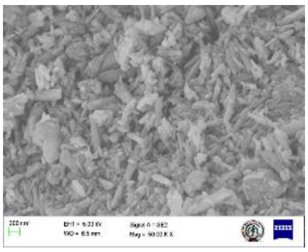
b)



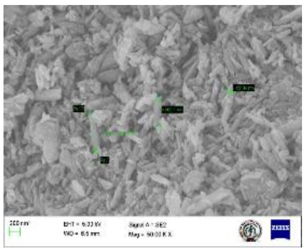
c)



d)



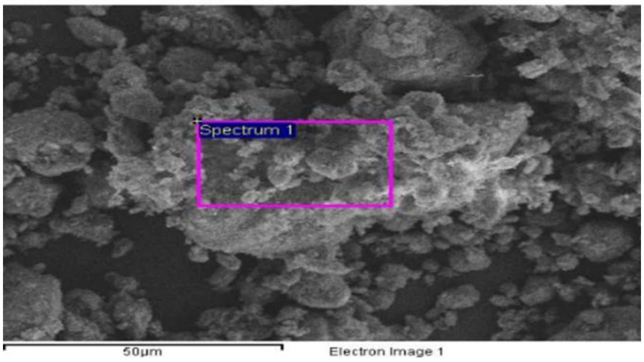
e)

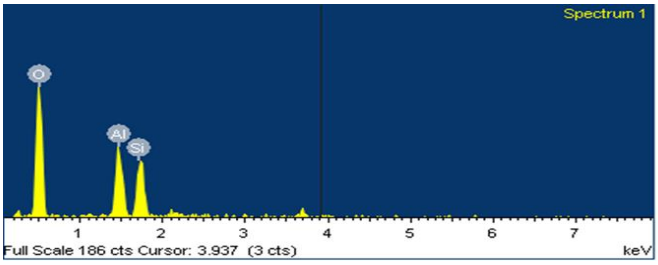


f)

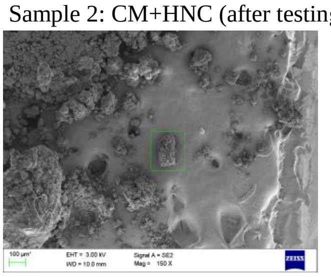
International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 05, May-2016

- Sample 2: EDX analysis (before testing)

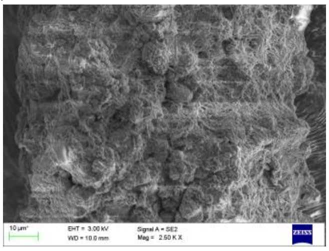




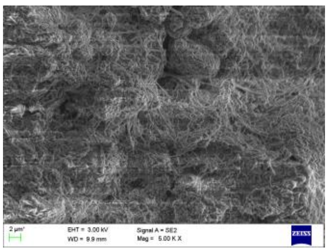
d) Sample 2: CM+HNC (after testing)



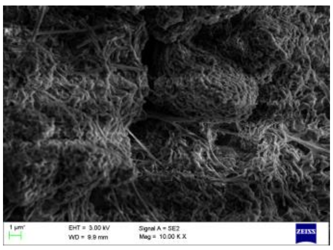
a)



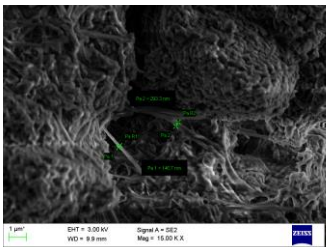
b)



c)

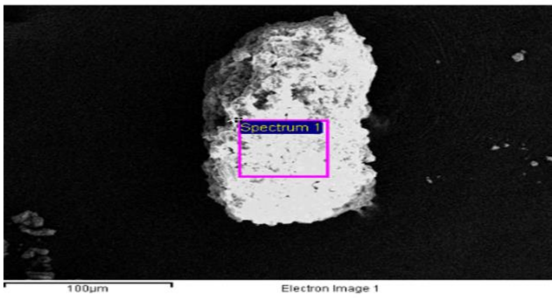


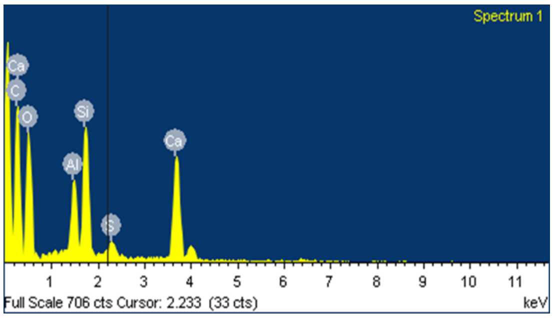
d)



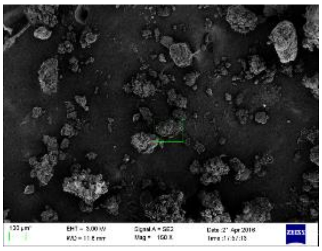
e)

- Sample 2: EDX analysis (after testing)

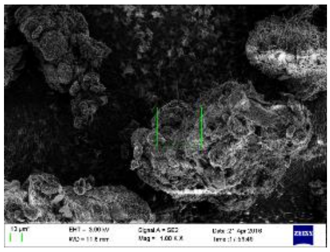




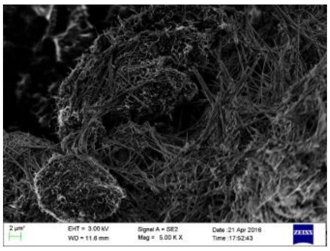
e) Sample 3: CM+MWCNT (before testing)



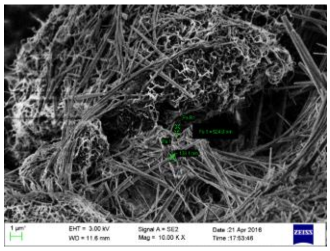
a)



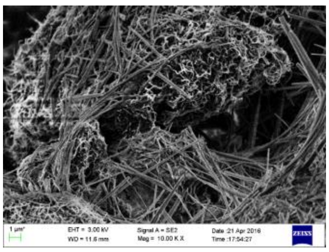
b)



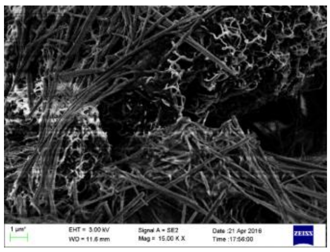
c)



d)



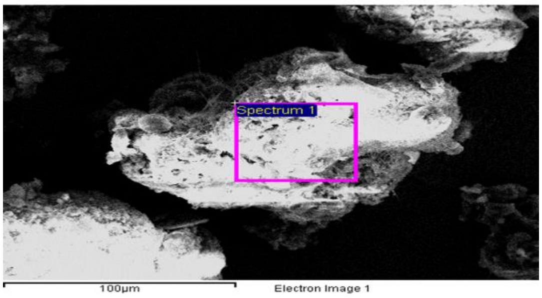
e)

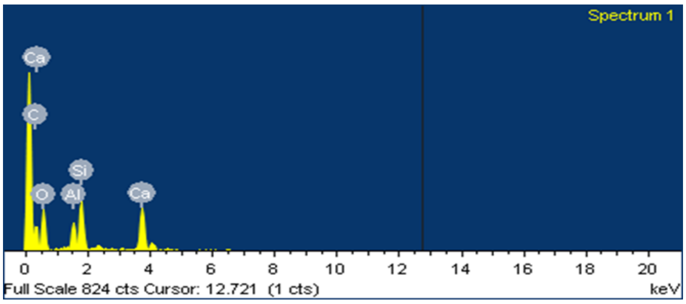


f)

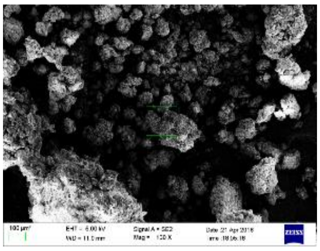
International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 05, May-2016

- Sample 3: EDX analysis (before testing)

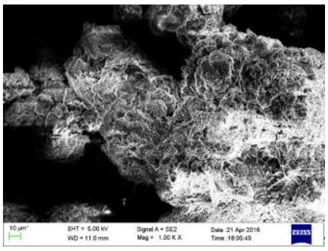




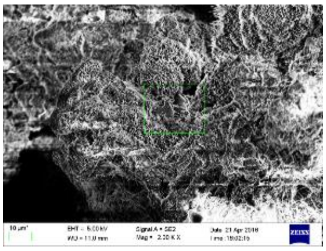
f) Sample 3: CM+MWCNT (after testing)



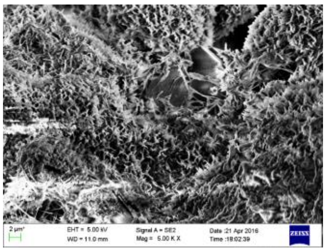
a)



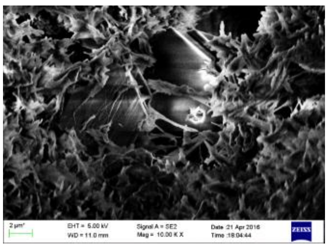
b)



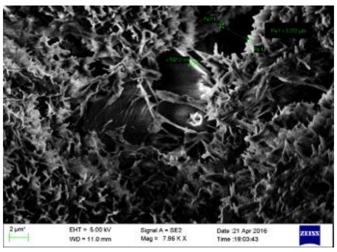
c)



d)

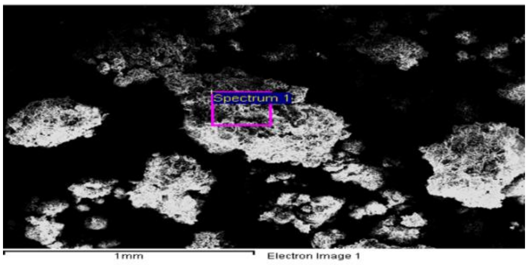


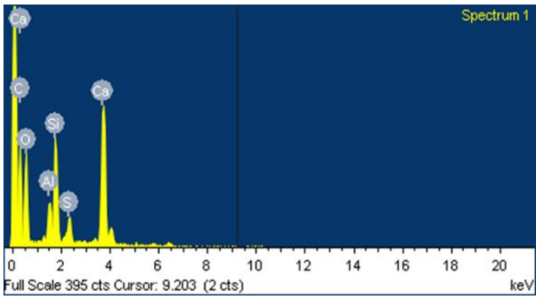
e)



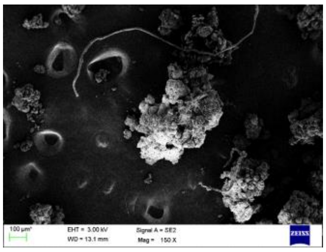
f)

- Sample 3: EDX analysis (after testing)

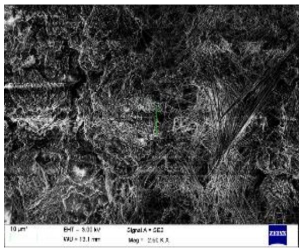




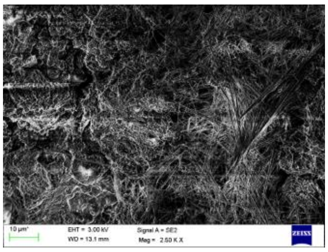
g) Sample 4: CM+SWCNT (before testing)



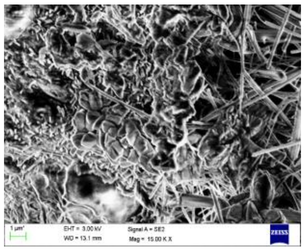
a)



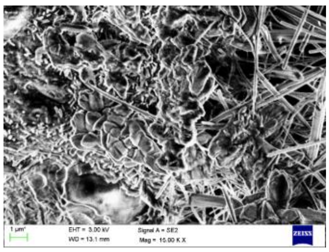
b)



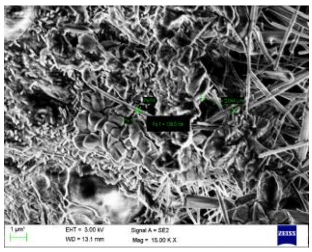
c)



d)



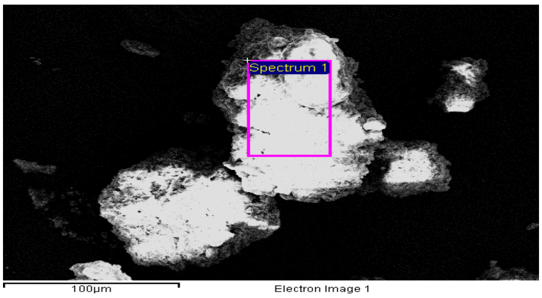
e)

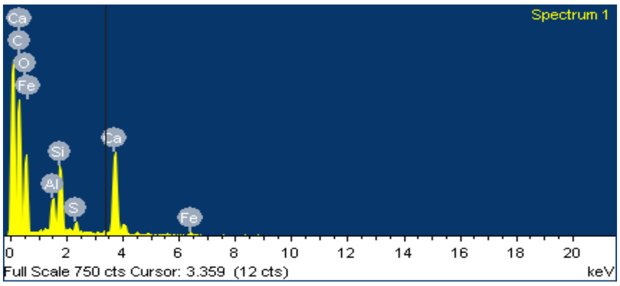


f)

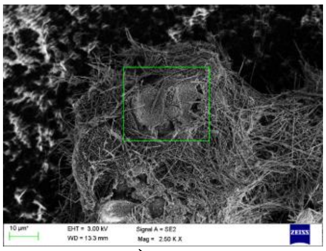
International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 05, May-2016

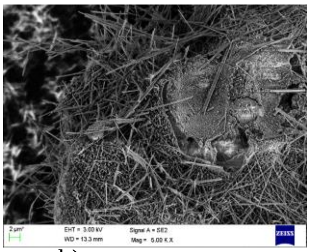
- Sample 4: EDX analysis (before testing)

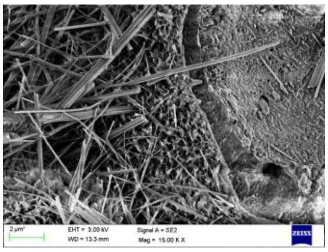


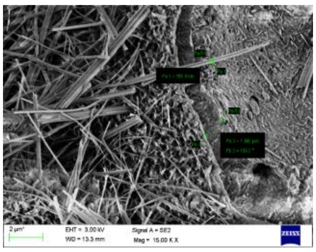


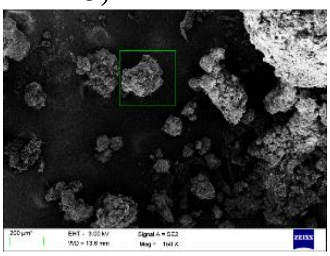
h) Sample 4: CM+SWCNT (after testing)

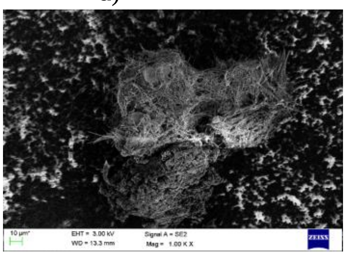

a)


b)

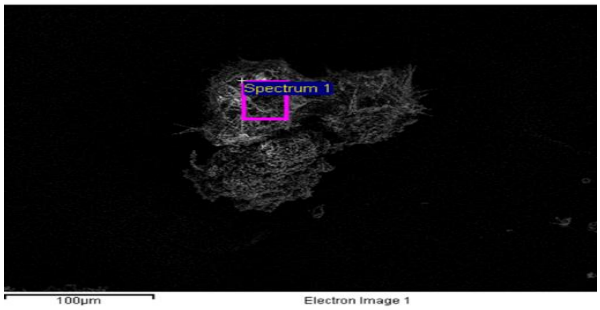

c)

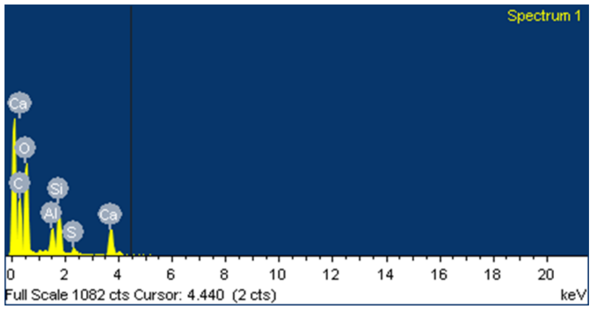

d)


e)


f)

- Sample 4: EDX analysis (after testing)





6. EXPERIMENTAL RESULTS

Compressive stress, N/mm^2 (28 day curing)		% of HNC
With HNC	Without HNC	
1.423	2.16	0.5
2.402	2.16	0.75
2.38	2.16	1

Fig 8: Stress values

Compressive stress, N/mm^2 (28 day curing)		% of SWCNT
With SWCNT	Without SWCNT	
1.163	2.16	0.5
0.795	2.16	0.75
2.295	2.16	1

Fig 9: Stress values

Compressive stress, N/mm^2 (28 day curing)		%of MWCNT
With MWCNT	Without MWCNT	
2.33	2.16	0.5
2.49	2.16	0.75
1.57	2.16	1

Fig 10: Stress values

Split tensile stress, N/mm^2 (28 day curing)		%of HNC
With HNC	Without HNC	
0.32	0.46	0.5
0.47	0.46	0.75
0.46	0.46	1

Fig 11: Split tensile strength values

Split tensile stress, N/mm^2 (28 day curing)		%of SWCNT
With SWCNT	Without SWCNT	
0.34	0.46	0.5
0.52	0.46	0.75
0.44	0.46	1

Fig 12: Split tensile values

Split tensile stress, N/mm^2 (28 day curing)		%of MWCNT
With MWCNT	Without MWCNT	
0.32	0.46	0.5
0.53	0.46	0.75
0.40	0.46	1

Fig 13: Split tensile strength

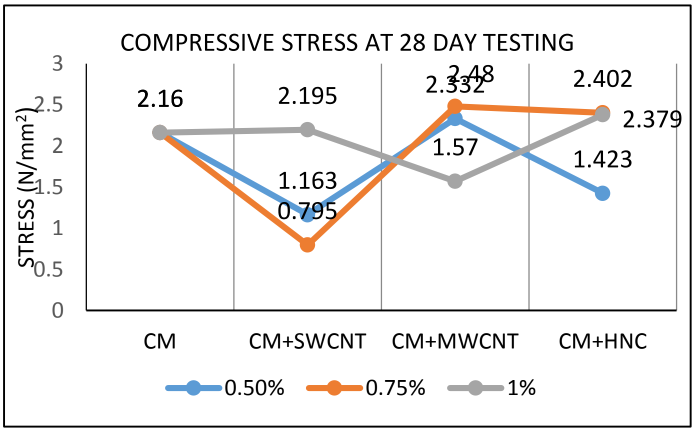


Fig 14: Compressive Stress at 28 day testing

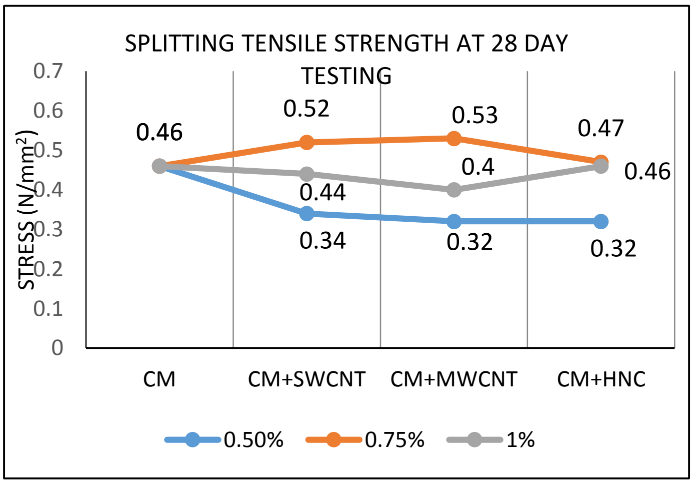


Fig 15: Splitting tensile strength at 28 day

7. CONCLUSIONS

a. After 28 days of curing, composite containing MWCNT showed 18.2% greater strength than nominal mix and HNC showed 14% more strength than nominal mix.
b. Compressive strength of composites with SWCNT showed 16.8% reduction in strength than nominal mix.
c. After 14 days of curing, composites with 0.75wt% SWCNT, MWCNT and HNC showed 13.1%, 15.3% and 2.7% of strength gain respectively as compared to nominal mix.
d. After 14 days of curing, composites with 0.5wt% SWCNT, MWCNT and HNC showed 34.3%, 43% and 44.8% reduction in strength respectively as compared to nominal mix.
e. The optimum dosage of nanotubes for compression testing was found to be 1wt% for HNC and 0.5wt% for both CNT's whereas for splitting tensile strength, optimum dosage was found to be 0.75wt% for all the nanotubes.

ACKNOWLEDGEMENT

Authors are highly indebted to VGST, Bangalore for funding MultiTest 25-i Nano under K-FIST, Level-2. We also thank Centre for Nano Science and Engineering, IISc, Bangalore for providing SEM and EDX analysis reports. We also thank Dr. Mohamed Haneef, Principal, GCE and Dr. Mohamed Ilyas Anjum, Vice- principal, HoD, Dept. of Civil Engg, GCE, Ramanagaram for their continuous support.

8. REFERENCES

[1] Sanjeev Kumar, F.ASCE; Prabir Kolay, M.ASCE; Sunil Malla; and Sanjay Mishra. 'Effect of Multiwalled Carbon Nanotubes on Mechanical Strength of Cement Paste.' DOI: 10.1061/(ASCE)MT.1943-5533.0000350. © 2012 American Society of Civil Engineers .
[2] Tanvir Manzur and Nur Yazdani, 'Optimum Mix Ratio for Carbon Nanotubes in Cement Mortar.' KSCE Journal of Civil Engineering (2015) 19(5):1405-1412 Copyright ©2015 Korean Society of Civil Engineers DOI 10.1007/s12205-014-0721-x
[3] Ravindra Kamble, Manasi Ghag, Sheetal Gaikawad, Bijoy Kumar Panda. 'Halloysite Nanotubes and Applications: A Review' Journal of Advanced Scientific Research**,** *Ravindra Kamble et al, J Adv Scient Res, 2012, 3(2): 25-29*
[4] Vindhya C.R, N.S Kumar. 'Experimental investigation on Halloysite Nano tubes & clay in infilled composite steel tube.' MTech thesis submitted to VTU in 2014.
[5] M. T Albdiry, B. F Yousif. 'Role of silanized Halloysite Nano tubes on structural, mechanical properties and fracture toughness of thermoset Nano composites.' Materials and design 57 (2014) 279-288, www.elsevier.com/locate/matdes
[6] Deepak Rawtani and Y.K. Agrawal. 'Multifarious applications of Halloysite Nanotubes: a review.' @2012 Advanced Study Centre Co. Ltd, Rev.Adv.Mater.Sci.30(2012)282-295.
[7] Ravindra Kamble, Manasi Ghag, Sheetal Gaikawad, Bijoy Kumar Panda. 'Halloysite Nanotubes and Applications: A Review' Journal of Advanced Scientific Research**,** *Ravindra Kamble et al, J Adv Scient Res, 2012, 3(2): 25-29*
[8] Naresh R. Shimpi, Prof. D. N Jadhav. 'Carbon Nano-Particle As A Composite Structure: A Technical Review.' International Journal of Engineering Research & Technology (IJERT)ISSN: 2278-0181 www.ijert.org Vol. 2 Issue 6, June – 2013.
[9] Tanvir Manzur and Nur Yazdani, 'Optimum Mix Ratio for Carbon Nanotubes in Cement Mortar.' KSCE Journal of Civil Engineering (2015) 19(5):1405-1412 Copyright ©2015 Korean Society of Civil Engineers DOI 10.1007/s12205-014-0721-x
[10] V. Kartik Ganesh. 'Nanotechnology in Civil Engineering.' Department of civil engineering, SRM University, Chennai.
[11] A. Hakamy, FUA Shaikh, I.M Low. 'Microstructures and mechanical properties of hemp fabric reinforced organoclay-cement Nanocomposites.' Construction and building materials 49 (2013) 298-307. www.elsevier.com/locate/conbuiltmat.
[12] M.L Nehdi. 'Clay in cement based materials: Critical overview of state of the art.' Construction and building materials 51 (2014) 372-382. . www.elsevier.com/locate/conbuiltmat.
[13] M.T Albdiry, H. Ku, B.F. Yousif. 'Impact fracture behaviour of silane treated Halloysite Nanotubes reinforced unsaturated polyster.' Engineering failure analysis 35 (2013) 718-725. Www. Elsevier.com/locate/engfailanal.
[14] Rashid K. Abu Al-Rub, Bryan M. Tyson, Ardavan Yazdanbakhsh, and Zachary Grasley. 'Mechanical Properties of Nanocomposite Cement Incorporating Surface-Treated and Untreated Carbon Nanotubes and CarbonNanofibers.' http://dx.doi.org/10.1061/(ASCE)NM.2153-5477.0000041 © 2012 American Society of Civil Engineers.

International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 05, May-2016

[15] K. Prashantha, M. F. Lacrampe, P. Krawczak. 'Processing and characterization of Halloysite Nanotubes filled polypropylene Nanocomposites based on a masterbatch route: effect of Halloysites treatment on structural and mechanical properties.' eXPRESS Polymer Letters Vol.5, No.4 (2011) 295–307 Available online at www.expresspolymlett.comDOI:10.3144/ expresspolymlett.2011.30
[16] Rashid K. Abu Al-Rub, Bryan M. Tyson, Ardavan Yazdanbakhsh, and Zachary Grasley. 'Carbon Nanotubes and Carbon Nanofibers for Enhancing the Mechanical Properties of Nanocomposite Cementitious Materials.' DOI: 10.1061/(ASCE)MT.1943-5533.0000266. © 2011 American Society of Civil Engineers.
[17] Micro & Nano Characterization Facility (MNCF) Center for Nano Science and Engineering (CENSE) IISc, Bangalore.
[18] R&D Lab, Dept of Civil Engineering, GCE, Ramanagaram.
[19] Sigma Aldrich® Bommasandra, Bangalore.

BIOGRAPHIES

Graduated in the year 2014 from VTU, Belgaum Presently perusing Master of Technology in Structural Engineering at Ghousia College of Engineering, Ramanagaram Also working on this topic for the dissertation under the guidance of Dr. N S Kumar

Involved in the Research field related to behavior of Composite Steel Column since a decade Presently guiding 5 Ph.D Scholars (Research under VTU, Belgaum) Has more than 25 years of teaching experience & 6 years of Research experience at Ghousia College of Engineering, Ramanagaram.