

# EXPERIMENTAL INVESTIGATION ON HALLOYSITE NANO TUBES & CLAY AN INFILLED COMPOSITE STEEL TUBE

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## Abstract

This research aims at Comparative experimental behavior of Halloysite Nano Tubes & Clay as an in-fill to Composite Steel tubes. Both materials are of Nano category and one is in the tubular form and the other amorphous. 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. The effects of the diameter, length of steel tube, grade of concrete & volume fractions of HNC's to concrete (0%, 0.5%, 1%, 1.5%, & 2%) on the behavior of Halloysite Nano Clay (HNC's) concrete filled steel tube columns under axial compression are presented and compared with that of Nano tubes. Also, Studies are carried out to know the effect of Diameter (D), Change in steel tube length (l), and Strength of infill ( $f_{CK}$ ) and to determine the ultimate load ( $P_{ult}$ ) & deflection ( $\Delta_{axial}$ ) in HNC's composite steel hollow tubes under monotonic loading and SEM (scanning electron microscope) image are taken during mixing, before testing and after testing and Fracture Analysis will be carried out for the buckled steel tubes using Radiographic Testing.

**Keywords:** Halloysite Nano Clay<sup>1</sup>, SEM<sup>2</sup>, Fracture Analysis<sup>3</sup>.

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## 1. INTRODUCTION

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 Clay (HNC's).

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.

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. HNCs are chemically similar to kaolinite and they used in the manufacture of high quality ceramic white-ware. HNTs have high mechanical strength and modulus and these features make it an ideal material preparing for different composites.

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

### 1.1 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

Experiment were carried out on four different specimens with four variations in each specimen, which varies in length 50mm, 75mm, 100mm, 125mm, 150mm and 200mm with constant diameter 25mm. And mixed with Cement, Sand & HNC's (0.5%, 1%, 1.5% and 2%).

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

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

Sample3- 1:2 (Cement: HNC)

Sample4- 2:3 (HNC: Sand)

Sample5- 1:1.5 (Cement: HNC)

Sample6- 1.5:3 (HNC: Sand)

Sample7- Cement

Sample8- HNC's

Sample9- Sand

Sample10- After compression testing

### 2.1 Halloysite Nanotubes



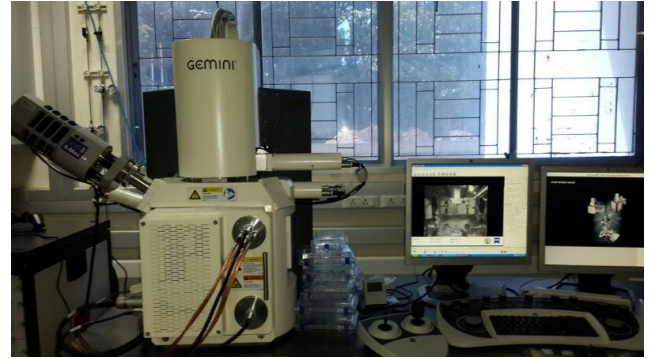
Source: Sigma Aldrich (manufacturer)-New Zealand [1]

Physical and chemical properties (provide by the supplier)

- ✓ Synonyms: Kaolin clay
- ✓ Appearance Form: powder
- ✓ Colour: White to Tan
- ✓ Relative density 2, 53 g/cm<sup>3</sup>
- ✓ Formula: H<sub>4</sub>Al<sub>2</sub>O<sub>9</sub>Si<sub>2</sub> · 2 H<sub>2</sub>O
- ✓ Molecular Weight: 294, 19 g/mol
- ✓ P<sup>H</sup> 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)



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.



Sample preparation before SEM testing [2]



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.

### 2.2.2 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 SCADA software, strain gauge SI -30 & strain indicator. To conduct the compression tests on all the specimens.



Concrete filled steel tube subjected to monotonic loading [4]

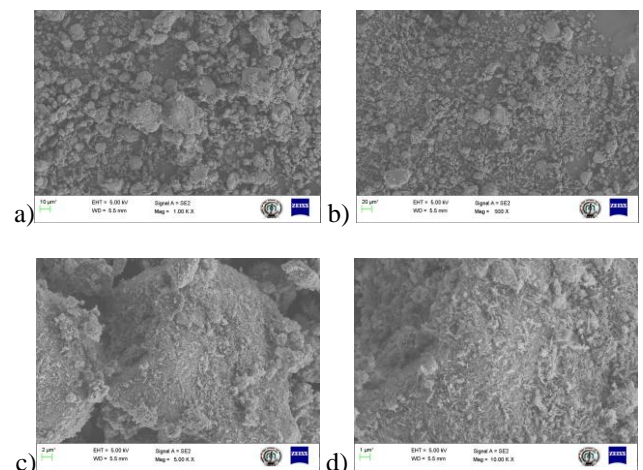


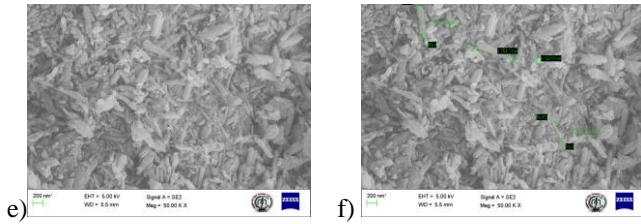
Composite steel tube under testing [4]

## 3. RESULTS & DISCUSSIONS

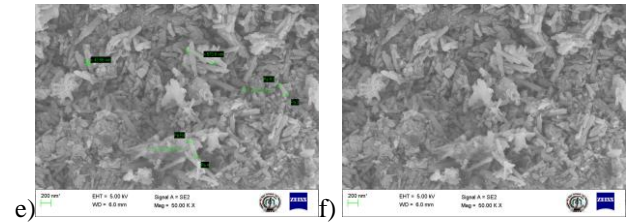
### 3.1 Scanning electron microscopy (SEM)

#### Sample1- 1:2:3(Cement: HNC: Sand)





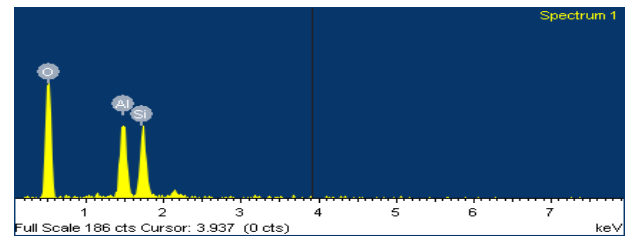
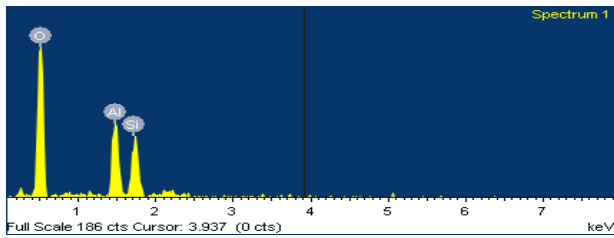
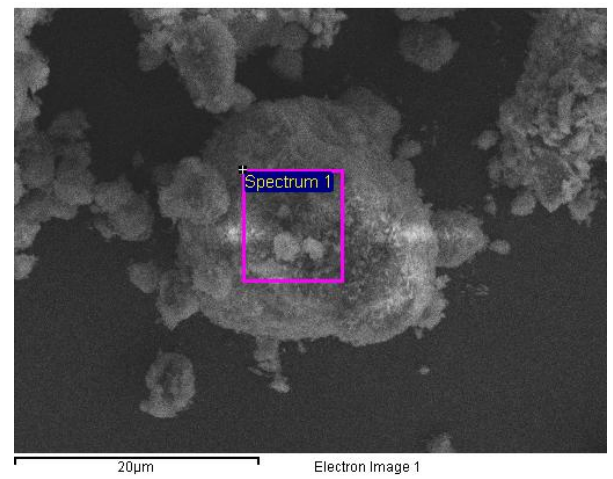
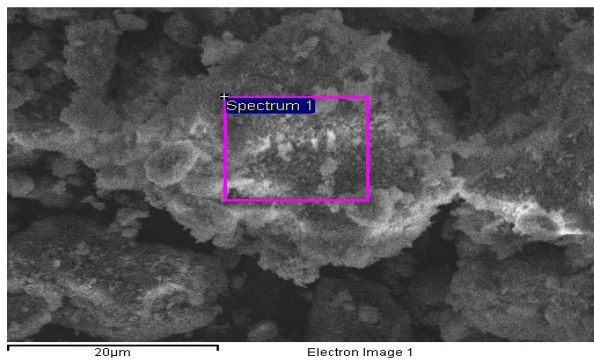
**Fig1.** SEM Images of fracture surface morphology from low to high magnification (a-f)



**Fig2.** SEM Images of fracture surface morphology from low to high magnification (a-f)

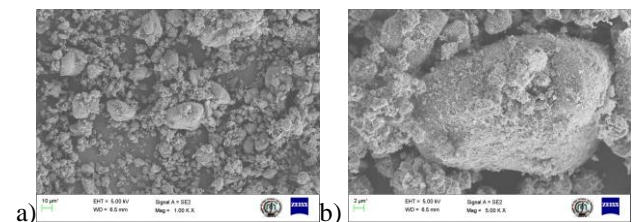
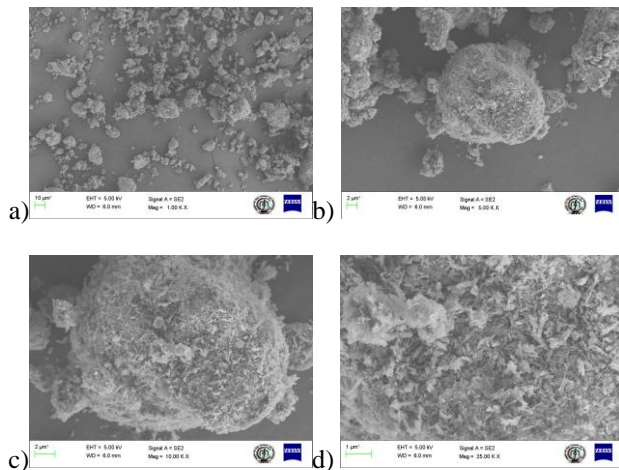
**Sample1- Energy-dispersive X-ray spectroscopy (EDX) Analysis**

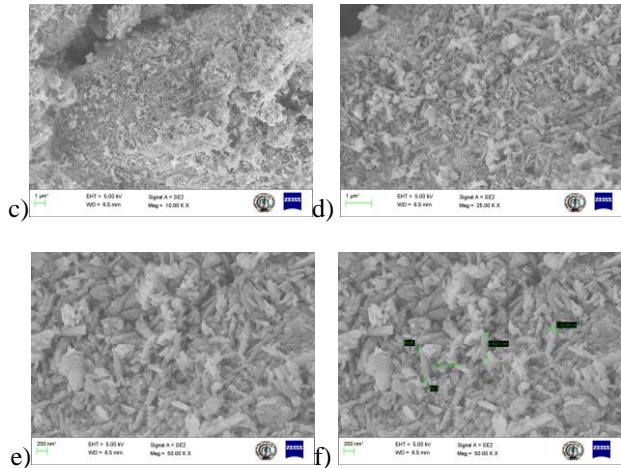
**Sample2- Energy-dispersive X-ray spectroscopy (EDX) Analysis**



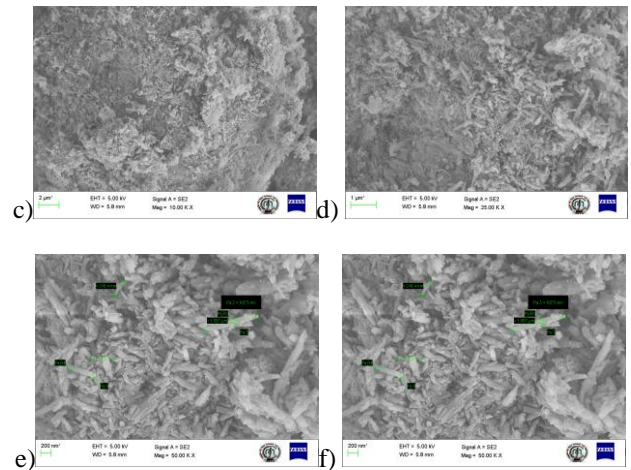
**Sample2- 1:1.5:3 (Cement: HNC: Sand)**

**Sample3- 1:2 (Cement: HNC)**





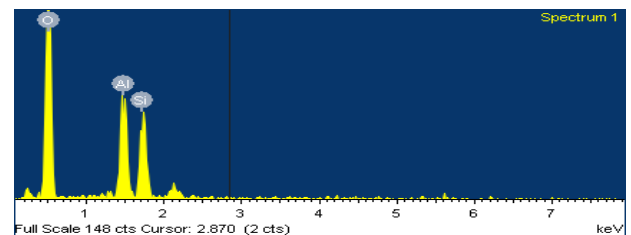
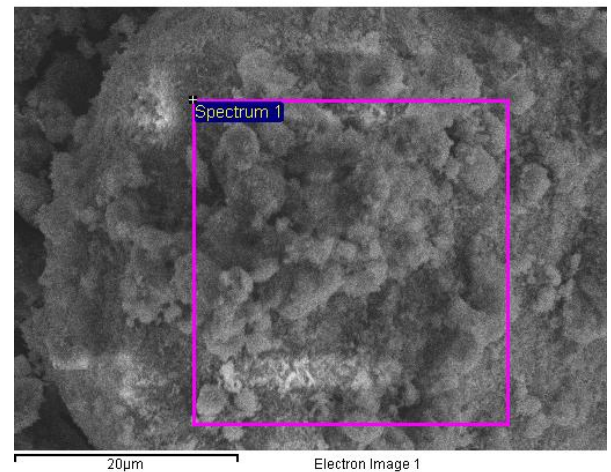
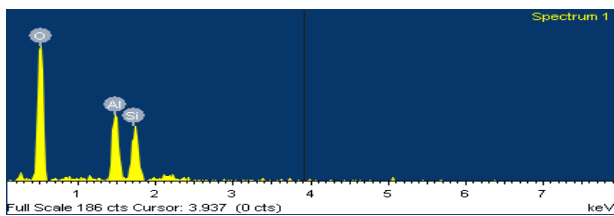
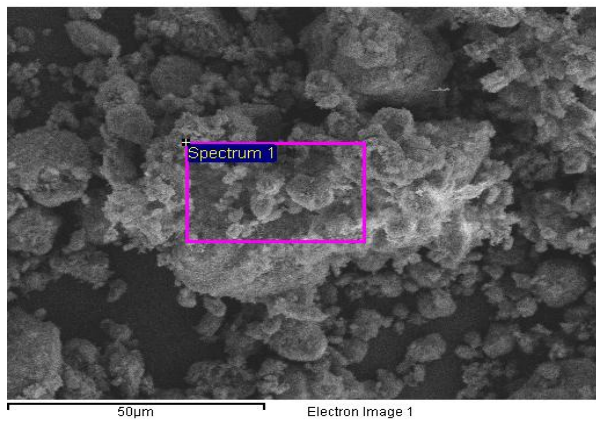
**Fig3.** SEM Images of fracture surface morphology from low to high magnification (a-f)



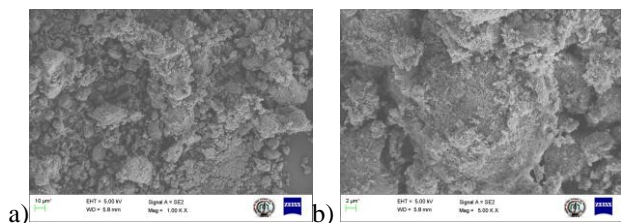
**Fig4.** SEM Images of fracture surface morphology from low to high magnification (a-f)

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

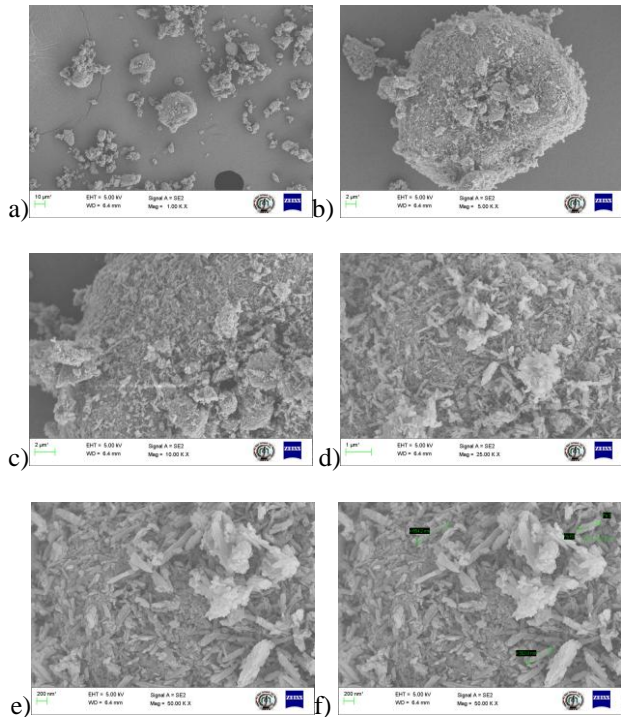
**Sample4- Energy-dispersive X-ray spectroscopy (EDX) Analysis**



**Sample4- 2:3 (HNC: Sand)**

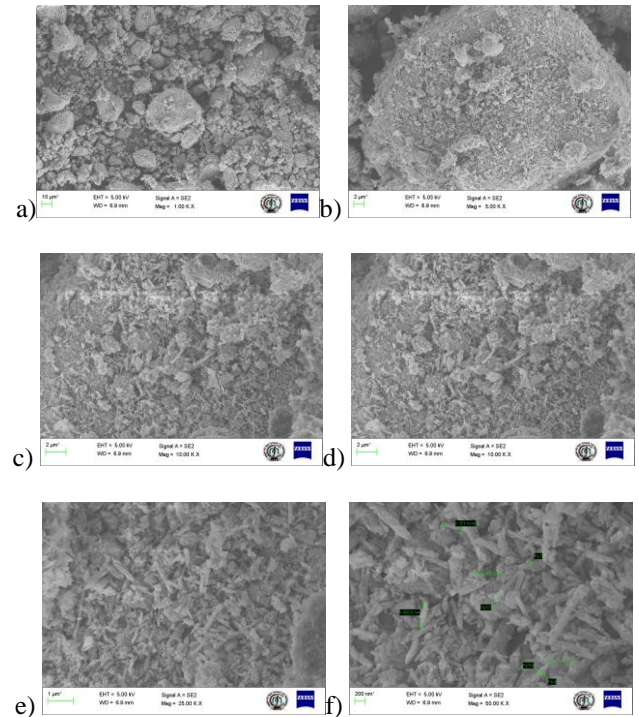


**Sample5- 1:1.5 (Cement: HNC)**



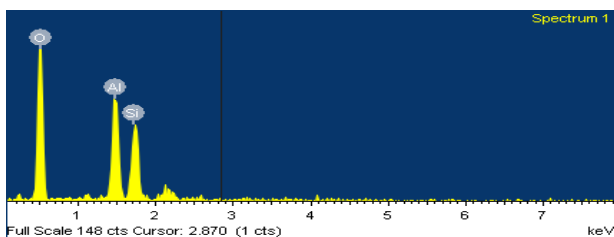
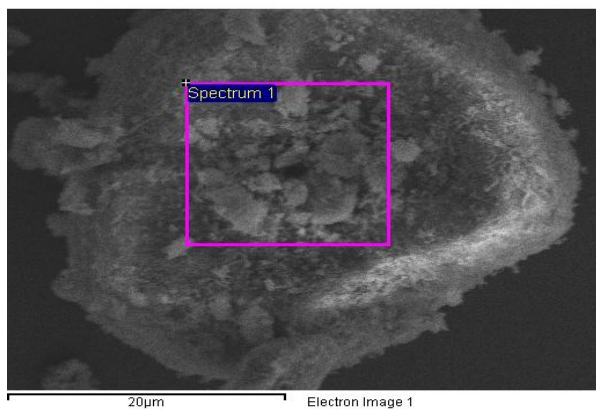
**Fig5.** SEM Images of fracture surface morphology from low to high magnification (a-f)

**Sample6- 1.5:3 (HNC: Sand)**

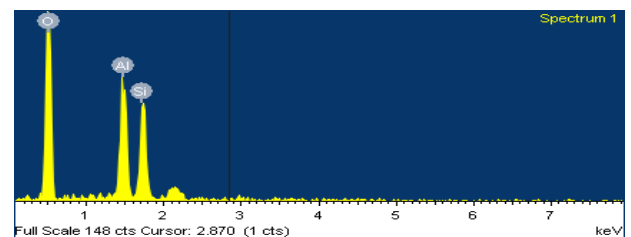
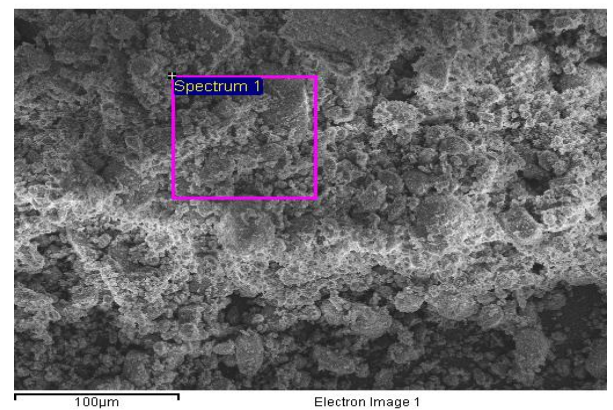


**Fig6.** SEM Images of fracture surface morphology from low to high magnification (a-f)

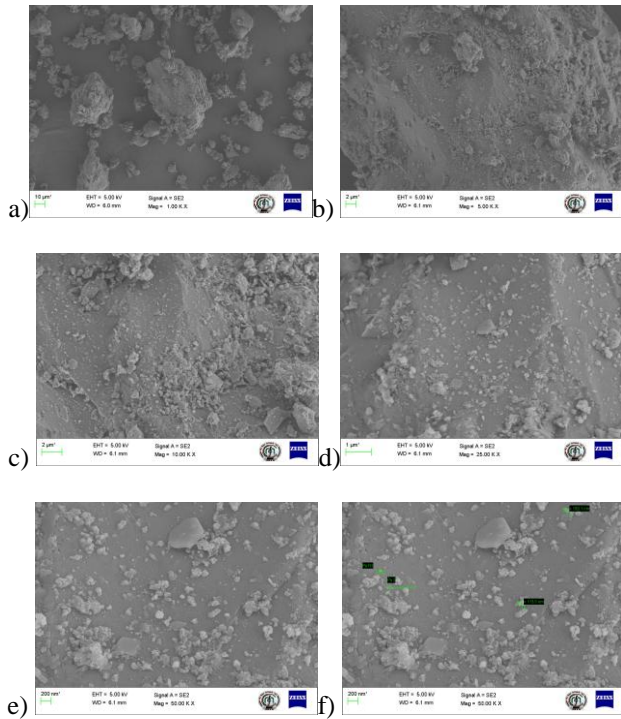
**Sample5- Energy-dispersive X-ray spectroscopy (EDX) Analysis**



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

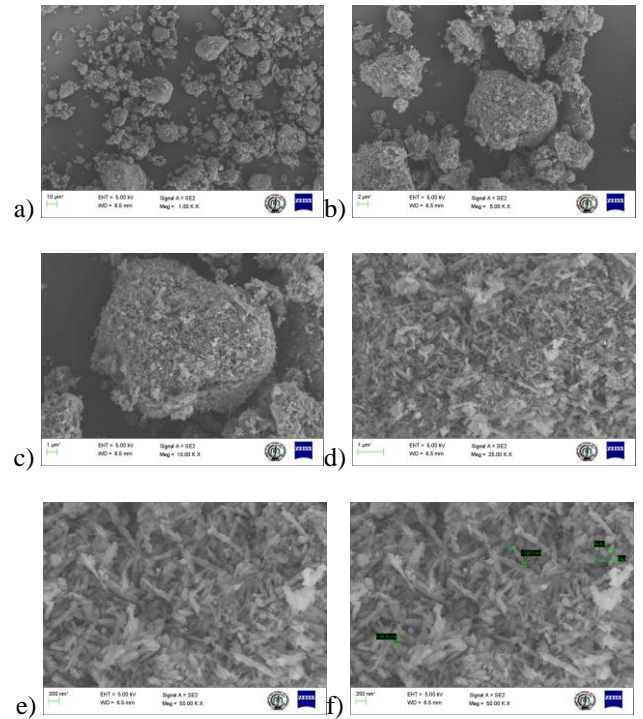


**Sample7- Cement**



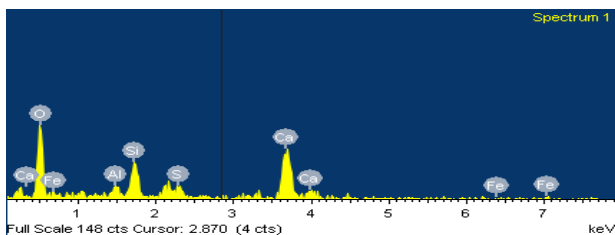
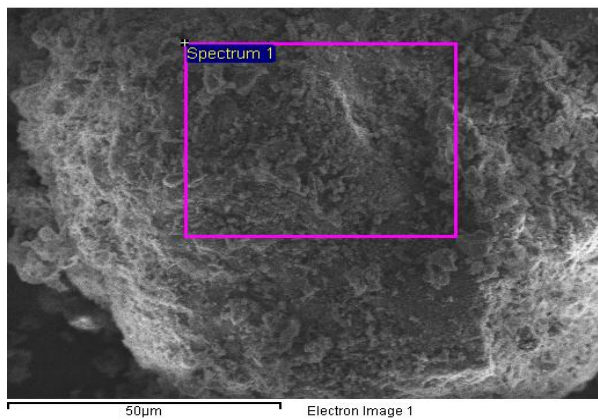
**Fig7.** SEM Images of fracture surface morphology from low to high magnification (a-f)

**Sample8- HNC's**

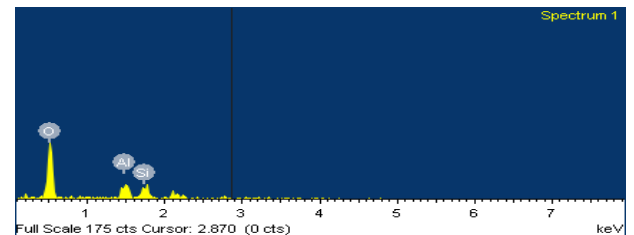
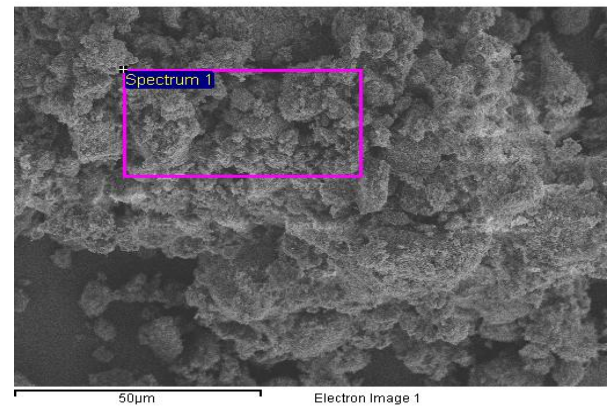


**Fig8.** SEM Images of fracture surface morphology from low to high magnification (a-f)

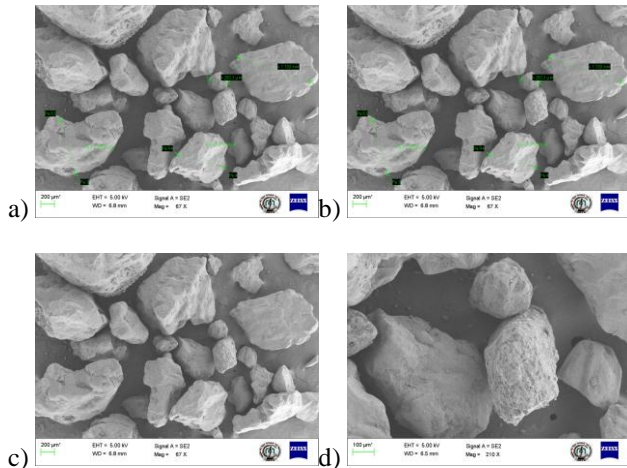
**Sample7- Energy-dispersive X-ray spectroscopy (EDX) Analysis**



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

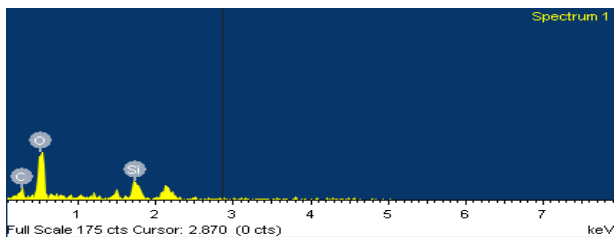
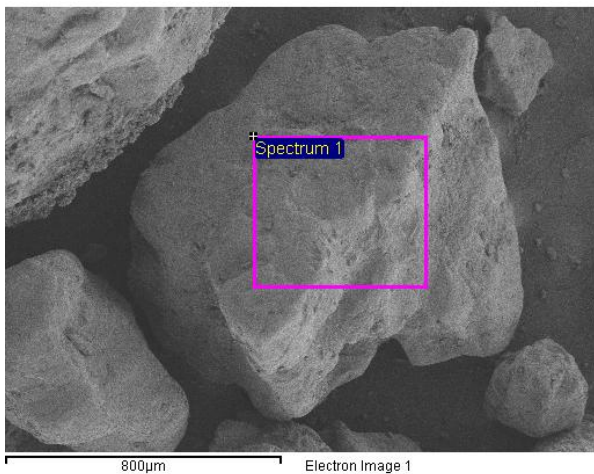


**Sample9- Sand**

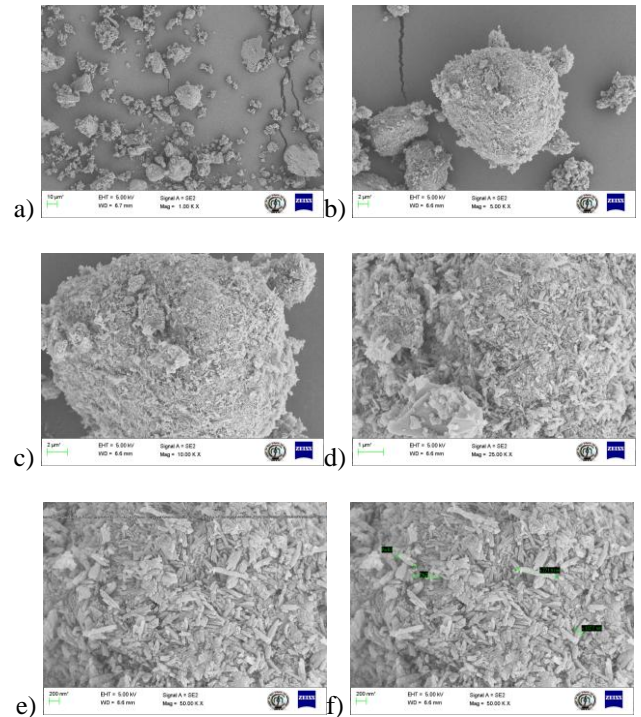


**Fig9.** SEM Images of fracture surface morphology from low to high magnification (a-f)

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

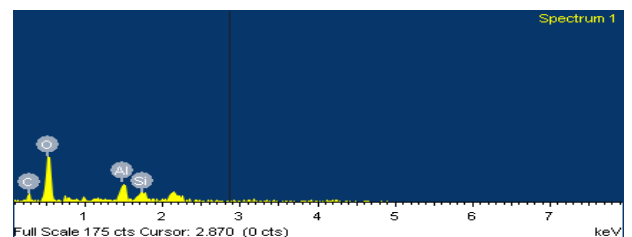
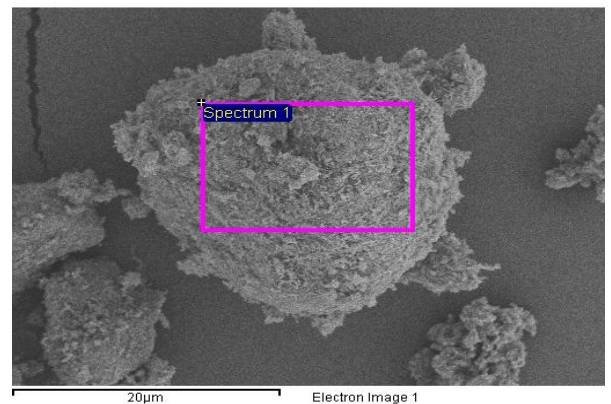


**Sample10- After compression testing**



**Fig10.** 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 11 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.

### 3.3 Experimental Results

Length 300mm, diameter 22mm, thickness 5mm & M20 (1:2:3) concrete.

Ultimate load ( $P_{ult}$ ) in KN		
Without HNC's	With HNC's	HNC's in %
84	85.68	0.5
86.10	88.25	1.0
86.52	89.11	1.5
87.36	82.32	2.0

Fig 12- Result Table

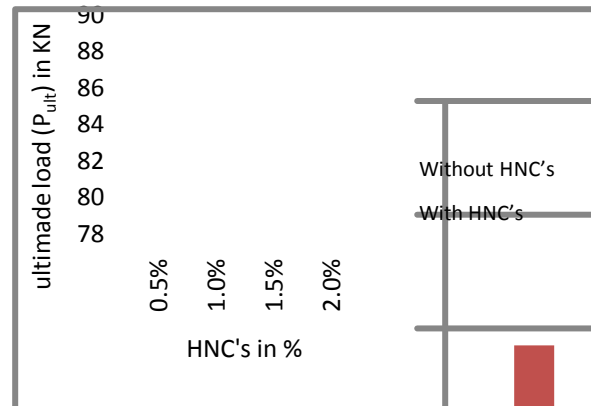


Fig 13- Ultimate load ( $P_{ult}$ ) vs. HNC's graph

### 4. CONCLUSIONS

Physical and chemical properties of the HNC'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 HNC's. When mixed with only cement, only sand & all the three. SEM & EDX analysis are show's that HNC's can lead to homogenous mixture which in turns enhances load caring capacity of composite steel column.

As shown in the fig 2 as percent of HNC's increase load also increase observed and ultimate load ( $P_{ult}$ ) is increased but reached optimum and started deceasing between 1.5 to 2.0%.

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## BIOGRAPHIES



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