



Experimental Investigation on Steel Columns Infilled With Halloysite Nano Tubes

KEYWORDS

Halloysite Nano Tubes¹, SEM², Fracture Analysis³.

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ABSTRACT Concrete filled steel tubes have been extensively used in the modern Structure mainly due to the combination of the advantage of Steel tube & Concrete core. The in-fill material inside Steel tubes is required to be of the quality as to increase the ductility, but not the strength of composite columns, many kind of in-fill materials were used to improve ductility of composite columns. Among the various in fill materials, we are using Halloysite Nano Tubes (HNT's). Naturally formed in the Earth over millions of years, Halloysite Nano Clay are unique and versatile Nano materials that composed of aluminum, silicon, hydrogen and oxygen and are mined from natural deposits in countries like China, New Zealand, America, Brazil, and France In this experimental research , the effects of the diameter, length of steel tube, grade of concrete & volume fractions of HNT's to concrete (0%,0.1%,0.2%,0.3%) on the behavior of Halloysite Nano Tubes(HNT's) concrete filled steel tube columns under axial compression are being investigated. Also, study being conducted on the effect of Diameter (D), Change in steel tube length (l), and Strength of infill (f_{CK}) and the ultimate load (P_{ult}) & deflection (Δ axial) in HNT's composite steel hollow tubes under monotonic loading and SEM (scanning electron microscope) images are taken during mixing, before testing and after testing along with the Steel tube -Fracture analysis carried out for the buckled steel tubes using Radiographic Testing.

1. INTRODUCTION

Composite Steel Concrete construction has been widely used in many Structures such as Building and Bridge .The concrete encased composite column is one of the common composite structural elements. At the same time, due to the traditional separation of structural Steel and Reinforced Concrete Design and Construction, this type of Column has not received the same level of attention as Steel or Reinforced Concrete Column.

Composite Structures from Concrete Steel section show considerable larger stiffness, stability and load carrying capacity in comparison with steel construction .An increase of corrosion and fire resisting is an addition advantage of concrete element.

Recent developments in nanoscience and nanotechnology opened fundamental and applied new frontiers in science and materials engineering. Advanced materials are being developed with enhanced chemical and physical properties with unique characteristics. The properties of these materials are determined not only by their composition and chemical bonds, but also by size and morphology. The nanotube (NT) term is recent; the idea of a small tubular structure is not new. In 1930, Linus Pauling (1930) proposed the existence of cylindrical structures formed by minerals in nature.

The formation of halloysite is due to hydrothermal alteration, and it is often found near carbonate. For example, halloysite samples found in Wagon Wheel Gap, Colorado and United are suspected to be the weathering product of rhylolite by downward moving waters. In general the formation of clay minerals is highly favored in tropical and sub-tropical climates due to the immense amounts of water flow. Halloysite has also been found overlaying basaltic rock, showing no gradual changes from rock to mineral formation. Halloysite occurs primarily in recently exposed volcanic-derived soils, but it also forms from primary min-

erals in tropical soils or pre-glacially weathered materials. Igneous rocks, especially glassy basaltic rocks are more susceptible to weathering and alteration forming halloysite.

1.2 Advantages of Halloysite Nanotubes

- Fine particle size, high surface area and dispersion.
- Implementable in many forms such as powders, creams, gels,
- Superior loading rates to other carriers, Fast adsorption rate
- High aspect ratio, high porosity and non swelling
- Regeneration ability and increased efficacy

2. EXPERIMENTAL PROGRAM

Experiment were carried out on seven different specimens with four variations in each specimen, which varies in length 50mm,75mm,100mm,125mm,150mm,175mm and 200mm with constant diameter 43mm. And mixed with Cement, Sand & HNT's (0.1%, 0.2%, 0.3% and 0.4%).

Sample1- 1:2:3 (Cement: HNT: Sand)

Sample2- 1:1.5:3 (Cement: HNT: Sand)

Sample3- 1:2 (Cement: HNT)

Sample4- 2:3 (HNT: Sand)

Sample5- 1:1.5 (Cement: HNT)

Sample6- 1.5:3 (HNT: Sand)

Sample7- Cement

Sample8- HNT's

Sample9- Sand

Sample10- After compression testing

2.1 Halloysite Nanotubes



Fig1.Source: Sigma Aldrich (manufacturer)-New Zealand [1]

Physical and chemical properties (provide by the supplier)

- ✓ Synonyms: Kaolin clay
- ✓ Appearance Form: powder
- ✓ Colour: White to Tan
- ✓ Relative density 2, 53 g/cm³
- ✓ Formula: H₄Al₂O₉Si₂ · 2 H₂O
- ✓ Molecular Weight: 294, 19 g/mol
- ✓ P^H Value 6.5-6.9
- ✓ Pore volume 1.26-1.34 ml/gm
- ✓ Diameter 30-70nm (nanometers)
- ✓ Length 0.25-4 microns.

2.2 Characterization

2.2 .1 Scanning electron microscopy (SEM)

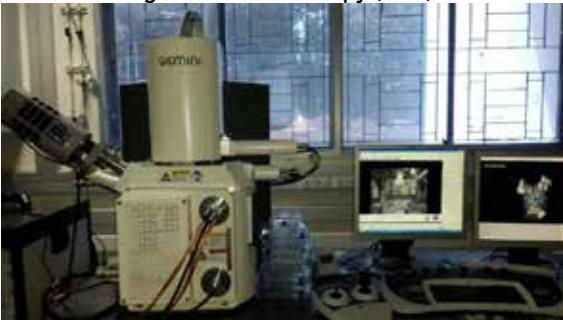


Fig2. SEM (Scanning electron microscopy)

A Scanning electron microscopy [2] imaging was obtained to investigate the microstructures and the fracture surfaces of composites. The samples were mounted on aluminum stubs using carbon tape. The samples were then coated with a thin layer of gold to prevent charging before the observation by SEM.

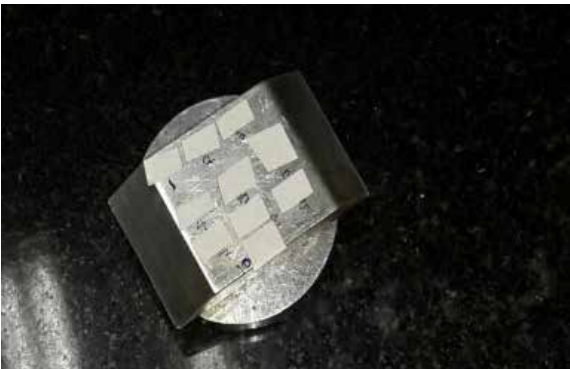


Fig3.Sample preparation before SEM testing [2]



Fig4.Gold coating after sample preparation before SEM testing [2]

Ultra high resolution scanning electron imaging coupled with material spectroscopy tools

The ULTRA 55 represents the latest development in GEMINI technology. Based on the SUPRA 55, the ULTRA 55 now comprises a fully integrated Energy and angle selective Backscattered electron (EsB) detector. The ULTRA 55 offers ultra high resolution for both SE to image surface information and BSE to present compositional information. The new EsB detector features an integrated filtering grid to enhance image quality and requires no additional adjustments. The EsB detector is less sensitive for edge contrast and charging effects which enables precise imaging and measurement of boundaries, particles, and features. Combined with the large multi-port analytical chamber, the fully motorised 5-axes motorised eucentric stage and the GEMINI high current mode the ULTRA 55 also offers superb analytical capabilities.

2.2 .1 Fracture Analysis

Radiographic Testing were examined using Iridium-192 diameter 2.7*1.2mm, Strength 30 Ci Gamma Ray machine





View of a Gamma Ray machine [3]

2.2.2 Energy-dispersive X-ray spectroscopy (EDX) Analysis

EDX is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray spectrum

Another way to use SEM/EDX is to make a quantitative chemical analysis of materials.

2.2.3 200 Ton cyclic Loading machine

Hydraulic press for testing load comprising Press frame; hydraulic cylinder (dia320Xdia 250X250mm stroke). Hydraulic power pack 100 it with electric motor 5hp X 1440rpm, electrical control panel operating with PLC SCA-DA software, strain gauge SI -30 & strain indicator. To conduct the compression tests on all the specimens.



Fig5. Concrete filled steel tube subjected to monotonic

loading [4]



Fig6. Composite steel tube under testing [4]

3. RESULTS & DISCUSSIONS

3.1 Scanning electron microscopy (SEM)

Sample1- 1:2:3(Cement: HNT: Sand)

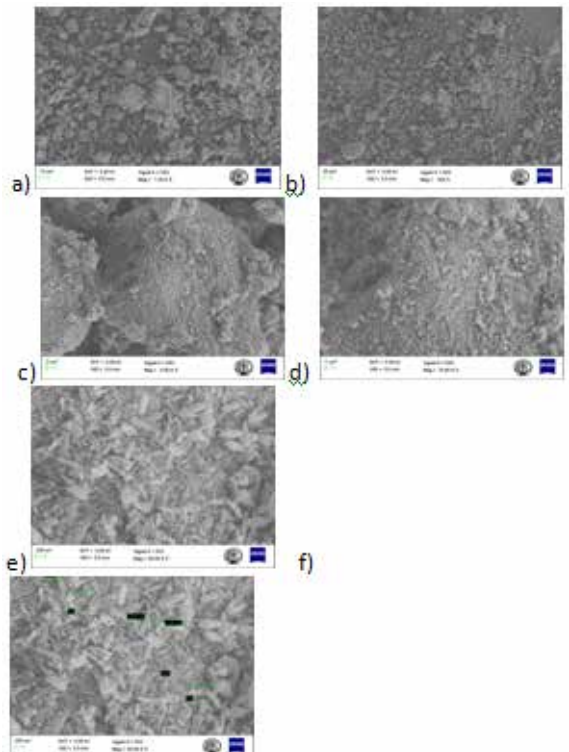
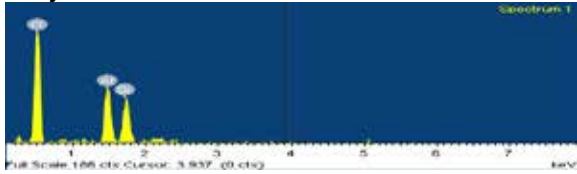


Fig7. SEM Images of morphology from low to high

magnification (a-f)
Sample1- Energy-dispersive X-ray spectroscopy (EDX) Analysis



Sample2- 1:1.5:3 (Cement: HNT: Sand)

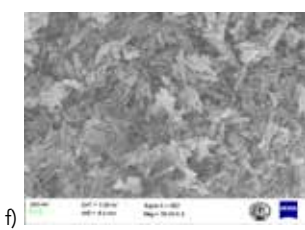
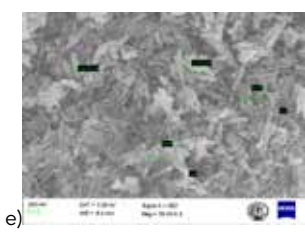
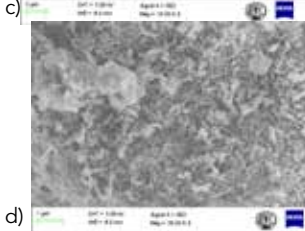
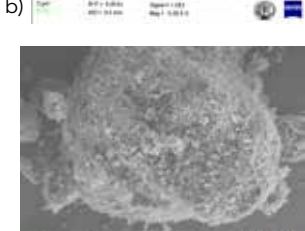
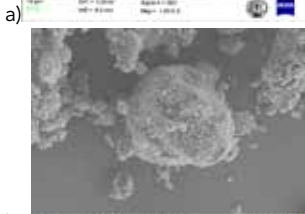
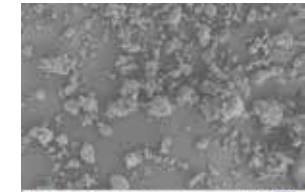
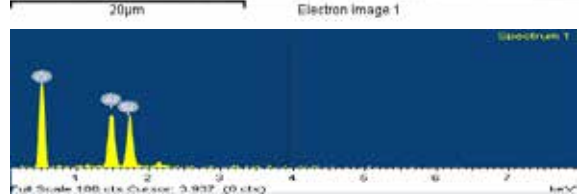
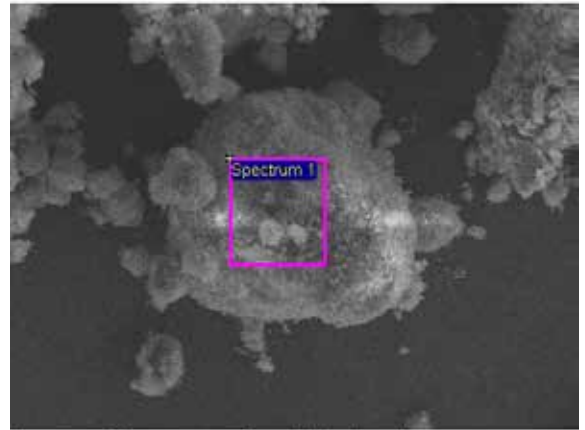
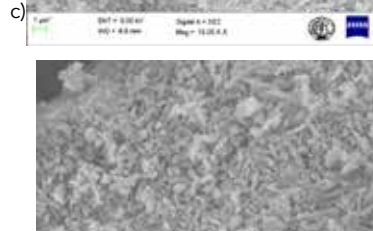
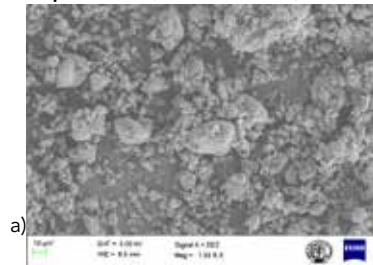


Fig8. SEM Images of morphology from low to high magnification (a-f)

Sample2- Energy-dispersive X-ray spectroscopy (EDX) Analysis



Sample3- 1:2 (Cement: HNT)



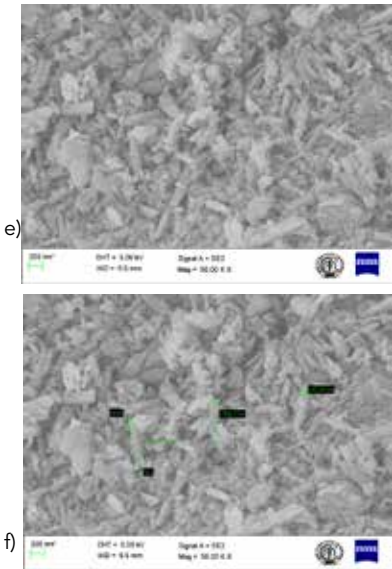
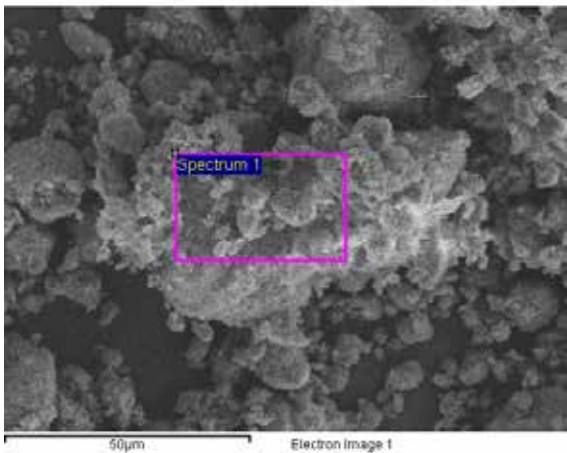


Fig9. SEM Images of morphology from low to high magnification (a-f)



Sample3- Energy-dispersive X-ray spectroscopy (EDX) Analysis

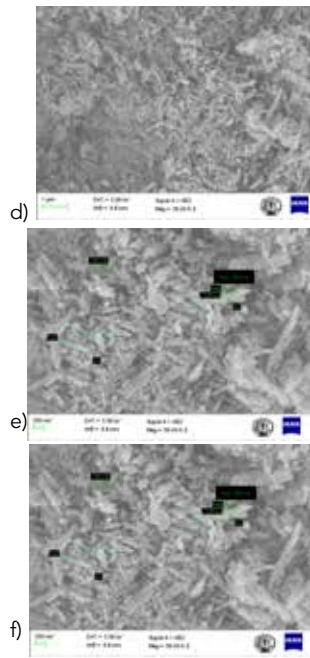
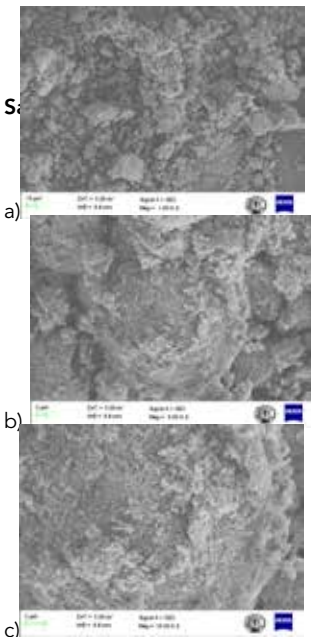
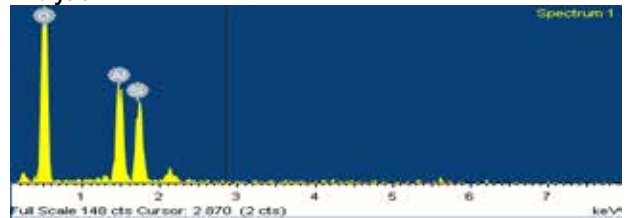
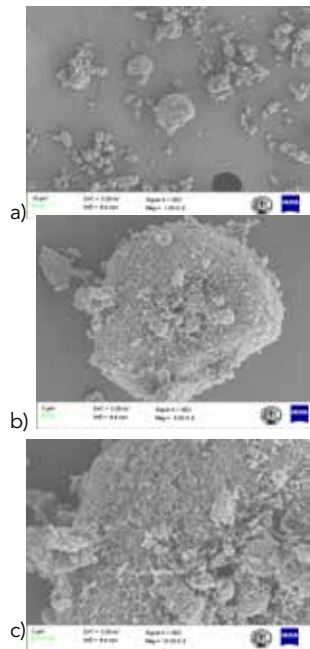


Fig10. SEM Images of morphology from low to high magnification (a-f)

Sample4- Energy-dispersive X-ray spectroscopy (EDX) Analysis



Sample5- 1:1.5 (Cement: HNT)



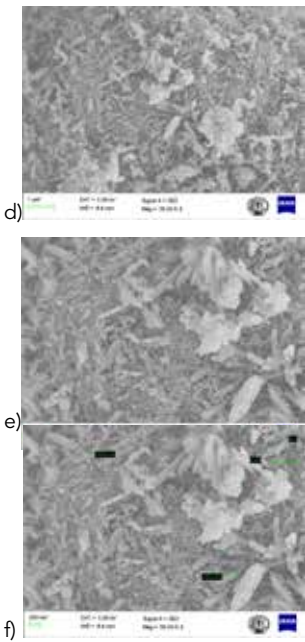
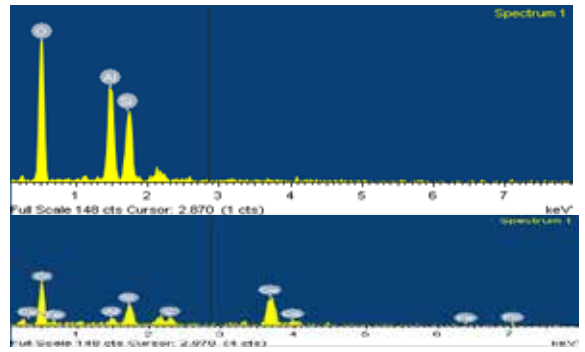
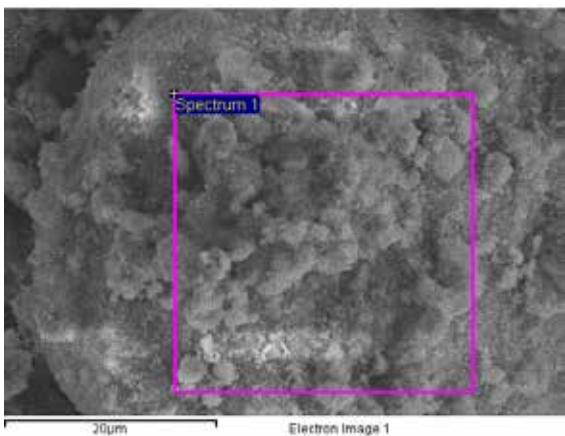
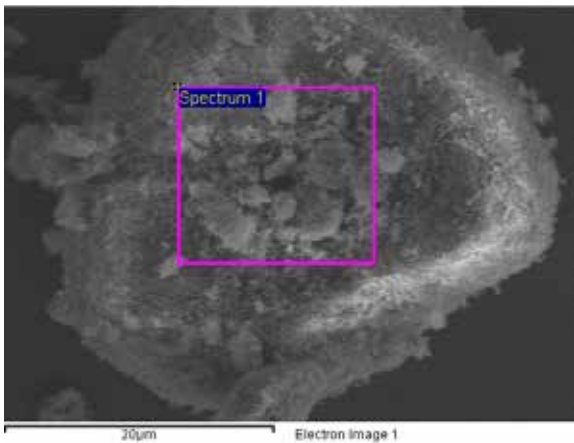


Fig11. SEM Images of morphology from low to high magnification (a-f)

Sample5- Energy-dispersive X-ray spectroscopy (EDX) Analysis



Sample6- 1.5:3 (HNT: Sand)

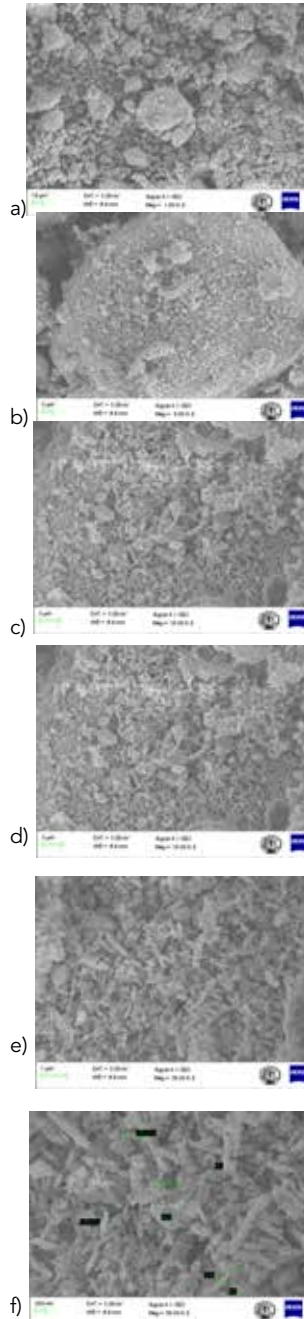
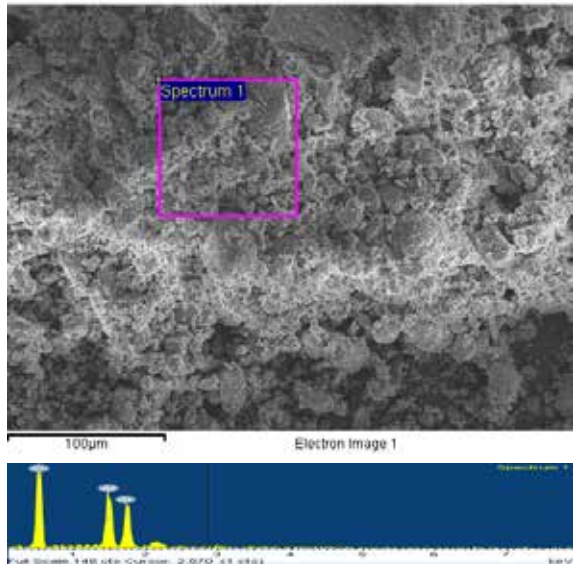
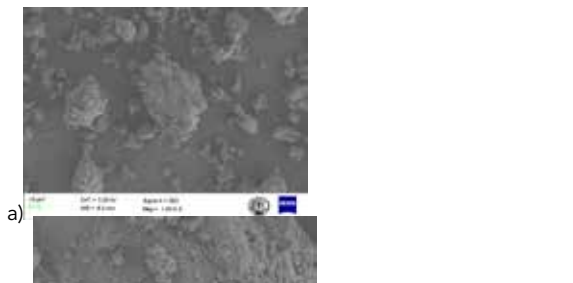


Fig12. SEM Images of morphology from low to high magnification (a-f)

Sample6- Energy-dispersive X-ray spectroscopy (EDX) Analysis



Sample7- Cement



Sample8- HNT's

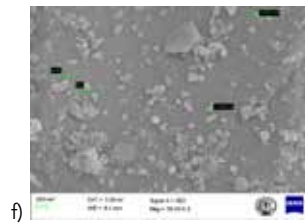
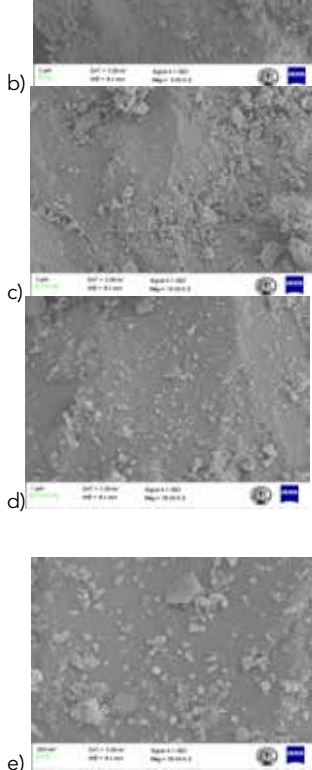
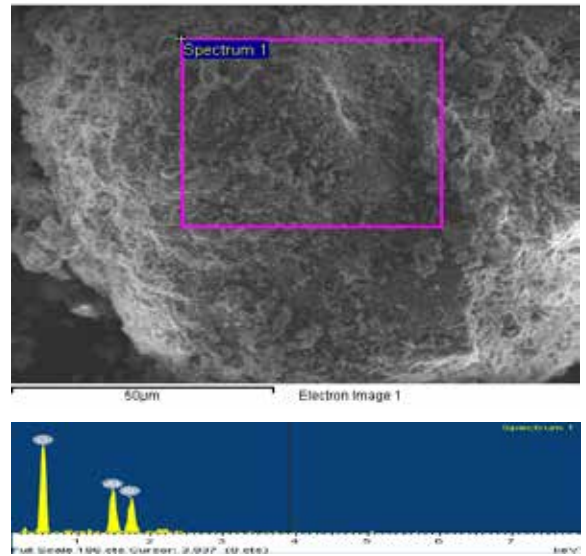
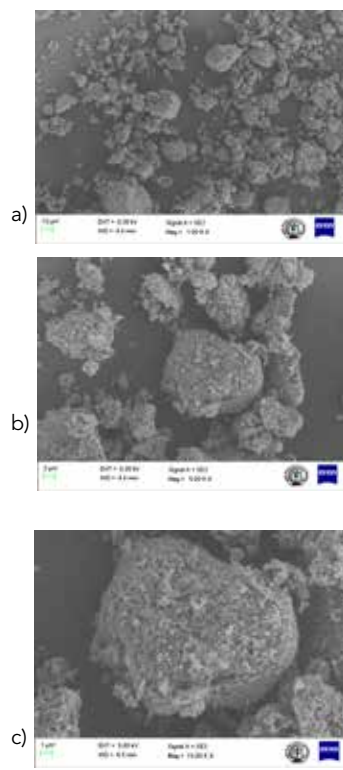


Fig13. SEM Images of morphology from low to high magnification (a-f)

Sample7- Energy-dispersive X-ray spectroscopy (EDX) Analysis



Sample8- HNT's



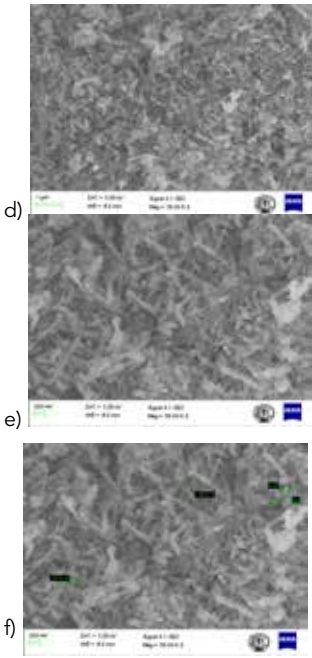
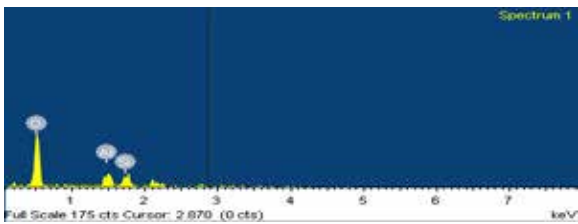
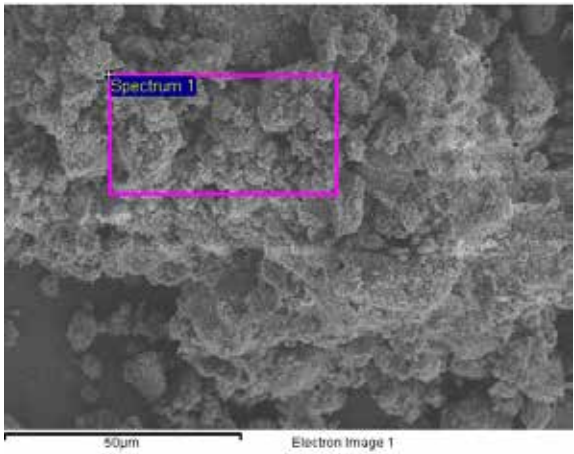
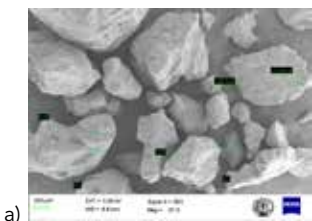


Fig14. SEM Images of morphology from low to high magnification (a-f)

Sample8- Energy-dispersive X-ray spectroscopy (EDX) Analysis



Sample9- Sand



a)

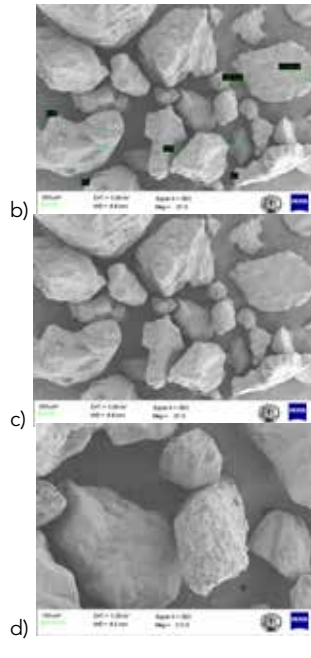
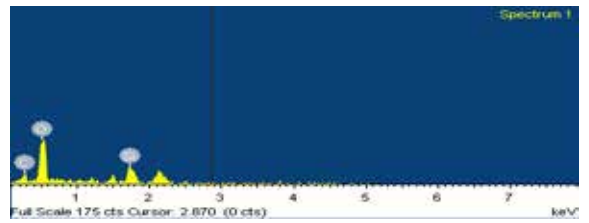
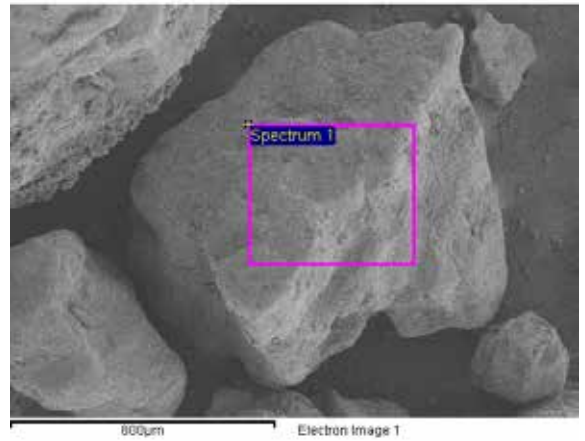
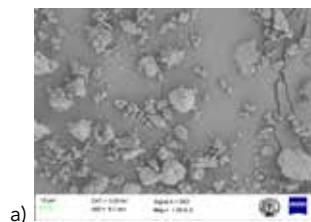


Fig15. SEM Images of morphology from low to high magnification (a-f)

Sample9- Energy-dispersive X-ray spectroscopy (EDX) Analysis



Sample10- After compression testing



a)

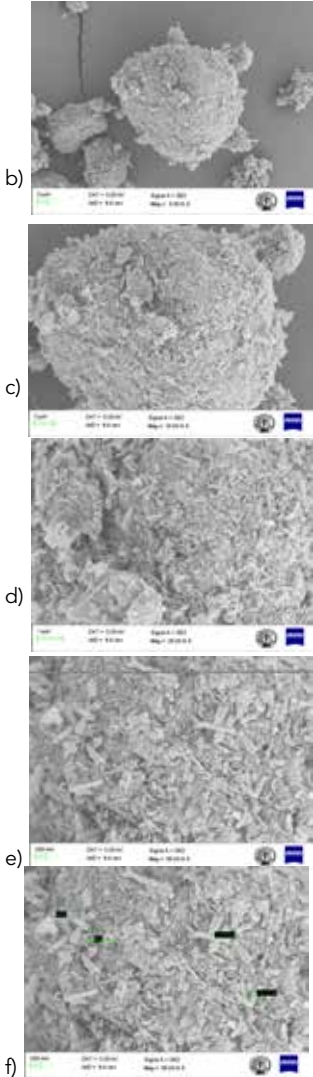
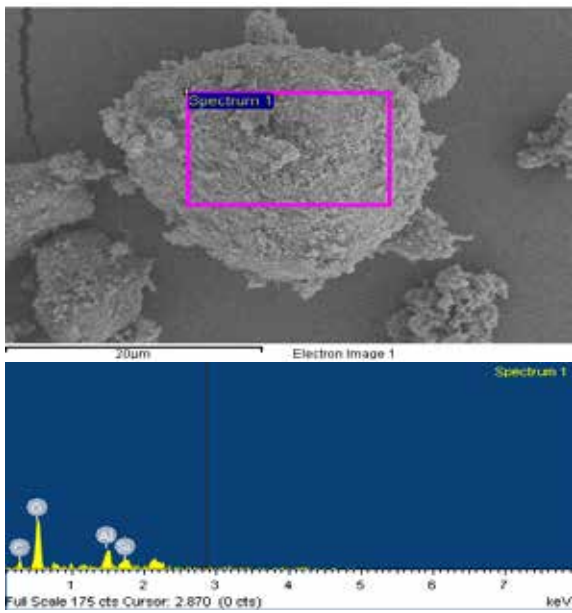


Fig16. SEM Images of fracture surface morphology from low to high magnification (a-f)

Sample10- Energy-dispersive X-ray spectroscopy (EDX) Analysis



3.2 Fracture Analysis

Radiographic Testing were examined for the samples

- 1) Length 300mm, c/s3*3mm -stainless steel
- 2) Length 300mm, diameter 23mm-middle steel
- 3) Length 280mm, diameter 22mm- middle steel
- 4) Length 30mm, diameter 30mm -middle steel

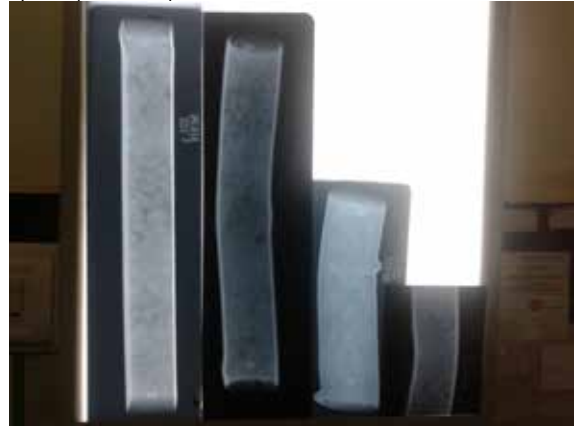


Fig 17 radiographic film

When the Tube was monotonically loaded it was observed that crack patters as shown in the fig1 & were obtained from the radiographic testing and further work is being carried out by the author for modeling the future using facture analysis approach and for further developing the mathematical modeling for facture.

5) Length 150mm, diameter 43mm -mild steel



Facture analysis test was conducted using ASM hand book method. There are two cracks are observed. Type of frac-

ture are Ductile fracture, Type of crack is cleavage crack, crack length are 13.5mm, depth 2mm, width 2.6mm, small crack length are 4mm,width 1.3mm , depth 1mm.

3.3 Experimental Results

Length 150mm, diameter 20mm, thickness 5mm & M20 (1:2:3) concrete.

| Ultimate load (P_{ult}) in KN | | |
|-----------------------------------|------------|------------|
| Without HNT's | With HNT's | HNT's in % |
| 86 | 86.68 | 0.1 |
| 88.10 | 89.25 | 0.2 |
| 89.52 | 90.11 | 0.3 |
| 90.36 | 91.32 | 0.4 |

Fig 18- Result Table

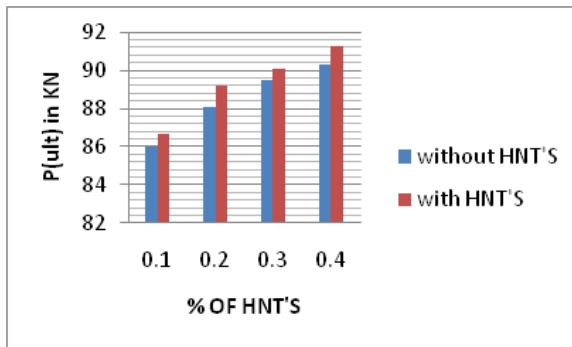


Fig 19- Ultimate load (P_{ult}) vs. HNT's graph

4. CONCLUSIONS

Physical and chemical properties of the HNT's tested were founded to be very positive for civil engineering applications. Especially it has light weight and is in the powder form which can mix well with the cement & sand. Tests are being conducted for the compressive strength of HNT's. When mixed with only cement, only sand & all the three. SEM & EDX analysis are show's that HNT's can lead to homogenous mixture which in turns enhances load carrying capacity of composite steel column.

As shown in the fig 19 as percent of HNT's increase load also increase observed and ultimate load (P_{ult}) is increased but reached optimum and started decreasing between 1.5 to 2.0%.

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- [1] Sigma Aldrich@ Bommasandra, Bangalore. | [2] Micro & Nano Characterization Facility (MNCF) Center for Nano Science and Engineering (CENSE) IISc, Bangalore | [3] Pallakki NDT Excellence Center, Peenya, Bangalore | [4] R&D Lab, Dept of Civil Engineering, GCE, Ramanagaram. | [5] Alamri H, Low IM. Microstructural, mechanical, and thermal characteristics of recycled cellulose .ber-halloysite-epoxy hybrid nanocomposites. *Polym | Compos* 2012;33(4):589-600. | [6] Churchman GJ, Davy TJ, Aylmore LAG, Gilkes RJ, Self PG. Characteristics of .nepores in some halloysites. *Clay Miner* 1995;30:89-98. | [7] Joussein E, Petit S, Churchman J, Theng B, Right D, Delvaux B. Halloysite clay minerals a review. *Clay Miner* 2005;40:383-426. | [8] Smith ME, Neal G, Trigg MB, Drennan J. Structural characterization of the thermal transformation of halloysite by solid-state NMR. *Appl Magn Reson* 1993;4:157-70. Cer | [9] Fix, D., Andreeva, D.V., Lvov, Y.M., Shchukin, D.G., Möhwald, H., 2009. | [10] Application of inhibitor-loaded halloysite nanotubes in active anti-corrosive coatings. *Advanced* | [11] *Functional Materials* 19 (11), 1720-1727. | 2006. Layer-by-layer assembled nanocontainers for self-healing corrosion protection. | *Advanced Materials* 18 (13), 1672-1678. | [12] Prashantha K, Lacrampe MF, Krawczak P. Processing and characterisation of halloysite nanotubes filled polypropylene nanocomposites based on a master batch route: effect of halloysites treatment on structural and mechanical properties. *Express Polym Lett* 2011;5(4):295-307. | [13] Handge UA, Hedicke-Höchstötter K, Altstadt V. Composites of polyamide-6 and silicate nanotubes of the mineral halloysite: in.uence of molecular weight on thermal, mechanical and rheological properties. *Polymer* | 2010;51(12):2690-9. | [14] Lin Y, Ng KM, Chan C-M, Sun G, Wud J. High-impact polystyrene/halloysite nanocomposites prepared by emulsion polymerization using sodium | dodecyl sulfate as surfactant. *J Colloid Interface Sci* 2011;358:423-9. | [15] Yuan Q, Misra RDK. Polymer nanocomposites: current understanding and issues. *Mater Sci Technol* 2006;22:742-55. | [16] Friedrich K, Fakirov S, Zhang Z. Polymer composites: from nano-to-macro-scale. (USA): Springer Science & Business Media, Inc.; 2005. | [17] Deng S, Zhang J, Ye L. Halloysite-epoxy nanocomposites with improved particle dispersion through ball mill homogenisation and chemical treatments. | *Compos Sci Technol* 2009;69:2497-505. | [18] Morote-Martinez V, Pascual-Sanchez V, Martin-Martinez JM. Improvement in mechanical and structural integrity of natural stone by applying | unsaturated polyester resin-nanosilica hybrid thin coating. *Eur Polym J* 2008;44:3146-55. |