

Experimental Studies on Development of Crack-Free Mortar Using Nano-composites (Nano Silica and Halloysite Nano Clay)

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ABSTRACT

Cement mortar is susceptible to micro-cracking, which reduces its durability and service life. This study investigates the effectiveness of Nano Silica (NS) and Halloysite Nanoclay (HNC) in enhancing the mechanical properties and crack resistance of cement mortar. Using a Taguchi L9 orthogonal array, three factors were evaluated: mix proportion (1:3, 1:4, and 1:5), water-cement ratio (0.40, 0.45, and 0.50), and nanomaterial dosage (5%, 7%, and 9% by weight of cement). A total of 18 mixes were tested for compressive, split tensile, and flexural strengths at curing ages of 3, 7, 14, and 28 days. The results showed that mix proportion was the most influential factor affecting compressive strength. The optimum combination for both nanomaterials was a 1:3 mix proportion with a 0.40 water-cement ratio. HNC achieved a maximum 28-day compressive strength of 51.5 MPa at 7% dosage, while NS achieved 42.51 MPa at 5% dosage. Compared with NS-modified mortar, HNC-modified mortar exhibited improvements of 21.1% in compressive strength, 0.9% in flexural strength, and 2.9% in split tensile strength. The superior performance of HNC is attributed to its hollow tubular structure, which enhances crack bridging and mechanical interlocking. Validation experiments confirmed the reliability of the Taguchi model with errors below 2.5%. The study concludes that Halloysite Nanoclay is a promising, cost-effective, and naturally abundant nanomaterial for producing crack-resistant cement mortar with improved mechanical performance and durability.

Keywords: Nano Silica (NS), Halloysite Nanoclay (HNC), Nanomaterials, Mechanical Properties.

1. Introduction

Cement mortar is one of the most widely used construction materials due to its versatility, ease of application, and cost-effectiveness. It serves as a binding material in masonry and plastering works and contributes significantly to the strength and durability of civil engineering structures. Cement mortar is primarily composed of cement, fine aggregate (sand), and water, which undergo hydration reactions to form a hardened matrix. Despite its extensive use, cement mortar is inherently susceptible to cracking caused by plastic shrinkage, drying shrinkage, thermal stresses, freeze-thaw cycles, and external loading

[01]. The formation and propagation of micro-cracks adversely affect the durability and service life of structures by facilitating the ingress of moisture and aggressive chemicals, leading to deterioration and reduced structural performance. To address these limitations, researchers have increasingly explored the incorporation of nanomaterials into cementitious composites [02]. Nanotechnology offers significant potential for enhancing the microstructure and mechanical performance of cement-based materials due to the unique properties of nanoparticles, including their high surface area, enhanced reactivity, and superior bonding characteristics. Nano materials can act as fillers, nucleation sites, and reinforcing agents, thereby reducing porosity, accelerating cement hydration, and improving strength and durability [03]. Among the various nano materials investigated, Nano Silica (NS) and Halloysite Nano clay (HNC) have attracted considerable attention. Nano Silica is a highly pozzolanic material that reacts with calcium hydroxide produced during cement hydration to generate additional calcium-silicate-hydrate (C-S-H) gel, resulting in a denser microstructure and improved mechanical properties [05-09]. Halloysite Nanoclay, a naturally occurring aluminosilicate clay mineral, possesses a unique hollow tubular morphology that enables effective crack bridging, enhanced mechanical interlocking, and improved resistance to crack propagation. Furthermore, HNC is abundant, environmentally friendly, and relatively economical compared to several synthetic nanomaterials [10-15]. Although previous studies have demonstrated the beneficial effects of NS and HNC individually, limited research has been conducted on their comparative performance in cement mortar. Additionally, optimization of nano-modified mortar mixtures using statistical techniques such as the Taguchi method has received relatively little attention. Therefore, a systematic investigation is required to identify the optimum combination of mix proportion, water-cement ratio, and nanomaterial dosage for maximizing mortar performance [16-18]. The present study evaluates and compares the effects of Halloysite Nanoclay and Nano Silica on the compressive strength, split tensile strength, and flexural strength of cement mortar. The experimental program was designed using the Taguchi L9 orthogonal array considering three factors at three levels, namely mix proportion, water-cement ratio, and nanomaterial dosage. Signal-to-Noise (S/N) ratio analysis and Analysis of Variance (ANOVA) were employed to determine the significance of each factor and to identify the optimum mix parameters. The study aims to develop a crack-resistant cement mortar with enhanced mechanical performance and improved long-term durability [20-26].

2. MATERIALS AND METHODOLOGY

2.1 General

This chapter provides a comprehensive description of the materials used in the study, including their physical and chemical properties, mix design calculations, experimental setup, and the methodology adopted for the preparation and testing of nano-modified cement mortar specimens. The research focuses on evaluating the mechanical properties of cement mortar incorporating two types of nanomaterials, namely Halloysite Nanoclay (HNC) and Nano Silica (NS). To optimize the mix proportions while minimizing the number of experimental trials, the Taguchi Design of Experiments (DOE) method was employed. The prepared mortar specimens were tested to assess their compressive strength, split tensile strength, and flexural strength, and the results were subsequently analyzed using Signal-to-Noise (S/N) ratio and Analysis of Variance (ANOVA) techniques [17].

2.2 Materials Used

2.2.1 Cement

Ordinary Portland Cement (OPC) 53 grade, conforming to **IS 12269:1987**, was used for the experimental investigation. The physical properties of the cement were determined through laboratory testing in accordance with the relevant Indian Standard specifications [11]. The cement was selected due to its high strength and suitability for structural applications. The physical properties of the cement used in this study are presented in **Table 1**.

Table 1: The physical characteristics of the cement – OPC 53

Sl. No.	Property	Test Result	Requirements as per I S 12269
1	Fineness (%)	7%	Not more than 10%
2	Soundness (mm)	1.2 mm	Not more than 10mm
3	Initial Setting Time (minutes)	167 min	Not less than 30 min
4	Final Setting Time (minutes)	377 min	Not more than 600 min
5	Standard Consistency (%)	31%	-----
6	Specific Gravity	3.15	-----

2.2.2 Fine Aggregate (M-Sand)

Locally available manufactured sand (M-Sand), conforming to Zone II grading as specified in IS 383:2016, was used as the fine aggregate in this study. The sand was air-dried and sieved through a 2.36 mm IS sieve to remove any oversized particles and organic impurities before use [23]. The selected M-Sand provided adequate grading and particle distribution for the preparation of cement mortar specimens. The physical properties of the fine aggregate used in this investigation are presented in Table 2

Table 2: Physical Characteristics of Fine Aggregate

Sl. No.	Property	Value
1	Specific Gravity	2.6
2	Moisture Content (%)	0.22%
3	Fineness Modulus	2.88

2.2.3 Halloysite Nanoclay (HNC)

Halloysite Nanoclay (HNC) is a naturally occurring aluminosilicate mineral characterized by its unique hollow tubular structure. It was procured from a commercial supplier and used as one of the nanomaterials in this study. The distinctive tubular morphology of HNC provides a high aspect ratio and large surface area, making it an effective reinforcing agent for cementitious composites. These

characteristics contribute to improved microstructural densification, enhanced crack-bridging capability, and better mechanical performance of the cement matrix at the nanoscale. The physical and chemical properties of Halloysite Nanoclay are presented in the subsequent sections.



Fig 1: Halloysite Nanoclay (HNC)-SIGMA Aldrich ,NEW ZEALAND

2.2.4 Nano Silica (NS)

Nano Silica (NS) is an amorphous form of silicon dioxide (SiO_2) with particle sizes in the nanometer range. It was used as the second nanomaterial in this study due to its high pozzolanic activity and excellent pore-refinement capability [14]. Nano Silica reacts with the calcium hydroxide released during cement hydration to produce additional calcium-silicate-hydrate (C-S-H) gel, which enhances the strength, density, and durability of the cementitious matrix.



Fig 2: Nanosilica–Supplied From Noida,India

2.3 Taguchi Experimental Design and Statistical Analysis

2.3.1 Design of Experiments

Design of Experiments (DOE) is a statistical approach used to evaluate the influence of multiple input factors on a response variable while minimizing the number of experimental trials. Compared with conventional trial-and-error methods, DOE provides a systematic framework for identifying significant factors and optimizing process parameters with improved statistical reliability and reduced experimental cost.

2.3.2 Taguchi Method

The Taguchi method was adopted in the present study to optimize the mix parameters of nano-modified cement mortar. This method utilizes orthogonal arrays to evaluate the effects of multiple factors simultaneously while significantly reducing the required number of experiments. In addition, Taguchi analysis incorporates Signal-to-Noise (S/N) ratios to determine the optimal parameter combinations that maximize performance and minimize variability [22].

2.3.3 Selection of Factors and Levels

Based on previous studies and preliminary investigations, three control factors were selected for the experimental program: mix proportion (cement ratio), water-cement ratio, and nanomaterial dosage. Each factor was evaluated at three levels, as presented in Table 3. The selected factors were considered critical in influencing the mechanical properties and microstructural characteristics of nano-modified cement mortar.

2.3.4 L9 Orthogonal Array

A full factorial design consisting of three factors at three levels would require 27 experimental runs (3^3). To reduce the experimental effort without compromising the quality of statistical analysis, a Taguchi L9 orthogonal array was employed. The L9 array enabled the investigation of the main effects of the selected factors using only nine experimental combinations. Separate L9 experimental matrices were developed for Halloysite Nanoclay (HNC) and Nano Silica (NS) modified mortar systems while maintaining identical factor levels to facilitate comparative evaluation.

2.3.5 Signal-to-Noise Ratio Analysis

The Signal-to-Noise (S/N) ratio was used to evaluate the robustness of the experimental responses and identify the optimum parameter levels. Since the objective of this study was to maximize compressive strength, split tensile strength, and flexural strength, the “Larger-the-Better” quality characteristic was adopted. The corresponding S/N ratio was calculated using:

$$S/N = -10 \log \left(\frac{1}{n} \sum_{i=1}^n \frac{1}{y_i^2} \right)$$

Where n represents the number of observations and y_i denotes the measured response value. Higher S/N ratios indicate superior performance and lower variability [09].

2.3.6 Analysis of Variance (ANOVA)

Analysis of Variance (ANOVA) was performed to quantify the statistical significance and percentage contribution of each factor to the measured responses. The technique partitions the total variation in the experimental data into contributions from individual factors and experimental error. The resulting percentage contributions were used to identify the dominant parameters influencing the mechanical properties of HNC- and NS-modified cement mortars and to validate the optimization results obtained from S/N ratio analysis [13].

2.6 Testing Procedures

2.6.1 General

The mechanical properties of nano-modified cement mortar specimens were evaluated using a Mecmesin MultiTest 25-i Universal Testing Machine equipped with Emperor software for load application and data acquisition. Prior to testing, the specimens were removed from the curing tank and

allowed to surface dry. For each mix composition and curing age, a minimum of three specimens were tested, and the average value was reported.

3.6.2 Split Tensile Strength Test

Split tensile strength tests were conducted on cylindrical specimens measuring 40 mm in length and 20 mm in diameter. The specimens were positioned horizontally between the loading platens of the testing machine, and load was applied continuously until failure occurred. The peak load recorded by the software was used to calculate the split tensile strength. The average value of three specimens was considered for analysis.

3.6.3 Flexural Strength Test

Flexural strength was determined using a three-point bending test on prism specimens measuring 100 mm × 40 mm × 20 mm. The specimens were centrally placed on the support span, and load was applied at the midpoint until fracture occurred.

3.6.4 Compressive Strength Test

Compressive strength tests were performed on square mortar specimens of dimensions 40 mm × 40 mm × 20 mm. The specimens were centrally aligned between the compression platens, and load was applied gradually until failure. The peak load recorded by the testing machine was used to determine the compressive strength. The average compressive strength of three specimens was reported for each mix and curing age.

3.1 EQUIPMENTS USED



Fig 3:MultiTest25-I NanoTesting Machine



Fig 4: Nano Material testing Machine connected to automatic data acquisition system

3.2 Experimental Set-up for the preparation of Specimens:





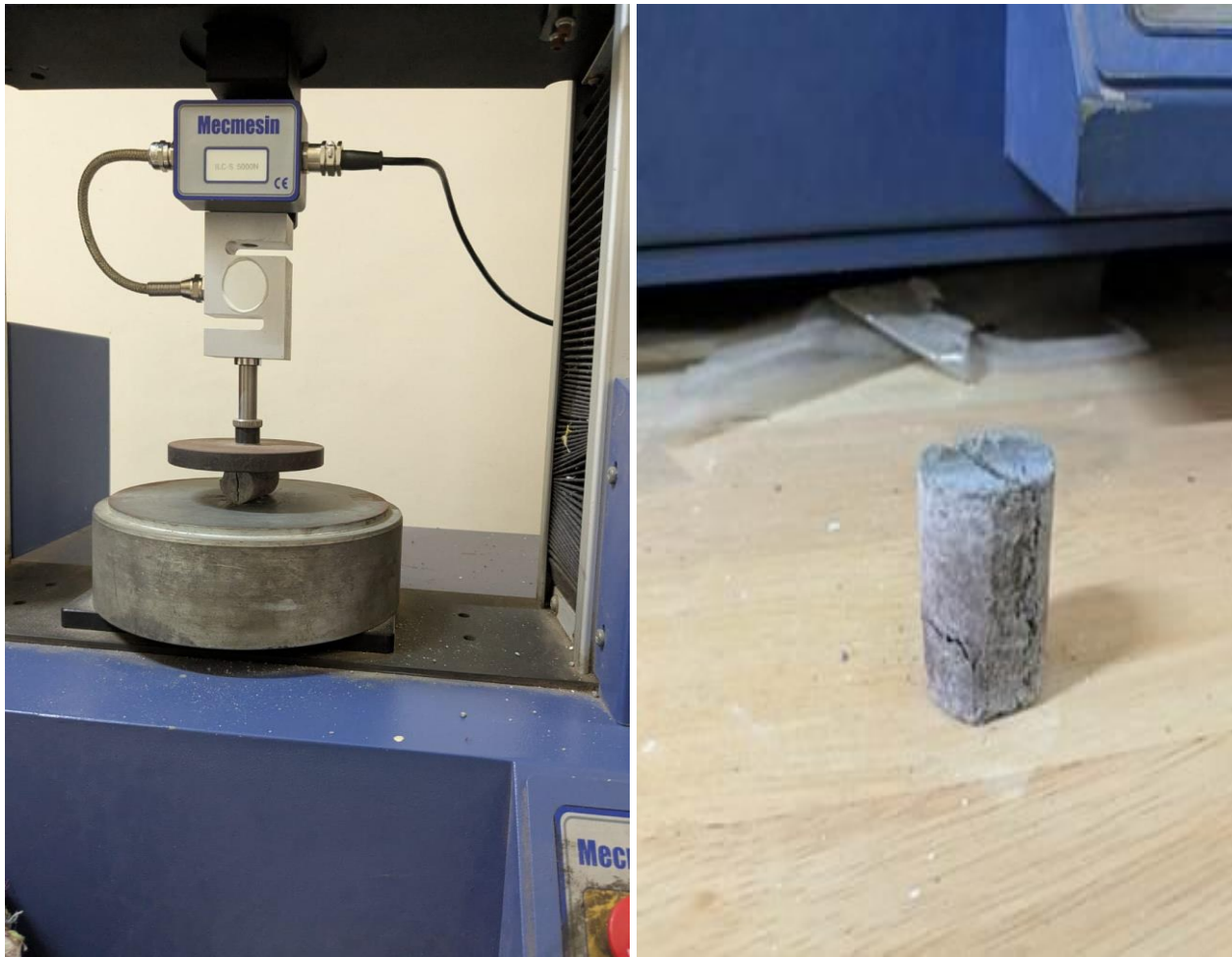


Fig 5: Experimental set up and different samples fabrication for mechanical test

1. Results and Discussion.

3.1.1 Evaluation of Split Tensile Strength of Conventional Cement Mortar

Table 3: Experimental Split Tensile Strength Results of Conventional Cement Mortar

Sl.No.	Proportion	W/C	3d	7d	14d	28d
1	1:3	0.4	3.10	3.65	4.21	4.50
2	1:3	0.45	2.71	3.42	3.96	4.14
3	1:3	0.5	2.42	3.13	3.55	3.69
4	1:4	0.4	2.65	3.36	3.76	4.07
5	1:4	0.45	2.36	3.05	3.45	3.65
6	1:4	0.5	2.11	2.76	3.16	3.36
7	1:5	0.4	2.26	2.95	3.26	3.54
8	1:5	0.45	2.07	2.63	2.95	3.24

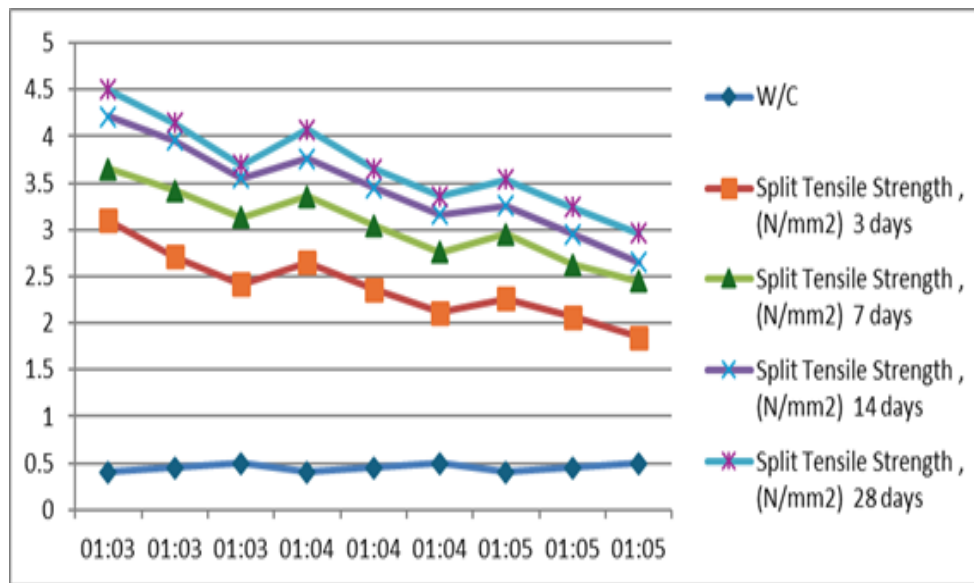


Fig 6: Experimental Split Tensile Strength Results of Conventional Cement Mortar

The split tensile strength results of conventional cement mortar at different curing ages are presented in Table 4.1. It can be observed that the split tensile strength increased consistently with curing age for all mix proportions due to the progressive hydration of cement and the consequent development of a stronger cementitious matrix. The highest 28-day split tensile strength of 4.50 MPa was achieved by the 1:3 mix proportion with a water-cement ratio of 0.40, while the lowest strength was recorded for leaner mixes with higher water-cement ratios. The results indicate that lower water-cement ratios and richer cement contents contribute to improved tensile performance by producing a denser and less porous mortar structure.

3.1.2 Flexural Strength Results of Conventional Cement Mortar

Table 4: Flexural Strength of Conventional Cement Mortar at Different Curing Ages.

Sl. No.	Proportion	W/C	3d	7d	14d	28d
1	1:3	0.4	4.21	5.47	6.23	7.24
2	1:3	0.45	3.96	5.11	5.97	6.82
3	1:3	0.5	3.61	4.76	5.51	6.23
4	1:4	0.4	3.71	4.84	5.64	6.61
5	1:4	0.45	3.42	4.56	5.21	6.08
6	1:4	0.5	3.11	4.17	4.87	5.67
7	1:5	0.4	3.21	4.28	5.09	5.87
8	1:5	0.45	2.95	3.96	4.61	5.43
9	1:5	0.5	2.61	3.51	4.25	5.06

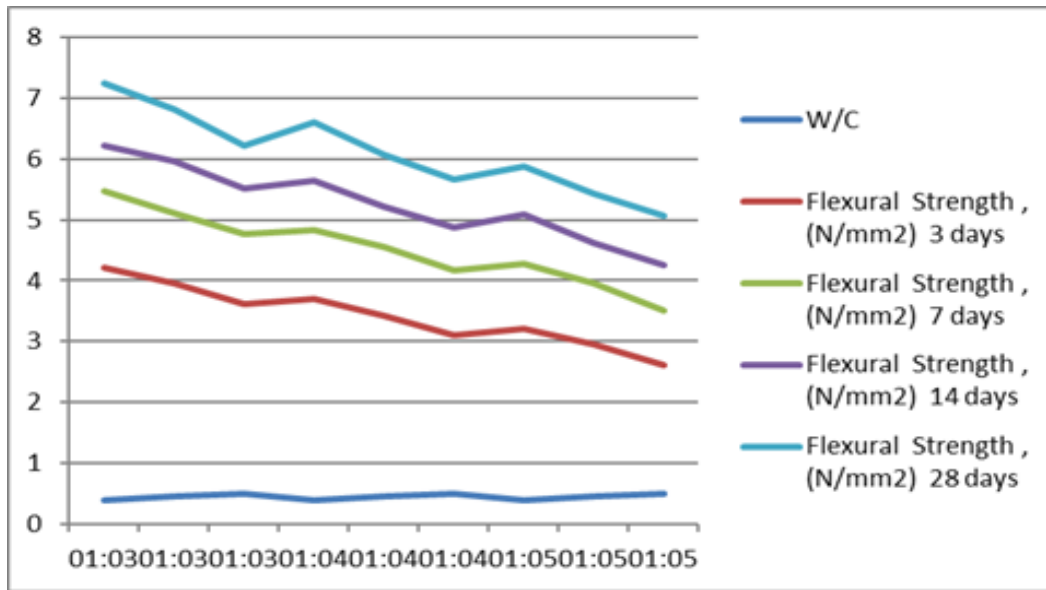


Fig 7: Flexural Strength of Conventional Cement Mortar at Different Curing Ages.

The flexural strength results of conventional cement mortar at different curing ages are presented in Table 4. A gradual increase in flexural strength was observed with increasing curing age for all mix proportions, indicating continuous cement hydration and improvement in bond strength within the mortar matrix. The maximum 28-day flexural strength of 7.24 MPa was obtained for the 1:3 mix proportion with a water-cement ratio of 0.40, whereas the minimum value of 5.06 MPa was recorded for the 1:5 mix proportion with a water-cement ratio of 0.50. The results demonstrate that richer cement content and lower water-cement ratios contribute to enhanced flexural performance by producing a denser microstructure and stronger interfacial bonding between cement paste and fine aggregate.

3.1.3 Compressive Strength Results of Conventional Cement Mortar

Table 5: Experimental Compressive Strength Results of Conventional Cement Mortar (N/mm²)

W/C Ratio	Proportion	3 days (N/mm ²)	7 days (N/mm ²)	14 days (N/mm ²)	28 days (N/mm ²)
0.4	1:03	18.54	26.51	32.05	38.52
0.4	1:04	15.54	22.56	27.54	33.59
0.4	1:05	13.54	19.54	24.06	28.57
0.45	1:03	16.07	23.06	28.16	34.18
0.45	1:04	13.54	19.58	24.67	29.58
0.45	1:05	12.76	17.64	21.56	25.76
0.5	1:03	14.87	20.15	24.57	30.76
0.5	1:04	11.51	17.41	21.05	26.47
0.5	1:05	10.51	15.38	18.64	21.52

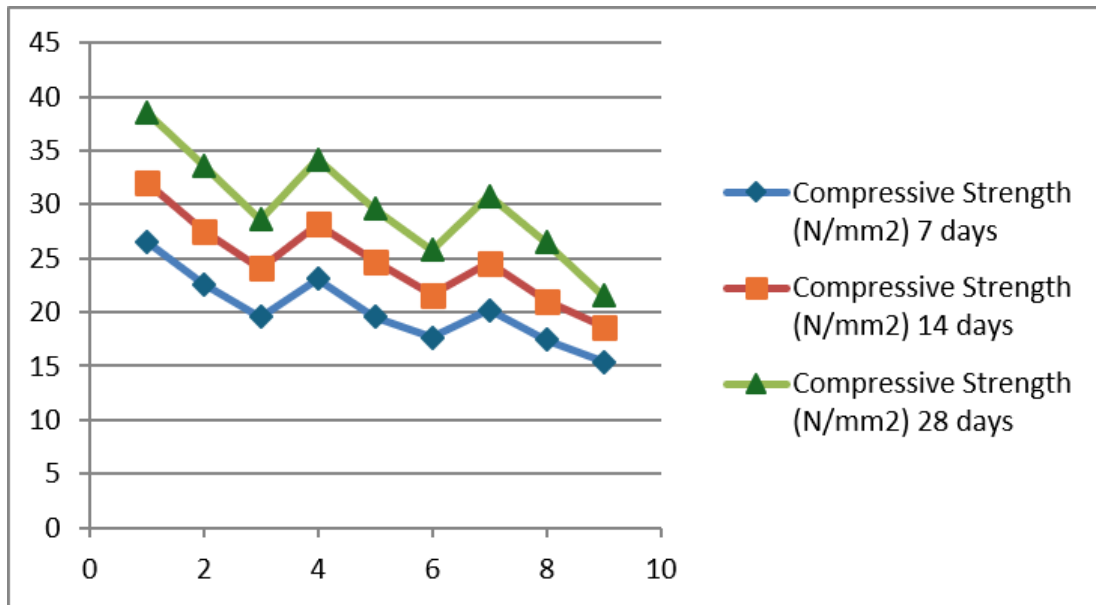


Fig 8: Experimental Compressive Strength Results of Conventional Cement Mortar (N/mm²)

The compressive strength results of conventional cement mortar at different curing ages are presented in Table 5. For all mix proportions, compressive strength increased progressively with curing age due to the continued hydration of cement and the consequent development of a denser and stronger cementitious matrix. The highest 28-day compressive strength of 38.52 MPa was achieved by the 1:3 mix proportion with a water-cement ratio of 0.40, whereas the lowest value of 21.52 MPa was recorded for the 1:5 mix proportion with a water-cement ratio of 0.50. The results clearly indicate that a lower water-cement ratio and higher cement content significantly enhance compressive strength by reducing porosity and improving the overall microstructure of the mortar.

3.2 Taguchi and ANOVA Analysis Using MINITAB 16

Table 6: Response Table for Means

Level	Proportion	W/CRatio	% HNC
1	33.85	32.56	31.83
2	32.94	32.17	32.12
3	29.25	31.30	32.09
Delta	4.61	1.27	0.29
Rank	1	2	3

Table 7: Response Table for Signal to noise ratio.

Level	Proportion	W/CRatio	% HNC
1	49.35	43.39	40.02
2	44.45	41.77	42.04
3	29.06	37.70	40.80
Delta	20.28	5.68	2.03
Rank	1	2	3

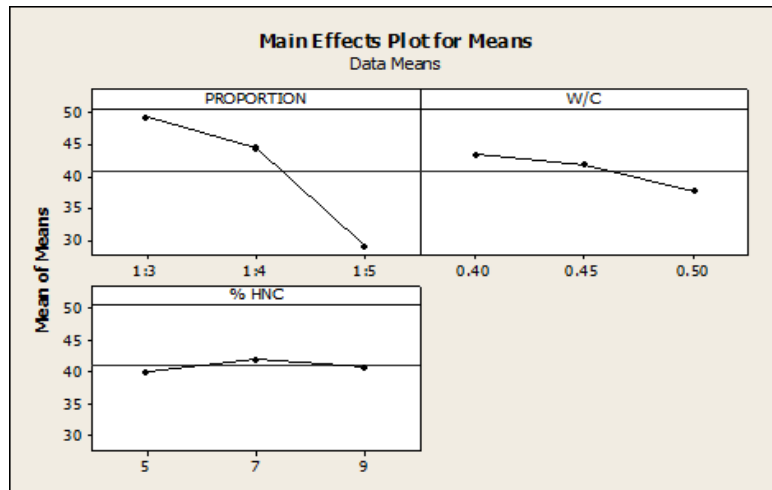


Fig 9: Main Effects Plot for Means

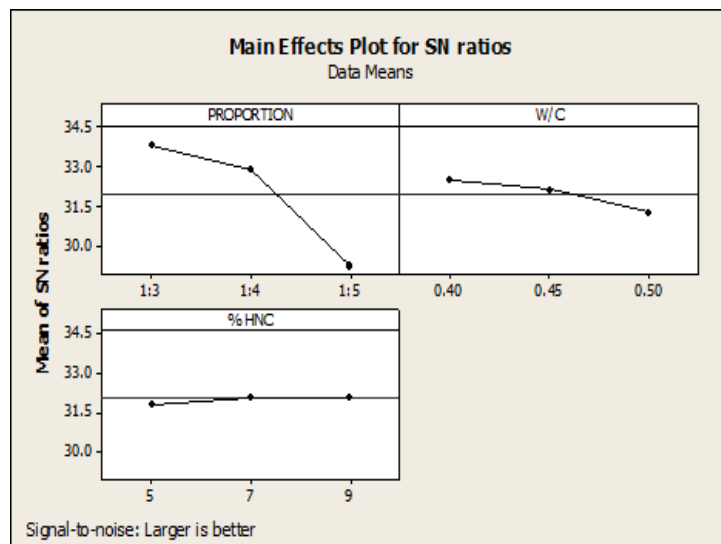


Fig 10: Main Effects Plot for SN ratios

The Taguchi analysis was performed to evaluate the influence of mix proportion, water-cement ratio, and Halloysite Nanoclay (HNC) dosage on the compressive strength of cement mortar. The Signal-to-Noise (S/N) ratio based on the Larger-the-Better criterion was employed to identify the optimum factor levels for maximizing compressive strength. The response table obtained from MINITAB 16 is presented in Table 6.

3.2.3 Comparison of Experimental and Regression-Predicted Compressive Strength of HNC-Modified Cement Mortar

Table 8: Comparison of Experimental and Regression-Predicted Compressive Strength Values for HNC-Modified Cement Mortar.

Sl.No.	Optimized Experiment from Taguchi	Compressive Strength from Regression (N/mm ²)	Compressive Strength from Experiment (N/mm ²)	% Error
1	Proportion 1:3, W/C 0.4, 5% HNC	51.74	50.50	2.40

2	Proportion 1:3, W/C 0.45, 7% HNC	49.35	51.50	4.36
3	Proportion 1:3, W/C 0.5, 9% HNC	46.90	46.04	1.83
4	Proportion 1:4, W/C 0.4, 7% HNC	47.32	48.07	1.58
5	Proportion 1:4, W/C 0.45, 9% HNC	44.84	44.76	0.18
6	Proportion 1:4, W/C 0.5, 5% HNC	41.22	40.51	1.72
7	Proportion 1:5, W/C 0.4, 9% HNC	32.31	31.59	2.23
8	Proportion 1:5, W/C 0.45, 5% HNC	28.68	29.04	1.25
9	Proportion 1:5, W/C 0.5, 7% HNC	26.22	26.56	1.30

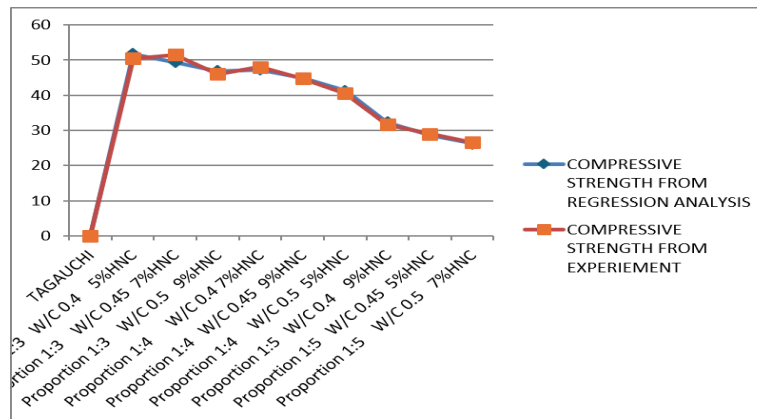


Fig 11: Comparison of Experimental and Regression-Predicted Compressive Strength of HNC-Modified Cement Mortar.

3.2.4 Comparison of Experimental and Regression-Predicted Compressive Strength of NS-Modified Cement Mortar

Table 9: Comparison of Experimental and Regression-Predicted Compressive Strength Values for NS-Modified Cement Mortar

Sl.No.	Optimized Experiment from Taguchi	Compressive Strength from Regression (N/mm ²)	Compressive Strength from Experiment (N/mm ²)	% Error
1	Proportion 1:3, W/C 0.4, 5% NS	42.51	42.15	0.85
2	Proportion 1:3, W/C 0.45, 7% NS	38.54	38.53	0.03
3	Proportion 1:3, W/C 0.5, 9% NS	34.54	34.90	1.03

4	Proportion 1:4, W/C 0.4, 7% NS	36.54	36.76	0.60
5	Proportion 1:4, W/C 0.45, 9% NS	33.71	33.14	1.72
6	Proportion 1:4, W/C 0.5, 5% NS	30.57	30.90	1.07
7	Proportion 1:5, W/C 0.4, 9% NS	30.11	53.66	43.89
8	Proportion 1:5, W/C 0.45, 5% NS	28.07	28.30	0.81
9	Proportion 1:5, W/C 0.5, 7% NS	25.08	24.60	1.95

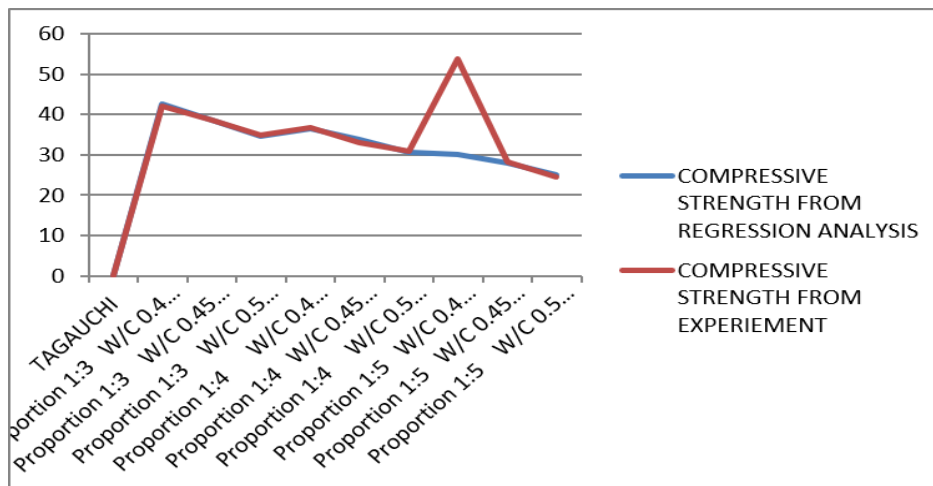


Fig 12: Comparison of Experimental and Regression-Predicted Compressive Strength Values for NS-Modified Cement Mortar

2. Conclusion:

This study evaluated the effects of Halloysite Nanoclay (HNC) and Nano Silica (NS) on the mechanical properties of cement mortar using the Taguchi L9 experimental design. The results demonstrated that the incorporation of nanomaterials improved the compressive and split tensile strengths of cement mortar, with HNC exhibiting superior performance compared to NS. The optimum mix proportion for both nanomaterials was identified as 1:3 with a water-cement ratio of 0.40. HNC-modified mortar achieved the highest 28-day compressive strength of 51.50 MPa at a dosage of 7%, representing a 33.7% improvement over conventional mortar, whereas NS-modified mortar attained a maximum compressive strength of 42.51 MPa at a dosage of 5%. Statistical analysis revealed that mix proportion was the most influential factor governing compressive strength, followed by water-cement ratio of 0.40. HNC-modified mortar achieved the highest 28-day compressive strength of 51.50 MPa at a dosage of 7%, representing a 33.7% improvement over conventional mortar, whereas NS-modified mortar attained a maximum compressive strength of 42.51 MPa at a dosage of 5%. Statistical analysis revealed that mix proportion was the most influential factor governing compressive strength, followed by water-cement ratio, while nanomaterial dosage had a comparatively lower contribution within the investigated range.

The enhanced performance of HNC was attributed to its hollow tubular morphology, which promotes crack bridging, improved mechanical interlocking, and microstructural densification of the cement matrix.

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