

# PERFORMANCE ENHANCEMENT IN CONCRETE USING NANO SILICA AND FLY ASH

A. Bharat <sup>a,\*</sup>, S. Kumar <sup>a</sup>

<sup>a</sup> Department of Civil Engineering, National Institute of Technology, Patna, India, ORCID ID 0009-0004-5282-9141 / ORCID ID 0000-0001-6497-9519, E-mails: \* [abhinav.ce17@nitp.ac.in](mailto:abhinav.ce17@nitp.ac.in), [sanjay@nitp.ac.in](mailto:sanjay@nitp.ac.in)

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## ABSTRACT:

To improve the concrete performance, combining nano materials with mineral admixtures is quite effective in refining the fresh as well as hardened properties. However, the combined effects of nano and micro level materials on the concrete performance needed to be investigated for better understanding of their synergistic effects. In this paper fly ash (FA) is blended with nano silica (NS) replacing ordinary Portland cement (OPC) grade 43 to produce high performance concrete (HPC). Experiment was conducted at fixed water binder ratio with varying percentage of superplasticizer to produce workable and cohesive mix. Ternary blending with NS gave better cube strength at early stage than the binary blended mix. Even at later stage there was enhancement in the strength at 15% FA and 2% NS replacement. This could be due to seeding effects of ultra fine NS particles providing favourable hydration conditions for FA. Nano modified concrete gave lower strength at higher proportions due to the inherent problem of agglomeration. Durability of the mix was investigated by the rapid chloride penetration test (RCPT). Also, ultrasonic pulse velocity (UPV) test was conducted to observe the homogeneity of the mix. Ternary mix of 1.5% NS and 20% FA with OPC was the optimum dosage as it showed enhanced strength and durability. The images obtained from scanning electron microscope (SEM) was analysed to understand the CSH gel formation and the hydration process. These results showed that the combined usage of NS and FA has synergistic effects which could be utilized for maximum performance.

## 1. INTRODUCTION

The development of high-performance concrete (HPC) is done for not only improving the strength but also the durability improvement becomes a key factor. Selection of binder materials becomes the most important part. Cement is obvious choice but for the sake of sustainable development, it is essential to replace cement with industrial wastes. Also looking at particle size distribution of cement which roughly varies from 1 micron to about 100 microns there is a need of materials of different sizes to improve packing density of the concrete. Sand will fill the sizes more than 100 microns but for better packing binder materials below 1 micron is also needed (Bentz et al. 1999; Meddah, Zitouni, and Belâabes 2010). Therefore, the utilisation of nano silica (NS) becomes mandatory not just for packing but also for their reactive silica which helps cement to do its job better (Singh et al. 2019; L. G. Li et al. 2017). NS serves as nucleation sites for the formation of secondary calcium silicate hydrate (CSH) gel (Bai, Guan, and Li 2022). This improves the early strength of the HPC significantly. Also due to its filler effect, the microstructural densification of the cementitious matrix results in durability improvement. But the use of NS at higher dosages leads to agglomeration due to its very high specific surface area (Tabish, Zaheer, and Baqi 2023). Also, the workability of the mix becomes a problem. Few researchers have investigated the effect of NS replacement on the HPC using the ultrasonic dispersion for reduction of agglomeration (Kim et al. 2013; Zyganitidis et al. 2011). But this raises the fabrication costs of the structure. Therefore, in this paper, NS is replaced at lower replacement ratio of 1.5%

and 2% so that the conventional mixing procedures are used and other costly special techniques should be avoided. Since the water binder ratio is kept lower (0.28) to develop HPC, it is reasonable to keep lower NS dosage. Also, fly ash (FA) is selected as other supplementary cementitious materials (SCMs) which complement the properties of NS in developing good workable mix (Yuan et al. 2018). Also, the inclusion of FA with NS reduces the heat of hydration, thereby reducing the shrinkage. Dosage of FA is decided according to the recommendation of IS 456 and IS 10262 (IS 456 2000; Indian standard 2019). Using large proportion of NS is not feasible considering the fabrication cost and agglomeration, therefore ternary blending of NS with FA is a viable option (Wang et al. 2020). So, in this paper the development of HPC mix is performed in such a way that the ordinary Portland cement (OPC), FA and NS acts synergistically at low water binder ratio of 0.28. Rapid chloride penetration test (RCPT) and ultrasonic pulse velocity (UPV) test were performed to investigate the durability and homogeneity of the mix. Also, the images obtained from the scanning electron microscope (SEM) were analysed to understand the CSH gel formation and the hydration mechanism.

## 2. METHODOLOGY

### 2.1 Materials

Selection of materials and proper mix design of the constituents of concrete is necessary for the development of HPC. The cement used as the main binder was OPC 43 as per Indian standards (IS) code (Bureau of Indian Standard (BIS) 2013). NS

powder was provided by Nano research lab, Jamshedpur having 99% purity. Class F type of FA was available locally of dark greyish colour. The physical properties and its composition are presented in table 1. Locally available sand of zone II was used along with crushed stone aggregate of size 12.5 mm. High range water reducing admixture (HRWR) was polycarboxylic ether based (chryso optima S6123) which permits the design of HPC at lower water binder ratio. Relative density of materials

involved in the experiment using water as reference material are mentioned in table 1 and table 2. The resistance of aggregate to crushing under a gradually applied compressive load is measured by the aggregate crushing value test. Properties of HRWR and aggregates are given in table 2.

| Specifications                     | OPC 43 | FA   | NS     |
|------------------------------------|--------|------|--------|
| CaO (%)                            | 63.1   | 2.4  | -      |
| SiO <sub>2</sub> (%)               | 17.2   | 53.2 | 99     |
| Al <sub>2</sub> O <sub>3</sub> (%) | 6.4    | 28.4 | -      |
| Fe <sub>2</sub> O <sub>3</sub> (%) | 4.7    | 13.4 | -      |
| MgO (%)                            | 0.7    | 0.7  | -      |
| SO <sub>3</sub> (%)                | 2.8    | 0.4  | -      |
| Loss on Ignition (%)               | 2.4    | 0.6  |        |
| Alkali (%)                         | 0.6    | 0.5  | -      |
| Relative density                   | 3130   | 2180 | 1200   |
| Fineness(m <sup>2</sup> /kg)       | 250    | 340  | 300000 |

Table 1. Properties and compositions

| HRWR             | Values          | Fine Aggregates      | Values | Coarse aggregates            | Values |
|------------------|-----------------|----------------------|--------|------------------------------|--------|
| Relative density | 1118            | Relative density     | 2650   | Relative density             | 2740   |
| pH               | ≥6              | Fineness Modulus     | 2.76   | Water Absorption (%)         | 0.36   |
| Appearance       | Brownish liquid | Water Absorption (%) | 1.07   | Aggregate Crushing Value (%) | 24     |

Table 2. Properties of aggregates and HRWR

## 2.2 Mix Proportions

Creating HPC requires a precise mix design and meticulous mixing procedure to achieve the desired properties. IS mix design(Indian standard 2019) was followed to proportion materials such that M70 grade concrete is achieved. N0F0 denotes the control mix where N stands for nano silica and F stands for fly ash. Four binary mixes with 15% and 20% FA and 1.5% and 2% NS were performed at constant w/b ratio of 0.28. Then four ternary mixes involving NS and FA was conducted. Use of HRWR was done accordingly to attain the

desired workability. The quantity of fine and coarse aggregates is different due to the variation of HRWR for different mixtures taken in the calculations. This has been done to get the slump value closer to the stipulated value of 150 mm. Also, the change in the quantity of SCMs changes the amount of fine and coarse aggregates in each mixture. The compositions of the mixes to prepare 1m<sup>3</sup> of HPC are tabulated in table 3.

| Mix     | Cement (kg) | NS (kg) | FA (kg) | Fine Aggregate (kg) | Coarse aggregate (kg) | Total mass (kg) |
|---------|-------------|---------|---------|---------------------|-----------------------|-----------------|
| N0F0    | 550         | -       | -       | 803                 | 992                   | 2345            |
| N0F15   | 467.50      | -       | 82.50   | 787                 | 970                   | 2307            |
| N0F20   | 440         | -       | 110     | 782                 | 965                   | 2297            |
| N1.5F0  | 541.75      | 8.25    | -       | 789                 | 974                   | 2313            |
| N2F0    | 539         | 11      | -       | 788                 | 972                   | 2310            |
| N1.5F15 | 459.25      | 8.25    | 82.50   | 779                 | 961                   | 2290            |
| N1.5F20 | 431.75      | 8.25    | 110     | 774                 | 955                   | 2279            |
| N2F15   | 456.50      | 11      | 82.50   | 776                 | 957                   | 2283            |
| N2F20   | 429         | 11      | 110     | 771                 | 951                   | 2272            |

Table 3. Mix proportion

### 2.3 Tests involved

Slump test was performed in accordance with IS code (IS 1199 (Part 2) 2018; IS 456 2000) to ensure the mix meets the required consistency. Tests were conducted on standard cubes at 3, 7 and 28 days to find out the strength of the sample as per the IS code. RCPT were carried out on 28 days sample based on ASTM C1202 (ASTM C1202 2012) to determine the chloride permeability of the specimen. UPV is a non-destructive in-situ test conducted to evaluate the homogeneity and quality of the hardened mix. SEM images were observed to study the reactions of SCMs with calcium hydroxide and

identify the hydration products obtained during pozzolanic reactions.

### 3. RESULTS AND DISCUSSION

The results of the experimental program in accordance with Bureau of Indian Standards (BIS) are discussed in this section and are tabulated in table 4. The average value of 3 samples were taken for each test results. Images from SEM are also examined and analysed to get an idea of the morphology of nano modified concrete.

| Mix     | Slump (mm) | Compressive strength (MPa) |        |         | RCPT (C) | UPV (m/s) |
|---------|------------|----------------------------|--------|---------|----------|-----------|
|         |            | 3 days                     | 7 days | 28 days |          |           |
| N0F0    | 135        | 28.60                      | 40.30  | 67.80   | 1851     | 3894      |
| N0F15   | 165        | 26.72                      | 36.64  | 74.60   | 1363     | 4880      |
| N0F20   | 170        | 24.26                      | 34.40  | 72.80   | 1305     | 4860      |
| N1.5F0  | 110        | 34.10                      | 46.85  | 80.54   | 980      | 5016      |
| N2F0    | 105        | 38.90                      | 47.30  | 80.80   | 860      | 5085      |
| N1.5F15 | 150        | 36.80                      | 45.46  | 87.24   | 415      | 5245      |
| N1.5F20 | 150        | 34.84                      | 48     | 91.20   | 360      | 5154      |
| N2F15   | 145        | 35.44                      | 46.76  | 86.32   | 368      | 5099      |
| N2F20   | 150        | 35.80                      | 48.45  | 84.51   | 356      | 5068      |

Table 4. Results of the experiments

#### 3.1 Slump Test

The design was performed such that the slump stipulated for the mix was 150 mm. As per IS code, workability is high as the slump values are in the range of 100-170 mm as shown in figure 1. FA addition increased the workability beyond 150 mm due to its ball bearing effect and glassy surface. It was noted that the inclusion of NS reduced the workability considerably and the mix was sticky due to its fineness and very high specific surface

area. Also due to the agglomeration of NS particles lead to loss in slump. Ternary blending of FA and NS gave very good cohesive mix. Paste volume was increased by FA and the use of NS stopped bleeding giving workable mix. 1.2 % HRWR was used in all the mixes.

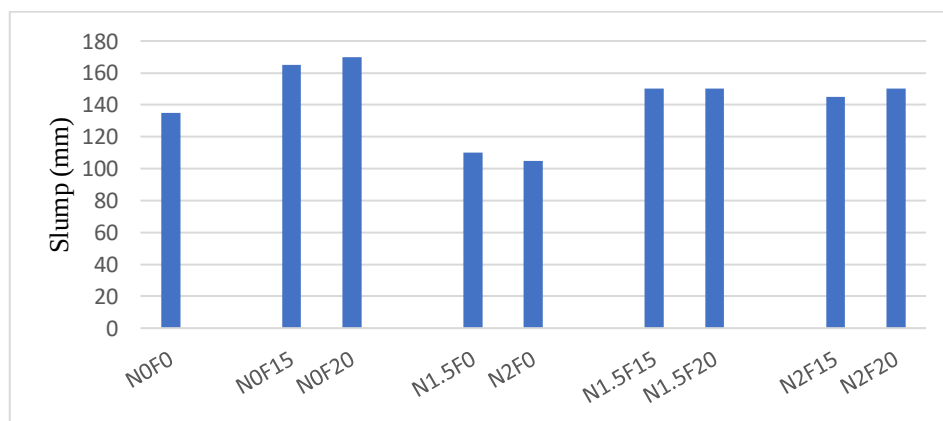


Figure 1. Slump test results

#### 3.2 Compressive strength test

Variation of compressive strength for different mixes are shown in figure 2. As the content of FA was increased, the compressive strength also starts decreasing. 3-day strength of FA blended specimen gave very poor results. As compared to control mix N0F15 mix gave 15.17% and 14.64% lesser 3 day and 7-day strength respectively. But after 28 days strength is higher than the control mix which indicates that the pozzolanic materials

like FA works well with continuous curing for longer time (Dave et al. 2017). This loss was compensated by the incorporation of NS into the mix which increased the early strength considerably. Adding 1.5% NS to 20% FA increased the 3 day and 28-day strength to 21.8% and 34.5% respectively as compared to control mix. The later strength of N1.5F20 was more than N1.5F15 as the contribution of FA to the strength gain is more with time due to the slow pozzolanic reaction rate of the particles. NS particles act as an activation agent for

hydration reaction with FA producing a synergy between these materials. But the dosages more than 2% creates agglomeration of finer particles leading to decrease in strength as well as workability (G. Li 2004; Isfahani et al. 2016). Also, the water

demand of NS at 2% dosage increased which is reflected in loss of strength at 20% FA. Therefore, the incorporation of 1.5% NS with 20% FA gave the optimum mix.

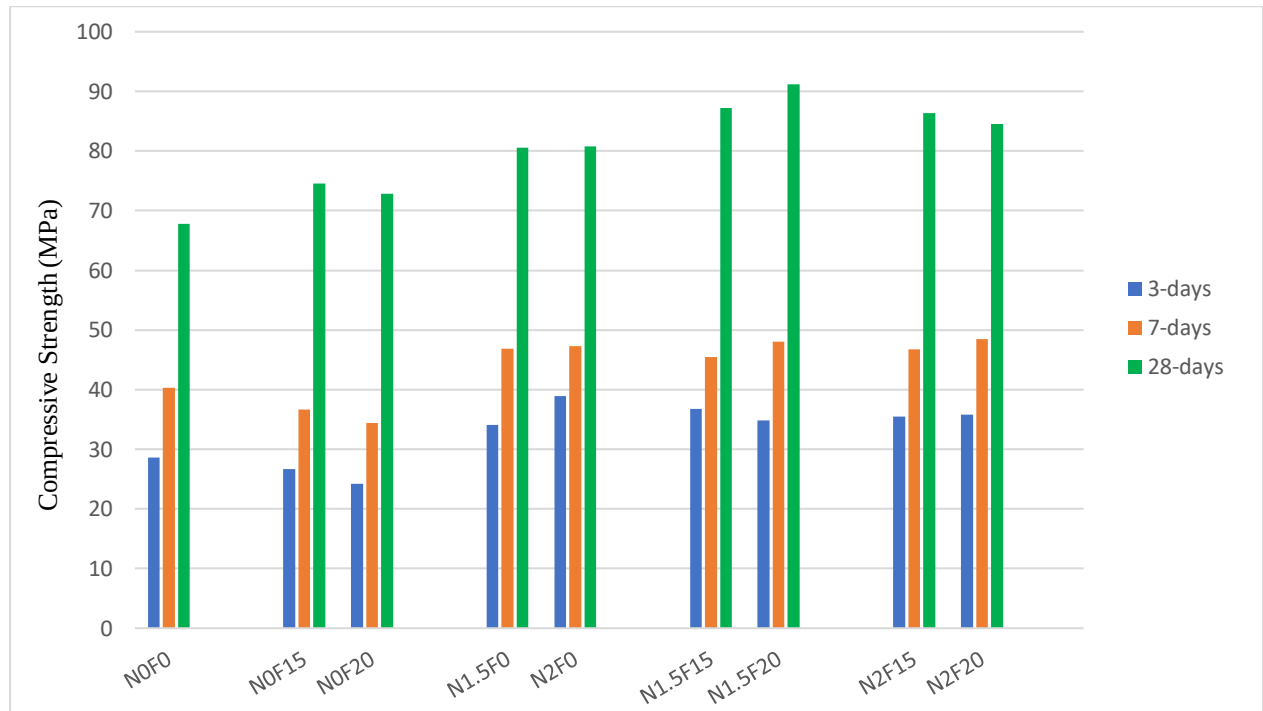


Figure 2. Compressive strength of M70 grade concrete

### 3.3 Rapid chloride penetration test

According to published research, a decrease in chloride permeability indicates an increase in resistance against the entrance of chloride ions. The fast chloride permeability was significantly reduced when pozzolanic material was substituted with cement (Ahmed, Kayali, and Anderson 2009). In comparison to binary concrete, ternary blend containing 20% FA and 2% NS shown reduced chloride permeability as shown in figure 3. The pozzolanic action of NS arrests all the  $\text{Ca}(\text{OH})_2$

created during hydration and hence charges passed are significantly lower than the binary mix with only FA as SCM as well as control mix (Kong et al. 2012; Zhang and Islam 2012). Reduction in capillary voids due to inclusion of very fine NS particles is directly related to the reduction in passage of chloride ions (Güneyisi et al. 2015). The addition of SCMs reduced the chloride penetration consistently giving coefficient of correlation,  $R^2 = 0.9252$  which gives a good linear relation between the charges passed and the pozzolanic action of SCMs.

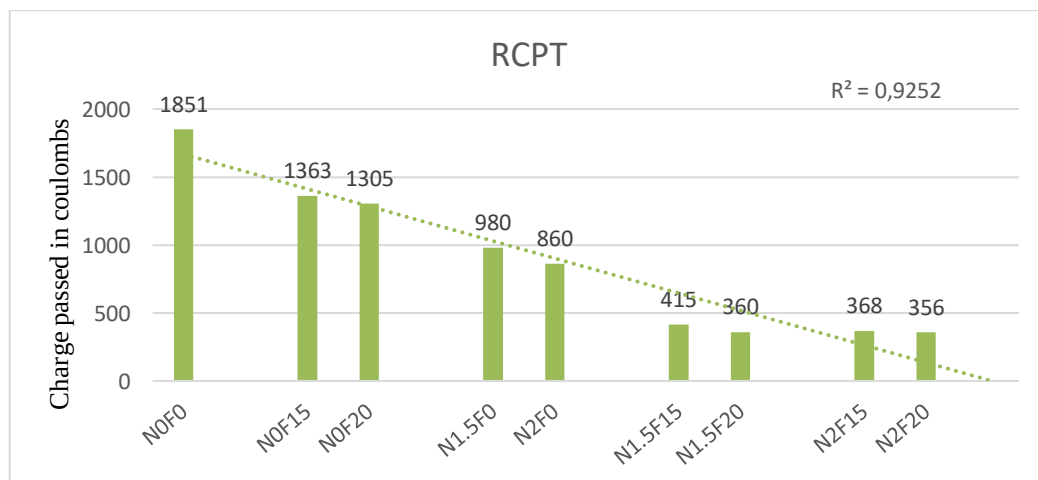


Figure 3. Charges passed through sample

### 3.4 UPV test

The measured UPV values showed excellent quality of concrete barring control mix. The ternary HPC mix had a higher pulse velocity than the binary mix because of the addition of NS. The substantial smaller size of NS filled the spaces between the cement particles resulting in dense mortar matrix, facilitating the quick passage of pulses. UPV of the mix N1.5F15 was highest among all the mixes. Figure 4 displays the best fit plot between the compressive strength and the UPV values at 28

days of the curing time with a value of  $R^2 = 0.8282$  which shows a good linear relation. Thus, the UPV values can be utilized to estimate the compressive strength of a given mix without destroying the cube sample (Choudhary et al. 2021).

$$y = 0.0441x - 140.58 \quad (1)$$

Where: y = Compressive strength in MPa  
x = UPV value in m/s.

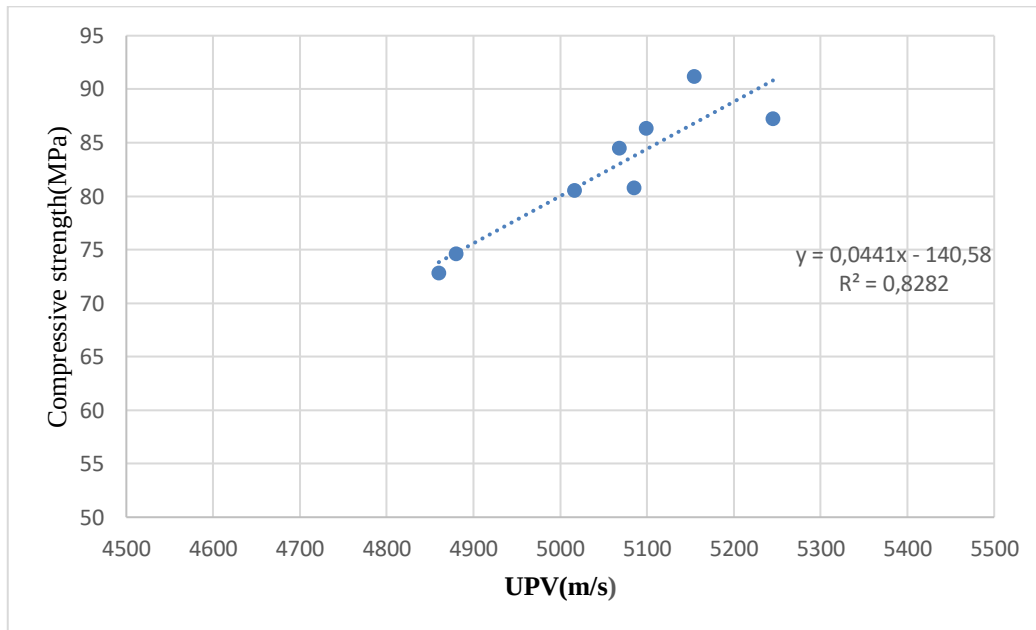
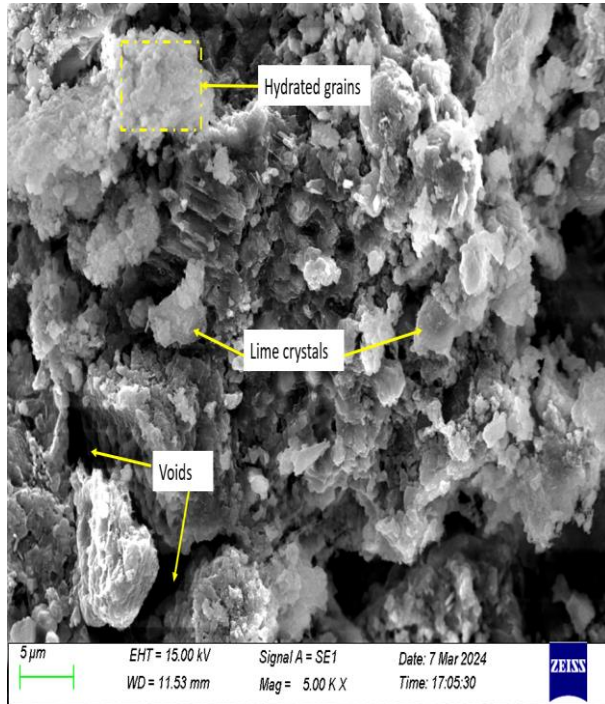


Figure 4. Trend line of compressive strength with UPV

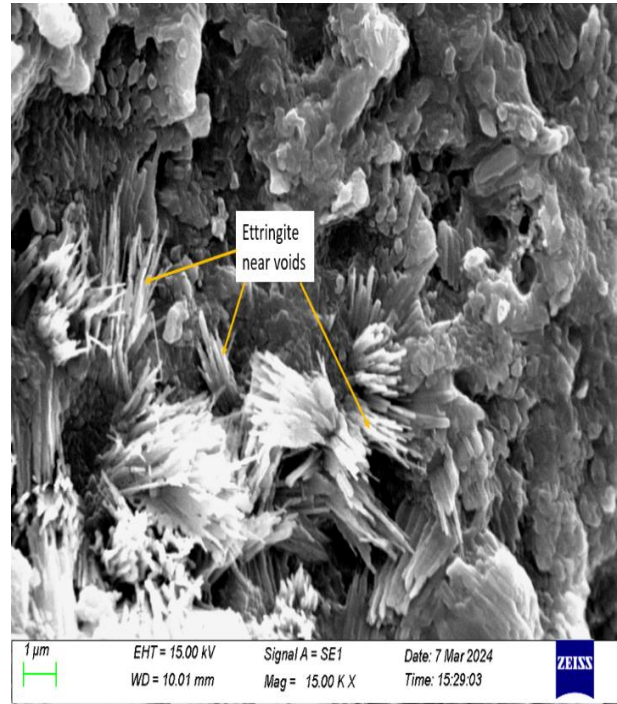
### 3.5 Microstructural study

The production of cementitious substances in the form of hydrated grains is seen in the microstructure. When water and the calcium silicate found in OPC combine, crystalline  $\text{Ca}(\text{OH})_2$  and CSH are produced. In the control mix microstructure, the pores were clearly visible and lime crystal were also present in large quantity as shown in figure 5a (Givi et al. 2011; Naji Givi et al. 2010). The formation of hydrated grains is not enough to reduce voids and densify the matrix (Bharat and Kumar 2024). Similar features were visible when binary blending using FA was performed. Some voids were reduced due to the pozzolanic reaction of FA as seen in 5b. In binary mix when NS was used as 2% replacement, large crystals of lime were consumed to produce secondary CSH gel. But as seen from figure 6a, deposits of ettringite crystals near a void having needle like features were clearly visible. The hydrated grains do not interlock as much as they should, despite the overall compact morphology. It is easy to find the empty spaces that exist between the hydrated particles. However, an extremely dense network of hydrated particles is observed in the case of ternary blending of nano silica with fly ash as seen in figure 6b. Very few pores result in better connectivity among the hydrated particles (Supit and Shaikh 2014). The increased strength of N1.5F20 mix is due to the presence of very fine NS particles resulting in the densification of the microstructure which was responsible for improved mechanical performances and

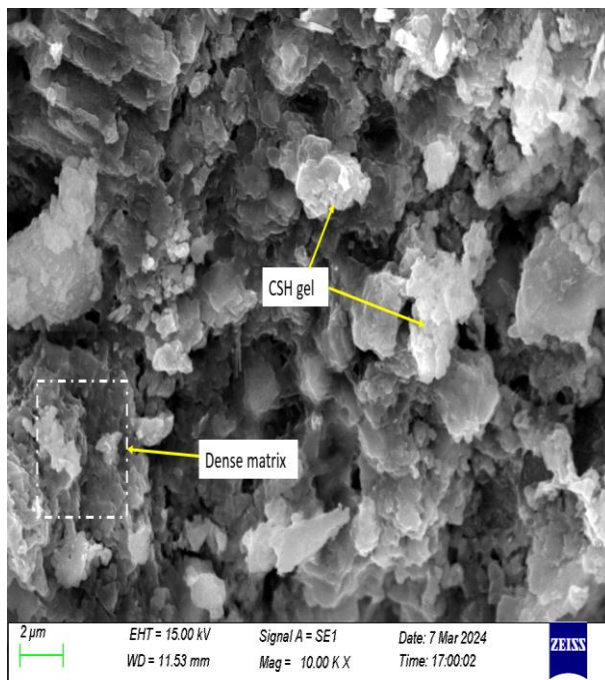
durability as evident from table 4 and figure 5b. The thick microstructure due to filling effect and the pozzolanic activity of latent hydraulic materials like NS and FA leads to lower porosity after the completion of hydration. The superior parameters like compressive strength, durability and resistance to thermal and chemical attacks are a consequence of lower porosity in the hardened state (Du, Du, and Liu 2015; L. G. Li et al. 2018).



(a)



(a)

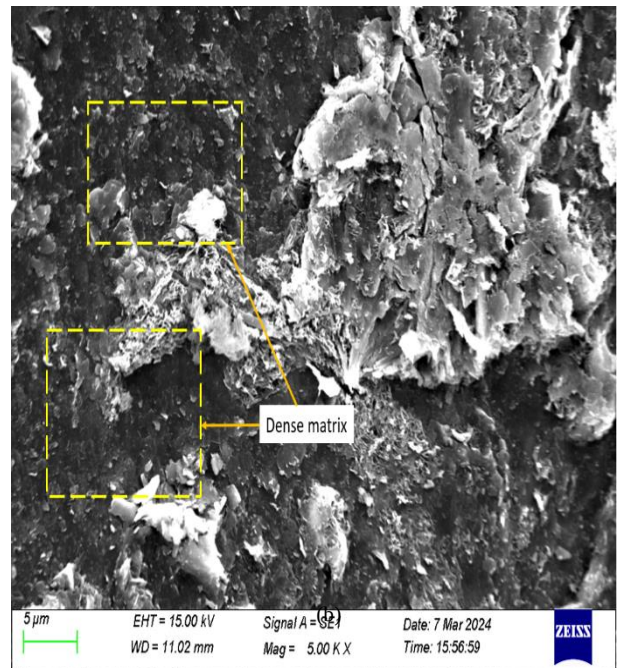


(b)

Figure 5. (a) control mix (b) binary blended mix @ 20% FA

#### 4. CONCLUSIONS

1. The NS dosage beyond 2% was not taken as the mix become unworkable and less cohesive due to agglomeration. FA addition increased the workability beyond 150 mm due to its ball bearing effect and glassy surface. Ternary blending gave very good cohesive mix as the paste volume was increased by FA and the use of NS stopped bleeding.



(b)

Figure 6. (a) binary blended @ 2% NS mix (b) ternary blended @ N1

2. As compared to control mix, N0F15 mix gave 15.17% and 14.64% lesser 3-day and 7-day strength respectively. As the dosage of FA increased early strength decreases. This loss was compensated by the incorporation of NS into the mix which increased the early strength considerably. Adding 1.5% NS to 20% FA increased the 3 day and 28-day strength to 21.8% and 34.5% respectively as compared to control mix.

3. Using lower w/b ratio with SCMs produced denser concrete structure having UPV values more than 5000 m/s. Also, a correlation has been established between compressive strength and UPV.
4. In comparison to binary concrete, ternary blend containing 20% FA and 2% NS shown reduced chloride permeability. Intrusion of aggressive ions considerably reduced due to creation of more stable products during the pozzolanic reactions of NS in the presence of FA.
5. Better packing of micro and nano silica particles produces a filler effect between the cement grains resulting in higher packing density. The secondary gel formation contributes to denser packing and fills the voids left by the primary hydration products as seen from the SEM images. Also, the large crystals of lime have been consumed by the pozzolanic action of NS and SCMs.
6. The benefits of NS could be efficiently utilised when it is used with SCMs such as FA and MS. This synergy leads to increased strength and improved durability while promoting sustainability and cost effectiveness which can be efficiently utilised in the construction of high-rise buildings and dam.

## 5. LIMITATIONS AND FUTURE WORK

1. The flexural and splitting tensile strengths has not been taken as only the durability and microstructural behavior were studied with few mechanical strength parameters. These tests could be performed in future with the inclusion of glass fibres.
2. IS code should recommend optimum dosage of NS to produce HPC.
3. Thorough studies on the impact of NS on the drying shrinkage, bond strength, and creep need to be done.

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