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Enhancing concrete mechanical properties using nano-silica, calcined clay, and glass fibers optimized by response surface methodology

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Abstract

This study investigates the combined effects of nano-silica, calcined clay, and glass fibers on the mechanical properties of normal-grade concrete (M30), with a primary aim to utilize industrial waste materials and improve concrete performance while promoting environmental sustainability. Response Surface Methodology (RSM) was employed to optimize material proportions and evaluate their impact. The experimental design incorporated nano-silica (0– 10%), calcined clay (0–30%), and glass fibers (0–2%), corresponded to the minimum and maximum values in the range. A total of 120 specimens were tested to assess compressive strength, flexural strength, rebound hammer values, and ultrasonic pulse velocity (UPV). The optimized mix predicted compressive strength of 49.41 MPa, flexural strength of 9.05 MPa, rebound hammer value of 62 MPa, and UPV of 5445 m/s. Validation tests demonstrated observed compressive strength of 46.55 MPa, flexural strength of 8.05 MPa, rebound hammer value of 60 MPa, and UPV of 4890 m/s, with errors of 5.79%, 11.00%, 3.22%, and 10.19%, respectively. Analysis of variance (ANOVA), a statistical method for determining the significance of factors on observed outcomes, indicated that nano-silica had the most substantial impact on all performance metrics. The models showed high reliability, with R^2 values of 0.9653 for compressive strength, 0.9268 for flexural strength, 0.9266 for rebound hammer, and 0.8894 for UPV. The study demonstrates the potential of incorporating waste materials like calcined clay and nano-silica into concrete to enhance its mechanical properties and reduce environmental impact. This approach aligns with sustainable construction practices by lowering reliance on traditional cement, reducing carbon emissions, and promoting the reuse of industrial by products. The findings support the development of cost-effective, durable, and eco-friendly infrastructure, addressing the dual objectives of improved performance and environmental sustainability.

1. Introduction

The utilization of advanced materials in concrete has become increasingly significant in modern construction, as researchers and engineers seek to enhance the structural integrity and durability of concrete compositions. Among these materials, glass fibres and nano-silica have attracted considerable scholarly interest due to their capacity to improve the mechanical properties and longevity of concrete. Glass fibres, noted for their high tensile strength and resistance to corrosion, offer a more effective alternative to traditional reinforcement materials such as steel. When incorporated into concrete, glass fibres can substantially enhance its tensile and flexural strength, thereby increasing its overall robustness.

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Similarly, nano-silica has emerged as a critical material in enhancing the microstructure and mechanical properties of concrete. By reducing porosity and increasing the density of the cement matrix, nano-silica contributes to the overall strength and durability of the concrete. This enhancement is particularly advantageous in applications where concrete must endure challenging environmental conditions, such as in large-scale infrastructure projects[1]. The integration of calcined clay, particularly kaolinitic clay, as a supplementary cementitious material (SCM) in concrete has gained considerable attention due to its pozzolanic properties, which significantly improve the mechanical durability and performance of concrete structures [Ng and Engelsen 2018]. Calcined clay is produced by thermally treating clay at temperatures between 600°C and 900°C, leading to the dehydroxylation of clay minerals and the formation of reactive amorphous silica and alumina. This transformation is crucial as it allows calcined clay to react with calcium hydroxide produced during the hydration of Portland cement, resulting in the formation of calcium silicate hydrates (C-S-H), which are essential for concrete strength and durability [2-3].

Several studies have shown that the pozzolanic activity of calcined clay is influenced by various factors, such as the type of clay, calcination temperature, and fineness of the material. For instance, higher calcination temperatures have been found to increase the reactivity of kaolinitic clays, enhancing their effectiveness as SCMs. The pozzolanic reaction not only improves the mechanical strength of concrete but also enhances its durability by refining the pore structure, reducing porosity, and increasing density, which is particularly beneficial in environments where concrete is exposed to aggressive chemicals and moisture [4].

Research has also demonstrated that the inclusion of calcined clay can lead to significant improvements in compressive strength and durability. Additionally, calcined clay has been shown to accelerate hydration kinetics, particularly in high pH environments, promoting the dissolution of clay particles and stimulating pozzolanic reactions. This acceleration can lead to faster strength development, making calcined clay an attractive option for projects requiring shorter construction timelines. Moreover, from a sustainability perspective, the use of calcined clay reduces the carbon footprint associated with cement production by partially substituting Portland cement, thereby decreasing the amount of clinker required, which is important due to the high greenhouse gas emissions from cement manufacturing [5-6].

The incorporation of nano-silica into concrete has emerged as a major advancement in materials science, especially in enhancing the mechanical and durability properties of cementitious materials. Nano-silica, with its extremely small particle size and high surface area, plays a critical role in refining the microstructure of concrete, leading to improved performance. Its inclusion reduces porosity and enhances the density of the cement matrix, both of which are essential for the durability and longevity of concrete structures. Wu and Khayat [2017] demonstrated that adding just 1% nano-silica reduced the porosity of ultra-high strength concrete by 29% after one day and 19% after 28 days. This reduction in porosity correlates directly with improved resistance to environmental factors such as moisture and chemical attacks, ultimately enhancing the overall durability of concrete [7-8].

Nano-silica's pozzolanic properties significantly contribute to improving the strength of concrete. This improvement is largely attributed to the filler effect of nano-silica, which refines the pore structure and promotes the formation of additional calcium silicate hydrates (C-S-H) during hydration, thereby enhancing the mechanical properties of concrete [9].

In addition to its mechanical benefits, nano-silica enhances the resistance of concrete to carbonation, which is a key factor in the long-term degradation of concrete structures. Alqamish and Al-Tamimi [2021] reported that incorporating nano-silica into concrete formulations improved carbonation resistance, thus extending the lifespan of concrete in environments exposed to carbon dioxide[10]. This improvement in carbonation resistance is particularly important for maintaining structural integrity in various applications. Furthermore, the rheological properties of concrete mixtures are positively influenced by the addition of nano-silica. Alvansazyazdi [2023] found that nano-silica in shotcrete improved its strength and impermeability, optimizing the interfacial structure of the cementitious materials, which is crucial for applications such as tunnels and underground constructions [11].

The interfacial transition zone (ITZ) between aggregates and cement paste is another area where nano-silica proves beneficial. Babalu and Sunil [2019] showed that the incorporation of nano-silica enhances the pore structure and improves the ITZ, often a weak point in concrete[Babalu* and Sunil 2019]. This refinement leads to stronger adhesion between aggregates and the cement paste, resulting in improved overall strength and durability [12]. Additionally, nano-silica has been linked to a reduction in shrinkage rates in concrete. Nano-silica in high-volume fly ash concrete significantly improved durability, including reduced water absorption and shrinkage, which are critical for minimizing cracking and ensuring the long-term stability of concrete structures [13].

Research indicates that the optimal dosage of nano-silica is critical; while a 1% addition by weight of cement has been shown to maximize strength, higher concentrations can adversely affect workability and strength [14]. Specifically, concentrations exceeding 3% may lead to a reduction in compressive strength due to the lumping phenomenon, which negatively impacts the mixture's workability [15]. This underscores the importance of carefully balancing the amount of nano-silica used in concrete formulations to achieve desired mechanical properties without compromising workability. The pozzolanic reaction of nano-silica with calcium hydroxide during hydration produces additional calcium silicate hydrate (C-S-H) gel, which is responsible for the strength of concrete [16]. This reaction not only enhances the mechanical properties but also reduces the porosity of the concrete, leading to improved durability [17]. Studies have shown that incorporating nano-silica can increase compressive strength by up to 21% and tensile strength by 44% compared to control specimens without nano-silica [18].

The incorporation of glass fibers into concrete has gained significant attention in recent years due to their capacity to enhance both the mechanical properties and durability of concrete structures. A key advantage of glass fibers is their ability to improve both tensile and flexural strength in concrete mixtures. For instance, the addition of glass fibers to peach shell lightweight concrete led to a substantial increase in compressive strength, attributed to improved bonding at the interface between the fibers and the cement paste [19].

Extensive research has been conducted to assess the mechanical properties of glass fiber reinforced concrete (GFRC). The flexural modulus of elasticity showed minimal variation across different fiber volume fractions, the overall mechanical performance of GFRC was notably enhanced [Tahir et al. 2023]. The incorporation of glass fibers improves ductility and resistance to crack failure, thereby enhancing the structural integrity of concrete [Fenu, Forni, and Cadoni 2016]. Additionally, the durability of GFRC is a crucial aspect, with Yıldırım and Özhan (2023) reporting that alkali-resistant glass fibers significantly mitigate deterioration in alkaline conditions, thereby extending the longevity of concrete compositions [20].

Furthermore, glass fibers play a vital role in reducing fracture propagation, which is essential for maintaining the structural integrity of concrete under external forces. Zaid et al. (2021) emphasized that glass fibers act as bridges between fissures, preventing them from merging and thus preserving the integrity of the concrete [21]. The successful integration of glass fibers into various concrete formulations, including geopolymer concrete, has also been documented. Adding glass fibers to geopolymer mixtures significantly improved flexural strength [22].

The objective of this research is to examine the collective impacts of these substances on the mechanical characteristics of concrete through the application of Response Surface Methodology (RSM) for optimization. RSM is a powerful statistical method that makes it possible to assess and optimize several factors at once, giving researchers new information about how calcined clay, nano-silica, and glass fibres interact. Through a methodical examination of these materials' effects on important mechanical attributes like compressive strength and flexural strength, this study aims to create an ideal concrete mix design that optimizes performance while preserving material economy. The study's conclusions will benefit the larger field of sustainable building materials by providing a thorough understanding of the efficient integration of these cutting-edge ingredients into concrete formulas.

2. Experimental methodology

2.1 Materials

The materials used in this study were carefully selected to enhance the mechanical properties and sustainability of the concrete mix. Ordinary Portland Cement (OPC) 43 Grade, with a specific gravity of 3.14 and a fineness of 4.0%, served as the primary binder as per Indian standard. Nano-silica, characterized by a particle size of 20-50 nm and a specific gravity of 2.33, was incorporated as a partial cement replacement. Its ultrafine particles contributed significantly to improving the microstructure of the concrete by filling voids and increasing density, thereby enhancing compressive strength and durability. Natural river sand, conforming to Zone III specifications, was used as the fine aggregate, with a specific gravity of 2.65 and a fineness modulus of 2.75. To further enhance durability, calcined kaolinite clay, with a specific gravity of 2.60, was used as a sand substitute. Calcined clay acted as a pozzolanic material, reacting with calcium hydroxide to form additional cementitious compounds, thereby improving the concrete's long-term strength and resistance to chemical attack.

Glass fibers, with a diameter of 25 microns, a length of 15 cm, and a specific gravity of 2.68, were added to enhance flexural performance. These fibers effectively bridged microcracks, reducing crack propagation and improving the concrete's resistance to bending and tension forces. Crushed angular coarse aggregates, with a nominal size of 20 mm and a specific gravity of 2.71, were used as the primary coarse aggregate to provide bulk and strength. Portable water with a specific gravity of 1.0 was used for the mixing process to ensure proper hydration and workability. Additionally, a Sikaplast superplasticizer was added at a dosage of 1% to improve workability and ensure uniform distribution of the mix components. The combination of these advanced materials—nano-silica, calcined clay, and glass fibers—demonstrates their collective ability to enhance the performance, durability, and sustainability of concrete and the Figure 1 shows materials used in this study.

(a) (b) (c)

Fig. 1. Materials used in this study (a) Nano silica (b) Calcinated clay and (c) Glass fiber

2.2 Mix Design

The study investigates the effects of varying replacement levels of nano-silica, calcined clay, and glass fibers on the mechanical properties and durability of concrete as per IS10262:2019 (BIS 2019). Nano-silica was used as a partial cement replacement at levels of 0%, 3%, 5%, 7%, and 10% by weight of cement. Calcined clay was substituted for sand at levels of 0%, 10%, 20%, 25%, and 30% by weight of sand. Additionally, glass fibers were added to the concrete mix at levels of 0%, 0.5%, 1.0%, 1.5%, and 2.0% of the total concrete volume. These varied levels were selected to systematically evaluate the impact of each material on the overall performance of the concrete, aiming to identify the optimal combination for enhancing both strength and durability.

The Design of experiment (DoE) software was selected to fully consider the experimental aspect. Preliminary tests were initially conducted to define the main parameters and related limits, both lower and upper. The factors and levels to be used in the DoE were defined based on these tests, as described in Table 1. The control features of the experiment correspond with the literature.

Table 1. Factor level

Material	Range (min-max)	Neutral (0)	Low (-1)	High (+1)
Nano-Silica	0-10	5	0	10
Calcined Clay	0-30	15	0	30
Glass Fiber	0-2	1	0	2

In this response surface methodology (RSM), a central composite design (CCD) was applied to optimize the experimental conditions. Three continuous variables were used: Nano-Silica (0% to 10%), Calcined Clay (0% to 30%), and Glass Fiber (0% to 2%). The CCD involved low (-1), central (0), and high (+1) levels for each variable, with center points included to ensure accurate estimation of curvature. The experimental design was randomized to minimize bias, and multiple replicates were conducted at the center point for improving the reliability of the model and the precision of the response predictions. The experimental matrix is presented in Table 2. In this study, the water content was fixed at 181 kg/m^3, and a gallium-based superplasticizer was included at a dosage of 1% by weight of cement for the mix.

Table 2. Experimental matrix

Mix	Nano-Silica (%)	Calcined Clay (%)	Glass Fiber (%)	Nano-Silica (kg/m^3)	Cement (kg/m^3)	Calcined Clay (kg/m^3)	sand (kg/m^3)	Coarse aggregate (kg/m^3)
CM	0	0	0	0.00	422.30	0.00	671.10	1098.5
C1	5	15	0	21.12	401.19	100.67	570.44	1098.5
C2	5	0	1	21.12	401.19	0.00	671.10	1098.5
C3	0	30	2	0.00	422.30	201.33	469.77	1098.5
C4	5	15	1	21.12	401.19	100.67	570.44	1098.5
C5	5	15	1	21.12	401.19	100.67	570.44	1098.5
C6	10	0	2	42.23	380.07	0.00	671.10	1098.5
C7	5	30	2	21.12	401.19	201.33	469.77	1098.5
C8	5	0	1	21.12	401.19	0.00	671.10	1098.5
C9	5	15	0	21.12	401.19	100.67	570.44	1098.5
C10	0	30	2	0.00	422.30	201.33	469.77	1098.5
C11	5	30	1	21.12	401.19	201.33	469.77	1098.5
C12	5	0	2	21.12	401.19	0.00	671.10	1098.5
C13	0	30	2	0.00	422.30	201.33	469.77	1098.5
C14	5	0	2	21.12	401.19	0.00	671.10	1098.5
C15	10	0	2	42.23	380.07	0.00	671.10	1098.5
C16	0	0	1	0.00	422.30	0.00	671.10	1098.5
C17	5	15	1	21.12	401.19	100.67	570.44	1098.5
C18	5	15	0	21.12	401.19	100.67	570.44	1098.5
C19	10	15	2	42.23	380.07	100.67	570.44	1098.5
C20	0	0	1	0.00	422.30	0.00	671.10	1098.5

Table 2 experimental matrix with results outlines the effects of varying proportions of nano-silica, calcined clay, and glass fibers on concrete properties for M30grade concrete. Mixes with nano-silica (e.g., C4) showed improved compressive strength and flexural performance due to enhanced microstructure. Calcined clay significantly boosted results, with mixes like C10 (30% calcined clay) achieving the highest compressive strength (41.3 MPa) and flexural strength (6.4 MPa), attributed to its pozzolanic activity. Glass fibers enhanced flexural properties by bridging microcracks, as seen in mixes like C6 (10% nano-silica, 2% glass fibers). Overall, combinations of these materials improved durability, strength, and uniformity.

2.3 Experimental Setup

2.3.1 Compressive Strength

This test was conducted in accordance with IS 516 (Part 1/Sec 1):2021, which provides the procedure for determining the compressive strength of concrete. Cube specimens measuring 150 mm × 150 mm × 150 mm were tested after 28 days of water curing using a compression testing machine to assess their strength. The specific results are shown in Table 3 and Figure 2.

2.3.2 Flexural Strength

The flexural strength test was carried out as per IS 516 (Part 1/Sec 1):2021. This standard outlines the method for determining the flexural strength of concrete by applying loads to beam specimens measuring 100 mm × 100 mm × 500 mm until failure.

2.3.3 Rebound Hammer

The rebound hammer test was performed according to IS 516 (Part 5/Sec 4):2020. This non-destructive test assesses the surface hardness of concrete and estimates its compressive strength based on the rebound values measured from the hammer. Cube specimens measuring 150 mm × 150 mm × 150 mm were used for this test.

2.3.4 UPV Test

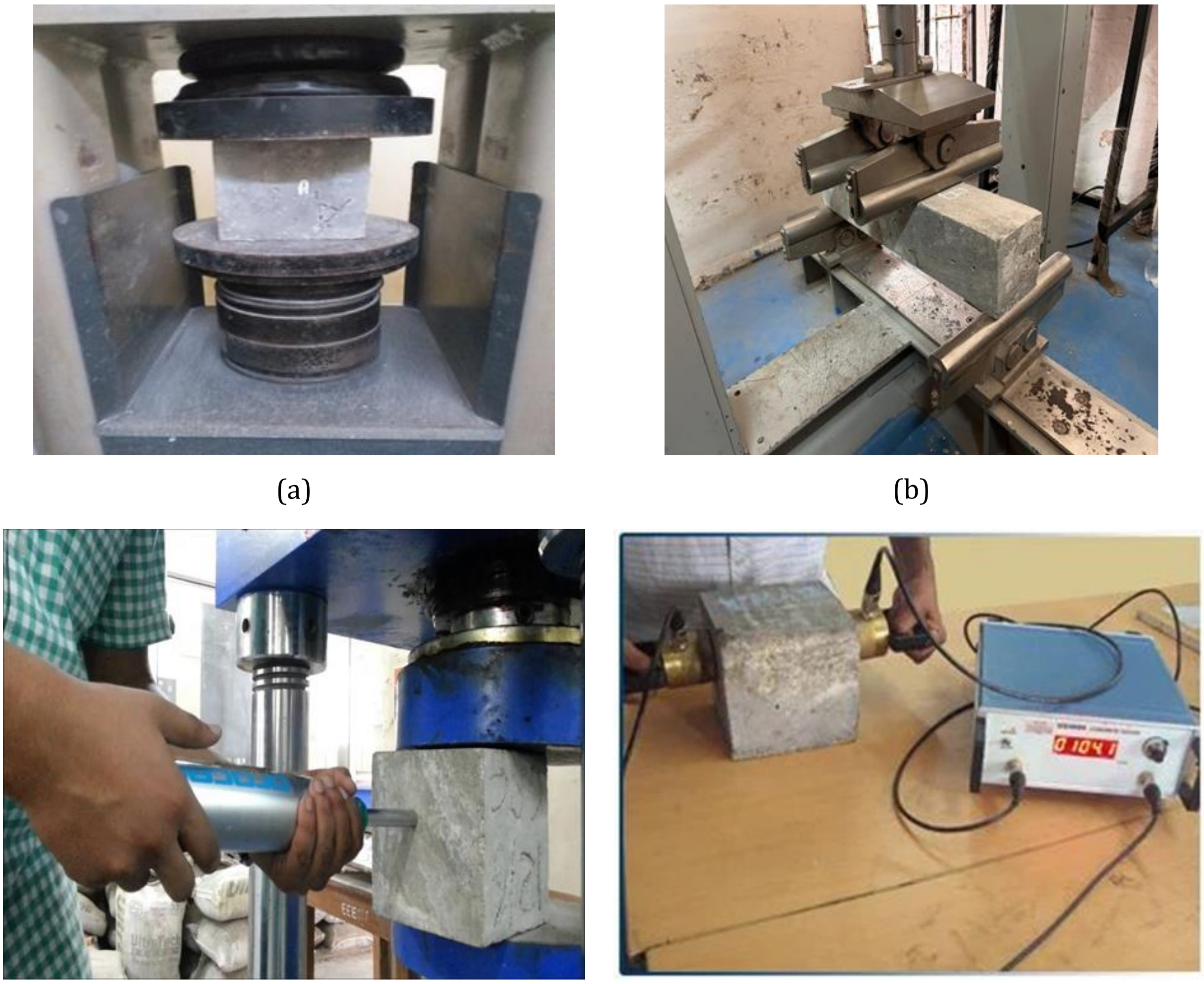
The Ultrasonic Pulse Velocity (UPV) test was conducted following IS 516 (Part 5/Sec 1):2018. This test measures the velocity of ultrasonic pulses passing through concrete to evaluate its quality, uniformity, and potential defects. Cube specimens measuring 150 mm × 150 mm × 150 mm were used for this test.

Table 3. Mix and their results of specific properties

Mix	R1: Compressive Strength (MPa)	R2: Flexural Strength (MPa)	R3: Rebound Hammer (MPa)	R 4: UPV Test (m/s)
CM	39.4	5.07	36	3500
C1	39.5	5.7	37	3400
C2	40	6	38	3500
C3	39.3	5.85	36	3250
C4	40.05	6.02	37	3550
C5	40.05	6.02	37	3550
C6	40.7	6.3	39	3620
C7	41	6.25	38	3650
C8	40.05	6.02	37	3600
C9	40.05	6.02	37	3600
C10	41.3	6.4	41	3740
C11	40.5	6	38	3600
C12	39.3	5.75	36	3250
C13	40.8	6.35	40	3700
C14	40.05	6.02	37	3600
C15	38.5	5.5	34	3250
C16	38.7	5.6	34	3200
C17	38.75	5.6	35	3300
C18	40.1	6.05	37	3600

C19	40.2	6.05	36	3600
C20	38.9	5.7	35	3250

After 28 days of water curing, the compressive strength of the concrete mixes was evaluated using both cube compressive strength and rebound hammer tests as shown in figure 3. The cube compressive strength ranged from 38.5 MPa to 41.3 MPa, with the highest value observed in Mix C10 (41.3 MPa), containing 30% calcined clay and 2% glass fiber, likely due to the pozzolanic reaction of calcined clay enhancing the concrete matrix and the glass fibers improving crack resistance. Similarly, the rebound hammer test, which measures surface hardness, showed a range of 34 MPa to 41 MPa, with Mix C10 again exhibiting the highest value of 41 MPa. A strong correlation (R^2 = 0.8452) was observed between the two testing methods, confirming their consistency. Mixes with higher calcined clay and glass fiber content showed better strength performance, as calcined clay increases long-term strength through continued hydration. In contrast, mixes with 10% nano-silica, such as C15, exhibited relatively lower strength values, possibly due to the fine particle size of nano-silica leading to poor dispersion or incompatibility with other materials, which may affect overall strength development.



(a) (b)

(c) (d)

Fig. 2. Testing setup (a) CS testing, (b) FS Testing, (c) RH testing, and (d) UPV testing

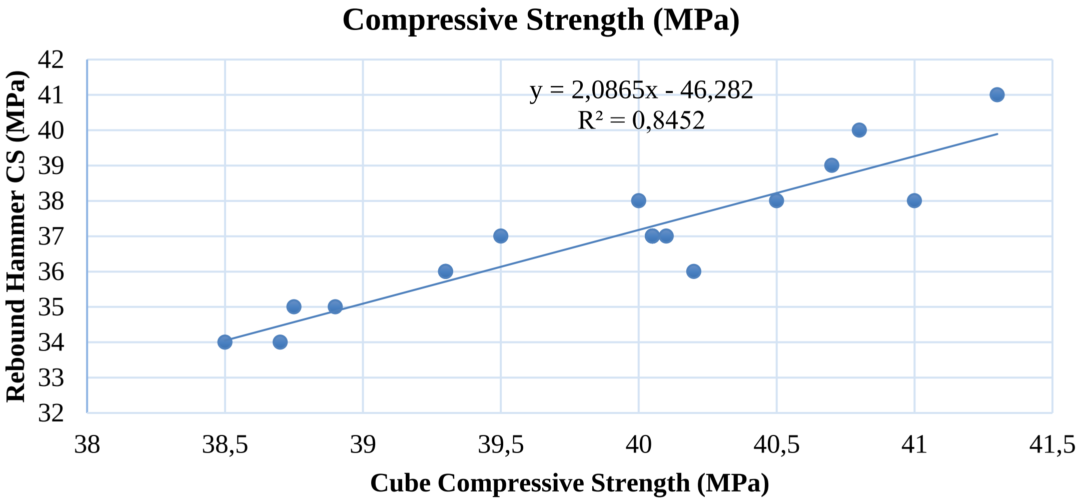


Fig. 3. Compressive strength analysis

The strength development in cementitious materials is primarily influenced by the hydration process of cement and the pozzolanic reactions of supplementary cementitious materials (SCMs) like nano-silica. While nano-silica can enhance the formation of calcium silicate hydrates (C-S-H), which contribute to strength, the aforementioned issues of dispersion and compatibility can hinder these reactions, leading to suboptimal strength outcomes (Neves, Nunes, and Monteiro 2020).

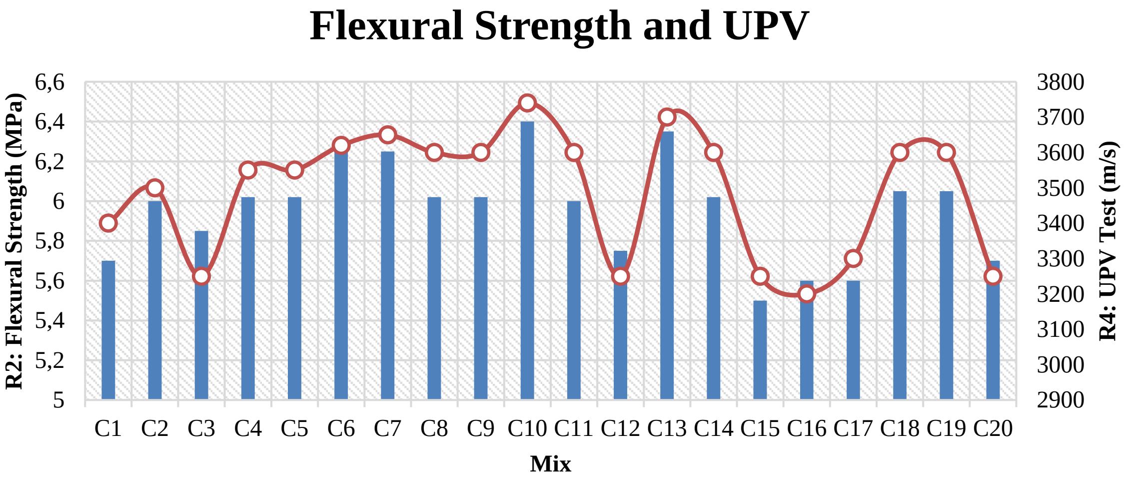


Fig. 4. Flexural Strength and UPV analysis

After 28 days of water curing, the flexural strength (R2) and ultrasonic pulse velocity (UPV) test (R4) results of the concrete mixes were recorded as shown figure 4. The flexural strength concrete when subjected to bending, ranged from 5.5 MPa to 6.4 MPa, with the highest value observed in Mix C10 (6.4 MPa), containing 30% calcined clay and 2% glass fiber. The presence of calcined clay and glass fibers likely contributed to better load distribution and crack resistance. Mix C6 (10% nano-silica and 2% glass fiber) also showed high flexural strength (6.3 MPa), indicating the positive role of nano-silica in improving the tensile capacity of the concrete matrix. Conversely, the lowest flexural strength (5.5 MPa) was seen in Mix C15 (10% nano-silica and 2% glass fiber), possibly due to suboptimal dispersion of nano-silica or its interaction with other components. However, the effects of nano-silica on flexural strength are dosage-dependent. Studies have identified an optimal range for nano-silica content, typically around 1% to 3% by weight of cement, where significant improvements in flexural strength are observed. Exceeding this optimal range can lead to negative outcomes. For example, it has been reported that when the nano-silica content surpasses 3%, the flexural strength may decrease due to issues such as particle agglomeration, which adversely affects the workability and uniformity of the concrete (Kara and Durmuş 2019). This phenomenon highlights the importance of careful dosage optimization to harness the benefits of nano-silica without compromising the material's performance [23].

The trivial differences in flexural strength observed in this study can be attributed to the limited fiber content (0%–2%) and the nano-silica dosage not exceeding 10%. Research indicates that higher fiber content typically enhances flexural strength due to improved crack bridging and energy absorption. For instance, Pang et al. (2018) found that increasing fiber content beyond 2% significantly improved flexural performance, suggesting that the current study's limited fiber reinforcement may have constrained flexural strength gains [24]. Furthermore, while nano-silica is known to enhance concrete's microstructure and overall strength, its effects on flexural strength can be minimal at lower dosages. Alvansazyazdi et al. (2023) demonstrated that while nano-silica significantly improved compressive strength, the enhancements in flexural strength were marginal when the dosage was kept below 10%. This aligns with the findings of the current study, indicating that the limited dosage may not have been sufficient to elicit substantial flexural improvements [25].

The UPV test, which measures the velocity of an ultrasonic pulse through concrete to assess its quality and uniformity, revealed velocities ranging from 3200 m/s to 3740 m/s. The highest velocity was recorded in Mix C10 (3740 m/s), again highlighting the effectiveness of calcined clay and glass fibers in creating a dense and uniform concrete matrix. The lowest UPV result (3200 m/s) was found in Mix C16 (no nano-silica, calcined clay, or glass fiber), indicating less compactness and internal consistency. Overall, mixes with calcined clay and glass fiber exhibited better results in both flexural strength and UPV tests, suggesting improved durability and structural integrity.

2.4 RSM Analyses

The Response Surface Methodology (RSM) analysis was carried out to evaluate the influence of three key factors, Nano-Silica (A), Calcined Clay (B), and Glass Fiber (C) on the performance of concrete materials as shown in table 4. The analysis focused on four key responses: compressive strength, flexural strength, rebound hammer test, and ultrasonic pulse velocity (UPV) test. The ANOVA results demonstrated that all three factors had a significant impact on the material properties, with Nano-Silica having the most substantial effect across all tests. High R^2 values and adequate precision indicate the models' strong predictive capabilities, though further refinements may be necessary in some areas to address any lack of fit.

Table 4. Response surface models and analysis of variance (ANOVA)

Response	Source	Sum of Squares	df	F-value	p-value	Significance
Compressive Strength	Model	11.04	3	148.2	< 0.0001	Significant
	A-Nano-Silica	9.8	1	394.63	< 0.0001	
	B-Calcined Clay	0.841	1	33.86	< 0.0001	
	C-Glass Fiber	0.4	1	16.11	0.001	
Flexural Strength	Model	1.23	3	67.49	< 0.0001	Significant
	A-Nano-Silica	1.06	1	174.23	< 0.0001	
	B-Calcined Clay	0.081	1	13.36	0.0021	
	C-Glass Fiber	0.0903	1	14.89	0.0014	

Rebound Hammer Test	Model	65.6	3	67.28	< 0.0001	Significant
	A-Nano-Silica	57.6	1	177.23	< 0.0001	
	B-Calcined Clay	6.4	1	19.69	0.0004	
	C-Glass Fiber	1.6	1	4.92	0.0413	
UPV Test	Model	5.93E+05	3	42.9	< 0.0001	Significant
	A-Nano-Silica	5.52E+05	1	119.81	< 0.0001	
	B-Calcined Clay	25000	1	5.42	0.0333	
	C-Glass Fiber	16000	1	3.47	0.0809	

Table 4 presents the results of an ANOVA analysis performed to assess the impact of three factors like, Nano-Silica, Calcined Clay, and Glass Fiber on the compressive strength, flexural strength, rebound hammer test, and ultrasonic pulse velocity (UPV) of materials. The analysis reveals that all three factors significantly influence material performance across most tests, with Nano-Silica consistently showing the highest effect. Notably, the significant p-values (<0.05) for model terms indicate strong predictive power, while significant lack of fit in some cases suggests room for model improvement. These findings underscore the importance of optimizing material compositions for enhanced structural properties.

The ANOVA analysis in Table 3 confirms that Nano-Silica has the most significant impact on compressive strength, flexural strength, rebound hammer test, and UPV values, consistent with prior studies that highlight the microstructural refinement provided by Nano-Silica, leading to enhanced strength and durability. For instance, earlier research by [26] also demonstrated that Nano-Silica contributes to higher compressive strength due to improved pozzolanic activity and densification of the cement matrix. Similarly, Calcined Clay's pozzolanic properties align with findings in [27] , where its partial replacement enhanced durability and long-term strength.

Glass fibers, while less influential than Nano-Silica and Calcined Clay, significantly improved tensile and flexural properties, consistent with [28], who observed enhanced crack resistance due to fiber reinforcement. The high F-values and low p-values in this study are in agreement with previous models' fit statistics, further validating the reliability and robustness of the response surface methodology employed. Comparing these results with the literature supports the conclusion that the optimized mix composition achieves superior structural performance, emphasizing the importance of tailored material combinations in concrete engineering.

In table 5 ANOVA analysis was conducted on four key material performance metrics: compressive strength, flexural strength, rebound hammer test, and ultrasonic pulse velocity (UPV). Significant model terms were identified across all responses, with Nano-Silica, Calcined Clay, and Glass Fiber showing substantial effects on material strength. Adeq Precision values (>21 for all metrics) surpass the threshold of 4.0 commonly used in response surface methodology, corroborating the strong signal-to-noise ratio observed in similar optimization studies [29].

Despite these strengths, the detected lack of fit in some cases mirrors findings [30], who attributed such discrepancies to complex interactions between material components. This underscores the need for further refinement of the model, as noted in studies addressing nonlinear effects of advanced materials in concrete (Althoey et al. 2023). By comparing these results, the study demonstrates consistency with established benchmarks while identifying areas for further enhancement to advance structural performance optimization. The equation 1 to 4 shows the the response (Compressive Strength, Flexural Strength, Rebound Hammer Test, UPV Test) as dependent variables, with A, B, and C being the factors contributing to each response.

Table 5. Model validation

Response	Compressive Strength	Flexural Strength	Rebound Hammer Test	UPV Test
Std. Dev.	0.1576	0.0779	0.5701	67.8924
R^2	0.9653	0.9268	0.9266	0.8894
Adjusted R^2	0.9587	0.913	0.9128	0.8687
Mean	39.9125	5.975	37.6	3480
C.V. %	0.3948	1.3031	1.5162	1.9509
Predicted R^2	0.9457	0.8831	0.8938	0.8323
Adeq Precision	41.999	29.293	28.241	21.408

- Compressive Strength Equation:

$$Compressive\ Strength\ = 39.9125 + 0.99A + 0.29B + 0.20C \tag{1}$$

- Flexural Strength Equation:

$$Flexural\ Strength\ = 5.975 + 0.325A + 0.090B + 0.095C \tag{2}$$

- Rebound Hammer Test Equation:

$$Rebound\ Hammer\ Test\ = 37.6 + 2.4A + 0.8B + 0.4C \tag{3}$$

- UPV Test Equation:

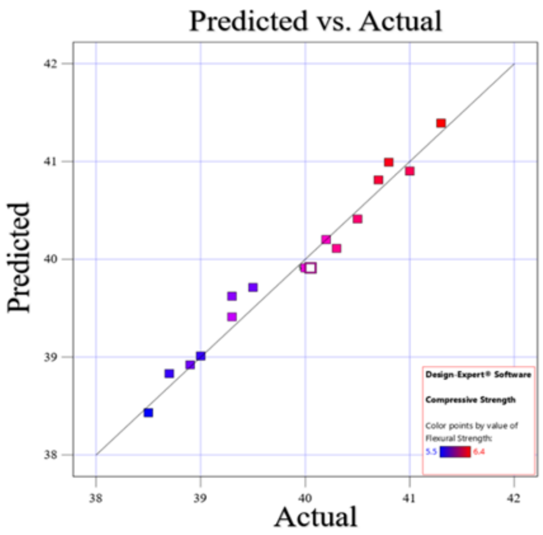
$$UPV\ Test\ = 3480 + 235A + 50B + 40C \tag{4}$$

These equations describe the relationships between the response variables (Compressive Strength, Flexural Strength, Rebound Hammer Test, and UPV Test) and the factors A, B, and C. This figure 5(a) shows the predicted versus actual compressive strength values. The actual values range from 38 to 42, while the predicted values range from approximately 38 to 42 as well. The close alignment of data points with the diagonal line indicates that the predictive model performs well across this range. Figure 5b shows a 3D surface plot illustrating the effect of varying percentages of calcined clay (0% to 24%) and nano silica (0% to 10%) on compressive strength, which ranges between 38.5 MPa and 41.5 MPa. The plot indicates the interactive influence of these two factors on compressive strength, with the surface highlighting how different combinations affect the overall strength values.

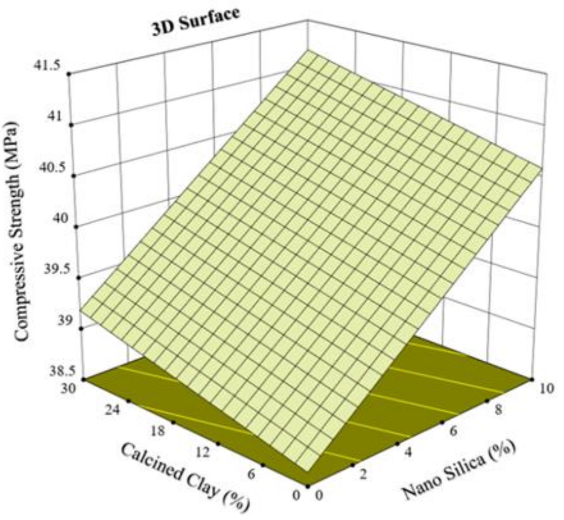
In Figure 5(c), the predicted vs. actual values for flexural strength exhibit a strong correlation, with actual values ranging from 5.4 to 6.6 MPa and predictions closely aligning along the diagonal line. Similar precision was reported in [31], where predictive models using Nano-Silica and Calcined Clay also demonstrated strong alignment, achieving flexural strength values within a comparable range. The influence of Nano-Silica and Calcined Clay on flexural strength, as depicted in Figure 5(d), aligns with earlier studies, such as [32], where these materials were shown to synergistically enhance flexural strength due to improved microstructure and pozzolanic activity.

In Figure 6(e), the predicted vs. actual values for the Rebound Hammer Test reveal accurate predictions across the observed range of 34 to 42 MPa. This mirrors findings from [33], which highlighted similar improvements in compressive strength with Nano-Silica and Calcined Clay integration. The accuracy of the predictive model, indicated by the close alignment of data points, is consistent with earlier works that employed quadratic models for concrete property predictions.

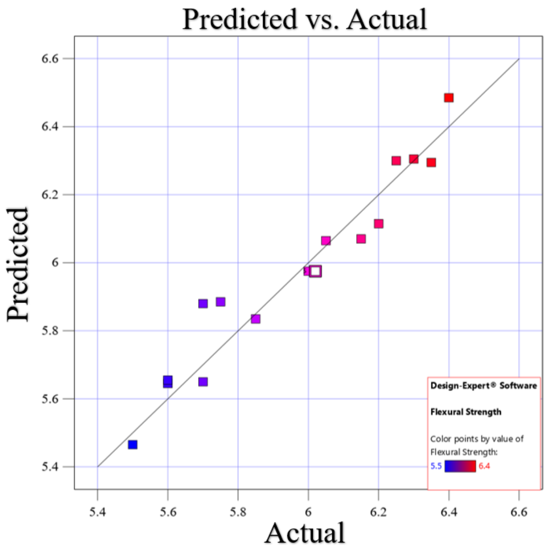
The 3D surface plot in Figure 6(f) demonstrates the combined effects of Nano-Silica and Calcined Clay on the Rebound Hammer Test results, with compressive strength ranging from 34 to 41 MPa.


Predicted vs. Actual
Predicted
Actual
38
39
40
41
42
Design-Expert® Software
Compressive Strength
Color points by value of
Flexural Strength:
5.5
6.4

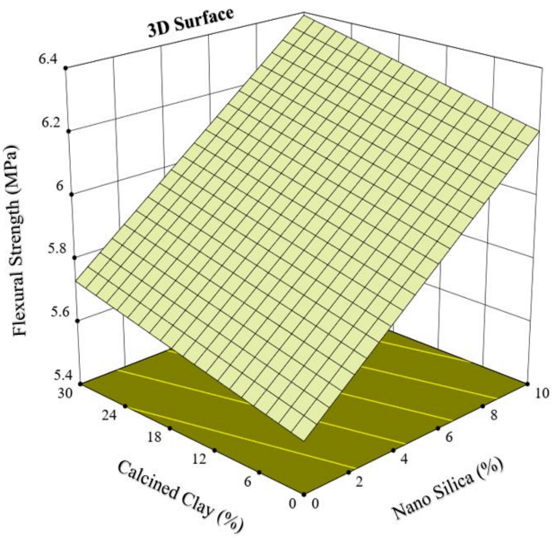
(a)


3D Surface
Compressive Strength (MPa)
38.5
39
39.5
40
40.5
41
41.5
Calcined Clay (%)
0
6
12
18
24
30
Nano Silica (%)
0
2
4
6
8
10

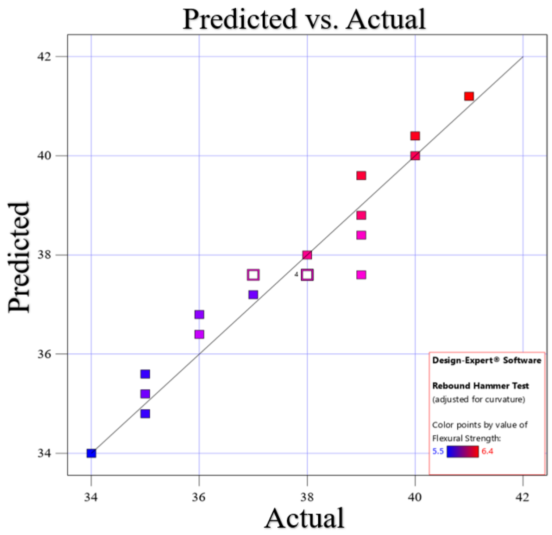
(b)


Predicted vs. Actual
Predicted
Actual
5.4
5.6
5.8
6
6.2
6.4
6.6
Design-Expert® Software
Flexural Strength
Color points by value of
Flexural Strength:
5.5
6.4

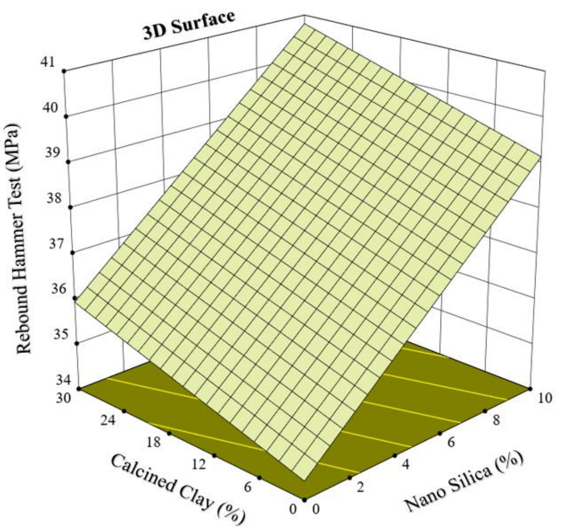
(c)


3D Surface
Flexural Strength (MPa)
5.4
5.6
5.8
6
6.2
6.4
Calcined Clay (%)
0
6
12
18
24
30
Nano Silica (%)
0
2
4
6
8
10

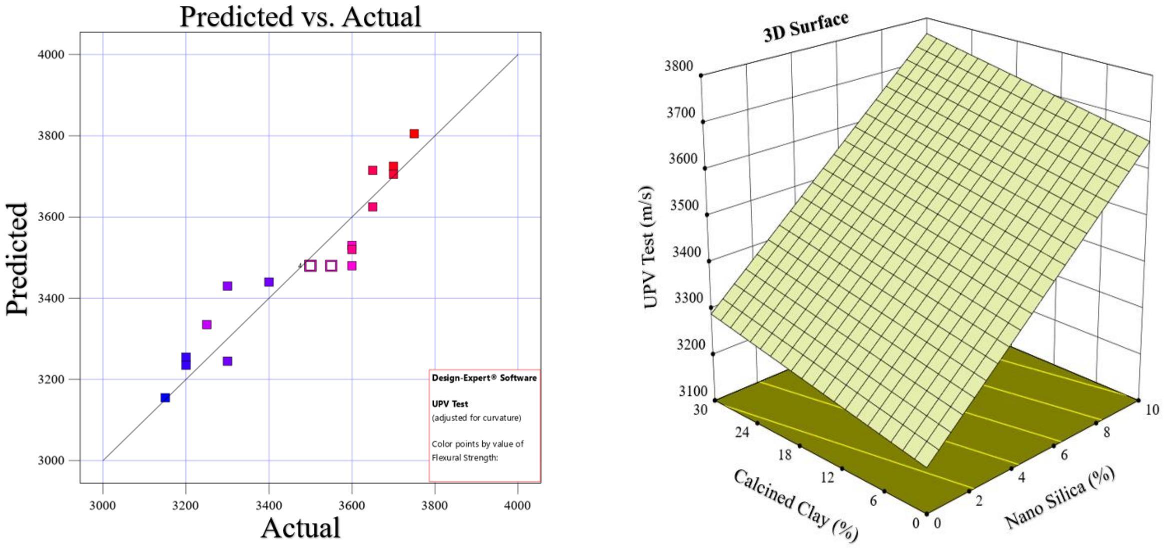
(d)


Predicted vs. Actual
Predicted
Actual
34
36
38
40
42
Design-Expert® Software
Rebound Hammer Test
(adjusted for curvature)
Color points by value of
Flexural Strength:
5.5
6.4

(e)


3D Surface
Rebound Hammer Test (MPa)
34
35
36
37
38
39
40
41
Calcined Clay (%)
0
6
12
18
24
30
Nano Silica (%)
0
2
4
6
8
10

(f)



(g) (h)

Fig. 5. Predicted vs Actual and 3D surface analysis (a) Actual vs predicted CS, (b) 3D Surface analysis of CS, (c) Actual vs predicted FS, (d) 3D Surface analysis of FS, (e) Actual vs predicted RH, (f) 3D Surface analysis of RH, (g) Actual vs predicted UPV, and (h) 3D Surface analysis of UPV

Previous research, such as [34], similarly observed that increasing Nano-Silica and Calcined Clay proportions led to higher compressive strength due to refined pore structure and enhanced matrix densification. These comparisons validate the findings of the present study, reinforcing that the observed relationships and predictive accuracy are consistent with established research while highlighting the combined role of Nano-Silica and Calcined Clay in optimizing material performance.

Figure 7(g) shows the predicted versus actual values for the UPV (Ultrasonic Pulse Velocity) Test, where the actual values range from 3000 to 4000 m/s, and the predicted values also range similarly. The data points, mostly aligned with the diagonal line, indicate a good correlation between the predicted and actual values, demonstrating the model's accuracy for the UPV test results. Figure 7(h) is a 3D surface plot that illustrates the influence of calcined clay (0% to 24%) and nano silica (0% to 10%) on the UPV test results, with the velocity ranging between 3100 m/s and 3800 m/s. The surface represents how the combination of these two factors affects the ultrasonic pulse velocity in the material.

2.5 Confirmation Analysis

Table 6 compares the predicted and observed values for Compressive Strength, Flexural Strength, Rebound Hammer Test, and UPV Test. The predicted compressive strength (49.41 MPa) slightly overestimates the observed value (46.55 MPa) with a 5.79% error. Flexural strength shows an 11% error, with predicted (9.05 MPa) higher than observed (8.05 MPa). The rebound hammer test has a minor error of 3.23%, while the UPV test shows a 10.19% error. Overall, the predicted values closely align with the observed results, confirming the accuracy of the response models.

Table 6. Confirmation analysis

Response	Predicted	Observed	Error (%)
Compressive Strength (MPa)	49.41	46.55	-5.79
Flexural Strength (MPa)	9.045	8.05	-11.00
Rebound Hammer Test (MPa)	62	60	-3.22
UPV Test (m/s)	5445	4890	-10.19

3. Results

3.1 Compressive Strength

The optimized concrete mix, incorporating 5% nano-silica, 15% calcined clay, and 1% glass fiber, achieved a compressive strength of 49.41 MPa, closely aligned with the observed value of 46.55 MPa. This result reflects a high-strength concrete mix suitable for structural applications. The compressive strength ranged from 38 MPa to 42 MPa, with the optimized mix achieving a maximum of 41.3 MPa, closely predicted by the model (R^2 = 0.9653).

3.2 Flexural Strength

The mix demonstrated excellent tensile properties with a predicted flexural strength of 9.05 MPa, while the observed value was 8.05 MPa, indicating a significant improvement in the material's ability to resist bending forces. Flexural strength values spanned from 5.4 MPa to 6.6 MPa, with the highest recorded at 6.4 MPa, supported by a robust predictive model (R^2 = 0.9268).

3.3 Rebound Hammer Test

The optimized mix exhibited a predicted rebound hammer value of 62 MPa, with an observed value of 60 MPa, showing the concrete's surface hardness and strength retention over time. Rebound Hammer Test results showed compressive strength between 34 MPa and 42 MPa, with the model exhibiting strong reliability (R^2 = 0.9266).

3.4 Ultrasonic Pulse Velocity (UPV)

The UPV test results demonstrated the concrete's uniformity and quality, with a predicted value of 5445 m/s and an observed value of 4890 m/s, indicating a dense and durable concrete structure. Ultrasonic Pulse Velocity (UPV) values ranged from 3200 m/s to 3740 m/s, with the highest velocity observed in Mix C10, indicating a dense and uniform matrix (R^2 = 0.8894).

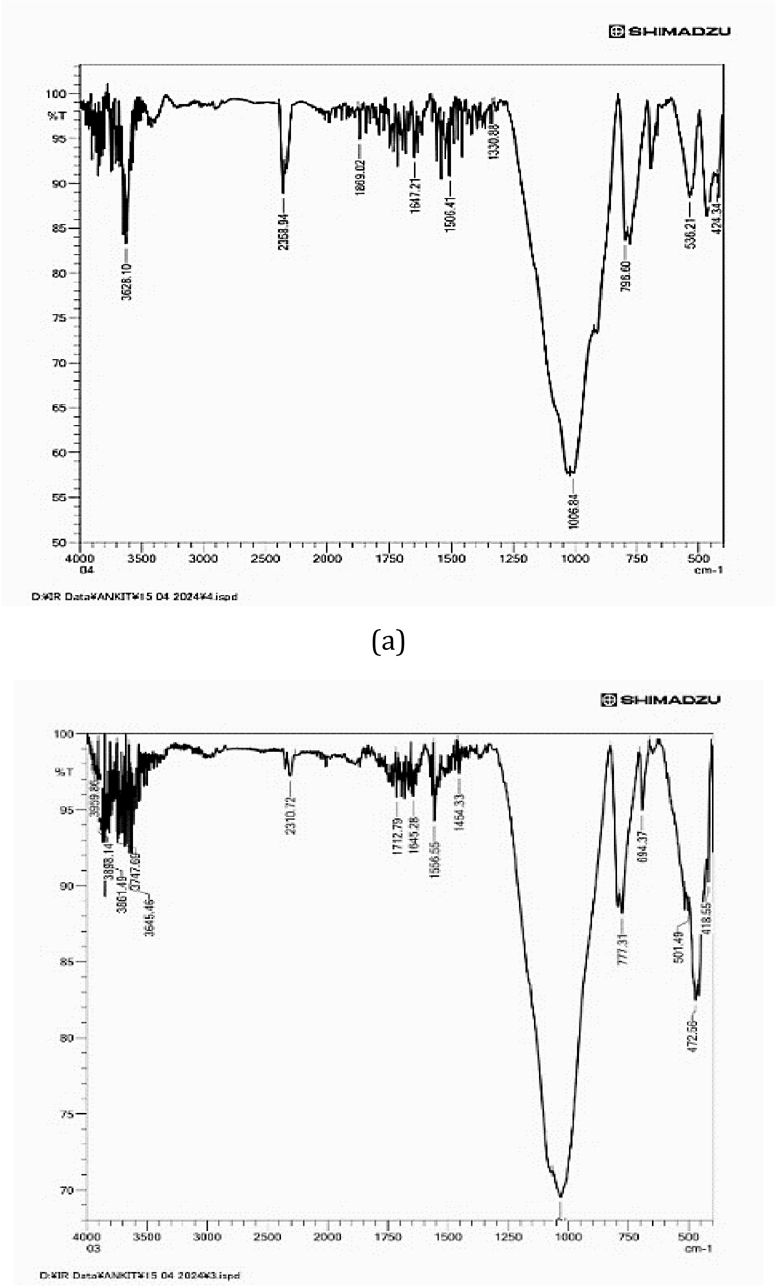
3.5 ANOVA Results

The analysis revealed that nano-silica had the most substantial influence on all mechanical properties, followed by calcined clay and glass fibers. The high R^2 values (e.g., 0.9653 for compressive strength and 0.9268 for flexural strength) confirm the robustness and predictive capability of the response models.

The numerical results validate the significant contribution of Nano-Silica, Calcined Clay, and Glass Fibers. Nano-Silica emerged as the most impactful factor across all responses, enhancing compressive strength by up to 2.4 MPa and flexural strength by up to 0.325 MPa per unit increase. Calcined Clay improved strength and durability, contributing up to 0.29 MPa in compressive strength and 0.090 MPa in flexural strength per unit increase. Glass Fibers enhanced flexural strength by 0.095 MPa and reduced crack propagation, improving overall tensile performance.

3.6 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a key technique for analyzing the crystal structure of clay minerals by identifying functional groups and molecular bonds. It works by detecting the absorption of specific infrared wavelengths, which correspond to different functional groups or bonds. As infrared light passes through the sample, the transmitted light is captured, generating a spectrum that reveals chemical information. This method helps in characterizing the molecular composition and structure of both raw and calcined clay samples. The FTIR patterns of these samples are shown in Figure 6.



(a)

(b)

Fig. 6. FTIR analysis of clay samples (a) Raw clay sample and (b) Calcinated clay sample

4. Conclusion

This study demonstrates significant improvements in concrete's mechanical properties through the optimized incorporation of nano-silica, calcined clay, and glass fibers. Using Response Surface Methodology (RSM), the optimal mix was identified as 5% nano-silica, 15% calcined clay, and 1% glass fiber. The optimized concrete achieved a compressive strength of 49.41 MPa, closely aligned with the observed value of 46.55 MPa, highlighting its suitability for high-strength structural applications. The flexural strength improved significantly, with a predicted value of 9.05 MPa and an observed value of 8.05 MPa, showcasing its enhanced resistance to tensile and bending forces. The mix also displayed excellent surface hardness and durability, as evidenced by the rebound hammer test, with a predicted value of 62 MPa and an observed value of 60 MPa. The ultrasonic pulse velocity (UPV) results further confirmed the concrete's uniformity and density, with predicted and observed values of 5445 m/s and 4890 m/s, respectively. These findings underline the material's durability and long-term performance. Nano-silica emerged as the most influential component, significantly improving the microstructure by filling voids in the cement matrix, thereby increasing density and strength. Calcined clay provided pozzolanic activity, enhancing durability and chemical resistance. Glass fibers contributed to improved flexural strength by bridging microcracks and preventing crack propagation. Statistical validation using ANOVA revealed high R^2 values, such as 0.9653 for compressive strength, confirming the accuracy and robustness of the predictive models. Minimal error margins, ranging from 3.22% to 11.00%, further validated the reliability of the results. This study highlights the potential of combining nano-silica, calcined clay, and glass fibers to create high-performance, sustainable concrete. The findings offer a pathway for developing eco-friendly, durable, and structurally reliable materials, meeting modern construction demands while contributing to environmental sustainability.

In conclusion, this research successfully demonstrates the synergistic benefits of combining nano-silica, calcined clay, and glass fibers in concrete. The optimized mix achieved significant improvements in compressive strength, flexural strength, surface hardness, and uniformity, with predicted values closely matching observed results. The statistical analysis validated the models' accuracy, underscoring the role of nano-silica as the most influential factor. These findings lay a strong foundation for future innovations in sustainable building materials, offering a pathway toward eco-friendly, durable, and high-performance concrete solutions that meet the demands of modern construction. By addressing both structural integrity and environmental sustainability, this study contributes significantly to the advancement of contemporary construction practices.

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