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A STUDY ON THE DURABILITY CHARACTERISTICS OF CONCRETE USING NANO SILICA AS PARTIAL REPLACEMNT OF CEMENT

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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. The nanoconcrete cubes were then immersed in 5% conentrated sulphuric acd for a period of 28 and 56 days. The change in the strength of concrete was then noted. 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 Concrete, Nano Silica, Flexure Strength, Scanning Electron Microscope (SEM).

1. INTRODUCTION

Concrete is a widely utilized building material around the world, however its tensile strength is lowest in comparison to its compressive strength. Concrete has many pores of different sizes and shapes, which directly affect the various physical, chemical and mechanical properties. In today's construction scenario the use of nanomaterials as repacement of cement is gaining importance. There are many nano materials such as nano-silica, carbon nano tubes, nano titanium dioxide, nano- alumina, nano clays, graphene, etc. Nano silica is the most widely used nano material.It reacts with Calcium hydroxide to form C-S-H gel, improves strength and durability. Nano-silica and other reactive nanomaterials consume calcium hydroxide (CH), forming additional C-S-H gel and improving the matrix. Nanoparticles act as nucleation sites for hydration products, accelerating cement hydration. Extremely fine nanoparticle fill voids between cement grains, reducing porosity and permeability. Some nanomaterials (CNTs, graphene) bridge microcrack and delay crack propagation. There are many benefits of concrete such high strength, enhanced durability, improved workability, early strength gain.Various types of nanoconcretes has numerous applications such as in High-performance concrete (HPC) and high-strength concrete, self compacting concrete(SCC), marine and aggressive chemical environments, pavements, bridges, orecast structural elements.

The concrete structures needs to have a long life and should also be resistant to external agents like wind, water, chemicals, frost action, freezing and thawing. One of the main compositions of concrete is cement that imvolves high amount of CO_2 gas on production, which occur nearly 7% of the earth total CO_2 emissions (Isaboke et al. 2023). Several researches have been conducted for finding the alternatives for cement such as by the incorporation of fly ash, rice husk, metakaolin or nanoparticles (NPs) (Ghosal and Chakraborty 2021;Monteiro et al 2022;Yehualaw 2023). NPs used in concrete serves not only as additives but also as replacement of cement at optimum percentage to improve different properties of concrete due to their higher surface area and their chemical composition. Cement can be partly replaced with nano materials such as nano silica, nano titanium, carbon nano tubes, graphene oxide. The resulting concrete will have high strength, enhanced durability, improved workability, early strength gain, self cleaning, self-healing capabilities. Numerous studies have been done on the durability of concrete. Experimental study on concrete with the use of recycled sand andnano-materials on ultra high performance concrete by adding recycled sand 0%, 25% and 50% and different nano material 1%, 2%, 3% and 4% respectively. The results showed that use of 50% recycled sand reduced compressive sttrength but by using nanomaterial with recycled sand in concrete increased increased mechanocal and microstructural property(Feng et al.2023).

Li et al. (2006) reported that adding 1-3% nano-silica reduced water absorption and sorptivity significantly due to pore refinement. Jo et al. (2007) found that nasno-silica blended concrete had 20-30% lower permeability than control mix because of additional C-S-H gel formation.

Various chloride ion penetration studies such as Sanchez & Sobolev (2010) noted that nano concrete showed 35-50% reduction in chloride permeability measured by Rapdi Chloride Permeability Test. Mukharjee et al. (2014) demonstrated that 2% nano silica replacement decreased chloriode ion diffusion coefficient by nearly 40%, enhancing corrosion resistance. Fivi et al.(2010) concluded that nano-silica concrete lost 25-40% less weight after ommersion in sulphuric acid solution compared ton normal concrete. Abhilasah et al. (2018) found improved surface integrity of nanosilioca concrete exposed to 5% sulphuric acid for 60 days; SEM micrographs showed denser matrix and reduced microcracks. Sulphate attack resistance studies such as by Querica et al.(2012) reported lower expansion and mass loss in nano-silica modified concrete under sodium sulphate exposure. Sharma & Bhattacharjee (2015) concluded that the reduction in CH content due to pozzolanic reaction improves sulphate resistance. Freeze –Thaw and Carbonation resistance studies such as Mikhaik et al (2013) observed that nano-alumina and nano silica vlended concrete showed reduced freeze – thaw scaling and better reidual strength after 300 cycles. Patil et al. (2019) fond carbonation depth reduced by up to 40% in concrete with 2% nano-silica after 90 days accelerated carbonation test.

In the present study, workability, density and water absorption tests were performed on nano SiO_2 concrete without fiber. Mechanical strengths such as flexural strength, split tensile strength and compressive strength were determined after acidic curing of nanoconcretes. Microstructural analysis, including crystallite size, single phase, morphology, particle size and purity of nanoconcrete in acidic exporure condition, has been investigated through XRD, followed by scanning electron microscope (SEM) with EDS.

2. MATERIALS AND METHODS

2.1 Materials used

Ordinary Portland Cement (OPC 43), which conforms with IS 8112 (IS 8112 2013) and IS 4031 (IS 4031 1996) part (1-6) was used for this investigation. Fine and coarse aggregate were used in research and tested as per code recommendation (IS 2386 1963; IS 383 2016). Sieve analysis of fine aggregate showed in Fig 1. The nano siica has a pH of 7.5 and a particle size distribution of 40-400 nm. Superplasticizer (SP) 430 was used as a water-reducing agent.

2.2 Mix proportion

A standard concrete with a compressive strength of 45MPa for 28 days is designed as per IS code10262 (IS 10262 2019). A proportion of five different mixtures are denoted as RC, C1, C2, C3 and C4 with wt. % 0, 1, 2, 3 and 4 of the cement substituted with nano SiO_2. The concrete were put to water curing for 28 days and then for 5 % sulphutic acid curing for 28 days and 56 days. Mechanical tests such as compressive strength test, split tensile strength test and flexural strength was do ne on concrete mixes after acid curing. No fibers were used in this researech. The superplazticizer accounted for 1% of the binder's weight . The water-to-cement ratio (w/c) for every mixture remained constant at 0.40. Concrete specimens cured for water abd acid curing under controlled temprature of 20 and 25 degree celcius 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. After testing all mechanical strength, a microstructural test was performed , and a fine particle of concrete samples was taken for the XRD and SEM test.

Table 2.1: 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

The slump cone test was employed in accordance with IS Code 1199 part 2(2018) to verify the flowability of concrete samples. A steel frustum with dimensions of 300 mm in height, 200 mm in lower diameter and 100 mm in upper diameter was filled with fresh concrete. The concrete has to be carefully and gently lowered vertically to remove the mould as soon as possible. This allows the concrete to sink, and the slump may be easily measured by figuring out how much the concrete has risen over the mould. The concrete cube's weight was determined after 28 days. The weight of the test specimen was first ascertained, and its volume was then divided by it's weight using eq 1 to find the density.

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

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

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

2.3.2 Compressive strength

Compressive strength is the ability of a material or structure to bear loads on its surface without breaking or deflecting. A compression testing machine was used to conduct compression tests on concrete cube specimens measuring 150 mm at 7,14 and 28 days in accordance with IS 516-part 1standard. The compressive strength test results for the three cubic specimen were averaged to determine the compressive strength of each mixture.

2.3.3 Split tensile strength

The split tensile was performed using cylindrical specimens having a diameter of 100 mm and a height of 200 mm, in compliance with IS 516 part 1 standard. Concrete tensile strength is far less than in compressive strength, tension stresses are carried byfibers in the concrete. The three samples were tested at ages 7, 14 and 28 days, with each sample being tested on acompressive testing machine according to eq 3.

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

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

2.3.4 Flexural Strength

The concrete beam specimen for concrete mixes measured 150 mm *150 mm*700 mm. Concrete has a far lower tensile strength than compressive strength., hence steek is used to bear the tensile forces in the material. It is believed that 10% of compressive strength is equal to tensile strength in concrete. The soecimens which had an effective span of 600 mm were tested as supported structures. According to equation 4 the load was given at two places that were each 100 mm from the centroid of the beam.

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

Where P: applied load maximum), B and D: lateral dimensions of sample and L: specimen length.

3. MICROSTRUCTURAL BEHAVIOUR OF CONCRETE

Concrete's microstructure is examined using X-ray diffraction (XRD). Scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) technkiques. Shards weregathered to ascertain the microstructura; properties of each concrete mix once its strength was ascertained. 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: Bruker, Germany and SEM, Model: JSM 6490 L.V.make JEOI. To ascertain the microstructure property of concrete. In XRD analysis samples are scanned continuously at a rate of 2ϴ per minute, ranging from 0 degree to 90 degree. The averafe particle size has been determined from the investigation using the Debye-Schrer, taking into account the peak at degrees. Bragg's Law is used to compute the d-space or inter planar separation between are shown in eq 5

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

D = average particle size (mm), ϴ= Diffraction angle, λ= wave length (0.1540 nm, β = FWHM (fullwidth at half maximum), d= interplanar spacing and n= order of reflection

The concrete's crystalline phases and mineralogical make up were examined using XRD which which aided in the identification of important phases such as calcium hydroxide(CH) and C-S-H s. It shed light on the hydration and the effect 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 microstructureof the concrete in high resolution concentrating on the interfacial transition zone, fracture and pore formation.

4. RESULT AND DISCUSSION

4.1 Flowability

Figure 4 shows the flowability test result for various bends. Reduces the wAdding nano silica reduced the flowabilityin fresh concrete mix. In comparison to the flowability (134 mm) of the reference concrete (RC) the addition of 1%, 2%,, 3% and 4% nano silica by weight of cement reduces the flowability by approximately 6.71%, 10.44%, 29.1% and 15.67% respectively.

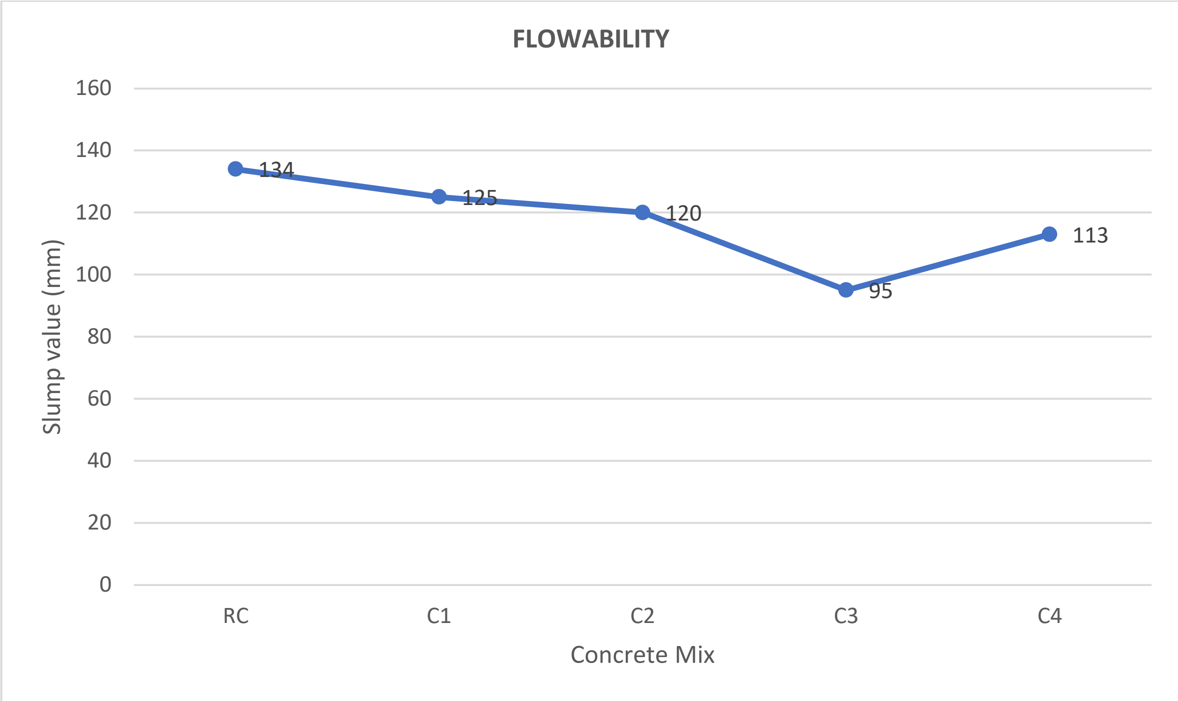


Fig 4.1: Flowability of concrete mix

4.2 Density

The density test results for the different concrete mixes are displayed in Fig 4.2. The density of hardened concrete increaed by approximately 1.96%, 3.44%, 3.53% and 3.58% respectively. Large surface area, micro size of nano SiO_2 and appropriately filled small pores in the concrete compared to the reference concrete, are the reasons for the higher density of the nanomaterial concrete.

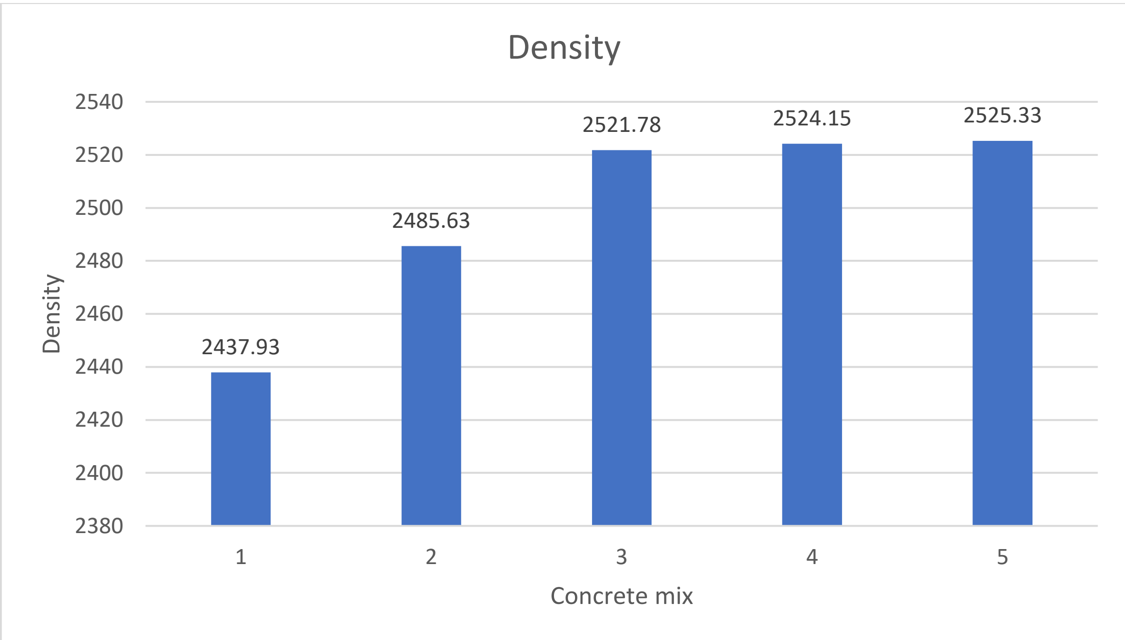


Fig 4.2: Density of concrete mix

4.3 Water absorption

Fig. 4.3 depicts the differences in water absorption among the various hardened concrete mixes. 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%. The small size of nano particle reduces water absorption, making the concrete more durable, resistant to damage and able to fill its tiny pores.

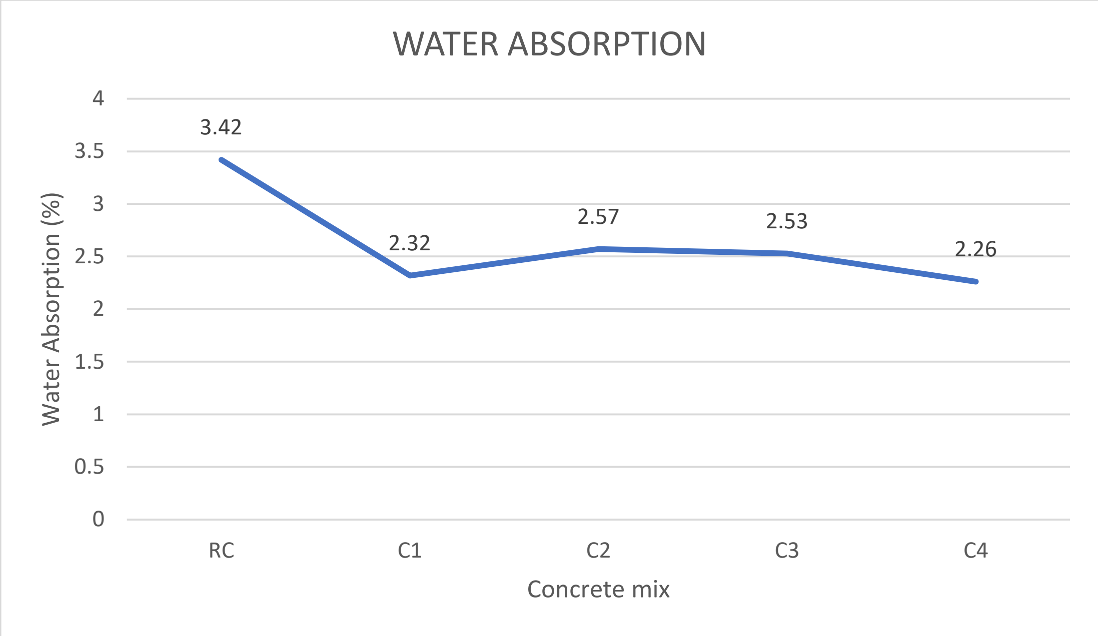


Fig 4.3: Water absorption of concrete mix

4.4 Compressive strength

The compressive strength of concrete is an indicator of its capacity to bear axial loads thtat compress or crush the material. Concrete mixes C1, C2, C3 and C4 with different percentages of nano SiO_2 increase the compressive strength of concrete cubes compared to reference concrete. When this concrete with nano silica was immersed in 5% sulphuric acid its 28 days compressive strength is found to decrease. This reduction in strength is due to the dissolving of C-S-H gel, the main strength giving conpound of concrete.The compressive strength of concrete when put to water curing was 53.70 MPa and when put to acidic curing was 14.44 MPa. This indicates that the compressive strength is reduced when put in acidic environment

Table 4.2: Compressive strength of concrete mix

S NO	28 DAY (MPA)	56 DAY (MPA)
C1	8.37	10.46
C2	10.12	9.65
C3	12.46	8.44
C4	14.44	8.22

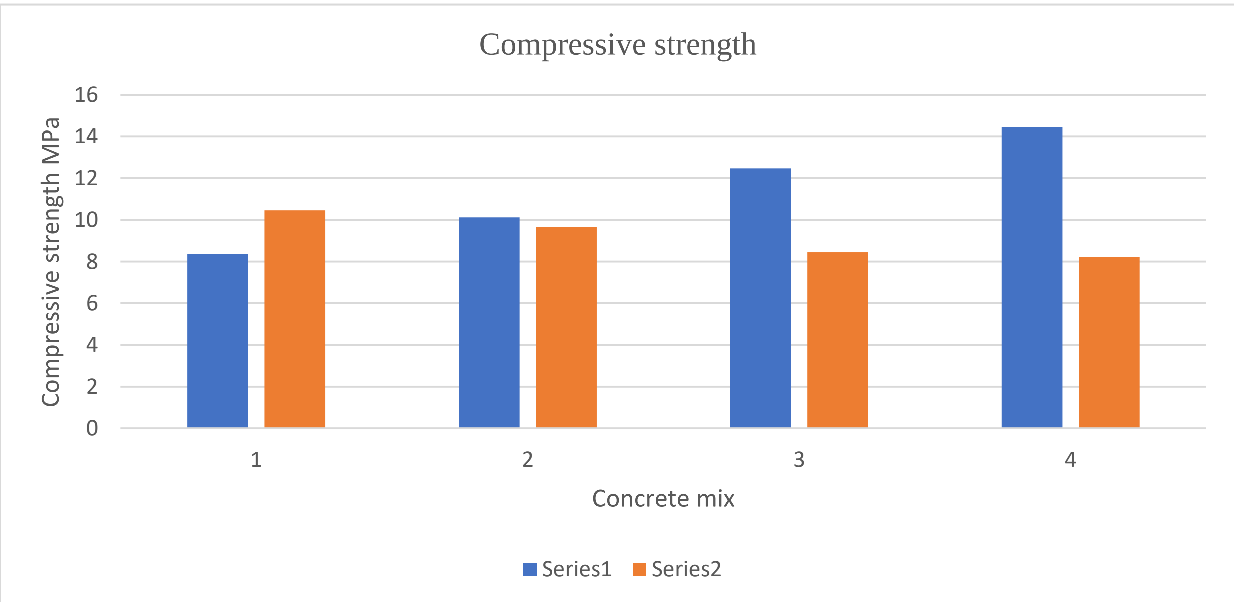


Fig 4.2: Compressive strength of different concrete mix

4.5 Split Tensile Strength

Split tensile strength evaluates concrete's resistance to tensile forces applied perpendicular to the loading axes ; figuring out the load when concrete members is critical. cylinders at 28 days, 56 days. The tensile strength of concrete mixes RC, C1, C2, C3 and C4 is found to decrease.The split tensile strength of nano silica based concrete was 3.0 MPa and when the nanoconcrete was put to acid curing and then split tensile strength was done on it, it was found to be 3.1 MPa.

Table 4.5: Split Tensile Strength of different concrete mix

S NO	28 DAY (MPA)	56 DAY(MPA)
RC	2.5	2.7
C1	2.8	2.9
C2	3.0	3.2
C3	3.1	3.3
C4	2.5	3.1

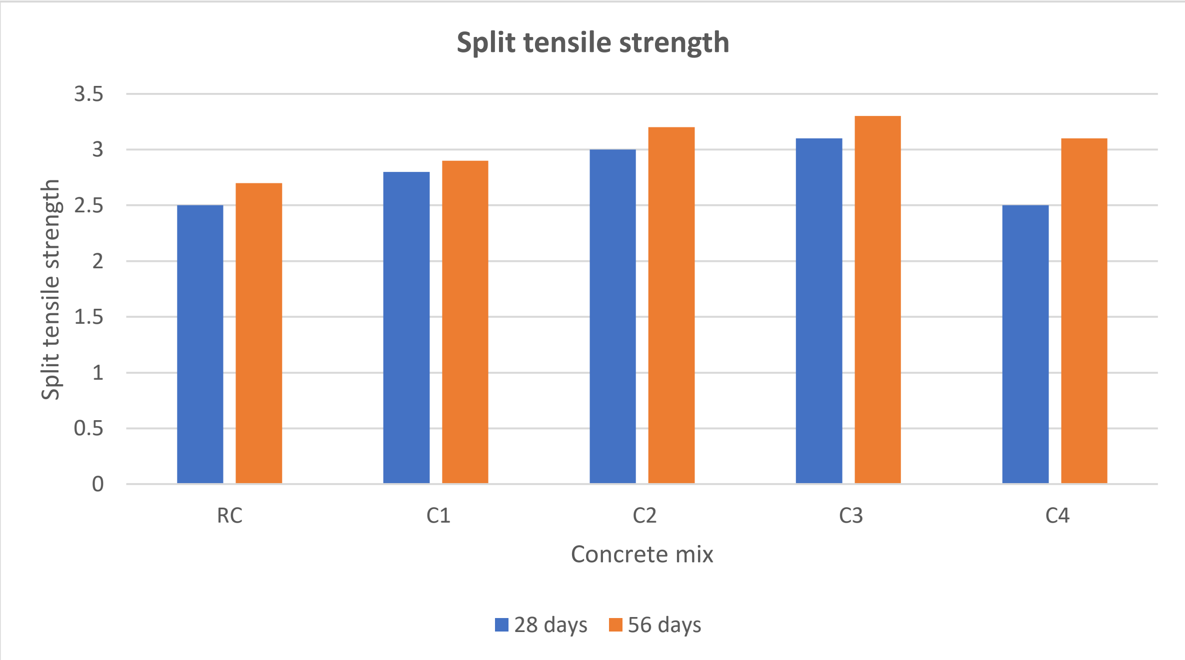


Fig 4.5: Split tensile strength of different concrete mix

4.6 Flexural Strength

Flexural strength is the ability of concrete to resist failure in bending. It is expressed as the modulus of rupture (f_r) and represents the tensile strfength of concrete in flexure. Beam size of 150 * 150 * 700 mm is used . The beam is first casted and then it is immersed in acid for acidic curing. The beam is then taken out of the tank after 28 days and 56 days and then put to test for flexure. The beam strength after 28 days and 56 days are noted and is found to decrease.The flexural strength is found to decrease when put in acidic curing and tested.

Table 4.6: Flexural strength of concrete mix

CONCRETE MIX	28 DAY (MPA)	56 DAY (MPA)
C1	4.6	3.2
C2	5.5	3.5
C3	4.5	3.7
C4	5.0	3.4

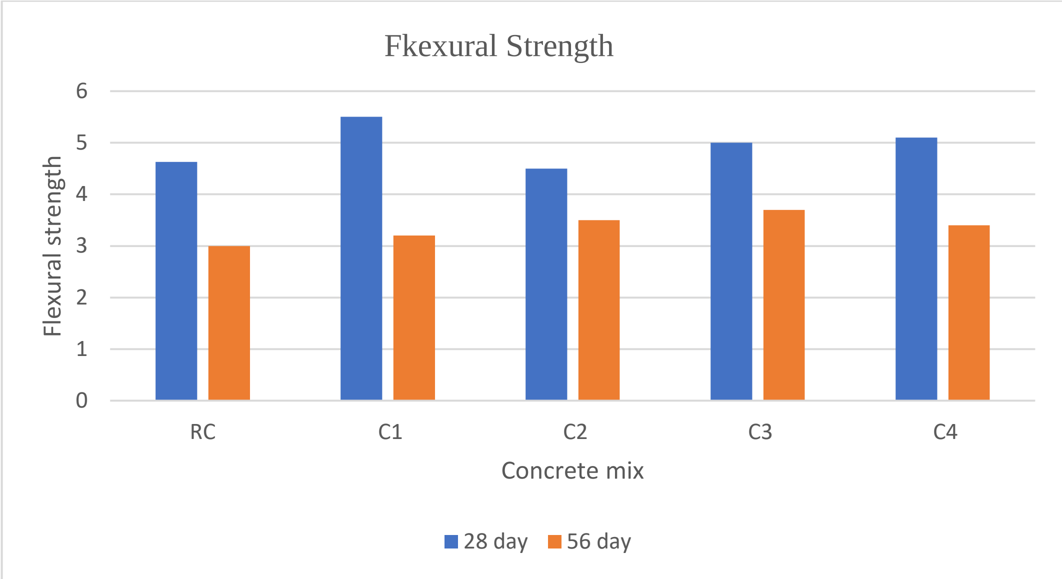


Fig 4.7: Flexural strength of different concrete mix.

5. MICROSTRUCTUTAL BEHAVIOUR

SEM micrographs of the concrete matrix C3 is shown in Fig 10.. Nano-silica acts as a pozzolanic and filler material. It reactsnwith calcium hydroxide (CH) to form additional C-S-H gel. This leads to arefined pore structure with reduced capillary porosity and improved packing density. After acid exposure the dense microtructure slows down acid ingress, delaying the dissolution of C-S-H and decalcification. This results in less internal damage compared to normal concrete. CH I the most vulmerable compound in cement oaste under acid attack because it readily dissolves. Nano-silica converts a large portion of CH into secondary C-S-H gel before exposure. As a result there is less Leachable CH, reducing the extent of acid induced voids and microcracks. In acidic conditions, calcium leaches out forming gypsum and then ettringite, causing expansion and cracking. Nano-silica concrete shows less gyosum and ettringite deposition because of lower CH content and denser matrix, minimizing internal stresses and microcracking. SEM(Scanning Electron Microscopy) .images of nano-silica concrete after acid exposure usually show fewere microcracks, more intact C-S-H gel, better interfacial transition zone (ITZ) around aggregates.

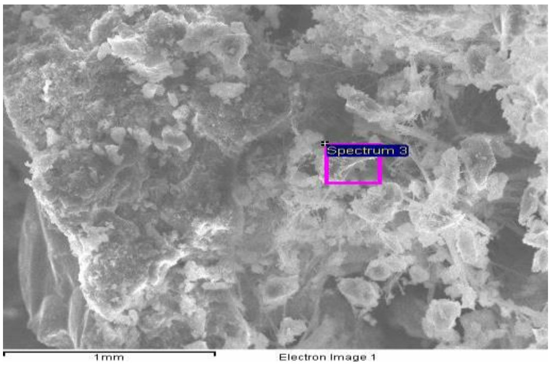


Fig 5 (a)

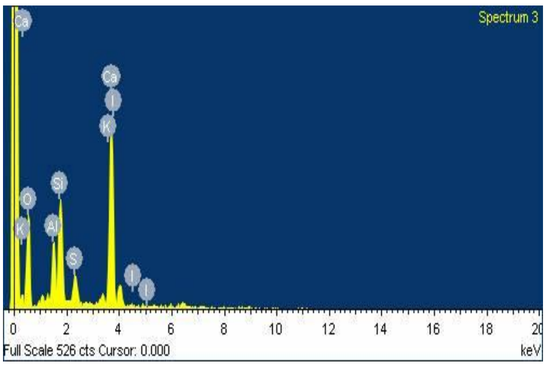
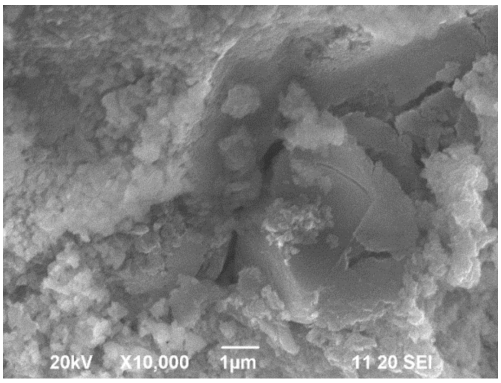
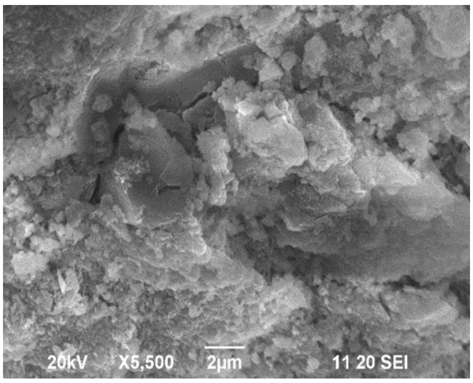


Fig 5 (b)

Fig 5.1 (a) & (b): Scanning Electron Microscope (SEM) images with Energy Dispersive Spectrometer (EDS)





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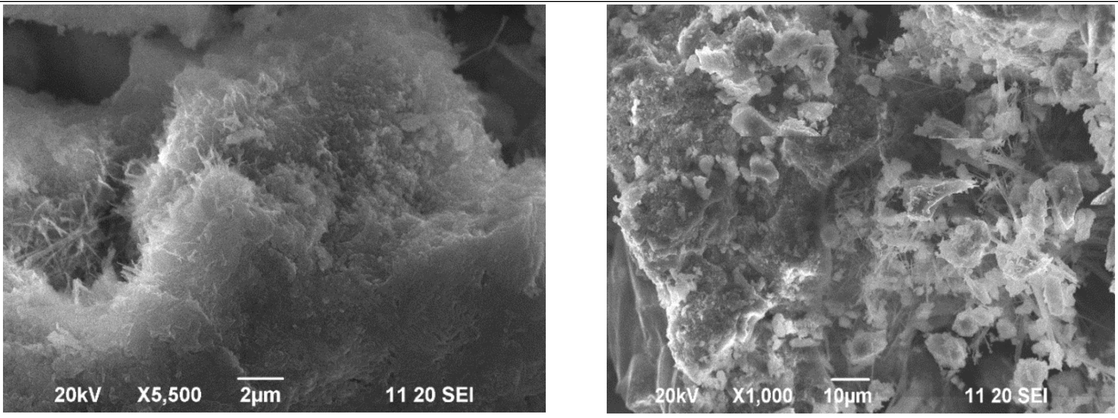

Fig 5.2: Showing Scsanning Electron Micropscope (SEM) images

5.1 X Ray Diffraction analysis

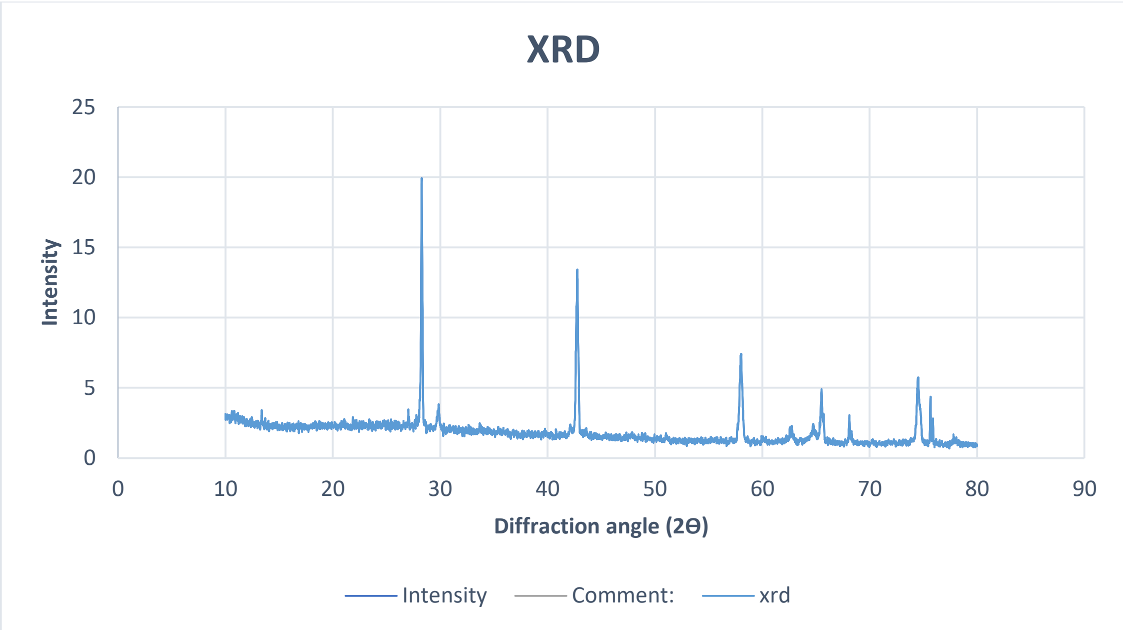

Fig 5.3: X ray Diffraction graph

XRD examination was performed on the powdered sample by pulverizing tiny fragments of nano-sized SiO_2 concrete samples. The primary mineral phases and the crystallite size and texture of the cementitious materialsnduring hydration could all be identified using the XRD pattern. The presence of significant hydration products in the form of titanium di-oxide, amorphous silica, calcium carbonate, aluminium oxide, iron titanium di-oxide, portlandite, quartz, alite, belite and C-S-H were represented by each of the peaks in he figure along with the varying intensity peaks at diffraction angles in between 10 and 90 degrees. As seen in fig 11, the prominent peaks at 26.71 degree, 37.06 degree, 58.35 degree and 65.1 degree signify the existence of SiO_2. Accompanying these peaks are amorphous silica and aluminium oxide. Strong diffraction peaks at 26.71 degree, 37.06 degree, 58.35 degree and 65.1 degree were seem in the XRD pattern. These patterns were taken on 28 day of curing and at an angle of 2Θ, which ranges from 10 to 90 degrees. The single ohase XRD pattern has been achieved for SiO_2 , TiO_2 and $CaCO_3$ with cubic hexagonal and rhombohedral structures respectively. The crystallite sizes and d-spacing values found in concrete specimen are crucial for their mechanical properties. A more finer microstructure is indiacated by smaller crystallites, which enhance hydration, denser C-S-H formation and 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 resisatance to deformation is indiacated 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.

Table 5.1: Composition of various elements

ELEMENT	WEIGHT %	ATOMIC %
O K	59.18	75.02
Mg K	1.15	0.96
Al K	1.49	1.12
Si K	8.63	6.23
S k	15.10	9.55
Ca K	13.14	6.65
Fe K	1.32	0.48
Total	**100**	

5.2 DURABILITY TEST

5.2.1 RAPID CHLORIDE PERMEABILITY TEST

The RCPT test refers to the Rapid Chloride Permeability Test, which is commonly used to evaluate the durability of concrete – specifically, its resistance to chloride ion penetration.The purpose of RCPT is to determine the ability of concrete to resist chloride ion penetration. Chloride ingress can lead to corrosion of steel reinforcement, reducing the service life of structres. The disc specimen is vaccum saturated. Specimen is placed between two cells: Cathode side- Sodium chloride (NaCl) solution and anode side –sodium hydroxide (NaOH) solution. A 60 V DC is applied acropss the specimen for 6 hours. The total charge passed (in Coulombs) is measured. A lower charge passed (measured in Coulombs) during the 6-hour test suggests better resistance to chlorides and lower permeability.

Table 5.2: Rapiod Chloride Permeability Test (RCPT)

S NO	CHARGE PASSED (IN COULOMBS)	AVERAGE CHARGE PASSED (IN COULOMBS)	CHLORIDE PERMEABILITY
1.	620.8	619.2	Very Low
2.	614.5		
3.	622.4		

A **very low** chloride permeability during the 6 hour test indicates better rresistance to chlorides. This test is crucial for quality control and for predicting the service life of concrete structures exposed to chloridce rich environments.

Fig 5.4: Concrete cube and cylinder compressive strength test and split tensile strength test after acid curing

6. CONCLUSION

Incorporation of nanomaterials such as silicon dioxide in concrete improves the compressive strength, split tensile strength, flexural strength. It reduces permeability, reduced setting time. When this nanoconcrete is exposed to 5 % sulphuric acid it causes some deterioration , nano silica modified concrete shows lower permeability, higher residual strength and reduced surfae erosion. Hence, nanoconcrete is a [romising solution for critical infrastructure such as sewerage system, wastewater treatment plants and industrial floors where chemical resistance is a primary requirement.

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