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EFFECT OF NANO SILICA ON STANDARD CONCRETE – AN EXPERIMENTAL APPROACH

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ABSTRACT

The mechanical properties of the concrete can be improved by adding nanoparticles. This research investigates the mechanical strength and durability of nanomaterial concrete using nano silicon dioxide as partial cement substitute. M45 grade concrete with 1% - 4% nano silicon dioxide and a water-to-cement ratio of 0.40 was tested. The optimal result was achieved with 4% nano silicon dioxide. Mechanical tests such as density, compressive, split tensile, flexural strength and microstructural analysis such as X-ray diffraction and scanning electron microscpe were conducted .

Keywords: Nano Silicon Dioxide, Flexural Strength, Split Tensile Strength, Scanning Electron Microscope.

1. INTRODUCTION

In 1959, during the Meeting of the American Physical Society at CalTech, the physicists Richard Feynman gave his famous speech entitled "There's plenty of room at the bottom," and thus, the new nanotechnology era begun. Feynman presented the idea of modifying and controlling matter at the scale of individual atoms and molecules. However, it was only in 1974 when the term "nanotechnology" was created by Norio Taniguchi and was defined as processing materials by one atom or by one molecule. Since then, the definition of "nanotechnology" has been modified several times over the years. Today, it can be defined as "the application of scientific knowledge to manipulate, control and restructure matter at the atomic and molecular level in the range of 1-100 nm to exploit the size-dependent and structure-dependent properties and phenomena distinct from those at different scales." Basically, nanotechnology is based on the statement that we can change any property of any material with reducing at latest one dimension of this material into the nano scale.

Nano technology hass the potential to enhance basic construction material such as cement, concrete and steel. By adding nanoparticles, concrete can become stronger, more durable, self healing, air purifying, fire resistant, easy to clean and compact more quickly.

Numerous studies have been conducted utilizing nano materials in concrete. Here are some key points of the previous research nanomaterial concrete. In the ultra–high performance concrete system, the C-S-H seeds demonstrated a stronger early strength enhancement than nanosilica. At the dosage of 0.3% weight, Nano silica and C-S-H seeds increased the 1 day compressive strengthof ultra high performance concrete samples by 25% and 57% respectively. The addition of nanomaterials led to an increase in the amount of hydration products in ultra-high performance concrete within one day. However, dut to the pozzalanic reaction of nano silica, the calcium hydrate content in the sample with nano silica was instead reduced (Zhongtau Luo et al. 2023). The effect of nanomaterials such as carbon nanotubes, nano silica and graphene oxide on bond behaviour of concrete and reinforcing bars. 0% Ground Copper Slag (GCS) increased by about 6.4% compared to OPC and 0.04% Ground Copper Slag. As a result of pull – out , it showed that slip decreased according to Graphene Oxide corporation (Dongsun Hwangbo et al. 2023). Using 2 % Double Hooked end (DHE) steel fibers led to 69% and 76% improvement in the splitting tensile strength in mixtures with 0% and 5% egg shell powder. The mixture with only 5% nano silica and with 2% double hooked end steel fibers depicted 89% higher splitting tensile strength (Osama Zaid et. al 2023). The optimal dosages for nano carbonate and nano silicon dioxide were 1.6% - 4.8% and 0.5% - 1.5%, respectively. If excessive content was added, the strength were even smaller than that of the reference samples. The nano silicon dioxide mainly contributed to the early strength development of UHSC before 7 days, while the nano carbonate led to significant strength increase between 7 and 28 days (R.Casas Zemei Wu et al. 2020). Silicon dioxide, titanium dioxide and alumina nanoparticle improves the bonding behaviour of concrete. The addtion of nanoparticles improves the microstructure of self-compacting concrete (Tien Dung Nguyen et al. 2017).

The addition of nanosilica can greatly enhance the strength and durability of concrete by improving the hydration of tricalcium silicate (C_3S) found in cement. This is achieved as the C-S-H gel expands into the capillary pores, binding the solid products and sealing any microcracks that may impact the structural integrity of the concrete . The exact mechanisms behind the improvement in hydration using nanomaterials have been studied extensively, and multiple theories have been proposed to explain their effectiveness. An example of a material with a significant pozzolanic impact is silica nanoparticles. They interact with calcium hydroxide (CH), which encourages the expansion of the C-S-H gel, resulting in an improvement. Additionally, because of its sizeable specific area, nanosilica can act as a reactive siliceous surface, aiding in early C-S-H growth. Moreover, silica nanoparticles can serve as seeds that facilitate cement hydration and densify the gel structure by blocking the voids between C-S-H particles and thereby increasing its stiffness .

Table 1: Test results of raw materials

Sr. No	Material	Property	Test results
1.	Cement	Initial Setting Time	44 min
		Final Setting Time	350 min
		Normal Consistency	27%
		Fineness through 90 micron sieve	7 %
		Specific Gravity	2.88
2.	Fine Aggregate	Zone	II
		Silt content	1.40%
		Specific Gravity	2.67
		Water absorption	0.90%
3.	Coarse Aggregate	Specific Gravity	2.69
		Water absorption	0.50%
4.	Nano Silicon dioxide	Si%	<95%
		Surface specific area	100 – 400 m^2/g
		Density	2.65 g/cm^3
		Alumina and Titanium dioxide present	Yes

Nanotechnology has the potential to enhance basic construction materials such as cement, concrete, and steel. By adding nanoparticles, concrete can become stronger, more durable, self-healing, air-purifying, fire-resistant, easy to clean, and compact more quickly.

2. MATERIALS AND METHODS

2.1 Ordinary Portland Cement (OPC 43), conforming to IS 8112 (IS 8112 2013) and IS 4031 (IS 4031 1996) part (1-6), was used for this investigation. Fine and coarse aggregates were used in research and tested as per code (IS 2386 1963 ; IS 383 2016). Sieve analysis of fine aggregate is shown in Fig.1 Superplasticizer (SP) 430 was used as a water-reducing agent. Table 1 represents the material data.

2.2 Mix- Proportion

A standard concrete with a compressive strength of 53.25 MPa for 28 days is designed as per IS code 10262 (IS 10262 2019). The methodology used for the present study is shown in Fig. 2. A proportion of five different mixtures was formed. The mixtures are denoted as C0, C1, C2, C3, C4 with weight % 0, 1, 2, 3 and 4 of the cement weight. The superplasticizer accounted for 1% of the binder's weight. The water-to-cement ratio (w/c) for every mixture remained constant at 0.40, as shown in Table 2. After casting all the mix specimen,it was cured for 28 days. Concrete specimens cured under controlled temperature of 20 and 25 °C and humidity conditions 95% or higher, which can be achieved by immersing them in water to avoid early-age cracking and to ensure that the desired mechanical properties are achieved. Compressive and split tensile strength tests were performed at 7, 14, and 28 days. Flexural strength, density and water absorption were measured of the samples at 28 days of curing. After testing the mechanical strength of all the samples, a microstructural test was performed, and samples of broken concrete was taken for the XRD and SEM test.

Table 2: Mix proportion of sample

Sr. No	Concrete	Cement (kg/m^3)	Coarse Aggregate (kg/m^3)	Fine Aggregate (kg/m^3)	W/C ratio	Water (kg/m^3)	Nano silicon dioxide	Super Plasticizer (kg/m^3)
1.	RC	450	1165.0	651.0	0.40	158.0	0.0	5.00
2.	C1	444	1165.0	651.0	0.40	158.0	4.5	4.1
3.	C2	430	1165.0	651.0	0.40	158.0	9.0	4.1
4.	C3	425	1165.0	651.0	0.40	158.0	13.5	4.1
5.	C4	415	1165	651.0	0.40	158.0	18.0	3.9

2.3 Experimental procedure

2.3.1 Workability, density and water absorption

As per IS code 1199 part 2 (IS 1199 (Part 2) 2018), the slump cone test was used to confirm the flowability of concrete samples. Fresh concrete was poured into a steel frustum that measured 300 mm in height, 200 mm in lower diameter and 100 mm in higher diameter. The mould must be removed from the concrete immediately by gently and cautiously vertically lowering it. This permits the concrete to sink, and the slump may be quickly quantified by calculating the difference between the mould's height and the concrete highest point (IS 7320 2008).

After 28 days, the weight of the concrete cube was measured. To calculate the density, the test specimen's weight was first determined and then divided by its volume as per eq. 1.

$$(1)\quad \text{Density} = \text{Mass} / \text{Volume}$$

A 150 mm cubic specimen that was 28 days old was employed in the water absorption test. After the wet concrete cubes were weighed, they were stored at 110 °C in an oven. Next, the dried sample weight was ascertained as per eq. 2

$$(2)\quad \text{Water absorption in \%} = (\text{Wet Concrete} - \text{Dry concrete}) / \text{Dry concrete}$$

2.3.2 Compressive strength

The capacity of a material or structure to support loads on its surface without cracking or deflecting is known as its compressive strength. According to IS 516-part 1, compression tests were performed on 150 mm concrete cube specimens at 7, 14 and 28 days using a compression testing machine. Each mixture's compressive strength was calculated by averaging the three cubic specimen's compressive test results (IS 516 2021).

2.3.3 Split Tensile Strength

The split tensile test was carried out in accordance with the IS 516-part 1 standard, utilizing cylindrical specimens that had a diameter of 100 mm and a height of 200 mm. Concrete tensile strength is far less tahn its compressive strength; tension stresses are carried by fibers in the concrete. The three samples were tested at ages 7, 14 and 28 days, with each sample being tested on a compression testing machine according to eq. 3.

$$(3)\ \text{Split Tensile Strength} = 2P / \pi LD$$

Where, P: maximum applied load, D: diameter of specimen and L: length of specimen.

2.3.4 Flexural Strength

The concrete beam specimens for concrete mixes measured 150 mm × 1 50 mm × 700 mm. Steel is employed to carry the tension forces in concrete because the material's tensile strength is significantly lower than its compressive strength. Tensile strength in concrete is thought to be equivalent to 10% of compressive strength (IS 456 2000) . The specimens were tested as supported structures with an effective span of 600 mm. The load was applied to two points, each 100 mm apart from the beam's centroid as per eq .4.

$$(4)\quad \text{Flexural strength} = PL/BD^2$$

where P: maximum applied load , B and D: lateral dimension specimen, and L: length of specimen.

2.3.5 Microstructural behaviour of concrete

XRD and SEM with energy dispersive spectrometer (EDS) techniques are used to analyse the microstructure of concrete. After determining the strength of each concrete mix, shards were collected to determine the microstructursl characteristics. The purpose of XRD is to figure out the structure of the phases of crystal-like substances. SEM gives a microscopic lens for exploring the complicated world of concrete, revealing concealed components, and helping to environmentally conscious building methods. We used XRD, Model : D8 Advance Eco, make: JEOL, to ascertain the microstructure property of concrete. Im XRD analysis, samples are scanned continuously at a rate of 2 theta per minute, ranging from 0 degree to 90 degree. The average particle size has been determined from this investigation using the Debye-Scherer formula, taking into account the peak at degrees. Bragg's Law is used to compute the d-space, or inter planar separation, between atoms are shown in eq. 5.

$$(5)\ \ D = 0.9\,\lambda/\,\boldsymbol{\beta}\cos\theta \ \text{ and } \ d = n\lambda\,/2\sin\theta$$

D = avg particle size (mm), Θ = diffraction angle, λ = wave length (0.1540 nm), β = FWHM (full width at half maximum), d = interplanar spacing, and n = order of reflection.

The concrete's crystalline phases and mineralogical makeup were examined using XRD, which aided in the identification of important phases such as calcium hydroxide (CH) and C-S-Hs. It shed light on the hydration process and the effects of additives such as nano silica, showing how they affected the development of denser, stronger phases, which were directly linked to better mechanical qualities. SEM also made it possible to image the microstructure of the concrete in high resolution, concentrating on the interfacial transition zone, fracture and pore formation, and fiber dispersion. SEM was able to clarify how effectively the fibers were implanted in the matrix and how they contributed to fracture bridging, which improved tensile and flexural strength, by analysing the fiber-matrix bonding.

3. RESULTS AND DISCUSSION

3.1 Flow ability

Figure 4 shows the flowability test results for the various blends. Adding nano silica reduced flowability in fresh concrete. In comparison to the flowability (134 mm) of the reference concrete, the addition of 1, 2, 3, and 4 wt. % nano silica reduces workability by approximately 6.71%, 10.44%, 29.1% and 15.67% respectively. This reduction in flowability is because the NP's tiny size an high surface area create agglomeration effects and raise the particle packing density (IS 1199 2018). The concrete's specific surface area has increased, which causes a decrease in flowability. The flowability progressively deteriorated as the dosage of nanomaterials rose.

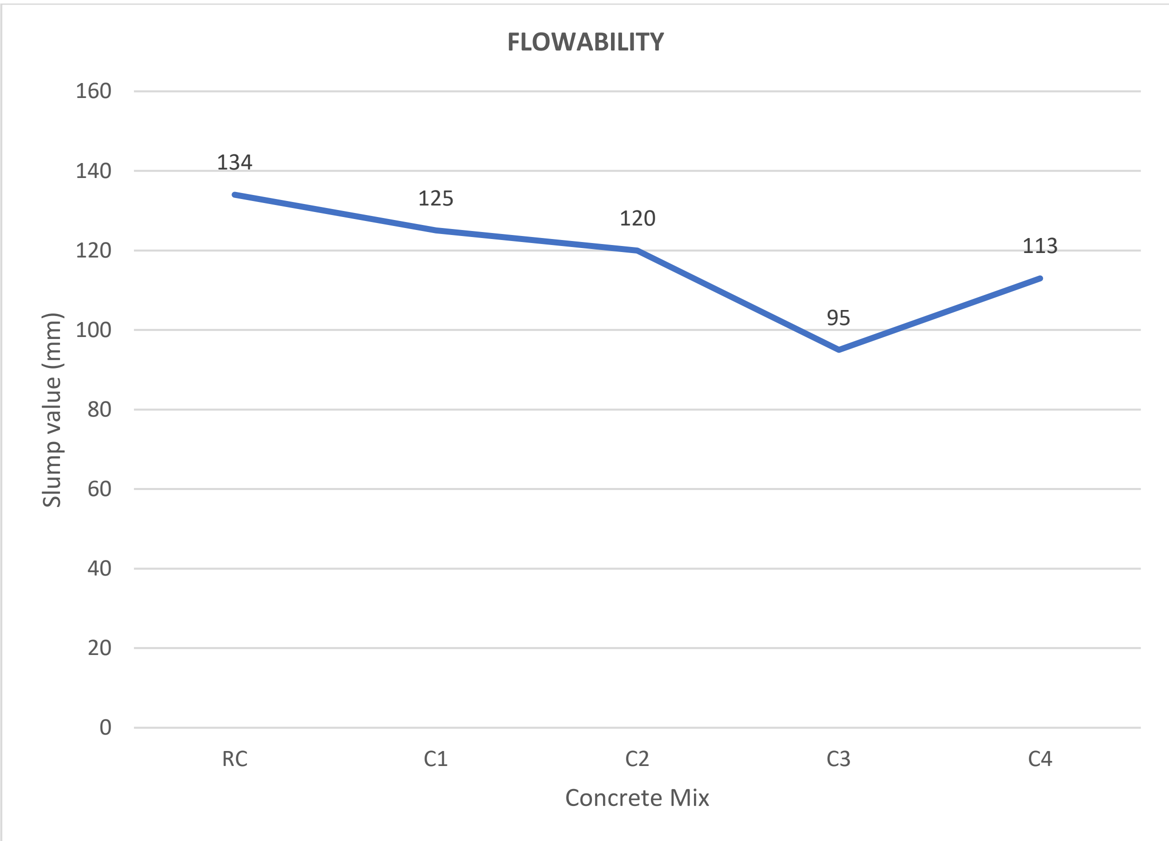

Fig 1: Workability of fresh concrete mix

The density test results for the different hardened concrete mixes are displayed i

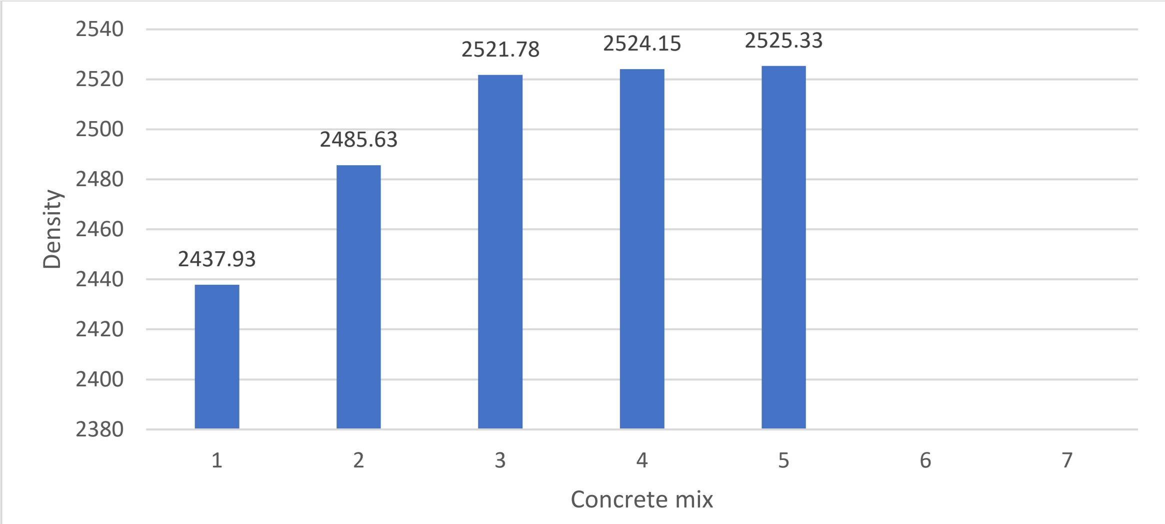

Fig 2: Density of hardened concrete mixes

Fig. The density of hardened concrete increased by approximately 1.96%, 3.44%, 3.53% and 3.58% respectively with the addition of nano silica 1%, 2%, 3% and 4%. Large surface area, micro size of nano silica and appropriately filled small pores in the concrete compared to reference concrete are the reasons for the higher density of naomaterial concrete (IS 1199 2018); Bhat and Vikram 2023).

3.2 Water Absorption

Figure depicts the differences in water absorption among the various hardened concrete mixtures. The observation is that there is a relationship between water absorption and compressive strength. With a decrease in compressive strength water absorption increases. Compared to reference concrete, the water absorption of concrete is reduced by approximately 32.16%, 24.9%, 25.98% and 33.84% when nano silica 1%, 2%, 3% and 4% are used. The small size of the NPs reduces water absorption, making the concrete more durable, resistant to damage, and able to fill its tiny pores.

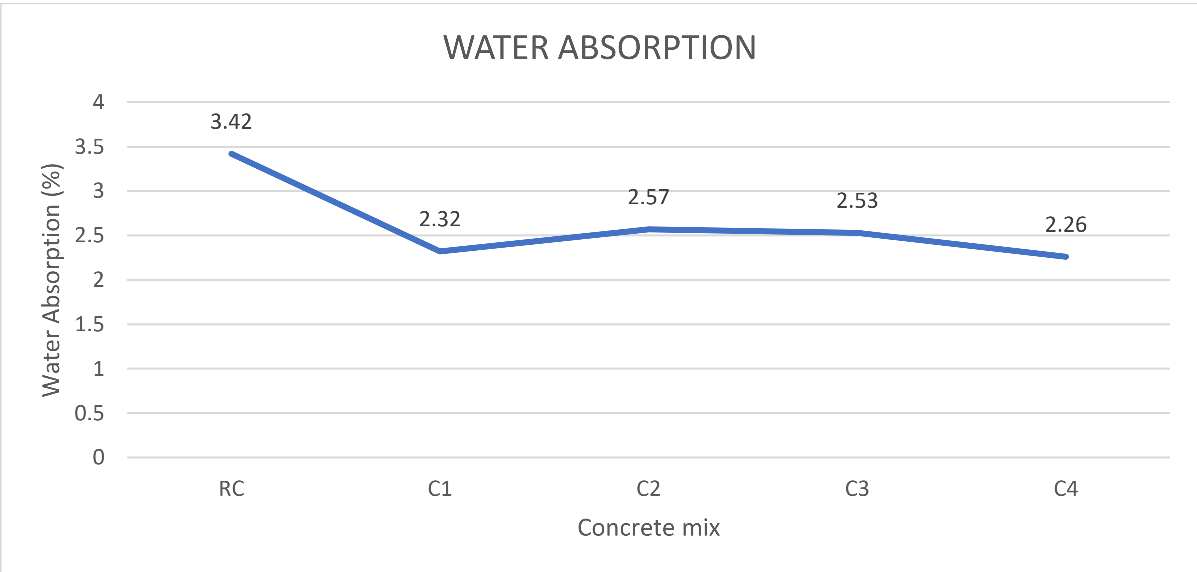

Fig 3: Water absorption of different concrete mix

3.3 Compressive strength results

The compressive strength of concrete is an indicator of its capacity to bear axial forces or pressures that compress or crush the material. The results of the compressive strength tests on concrete specimens containing nao silica at 7, 14 and 28 days were displayed in Table 3. Concrete mixes C1, C2 ,C3 and C4 with different percentages of nano silicon dioxide increase the compressive strength of concrete cubes compared to reference concrete. In comparison to reference concrete, it was found that concrete mixes C1, C2, C3 and C4 increased compressive load by approximately 15.61%, 18.85%, 14.14% and 3.83% respectively after 7 days. These strengths are enhanced by the much finer NP

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size, which fill the cement's tiny pores and increases density and weight, Seven days compressive strength is high when compared to 28 days strength because silicon dioxide immediately reacts with concrete and hardens it by forming CSH gel at an early stage.

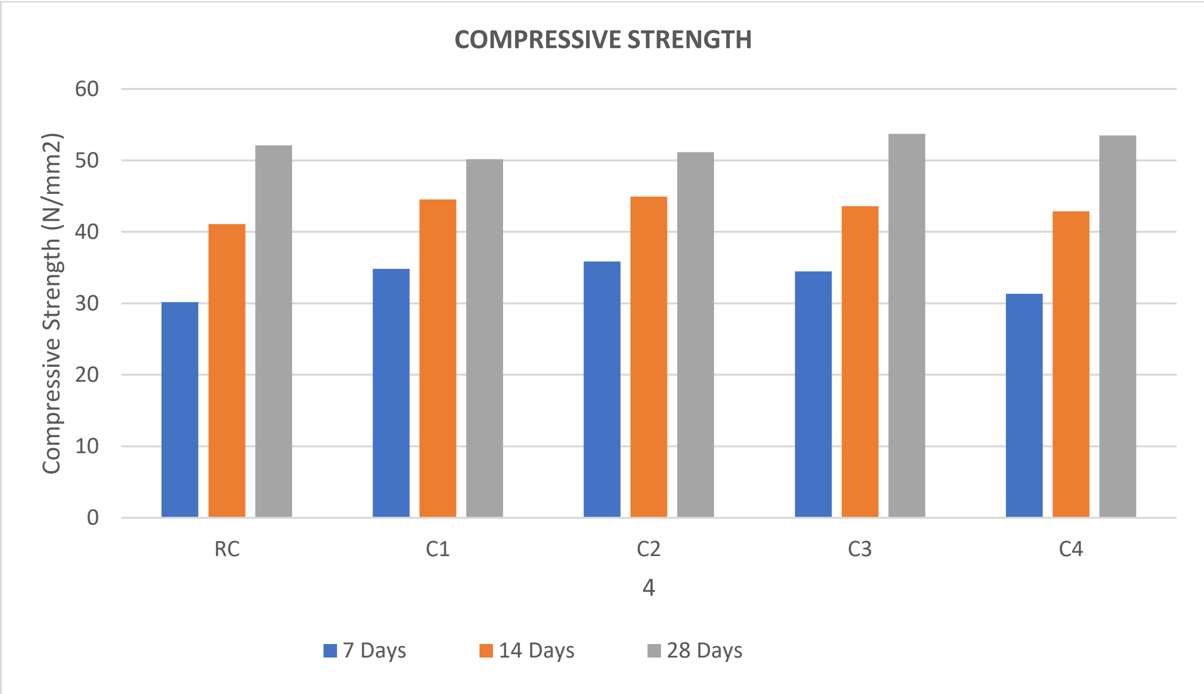

Fig 4: Compressive Strength of different concrete mixes

3.4 Split tensile strength results

Split tensile strength evaluates concrete's resistance to tensile forces applies perpendicular to the loading axis; figuring out the load when concrete members would fracture is critical (Table 4). Split Tensile Strength of different concrete mix illustrates the split tensile strength of the various concrete mix cylinders at 7,14, 28 days.

Table 4: Split tensile strength of concrete

	7 Days	14 Days	28 Days	7 Days	14 Days	28 Days
RC	5.88	6.01	6.98	0.00	0.00	0.00
C1	8.30	7.23	7.95	21.42	20.29	13.89
C2	8.30	8.33	8.67	16.24	15.21	9.05
C3	10.11	10.23	10.66	21.80	22.80	22.95
C4	10.04	10.17	10.12	-0.69	-0.58	-5.06

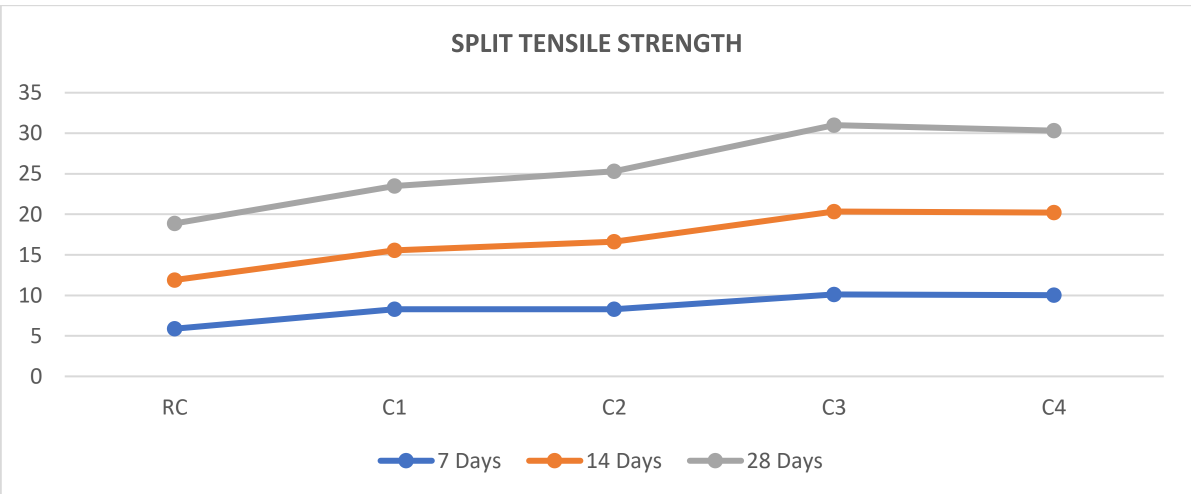

Fig 5: Split tensile strength of different concrete mix

According to the tensile strength data, adding nano silica to the concrete enhanced its split tensile strength. Concrete mixes C1, C2, C3 and C4 with increasing percentages of nano silica enhanced tensile strength by 2.31%, 5.38%, 4.62% and 3.85%, respectively, atv 28 days when compared to the reference concrete. Less pores and higher concrete cylinder density contribute to enhancing these strengths.

3.5 Flexural Strength

Table 5:

Concrete mix	Flexural strength (MPa) at 28 days	% increase in flexural strength
RC	4.63	0.00
C1	4.65	0.43
C2	4.66	0.21
C3	4.66	0.00
C4	4.65	-0.21

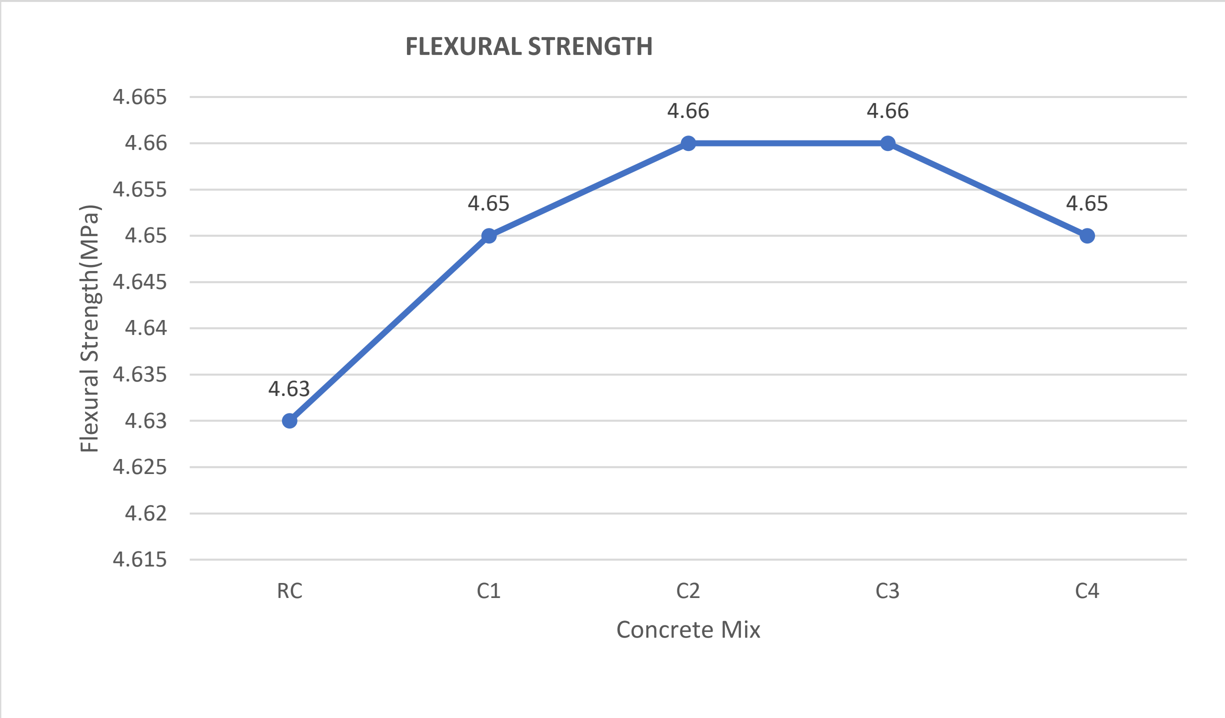

Fig 6: Flexural strength of different concrete mix

The modulus of rupture, also referred to as the flexural strength of concrete, is an indirect measure of the tensile strength or the stress in a material just before it yields in an unreinforced concrete flexure test. Understanding this strength is necessary to predict how concrete buildings behave under bending loads.

Concrete that has a high flexural strength can produce structures that are both sturdy and safe. Table 5 shows the concrete beam's flexural strength after 28 days of testing using a two point load flexural testing apparatus.

3.6 Microstructural behaviour

3.6.1 Scanning electron microscope with energy dispersive spectrometer

Numerous microstructure observation techniques (e.g., SEM, XRD, and TGA) were used to investigate the kinetics of nanomaterials in cementitious materials and their mechanism in densifying and compacting the concrete microstructure . The microstructure of concrete is primarily improved with nanoparticles through three effects, namely the nano-filler effect, pozzolanic reactivity, and the nucleation effect. The nanofiller effect is attributed to nanoparticle's ultra-small size and higher surface area, which helps them to occupy a higher percentage of fine voids in concrete. Nanoparticles work as crystal nuclei, promoting cement hydration. In addition, nanoparticles generate more C-S-H gel through their reaction with CH. These effects remarkably enhance the microstructural packing efficiency of concrete and subsequently improve the material's durability and mechanical strength SEM micrographs of the concrete matrice C4 are shown in Fig. that displays the elemental analysis of the concrete specimens. Concrete's porosity and water absorption were decreased by nano silica particles with small particle sizes. The mechanical characteristics of the concrete are directly correlated with the microstructural features shown in the SEM pictures. Higher compressive strength is associated with a denser matrix with smaller pores, as observed in specimens containing nano silica. This is because the reduced porosity improves the material's structural integrity and restricts the path for fracture propagation.

Several studies have shown that the addition of nanoparticles can greatly enhance the strength and durability of concrete by improving the hydration of tricalcium silicate (C_3S) found in cement. This is achieved as the C-S-H gel expands into the capillary pores, binding the solid products and sealing any microcracks that may impact the structural integrity of the concrete. The silica nanoparticles interact with calcium hydroxide (CH), which encourages the expansion of the C-S-H gel, resulting in an improvement. Because of its sizeable specific area, nanosilica can act as a reactive siliceous surface, aiding in early C-S-H growth. Moreover, silica nanoparticles can serve as seeds that facilitate cement hydration and densify the gel structure by blocking the voids between C-S-H particles and thereby increasing its stiffness.

3.6.2 X-ray diffraction analysis

XRD examination was performed on the powdered samples by pulverizing tiny fragments of nano sized silicon dioxide concrete samples. The primary mineral phases and the crystallite size and texture of cementitious materials during hydration could all be identified using the XRD pattern. The presence of significant hydration products in the form of amorphous silica, calcium carbonate, aluminium oxide, iron titanium oxide, portlandite, quartz, alite, belite and C-S-H with Inorganic Crystal Structure Database (ICSD) code numbers **************as represented by each of the peaks in the figure, along with the varying intensity peaks at diffrection angles between 10 and 90 degrees. As seen in Fig. 8, the prominent peaks **,**,**,**,** signify the existence of silicon dioxide. Accompanying these peaks are amorphous silica and aluminium oxide. Strong diffraction peaks at **,**,**,** were seen in the XRD patterns. These patterns were taken on day 28 of curing at an angle of 2Θ, which ranges from 10 to 90 degrees. The single phase XRD pattern

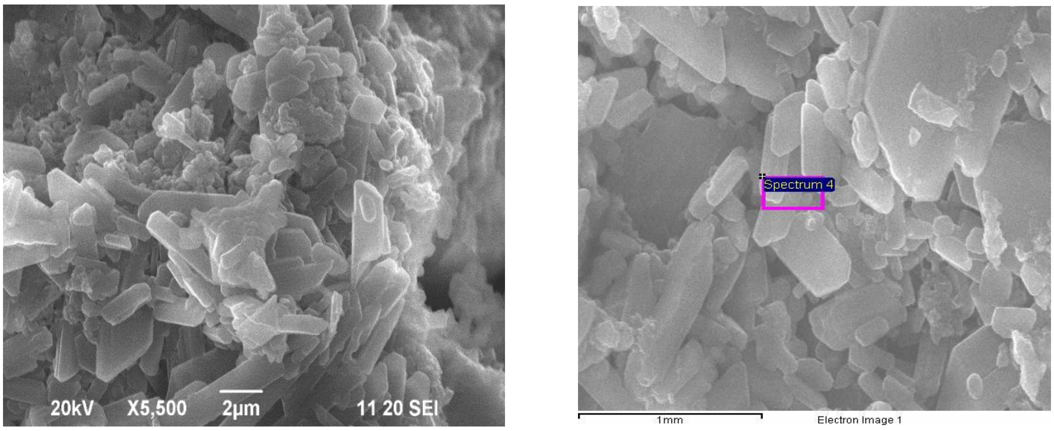

Fig a **Fig b**

Fig 7 a & b: Scanning Electron Microscope (SEM) images

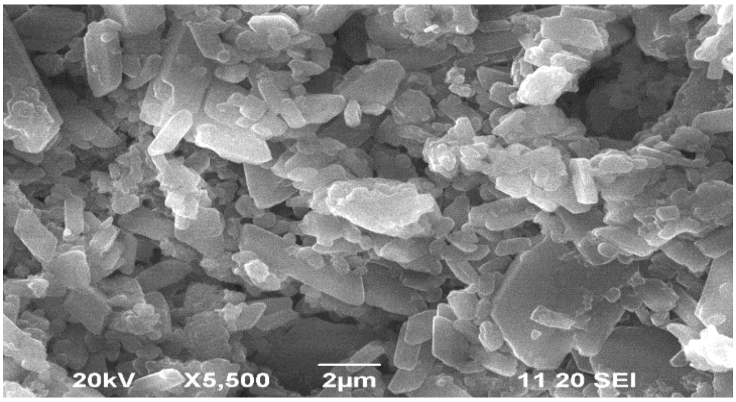

Fig 8: Scanning electron microscope of concrete sample

SEM has been achieved for silicon dioxide, titanium dioxide and calcium carbonate with cubic, hexagonal and rhombohedral structures respectively. The XRD analysis depicts the cryatallite size of C4 mix using Debye-Scherer formula as 59.9076, 59.39546, 70.40046, 76.9947 nm and d-spacing is 3.41445,4.157043, 1.378243 and 1.1823458 respectively. The crystallite sizes and d-spacing values found in concrete specimens are crucial for their mechanical

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properties (Niewiadomski et al 2017; Aguilar Rosero et al. 2023). A more finer microstructure is indiated by smaller crytallites, which enhance hydration, denser C-S-H formation, durability, flexural strength and compressive strength. Better stress distribution, decreased porosity and increased crack resistance are the outcomes of this. However larger crystallites indicate a more porous structure and incomplete hydration, which reduces tensile strength and increases the likelihood of breaking in concrete. A compact, well ordered structure that enhances stress transfer and resistance to deformation is indicated by smaller d-spacing values, whereas a weaker, more disordered structure that decreases strength and increases sensitivity to failure is suggested by larger d-spacing values.

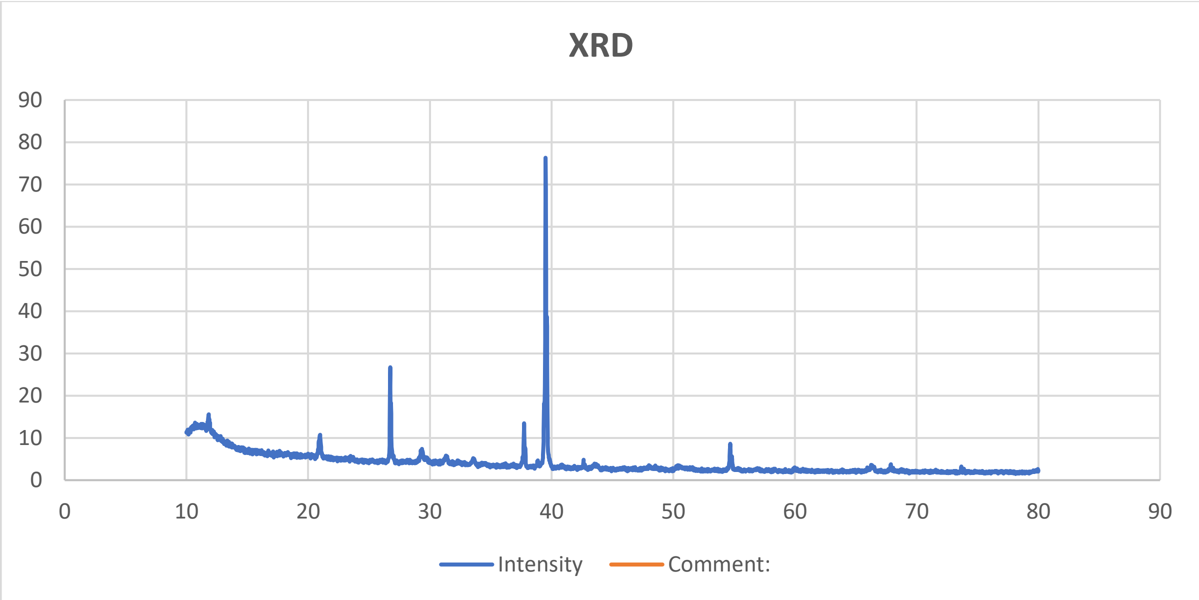

Fig 9: X-ray diffraction of Nano Silica

Fig 10: Casted concrete cubes

Fig. a **Fig. b**

Fig 11 a & b: Compresssive strength test of concrete sample

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Fig a **Fig. b**

Fig 12 a & b: Split Tensile Strength of concrete cylinders

Fig 13: Flexural Strength Test of Beam

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