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Effect of Halloysite Nano-clay on mechanical properties and workability of cement concrete

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Abstract: This paper investigates the effects of Halloysite Nano-clay on the compression and splits tensile strength of concrete. The cubes of size 10cm*10cm and cylinders specimens of size 10cm diameter and 20cm length are cast to test for compression and split tensile strength respectively and compared with the control specimens. The HNCs added to the specimens as a replacement for cement in the dosage of 0.5%, 0.75%, and 1wt% to cement. The slump cone test and compaction factor tests were carried out to check the effect of HNCs on the workability of concrete. The increment in the cement substitution by HNC caused decreasing the workability of concrete. It showed a 28.5% reduction of workability with 1% replacement as compared to plain concrete in the slump cone test. The optimum dosage of HNC for the compression strength is 0.75% and 0.5% for the splitting tensile strength.

Keywords: Halloysite Nano-clay, Workability, Compression strength, Split tensile strength

1. INTRODUCTION

Cementitious composites have great compression strength, but relatively low tensile strength, toughness, and as well as ductility. Therefore, additional reinforcing agents are used. Hence, in such situations, CNTs have been regarded as ideal reinforcing agents because of their great strength and binding properties which increase the compression strength as well as the flexural strength to a foremost level. However, they are considered jeopardous for humans as well as the environment because of their toxicology potencies. Furthering research in the Nano-technology field, another material has emerged as a safe option. Halloysites are naturally occurring eco-friendly nanotubes with low cost and are harmless to humans (Anjum & Kumar, n.d.). Nano Clay is unique and versatile and composed of aluminum, silicon, hydrogen, and oxygen and is mined from natural deposits in countries like China, New Zealand, America, Brazil, and France (Khan & Kumar, n.d.). Halloysite Nano-clay is an Aluminosilicate clay [Al2Si2O5(OH)4.H2O] with Nano tubular and hollow microstructure (Anjum & Kumar, n.d.). Chemically, the exterior surface of the halloysite nanotubes has properties similar to silica while the inner portion cylinder core is related to alumina which together may improve the cement matrix. The chemical composition and physical properties of halloysite Nano-clay may effectively enhance cementitious composites' performance. During hydration, kaolin clays react with cement resulting in property change. They are active as pozzolan during hydration due to dissociation, yielding ions leading to the formation of cementitious compounds(Farzadnia et al., 2013).

Advantages of using HNC

- Nano-clay particles act as an ultra-fine material, that promotes the filling action, rendering a denser microstructure.
- They are available in many implementable forms such as powders, creams, gels, etc
- Pozzolanic reaction of Alumino-Silicate, as an amorphous material, with lime elements of calcium oxide and hydroxide produces (C-S-H) gel enhanced bond strength, solid volume, and additional binding.
- They have a superior loading rate compared to other materials and also possess fast adsorption.
- Because of its remarkable strength and binding capabilities, which significantly increase the compressive and flexural strength.

OBJECTIVES OF STUDY

1. To add Halloysite Nano-clay as a partial replacement (0.5%, 0.75%, and 1 wt%) to cement.
2. To check the workability of the specimens with the addition of HNCs.

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3. Testing compression and split tensile strength into cubes and cylinders respectively.

2. LITERATURE REVIEW

1]. Ghazala Anjum, Dr. N. S. Kumar; "Experimental investigation on the influence of carbon nanotubes & halloysite nano-clay on strength of cement mortar": The experimental investigation carried out was to study the effect of Carbon nano-tubes and Halloysite Nano-clay on compression as well as the split tensile strength of cement mortar in comparison with the plain cement paste also known as the control specimen. The experiment was carried out with specimens of constant diameter and constant length of 20mm and 40mm respectively for both the tests. 53 grade of ordinary Portland cement and sand passing through IS sieve of 2.3mm was used the water-cement ratio fixed at 0.45. Carbon Nanotubes and Halloysite Nano-clay were added to the samples in dosages of 0.5, 0.75, and 1wt% of the cement. Tests were conducted after 7, and 14 & 28days of curing for both tests. Multi-Test 25-I nano Universal Testing machine was used for specimen testing and EMPEROR^TM software from MECMESIN was used to provide comments to the testing machine for test conduction. In the software, the load v/s deflection graph was plotted as the test was being conducted, and also calculations of maximum load & maximum displacement and also the average value along with sum, etc done in the software itself. After 28 days of compression testing, samples containing Multi-walled Carbon nano-tubes showed 18.2% greater strength, Nano-clay showed 14% greater strength a Single-walled Carbon nano-tube showed a 16.8% reduction in strength than the nominal mix and the optimum dosage found to be 0.5wt% for both Caron nano-tubes and 1wt% of HNC for compression strength whereas 0.75wt% for both the Nano-tubes and Nano-clay for split tensile strength.

[2]. Vindhya C.R, N. S. Kumar; "Experimental investigation on Halloysite Nanotubes & clay in infilled composite steel tube": An experimental study on Halloysite Nano-tubes and Nano-clay as an infill to the composite steel tube. The parameters considered were the diameter and length of the steel tube and volume fractions of Halloysite Nano-clay (0.5, 1, 1.5 & 2%) to concrete. HNC's concrete-filled CFST columns are tested for axial compression and compared with the samples containing nano-tubes. In addition to that, studies are carried out to know the effect of diameter, change in steel tube length, and the strength of infill to determine the ultimate load and deflection in HNC's CFST tubes under monotonic loading. Results showed that as the percentage of Halloysite Nano-clay increased, ultimate load increase was observed and reached optimum (1.5%) then started decreasing between 1.5% to 2%.

[3]. Nima Farzadnia, Abang Abdullah Abang Ali, Ramazan Demirboga, Mohammed Parvez Anwar; "Effect of halloysite nano-clay on mechanical properties, thermal behavior and microstructure of cement mortars": Another experimental study on Halloysite Nano-clay. This study aims at the mechanical properties, thermal behavior, flowability, and durability of mortar samples containing 1, 2 & 3% of HNC, and 2% Superplasticizer were constant throughout the research study. DSC was used to study the thermal behavior as well as XRD and SEM were used to study the microstructure and chemical composition respectively. The flow table test was done according to ASTM C 1437 and a 0.45 water to binder ratio was considered. Mortar mixing was done in compliance with ASTM C 305 with a 1:2.75 ratio of Cement to sand. For Compression strength, 5*5*5cm cubes were cast whereas 2*5cm cylinders were cast for the permeability test. The experiment was done for four groups of samples containing 0, 1%, 2% & 3% HNC. The result showed that compression strength improved up to 24% with the samples containing 3% HNC and Gas permeability improved up to 26% for 2%Nano-clay.

[4]. Heba Ahmed Gamal, M.S.El-Feky, Mohamed Kohail, El-Sayed A.R.Nasr; " Coupled effect of Nano-clay and carbon nanotubes on the mechanical properties of concrete": This study aims to investigate the coupled effect of Nano-clay and Carbon nano-tubes on the mechanical properties of concrete. The materials used in this study are ordinary Portland cement, Natural available clean sand with the size of particles lesser than 0.5nm; specific gravity of 2.58gram per cubic meter and fineness modulus of 2.25 and 12mm size coarse aggregate of specific gravity of 2.96 gram per cubic meter was used and the used Nano-clay is an off-white powder with particles size less than 150nm which is calcined at 850 degree Celsius for three hours and Carbon Nano-tubes are mainly graphene sheets folded in a cylindrical shape having length 10 to 100mm with inside diameter from 1.5 to 15 nano-meter and outside diameter from 1.5 to 50 nano-meter and also naphthalene sulfonate based superplasticizer was used for this study. Carbon Nano-tubes don't have any chemical affinity with the cement hydration product which is called hydrophobicity. The hydrophobicity, higher specific surface area, and lack of solubility of Carbon nano-tubes pose problems in dispersion and adhesion to the cement matrix so, to solve this problem, Nano-clay was used to improve the interaction between the Carbon Nano-tubes and the surrounding matrix. Cement was partially substituted by Nano-clay of 2.5, 5, and 7.5% and Carbon Nano-tubes of 0.01, 0.02, and 0.04% of cement. The compaction and curing of the samples were executed following ASTM C31 and a workability test was carried out to determine the consistency of concrete according to ASTM C 143. A total of six cubes were cast with the specimen size of 10cm for the compression test after 7 and 28 days of curing and 3 cylinders for the split tensile test with the dimension of 10cm diameter and 20cm height. The result showed the decreasing workability of concrete as the addition of nano-clay increases. The optimum dosage of Nano-clay was found to be 5% replacement with a 28.4%

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increase in compression strength and a hybrid mix of 5% Nano-clay and 0.01% Carbon Nano-tubes with the gain of 12.77% compression strength compared to the control specimen and the hybrid mix enhanced the tensile strength by 2.38% to 40.4% after 28 days as compared to 5% Nano-clay mix.

[5]. Sara Allalou, Rabia Kheribet, Abdelbaki Benmounah; "Effects of calcined halloysite nano-clay on the mechanical properties and microstructure of low-clinker cement mortar": The study on Calcined Halloysite Nano-clay to study the effect on the mechanical properties and microstructure of low-clinker cement mortar. The Nano-clay used in this study is a Halloysite Nano-clay with the formula of $H_4Al_2O_9Si_2.2H_2O$ and a molecular weight of 294.19gm/mol. The thermal treatment of HNC was performed by calcination for 2 hours at temperatures ranging from 650 to 800degree Celsius. The High-Volume Slag cement was (HVS) prepared by the OPC clinker blended with 70% of GBFS. A total of seven different samples were prepared. The first sample was prepared from 30% OPC with 70% GBFS to produce HVSC as a control specimen and the remaining six samples were prepared by the partial replacement CHNC ranging from 1 to 6%. Standard mortars were prepared with one part of cementitious materials with three parts of sand by mass with a water binder ratio of 0.5. A flexural test was carried out on prismatic specimens of size 40*40*160m at 2, 7, and 28 days of curing using central point loading under a controlled rate of 50+- 10N/s according to EN 2-1-015-1984. And compression test was carried out on the prism halves after the flexural strength test under a load control rate of 2400+- 200N/s according to FN 196-1 (2013). The water for standard consistency of mortar increased with increasing the addition of CHNC contents. The slag cement containing 5 % of CHNC possessed the highest improvement of the mechanical properties and microstructure of hardened cement pastes and mortars. It was suggested that the higher pozzolanic activity of CHNC particles and the nucleation of calcium hydro silicate (C–S–H) caused enhanced strength development.

3. EXPERIMENTAL WORK

3.1 MATERIAL PROPERTIES

Halloysite Nano-clay: Halloysite Nano-clay (HNC) is an Aluminosilicate clay. $Al_2Si_2O_5(OH)_4.H_2O$) is the chemical formula of HNC with a Nano tubular and hollow microstructure and this Nano-material is composed of Aluminum, Silicon, Hydrogen, and Oxygen. Halloysite nano-clay is a two-layered aluminosilicate with a predominantly hollow nanotubular structure. Chemically, the outer surface of the halloysite nanotubes had properties similar to SiO_2 while the inner cylinder core was related to Al_2O_3.

Fig 1: Halloysite Nano-clay

Properties (provided by the supplier)

- Kaolin clay
- Powder form
- Colour: White
- Formula: $Al_2Si_2O_5(OH) \cdot 2\ H_2O$
- Actual particle size (APS): <100nm

Concrete: The ordinary Portland cement of Grade 53 is used in this study. The designed strength for 28 days is a minimum of 53MPa. It sets quicker than OPC 43 and has a quite low initial setting time. The used manufacturing sand (M-sand) has a specific gravity of 2.56 with particles size smaller than 2.63mm used as a fine aggregate. The size of the aggregate smaller than the 10mm aggregate with a specific gravity of 2.95 is used as a coarse aggregate.
The water-cement ratio of 0.48 is considered for all the samples.

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Fig 2: (a) OPC 53 grade cement (b) M-sand (c) Coarse aggregate

Table 1: Mix proportion

Mix	M30
Cement (Kg/m^3)	434
Fine aggregate (Kg/m^3)	841
Coarse aggregate (Kg/m^3)	952
Water (lit/m^3)	208
0.5% HNC (Kg/m^3)	2.17
0.75% HNC (Kg/m^3)	3.22
1% HNC (Kg/m^3)	4.34

3.2. SPECIMENS PREPARATION

According to Indian Standard: 516-1959 METHODS OF TESTS FOR STRENGTH OF CONCRETE, from clause 2.8, mentioned if the largest nominal size of the aggregate is less than 2cm, 10cm cubes may be used as an alternative and cylindrical test specimens shall have a length equal to twice the diameter but the diameter of the specimen shall not be less than 7.5cm. For the compression strength test, three 10cm size cubes(100mm*100mm) are considered which contain Halloysite Nano-clay in the dosage of 0.5%, 0.75%, and 1wt% of cement and compared with the control specimen (without HNC). Three cylinders of size 10cm in diameter and 20cm in length are considered for the split tensile strength test. The results will be compared with the control specimen.

3.3. WORKABILITY OF CONCRETE

Concrete slump cone test: The test measures the consistency of fresh concrete before it sets. It is performed to check the workability of freshly made concrete. The test is carried out using Indian Standard: 1199-1959: Methods of sampling and analysis of concrete. The test is carried out using a conical frustum known as a slump cone which is open at both ends and has attached handles and has an inside diameter of 10cm at the top and 20cm at the bottom with a 30cm height.
Compaction factor test: It is more precise and sensitive than the slump test and is particularly useful for concrete mixes of very low workability. The test is carried out using Indian Standard: 1199-1959: Methods of sampling and analysis of concrete. The test was carried out by adding HNCs in the percentage of 0.5, 0.75, and 1wt% to cement to check the effect of HNCs on the workability of concrete.

3.4. COMPRESSION AND SPLIT TENSILE STRENGTH TEST: Compressive strength test is carried out to find the capacity of concrete to withstand loads before failure. The test was carried out on 10cm*10cm size cubes following the IS 516 – 1959 (Methods of tests for strength of concrete). After the specimen is fitted into the CTM, the load of 0.4kN/sec was applied until the specimen failure then noted down the dial gauge values to find out the failure load by using the below formula. Compression strength = maximum load applied/cross-sectional area of the specimen

The tensile strength can be defined as the maximum stress that a material can bear before breaking when it is allowed to be stretched or pulled. A method of determining the tensile strength of concrete using a cylinder that splits across the vertical diameter. It is an indirect method of testing the tensile strength of concrete. The test was carried out on 100mm (Diameter) * 200mm (Length) cylindrical specimens as per IS 5816:1999 (Splitting tensile strength of concrete – Method of test). The procedure is similar to compression testing but the load is applied along a line on the surface of the specimen. Splitting tensile strength = 2F/3.14*D*L

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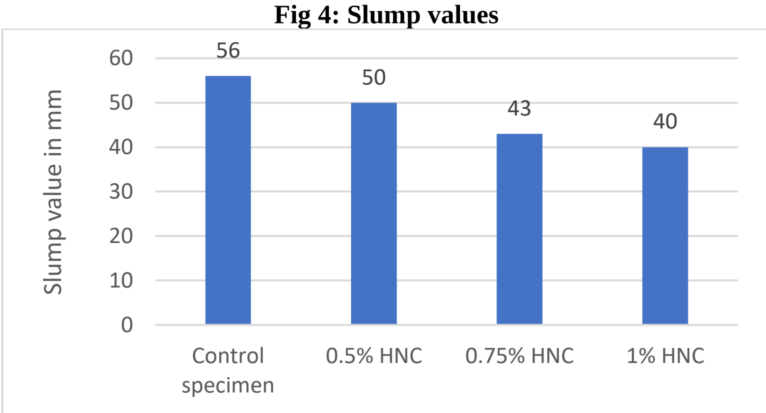
Fig 3: (a) Compression strength test **(b) Split tensile strength test**

4. RESULT AND DISCUSSIONS

4.1. WORKABILITY TEST

The tests are carried out using Indian Standard: 1199-1959: Methods of sampling and analysis of concrete.

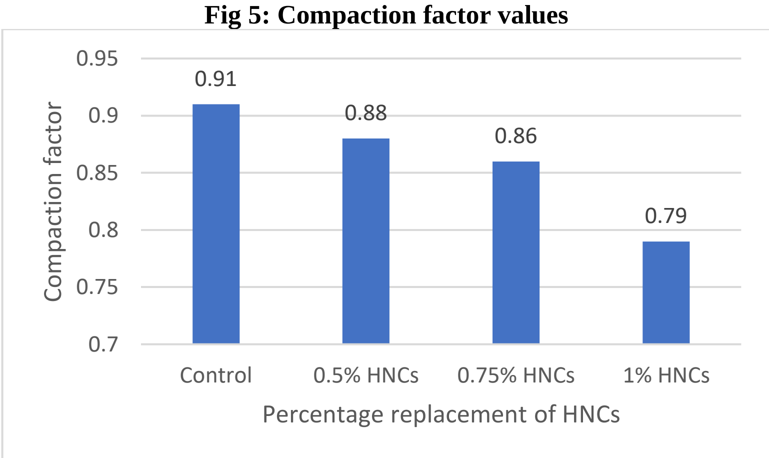
Fig 4: Slump values



For the control specimen: True Slump- 56 mm (Medium workability)

The influence of adding HNC on the consistency of concrete is shown in the below graph. Generally, the increment in the cement substitution by HNC caused decreasing the workability of concrete. The highest reading reached 40 mm with the cement replacement 1% HNC as compared to the plain concrete 56mm.

Fig 5: Compaction factor values



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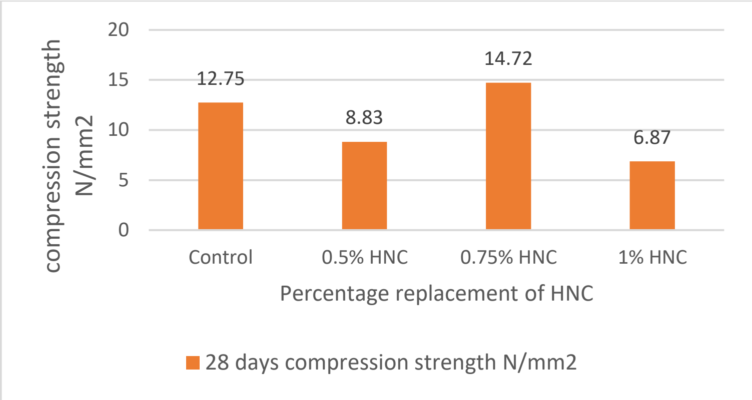
4.2. COMPRESSION STRENGTH TEST

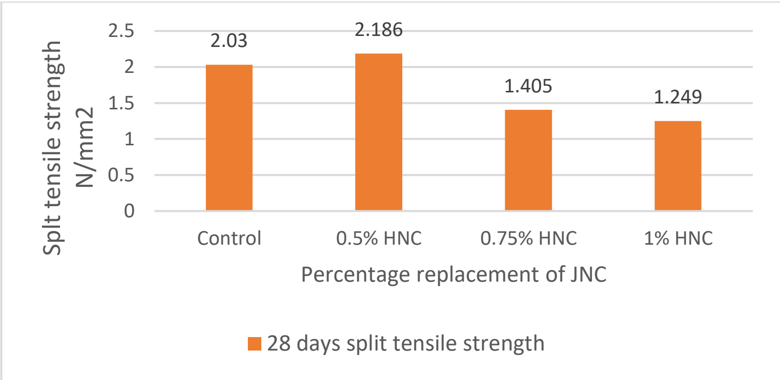
Fig 6: Compression strength after 28 days of curing



The influence of Halloysite Nano-clay (HNCs) on the compression strength is shown in (Fig). It is shown that the strength increases with increasing the percentage replacement of HNC reached a maximum and started decreasing between 0.75% and 1%. It is recorded that the peak of the compression strength after 28 days of curing with 0.75% HNC mix, the mix gained 13.38% more strength as compared to the control mix. Furthermore, the specimens with 1% HNC showed 46.15% less strength as compared to the control specimen.

4.3. SPLIT TENSILE STRENGTH TEST

Fig 7: Splitting tensile strength after 28 days of curing



The result of the tensile strength of the concrete with the influence of HNC is shown in (fig). Generally, the tensile strength increased with adding 0.5% HNCs and started decreasing as the percentage of HNCs increased. The result showed 7.14% more strength with the addition of 0.5% HNCs as compared to the control specimen. The specimens with 0.75% and 1% HNCs showed more than a 50% reduction in strength as compared to the specimens with 0.5% HNCs.

5. CONCLUSION

- Generally, the increment in the cement substitution by HNC caused decreasing the workability of concrete. The lowest reading reached 40 mm with the cement replacement 1% HNC. It showed a 28.5% reduction of workability with 1% replacement as compared to plain concrete (56mm).
- It is recorded that the peak of the compression strength after 28 days of curing with 0.75% HNC mix, the mix gained 13.38% more strength as compared to the control mix. Furthermore, It showed a 30.74% and 46.15% reduction in strength with the replacement of 0.5% and 1% HNC as compared to the control specimen respectively.
- The tensile strength increased with adding 0.5% HNCs and started decreasing as the percentage of HNCs increased. The result showed 7.14% more strength with the addition of 0.5% HNCs as compared to the control specimen. The specimens with 0.75% and 1% HNCs replacement showed 30.78% and 38.47% less strength as compared to the control specimen respectively.
- The optimum dosage of HNC for the compression strength is 0.75% and 0.5% for the splitting tensile strength.

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