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Experimental Investigation on Cement Mortar Blended with HNC and CNTs' [Hallyosite nanoclay and Carbon Nanotubes]-Taguchi's Approach

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ABSTRACT
This research focuses on the results of experimental investigation of the compressive strength of Cement Mortar cubes blended with Multi walled Carbon Nanotubes (MWCNTs) and Hallyosite Nanoclay (HNC's). The percentage of MWCNTs and HNC's was fixed to 0.5%, 0.75% and 1% by weight of cement. In this research, experiments were conducted on the 3 different grades of mortar(MM-3,MM-5,MM-7.5) and 3 different size of cube specimens (25x25x25, 50x50x50 and 75x75x75), out of which 50mm cube is the size as per IS 2250-1981 and remaining two (25 mm and 75mm) are trial mixes. Casted specimens were tested on 7th, 14th and 28th day of curing. Compressive strengths obtained from these experiments are analyzed through Taguchi's method of Optimization and ANOVA (Analysis of Variance) with the help of Commercial software MINITAB-16. Statistical Analysis is conducted to find the interaction of the factors and their effect on strength of mortar. Regression analyses are employed to obtain the optimal process parameters. Out of 0.5%, 0.75%and 1% of HNC added to the conventional cement mortar 0.75% resulted in Optimum Compressive Strength for 50mm size of cube specimen as per experiments and Regression Analysis. Also, it is observed that when MWCNTs are mixed, compressive strength increases as percentage increases.
Keywords: *Multi walled Carbon Nanotubes (MWCNTs), Hallyosite Nanoclay (HNC's), Compressive Strength, Tagauchi's approach, Regression Analysis.*

1 INTRODUCTION

CEMENT is a construction material commonly used due to its low cost and high compressive strength. An improvement in basic mechanical properties and durability of materials based on cement is of high important these days. Cement products are leading materials in the construction industry. Contemporary Civil engineering is permanently setting new conditions concerning the quality of engineering materials. Advanced technological aspects demand for higher performing cement-based materials with improved strength and toughness for use in civil engineering projects. Cementitious materials are typically characterized as quasibrittle materials, with low strength and low strain capacity, and hence affect long-term durability of structures.

Introduction of nanoparticles in cement based materials has gained popularity in recent years due to their excellent mechanical properties and application potential. Addition of carbon nanoparticles in the cementitious materials may provide extraordinary strength increase as well as controlling cracks. Remarkable improvements in the electrical and mechanical properties of cement composite materials, when carbon nanotubes (CNTs) and Hallyosite nano clay are used as fillers instead of other conventional materials, have been demonstrated by various research groups.

While the exploration for various applications of nanotechnology to develop innovative construction materials continues, it is already clear that the science of the very small is making big changes, with numerous economic benefits for the construction industry. Excellent mechanical, thermal, and electrical properties of carbon nanotubes (CNTs) and Hallyosite nano clay (HNC's) motivated the development of advanced nanocomposites with outstanding and multifunctional properties. After achieving a considerable success in utilizing these unique materials in various polymeric matrices, recently tremendous interest is also being noticed on developing CNT and Hallyosite nano clay (HNC's) blended cement-based composites.

However, the problems related to nano material dispersion also exist in case of cementitious composites, impairing successful transfer of nonmaterial's' properties into the composites. Performance of cementitious composites also depends on their microstructure which is again strongly influenced by the presence of nanomaterials.

CNTs can be classified as either single-walled CNTs (SWCNTs) or multi-walled CNTs (MWCNTs). The wall of a SWCNT is a seamlessly wrapped sheet of carbon atoms in a periodic hexagonal arrangement, with thickness equaling to a single atom and diameters ranging from 0.4 to 3 nm, while MWCNTs appear as multiple concentric SWCNTs, with diameters ranging from 1.4 to 100 nm. The length of these CNTs can be up to centimeters, which gives an aspect ratio exceeding 107; although the aspect ratio is typically 1000[1].

Halloysites are types of naturally occurring multiwall aluminosilicates with 1:1 sheet arrangement. The Halloysite layer structure is composed of octahedral coordinated Al3+ and tetrahedral coordinated Si4+ in a 1:1 arrangement with water molecules between the layers. Halloysites were firstly discovered by Berthier as a clay mineral of the kaolin group in 1826, and were named "Halloysite" after Omalius d'Halloy who analyzed the mineral first time. These nanoclays are found worldwide and their deposits have been reported in countries such as Australia, China, Belgium, Brazil, France, Spain, New Zealand, Mexico, America and others. Halloysites' aluminosilicate sheets are rolled into tubes and nanosized. Tubular Halloysite, also called Halloysite nanotube (HNT) is morphologically similar to multiwall carbon nanotubes.

In this experiments, compressive strength of 3 different grades of cement mortar blended with MWCNT's and HNC were studied. The factors related to compressive strength were determined by using Taguchi's experimental design method. Orthogonal arrays of Taguchi, the signal-to-noise (S/N) ratio, the analysis of variance (ANOVA), and regression analyses are employed to find the optimal levels and to analyze the effect of the %age of HNC and MWCNT's on compressive strength values.

1.1 Halloystite Nano tubes

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

Table 1: Typical properties of Halloystite Nanotubes[3]

Property	Value
Length	0.2–2 µm
Outer diameter	40–100 nm
Inner diameter	10–40 nm
Aspect ratio (L/D)	10–50
Elastic modulus (theoretical value)	140 GPa (230–340 GPa)
Mean particle size in aqueous solution	143 nm
Particle size range in aqueous solution	50–400 nm
BET surface area	22.1–81.6 m2/g
Pore space	14–46.8 %
Lumen space	11–39 %
Density	2.14–2.59 g/cm3
Average pore size	79.7–100.2 Å
Structural water release temperature	400–600 °C

1.2 Multi walled Carbon Nanotubes

Fig. 2. Source: Sigma Aldrich (manufacturer)-New Zealand [1]

Table 2: Typical properties of MWCNT

Name of the property	Characteristics
Length to diameter Ratio	60 (SWCNT's) ~300 m2/gm (MWNCT's)
Surface area	(2.60g/cm3 for MWNCT's)
Thermal conductivity	100% (CNT based interconnects which are stretchable)
Elongation	1.25 TPa (SWCNT's), 0.9 TPa (MWCNT's)
Young's Modulus	0.06-0.55 (SWCNT's)
Poission's Ratio	75 GPa (SWCNT's)
Tensile strength	<60 GPa (MWCNT's)
Compressive strength	100-150 GPa (MWCNT's)

2. Design of Experiment

Essentially, traditional experimental design procedures are too complicated and not easy to use. A large number of experimental works have to be carried out when the number of process parameters increases. To solve this problem, the Taguchi method uses a special design of orthogonal arrays to study the entire parameter space with only a small number of experiments. Taguchi methods have been widely utilized in engineering analysis and consist of a plan of experiments with the objective of acquiring data in a controlled way, in order to obtain information about the behavior of a given process.

Taguchi method is employed to design the experiment for compressive strength study in order to obtain optimum results with minimum number of experiments. The experimental design proposed by Genichi Taguchi involves the use of orthogonal arrays to organize the factors affecting the process. and also the levels these factors need to be varied logically in order to complete the experiment with bare minimum trials in order to save the time, money and resources instead of performing all possible combinations.

2.1 Plan of Experiments

An orthogonal array was constructed up on identifying the control factors, their levels and responses as shown in Table 1. This experiment has 3 variables at 3 different levels. A full factorial experiment would require (3^3) = 27 experiments. We conducted a Taguchi experiment with a L9 (3^3) orthogonal array (9 tests, 3 variables, 3 levels).The orthogonal array for the present work is as shown in Table 4.

Table 3: Controllable parameters with their levels

Factors	Levels		
	1	2	3
Mortar grade	3	5	7.5
%age of HNC	0.5	0.75	1
Cement grade	33	43	53

Table 4: L9 orthogonal array

Experiment No's	Column		
	1	2	3
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	2	2

3. Experimental Analysis

3.1 Equipments Used

Fig. 3: Universal Testing Machine [2]

Fig. 4: Multi Test 25-i Nano Universal Testing Machine (Funded by VGST, Bangalore) [2]

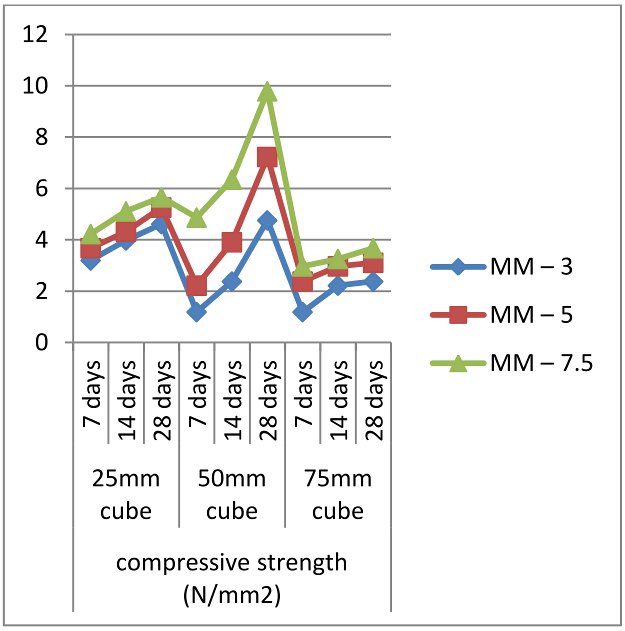
Fig. 5.1: Specimens under Curing

Fig. 5.2: Cracked Specimen

3.2 Compressive Strength of Conventional Cement Mortar Cubes

Cubes size in mm	Mortar grade	Compressive strength (N/mm^2)		
		7 days	14 days	28 days
25	MM – 3	3.2	3.98	4.62
	MM – 5	3.68	4.32	5.26
	MM – 7.5	4.23	5.11	5.66
50	MM – 3	1.19	2.38	4.76
	MM – 5	2.22	3.91	7.23
	MM – 7.5	4.87	6.35	9.79
75	MM – 3	1.19	2.22	2.38
	MM – 5	2.38	2.97	3.11
	MM – 7.5	2.97	3.26	3.68

Plots Obtained



3.3 Compressive Strength Of Conventional Cement Mortar Cubes With HNC

Experiement	Compressive Strength		
	7 days	14 days	28 days
1. CM-3 0.5% HNC 33CG	1.19	3.64	5.18
2.CM-3 0.75% HNC 43CG	1.89	3.23	6.92
3. CM-3 1% HNC 53CG	1.46	3.12	6.77
4.CM-5 0.5% HNC 43CG	2.32	3.89	8.18
5.CM- 0.75% HNC 53CG	2.98	4.11	9.76
6.CM-5 1% HNC 33CG	2.02	3.77	8.01
7.CM-7.5 0.5% HNC 53CG	3.45	5.17	11.12
8.CM-7.5 0.75% HNC 33CG	3.02	4.54	9.93
9.CM-7.5 1% HNC 43CG	2.87	4.06	9.17

3.4 Compressive Strength Of Conventional Cement Mortar Cubes With MWCNT

Experiement	Compressive Strength		
	7 days	14 days	28 days
1.CM-3,0.5% MWCNT 33CG	1.139	2.379	3.194
2.CM-3,0.75% MWCNT 43CG	2.224	4.987	6.77
3.CM-3,1% MWCNT 53CG	2.437	4.645	6.98
4.CM-5,0.5% MWCNT 43CG	3.654	4.913	9.81
5.CM-5,0.75% MWCNT 53CG	3.434	5.098	10.92
6.CM-5,1% MWCNT 33CG	4.097	5.787	11.21
7.CM-7.5,0.5% MWCNT 53CG	3.231	4.823	9.92
8.CM-7.5,0.75% MWCNT 33CG	4.823	5.991	11.32
9.CM-7.5,1% MWCNT 43CG	4.075	6.231	12.17

4. ANOVA (Analysis of Variance)

ANOVA can be useful for determining influence of any given input parameter from a series of experimental results by design of experiments for machining process and it can be used to interpret experimental data. Analysis of variance (ANOVA) is a collection of statistical models, and their associated procedures, in which the observed variance in a particular variable is partitioned into components attributable to different sources of variation. In its simplest form, ANOVA provides a statistical test of whether or not the means of several groups are all equal, and therefore generalizes *t*-test to more than two groups.

ANOVA is used in the analysis of comparative experiments, those in which only the difference in outcomes is of interest. The statistical significance of the experiment is determined by a ratio of two variances. This ratio is independent of several possible alterations to the experimental observations: Adding a constant to all observations does not alter significance. Multiplying all observations by a constant does not alter significance. So ANOVA statistical significance results are independent of constant bias and scaling errors as well as the units used in expressing observations. ANOVA is a statistically based, objective decision-making tool for detecting any differences in the average performance of groups of items tested. ANOVA helps in formally testing the significance of all main factors and their interactions by comparing the mean square against an estimate of the experimental errors at specific confidence levels. First, the total sum of squared deviations SST from the total mean S/N ratio nm can be calculated as

$$SST = \sum_{i=o}^{n} (ni - nm)$$

where n is the number of experiments in the orthogonal array and ηi is the mean S/N ratio for the ith experiment.

4.1 For HNC

Regression Equation

Compressive Strength = 1.06992 + 0.829617 Mortar Grade - 0.353333 %age of HNC + 0.0755 cement grade

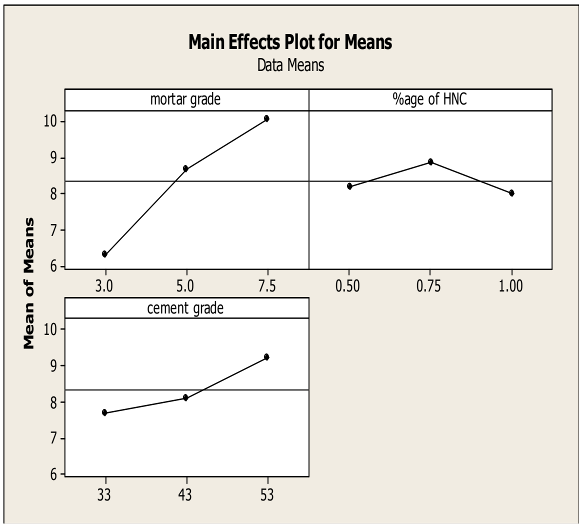
Table 4: Compressive strength values and S/N ratio values for experiments

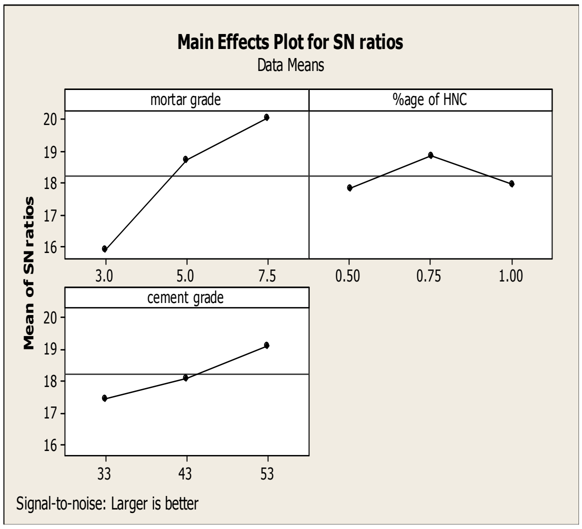
Mortar Grade	% age of HNC	Cement Grade	Compressive Strength	Signal to Noise ratio
3.0	0.50	33	5.18	14.2866
3.0	0.75	43	6.92	16.8021
3.0	1.00	53	6.77	16.6118
5.0	0.50	43	8.18	18.2551
5.0	0.75	53	9.76	19.7890
5.0	1.00	33	8.01	18.0727
7.5	0.50	53	11.12	20.9221
7.5	0.75	33	9.93	19.9390
7.5	1.00	43	9.17	19.2474

Table 5 S/N ratio values for Compressive strength by factor level

Level	Mortar Grade	% age of HNC	Cement Grade
1	6.290	8.160	7.707
2	8.650	8.870	8.090
3	10.073	7.983	9.217
Delta	3.783	0.887	1.510
Rank	1	3	2

Plots from minitab





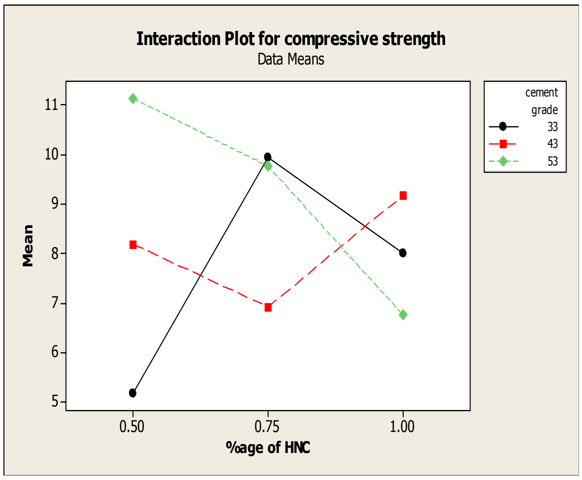


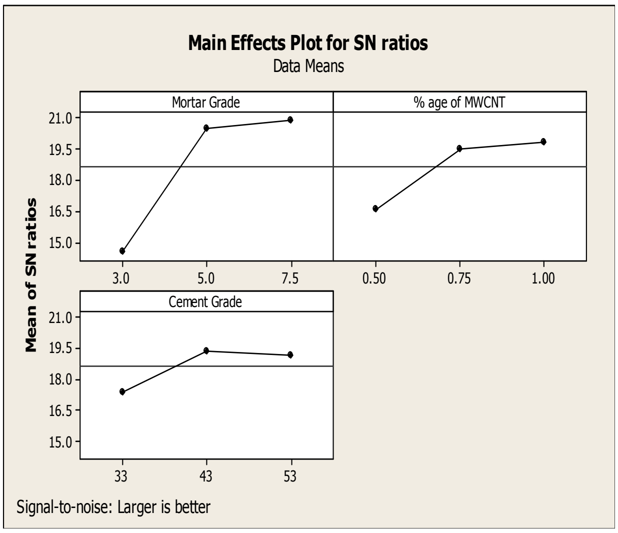
Table 6: Compressive strength values and S/N ratio values for experiments

Mortar grade	%age of MWCNT	Cement Grade	Compressive Strength	SNRA1
3	0.50	33	3.194	10.0867
3	0.75	43	6.770	16.6118
3	1.00	53	6.980	16.8771
5	0.50	43	9.810	19.8334
5	0.75	53	10.920	20.7645
5	1.00	33	11.210	20.9921
7.5	0.50	53	9.920	19.9302
7.5	0.75	33	11.320	21.0769
7.5	1.00	43	12.170	21.7058

Table 7: S/N ratio values for Compressive strength by factor level

Level	Mortar Grade	%age of MWCNT	Cement Grade
1	5.648	7.641	8.575
2	10.647	9.670	9.583
3	11.137	10.120	9.273
Delta	5.489	2.429	1.009
Rank	1	2	3

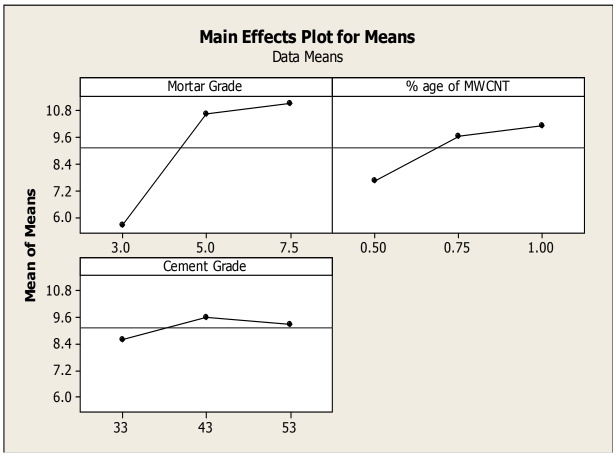
Plots from minitab

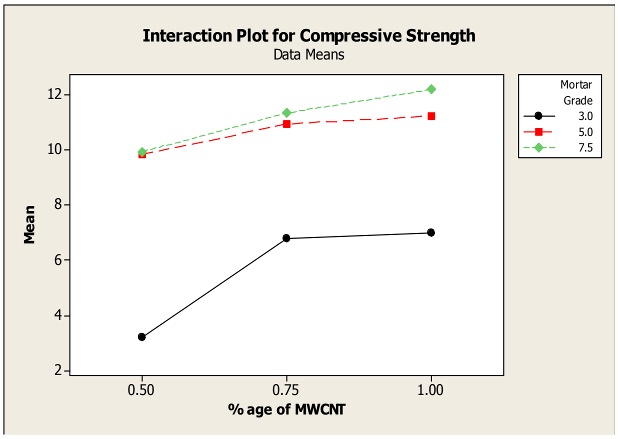


4.2 For MWCNT

Regression equation

Compressive Strength = - 2.16 + 1.18 Mortar Grade + 4.96 % age of MWCNT + 0.0349 Cement Grade





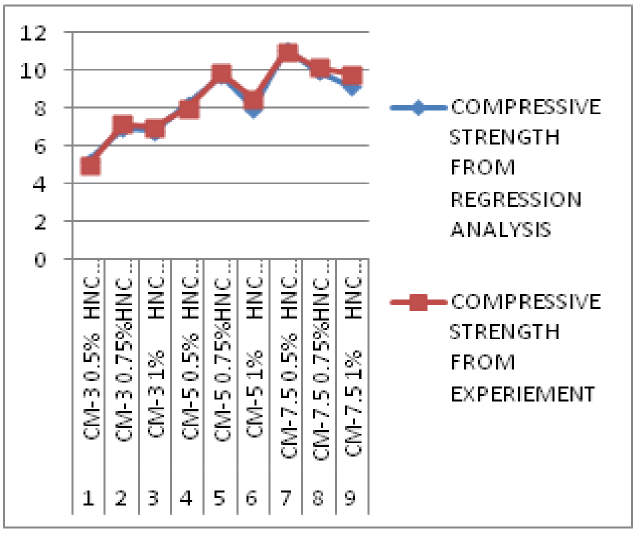
5. Results

5.1 Conventional Mortar

Sl. No.	Mortar Grade	Mix Ratio	σ_{code}	$\sigma_{expt.}$	% age Error
1	MM- 3	1: 6	3 To 5	4.76	a. 36.9 b. 15.96 c. -5.04
2	MM- 5	1: 5	5 To 7.5	7.23	a. 30.84 b. 17.01 c.-3.73
3	MM- 7.5	1: 4	>7.5	9.79	a. 18.28 b. 8.06 c.-2.14

Mortar Blended With Hallyosite Nano Clay

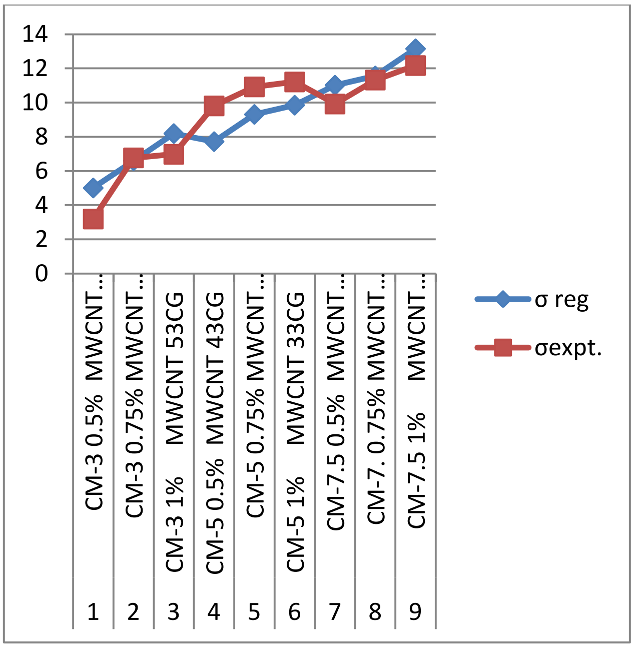
Sl No	Experiment	σ_{reg}	$\sigma_{expt.}$	% Age Error
1	CM-3 0.5% HNC 33CG	5.88	5.18	13.45
2	CM-3 0.75% HNC 43CG	6.54	6.92	5.75
3	CM-3 1% HNC 53CG	7.21	6.77	6.52
4	CM-5 0.5% HNC 43CG	8.29	8.18	1.36
5	CM-5 0.75% HNC 53CG	8.96	9.76	8.94
6	CM-5 1% HNC 33CG	7.36	8.01	8.81
7	CM-7.5 0.5% HNC 53CG	11.12	11.12	0.01
8	CM-7.5 0.75% HNC 33CG	9.52	9.93	4.26
9	CM-7.5 1% HNC 43CG	10.19	9.17	11.14



Plot showing comparsion between compressive strength from regression analysis and from experiments.

5.3 Mortar Blended With MWCNT

Sl No	Experiment	σ_{reg}	$\sigma_{expt.}$	% Age Error
1	CM-3 0.5% MWCNT 33CG	5.01	3.194	56.91
2	CM-3 0.75% MWCNT 43CG	6.60	6.77	2.56
3	CM-3 1% MWCNT 53CG	8.19	6.98	17.33
4	CM-5 0.5% MWCNT 43CG	7.72	9.81	27.06
5	CM-5 0.75% MWCNT 53CG	9.31	10.92	17.30
6	CM-5 1% MWCNT 33CG	9.85	11.21	13.79
7	CM-7.5 0.5% MWCNT 53CG	11.02	9.92	11.09
8	CM-7. 0.75% MWCNT 33CG	11.56	11.32	2.14
9	CM-7.5 1% MWCNT 43CG	13.15	12.17	8.06



Plot showing comparsion between compressive strength from regression analysis and from experiments.

5.4 Observation

Table 8: % age increase in compressive strength for HNC

Sl. No.	% age of HNC	% age increase in compressive strength	Cost in Rs
1	0.5	13.13	9
2	0.75	35	13
3	1	10.7	17

Plot A :

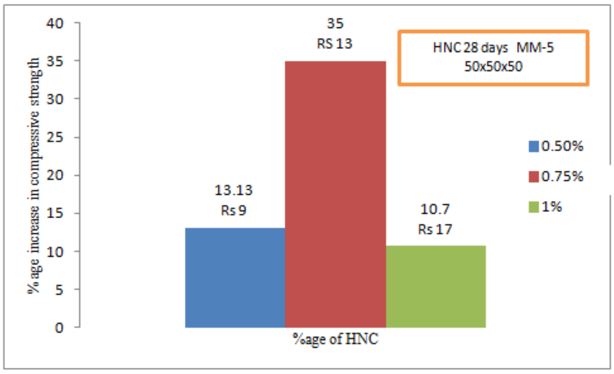


Table 9: %age increase in compressive strength for MWCNT

Sl. No.	% age of MWCNT	% age increase in compressive strength	Cost in Rs
1	0.5	35	495
2	0.75	51	742
3	1	55	990

Plot B:

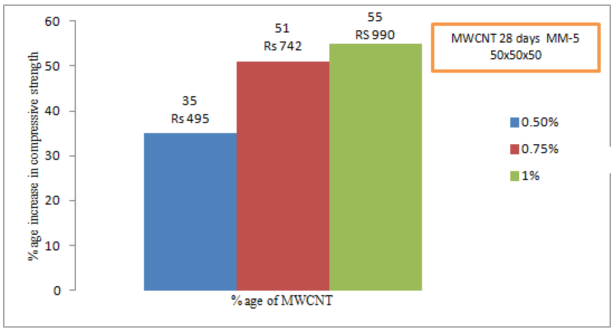


Table 10: Cost Analysis

HNC	1 gm= Rs 50	For 0.5% Rs 9	For 0.75% Rs 13	For 1% Rs 17
MWCNT	1 gm = Rs 3000	For 0.5% Rs 495	For 0.75% Rs 742	For 1% Rs 990

- Comparing plots A and B, optimum percentage increase in Compressive strength can be considered as 35%.
- Optimum percentage of HNC is 0.75% by weight of Cement from plot A.
- In case of MWCNT, optimum percentage cannot be arrived at due to continuous increase in strength v/s percentage increase in MWCNT. But it can be observed by plot B that Compressive strength decreases between 0.1% to 0.5%.
- Hence no need to conduct the test between 0.1% to 0.5% and 35% Compressive Strength obtained by 0.75% by weight of Cement can be taken as optimum.

Conclusion

- When mortar is blended with HNC, Compressive Strength increases with increases in %age of HNC and attained optimum value at 0.75% of HNC by weight of cement for 50mm cubes.

- Mortar blended with MWCNT, Compressive Strength increased with increase in %age of MWCNT..
- From Regression Analysis and from experiments Compressive Strength for MM-7.5 closely matches with Analytical results (differed by 0.01% only for HNC)
- In case of MWCNTs, MM-7.5 values from experiments and regression analysis closely matches and differed by 2.14% only.
- HNC of 0.75% by weight of cement found to be better for increase in Compressive Strength of MM-5 by 35% with respect to conventional mortar MM-5.
- MWCNT's of 0.75% by weight of cement found to be better for increase in Compressive Strength of MM-5 with respect to conventional mortar of MM-5 by 51%.
- From economical point of view , HNC is more suitable for 35% increase in compressive strength as it costs Rs 50 per gm whereas MWCNT's costs Rs 3000 per gm.

References

1. Sigma Aldrich® Bommasandra, Bangalore.
2. R&D Lab, Dept of Civil Engineering, GCE, Ramanagaram.
3. Ravindra Kamble*1, Manasi Ghag2, Sheetal Gaikawad1, Bijoy Kumar Panda3 Halloysite Nanotubes and Applications: A Review. J Adv Scient Res, 2012,3(2):25-29
4. Praveen Kumar K1, Radhakrishna2 Strength And Workability Of Cement Mortar With Manufactured Sand IJRET: International Journal of Research in Engineering and Technology 98.
5. *N* Ozcan Tan a,*, A. Sahin Zaimoglu a, Sinan Hinislioglu a, Selim Altun bTaguchi approach for optimization of the bleeding oncement-based grouts 0886-7798/$ - see front matter _ 2004 Elsevier Ltd
6. Cheah Chee Ban and Mahyuddin Ramli. Optimization of Mix Proportion of High Performance Mortar for Structural Applications American J. of Engineering and Applied Sciences 3 (4): 643-649, 2010.
7. Divyeshkumar D. Paradava*1, Prof. Jayeshkumar Pitroda2Experimental Studies on Mortar Using Polypropylene Fibers, 3(5): May, 2014]
8. Shubham Ashish Jha, Somesh Jethwani and Dheeraj Sangtiani "Carbon Nanotube Cement Composites" International Journal of Civil Engineering and Technology (IJCIET) Volume 7, Issue 5, September-October 2016, pp. 420–427
9. Sakthieswaran, M.Suresh. A Study on Strength Properties for Cement Mortar added with Carbon Nanotubes and ZeoliteVolume 4 Issue 6 June 2015.
10. Jyoti Bharj, Sarabjit Singh, Subhash Chander, Rabinder Singh Role of Dispersion of Multiwalled Carbon Nanotubes on Compressive Strength of Cement Paste International Journal of Mathematical, Computational, Physical, Electrical and Computer Engineering Vol:8, No:2, 2014
11. Vindhya C.R1, Hafeez Khan2, N.S Kumar3 Experimental Investigation On Halloysite Nano Tubes & Clay An Infilled Composite Steel Tubeijret: International Journal of Research in Engineering and Technology eISSN: 2319-1163
12. Pruthvi Serrao*, Ravikanth Prabhu, Chiranth B. P., Yazid Mohammed Application of Taguchi Method to Predict the Abrasive Wear Behavior of CP Titanium Journal of Mechanical Engineering and Automation 2016, 6(5A): 13-17
13. K. Mani lavanya, R.K.Suresh ,A.Sushil Kumar Priya, V.Diwakar Reddy. Optimization of Process Parameters in Turning Operation of AISI-1016 Alloy Steels with CBN Using Taguchi Method And Anova IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE) e-ISSN: 2278-1684,p-ISSN: 2320-334X,

Volume 7, Issue 2 (May. - Jun. 2013), PP 24-27 www.iosrjournals.

14. Abhijit Chatterjee, Debashis Das* Assessing Flow Response of Self-compacting Mortar byTaguchi Method and ANOVA Interaction*Materials Research*. 2013; 16(5): 1084-1091.