REVISTAMATÉRIA V.28 N.4
ISSN 1517-7076 articles e20230281, 2023

Characterization of nano fly ash and study on durability properties of nano fly ash embedded concrete

Manickam Harihanandh[1], Kathiresan Karuppanan[2], Kinipalayam Eswaran Viswanathan[3], Ammapalayam Ramasamy Krishnaraja[4]

[1]Marri Laxman Reddy Institute of Technology and Management, Department of Civil Engineering. Hyderabad, Telangana, India.

[2]Dire Dawa University, Institute of Technology, School of Civil Engineering and Architecture. Dire Dawa, Ethiopia.

[3]Builders Engineering College, Department of Civil Engineering. Kangeyam, Tamilnadu, India.

[4]Kongu Engineering College, Department of Civil Engineering. Perundurai, Tamilnadu, India.

e-mail: harivmk@gmail.com, kathiresan.Karuppanan@ddu.edu.et, keviswanathan@gmail.com, krajacivil@gmail.com

ABSTRACT

Fly ash is used partially as a replacement to cement to make the concrete. Fly ash contains SiO_2 and Al_2O_3, which contribute to increase the strength of concrete. In the current investigation, fly ash has been replaced with varying percentages of nanosize in concrete, and the strength of the resulting hardened concrete has been evaluated. The production of nanosized fly ash has been made by the ball milling process and the analyses has been completed using scanning electron microscope (SEM) and X-ray diffraction (XRD), in order to determine the chemical and physical composition of the nanoparticles of fly ash. The nano fly ash concrete (NFAC) have been undergone for the durability tests of acid, sulphate and chloride attack. The concrete with 23% of nano fly ash replaced with cement performs well against these adverse conditions. The comparison has been made between the conventional (CC), raw fly ash (RFAC) and NFAC. Nano fly ash makes concrete much denser and more resistant to cracking. So the durability behaviour of NFAC is much better than CC and RFAC. The percentage of reduction in strength after durability aspects is varies from 0.03% to 10.75% for the NFAC.

Keywords: Ball milling; Nano Fly ash; XRD; SEM; Durability properties.

1. INTRODUCTION

The study with the aim of determining the impact of nano particles on the mechanical properties of concrete at a range with respect to the ages of the concrete. Nano particles were used in the place of replacement of cement in this experiment. The samples were evaluated in accordance with the ASTM C666A standard by being frozen and thawed many times in water. After immersing the samples to a predetermined number of cycles of freezing and thawing, they were subjected to tests to determine, how the freezing and thawing affected their compressive strength, loss of mass, change in length, and water absorption. According to the results, the incorporation of nano particles into the concrete resulted in a significant improvement in the material's ability to withstand cold [1, 2]. An experiment is conducted to examine the feasibility to convert fly ash from micro size to nano size using electric ball mill and the sizes were confirmed through SEM analysis also the admixtures found through XRD analysis [3]. Totally 305 hydropower plants and 83 large coal-based thermal power plants are operating within the territory of India, as reported by the Central Electricity Authority of India (CEA) [4]. The purpose of the experiment was to determine the effect that the nanoparticles had on the mechanical properties of the concrete as well as the durability of the concrete [5, 6]. This work evaluates the performance of superplasticizer additive with colloidal nanosilica suspension and slurried silica fume with HPC and its macro- and micro-structural properties. The mechanical and microstructural properties of concretes with 1.5 and 3% nanosilica and 5 and 10% silica fume. The results indicate that, with the use of mineral admixtures, the compressive strength and elastic modulus of concrete increased significantly. The concrete with the use of 10% silica fume and 3% nanosilica resulted in a showed an increased strength over 75% at 28 days [7]. Researchers conducted research on the influence of nano silica has on the properties of concrete. For the purpose of this study, OPC and OPC mixed with class F fly ash were used in the production of concrete, and the effect of colloidal nano silica was analyzed. The results of this study provide evidence of the effect that nano silica and nano alumina have on the frost resistance

Corresponding Author: Kinipalayam Eswaran Viswanathan Received on 04/10/2023 Accepted on 23/11/2023

DOI: https://doi.org/10.1590/1517-7076-RMAT-2023-0281

in conventional concrete [8]. Researchers have considered a wide range of different mixtures, some of which include nano clay, nano silica, or both in varied quantities. According to the results of this study, nano particles have the potential to improving the mechanical properties of concrete, which is an area that requires significant improvement. In addition, the results suggest that nano silica is more beneficial than nano clay and that moist combinations are more effective than dry ones [9]. Researchers have conducted experiment on addition of nanosilica dispersed in polycarboxylate (NDP) in concrete has been improved the mechanical properties and the microstructural characteristics of cementitious composites. The NDP content (0.4%) showed better results at 28 days, presenting a 55% increase in compressive strength, 79% tensile test and a 40% decrease in the absorption of water, in relation to the reference, possibly through the NDPs performance [10]. Researchers proved that the use of nanotechnology in the construction industry leads to substantial breakthroughs that result in denser concrete. These advancements were brought about as a consequence of the use of nanotechnology [11]. This paper, a novel pretreatment method was employed on the fly ash binder in terms of thermal and mechanical means. Also, a cost-effective nano fly ash powder was synthesized and used as filler material on the geopolymer system. The efficiency of the fabricated geopolymer mortar was assessed by examining the workability, compressive strength, and resistance against chloride ion penetration [12]. The results of this study indicate that nano particles have the potential to become a highly effective tool for improving both the mechanical properties and the durability of concrete [13]. Nanoparticles such as nanoZrO_2, nanoFe_3O_4, nanoTiO_2, and nanoAl_2O_3 were added to high-performance concrete mixes in an experiment carried [14]. Researchers conducted a series of tests on a mortar cube that had been made by combining cement with 2.5 percent nano fly ash and nano lime. The results of these tests were published in the journal Scientific Reports. These tests were conducted with the intention of determining the mortar's capacity to endure the force of compression [15]. Study aims to analyze the influence of the addition of silica nanoparticles in cementitious composites, contents of 0.5%, 0.75% and 1.0% of nanosilica will be added in relation to the cement mass. The cementitious composites were analyzed using the compressive strength test at 3, 7 and 28 days, water absorption and acid etching, all at 28 days of curing. From the results, the densification causes improved compressive strength, reduced water absorption and acid attack [16]. The inclusion of Nano-silica as a supplementary cementitious materials (SCM) up to certain extent can enhance mechanical and durability properties of concrete. Concrete compressive strength increases as the content of Nano-silica increases. Nano-silica has a very high pozzolanic activity. Filling effect of Nano-silica notably influences the refinement of pore structure [17].

2. MATERIALS AND METHODS

2.1. Materials

Ordinary Portland Cement (OPC) of the 53 Grade, which obeys with IS 8112-2013 [18], is used here. The natural sand, was taken up from the Cauvery River used as fine aggregate. According to the results, the sand falls under Zone III of the IS: 383 – 1970 [19]. The properties of sand were found, based on the methods described in IS 2386, Part I, 1963 [20]. The coarse aggregate for this project has been acquired, from nearby granite quarries. Twenty millimetres is the largest allowable particle size for the coarse aggregate that may be utilized. The methods for testing coarse aggregate that are specified in the standard IS: 2386 (Part III)-1963 [21] are used in the analysis of the aggregate's properties. As per the specifications available in IS: 456–2000 [22], potable water, which is free from salt was utilized throughout the casting and curing of concrete.

This experiment used the fly ash that was procured from the Mettur Thermal Power Station. This fly ash is thought to be of the calcium kind, since it has a specific gravity of 2.2 and a fineness value of 5.5%. Figure 1 shows the raw fly ash and nano fly ash samples. As per the requirements of IS 10262–2009 [23], a mix has been developed with the aim of producing concrete for a grade of M20, with the ratio of 1:1.10:3.15. The quantities of each component to make one cubic meter of concrete are listed in Table 1.

2.2. Methodology

Conducting durability tests are more important to find the survivability of concrete in adverse weather conditions. The specimens have been undergone for acid, sulphate and chloride attack test.

2.2.1. Acid attack test

Concrete cubes are cast, cured in water at room temperature for a total of 28 days, and then immersed in a solution containing 5% hydrochloric acid (Hcl) that has been diluted with distilled water for 180 days in order to study the acid resistance of concrete. The solution is dumped after 15 days, when its concentration drops, and it is then supplied on a regular schedule. The samples are evaluated every thirty days, and the submerged samples are visually scrutinized on the surface for acid attack damage to the concrete. An examination is conducted once

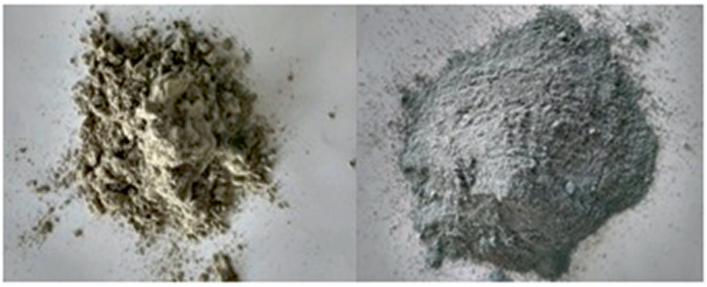

Figure 1: Nano fly and raw fly ash samples.

Table 1: Mix proportion of the various concrete.

MIX ID	CEMENT	F.A.	C.A.	W/C	RAW FLY ASH	NANO FLY ASH
CC	352	739.2	1108.8	0.45	–	–
NFAC 23	271.04	739.2	1108.8	0.45	–	80.96
RFAC 19	285.12	739.2	1108.8	0.45	66.88	–
RFAC 23	271.04	739.2	1108.8	0.45	80.96	–

every thirty days. Two examples of the tests that are performed on the specimen while it is immersed are weight loss and compression strength loss.

$$\text{Percentage of loss in weight} = (W_1 - W_2) / W_1 \quad (1)$$

Where, W_1 – Weight of cube at 28 days of normal water curing

W_2 – Weight of cube after 30, 60, 90, 120, 150, 180 days of immersion in acid medium

$$\text{Percentage of loss in compression strength} = (C_1 - C_2) / C_1 \quad (2)$$

Where, C_1 – Compression strength of cube after 28 days of normal water curing

C_2 – Compression strength of cube after 30, 60, 90, 120, 150, 180 days of immersion in acid medium

2.2.2. Sulphate attack test

The sulphate attack test is carried over the cube specimen after 28 days of normal water curing. After 28 days of water curing, the specimens have been immersed in 5% of sodium sulphate (H_2SO_4) solution which has been diluted with distilled water for 180 days. The solution is replaced to maintain its concentration level every 15 days of interval. The specimens are tested every 30 days of interval to observe its loss in strength and weight due to sulphate attack.

2.2.3. Chloride attack test

The cube specimen has been subjected to the chloride attack test, before that those cubes were submerged in normal water for curing for a period of 28 days. When the specimens have been cured in water for 28 days, they are next submerged for 180 days in a 5% sodium chloride (NaCl) solution that has been diluted with distilled water. After every 15 days, the solution was discarded and replenished in order to keep the concentration level constant. The specimens are examined at regular intervals of 30 days to track the strength and weight loss that have occurred as a result of the chloride attack.

3. RESULTS AND DISCUSSION

3.1. Ball milling process

Ball milling is a method that uses the impact and wear concept to decrease the size of minerals to a more manageable level. The force of the ball's impact on the particles will cause the particles to be shredded. Ball mills are used extensively throughout the mechanical alloying operations, in addition to the cold-welding process.

In addition, the production of alloys from powders may be accomplished by this procedure. The ball mill only has a portion of its capacity filled up. A ball formed of various materials such as steel, iron, graphite, etc. serves as the grinding medium [24].

3.2. X-ray diffraction analysis

Able to identify the fraction of the powder that was composed of crystalline structures by using this test. The X-ray diffraction analysis has the objective of qualitatively as well as quantitatively determining the chemical levels of the minerals and powders that are being assessed. This may be done by comparing the results of the study. A high-precision X-ray diffraction analysis may be used to determine the layer characteristics of semiconductor thin films, including the composition, thickness, roughness, and density of the layer. When measuring these metrics, the whole film is taken into consideration. Small-perspective X-ray scattering and pair distribution function (PDF) analysis are both beneficial methods that may be used to explore the structural features of nanomaterials. Figures 2 and 3 provide some examples of XRD.

The amount of fly ash that, when replaced with cement, results in an improvement in the strength of the concrete is the subject of a few pieces of research that have been conducted. Because certain peaks can be seen in Figure 2, it may be inferred that the fly ash contains chemical components such as silicon oxide and calcium magnesium aluminum silicate. This is supported by the fact that these peaks exist [25]. The existence of this data suggests that its incorporation may contribute to increased concrete strength. In a manner fairly similar to that described above, Figure 3 presents an illustration of the chemical components that may be found in nanofly ash. The peak that has the largest height implies that the material has the maximum amount of a certain chemical, such as calcium silicon oxide or silicon oxide. This may be determined by looking at the peak's elevation. According to the findings, nanoscale fly ash is capable of filling the pores that are already present in concrete, which results in an improvement in the material's inherent strength. This may be attributed to the material's ability to prevent water absorption.

3.3. Scanning electron microscope results on fly ash

For both conducting and non-conducting materials, the surface morphology (Nano scale analytics) has been determined using the Field Emission Scanning Electron Microscope (FE-SEM). With an X flash detector energy resolution ≥126 eV, the linked EDS detector provides precise qualitative elemental analysis. The SEM morphology of raw fly ash and nano fly ash samples are displayed in Figures 4 and 5. The outcomes of the aforementioned tests indicate that the size of the nanofly ash that was created after the eighth hour of the ball milling process has been confirmed with the assistance of SEM analysis. These findings were presented before. This evidence will guide the investigation in the direction of an experimental strategy and provide further support.

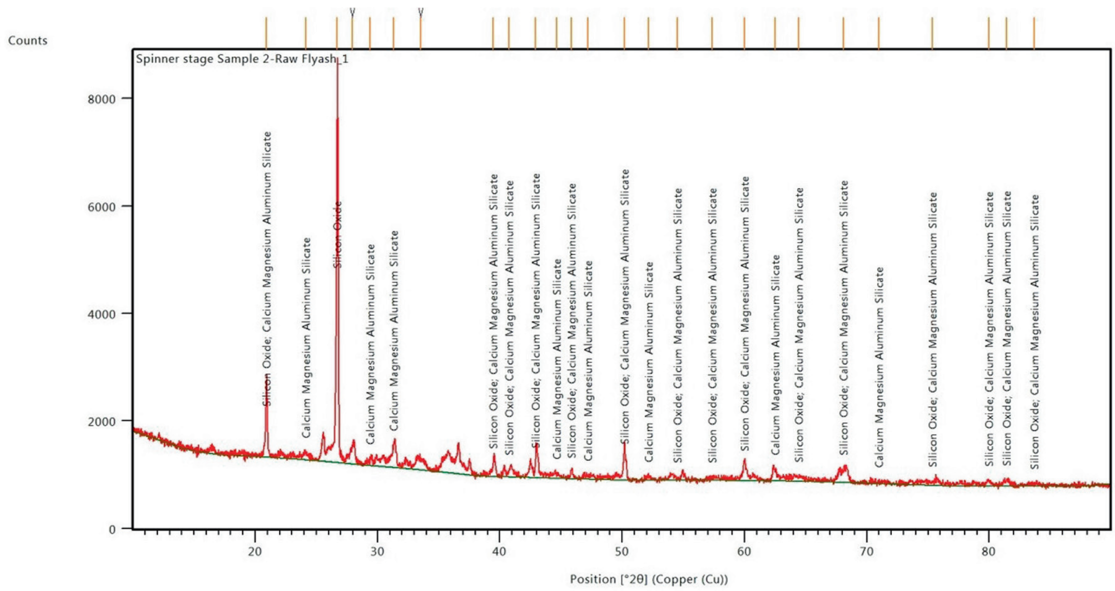

Figure 2: XRD image of fly ash.

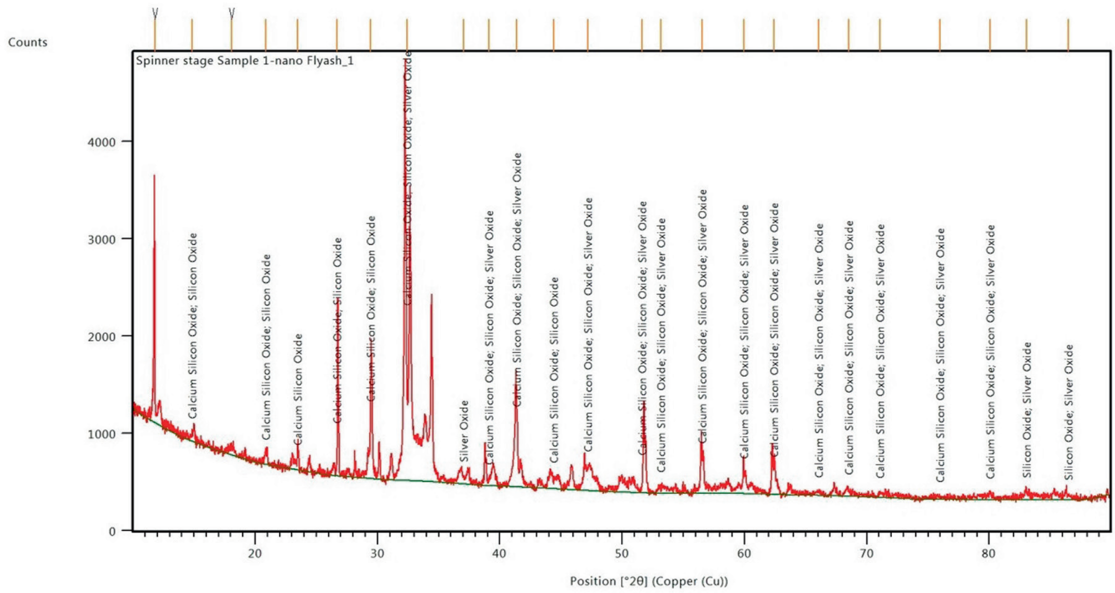

Figure 3: XRD Image of nano fly ash.

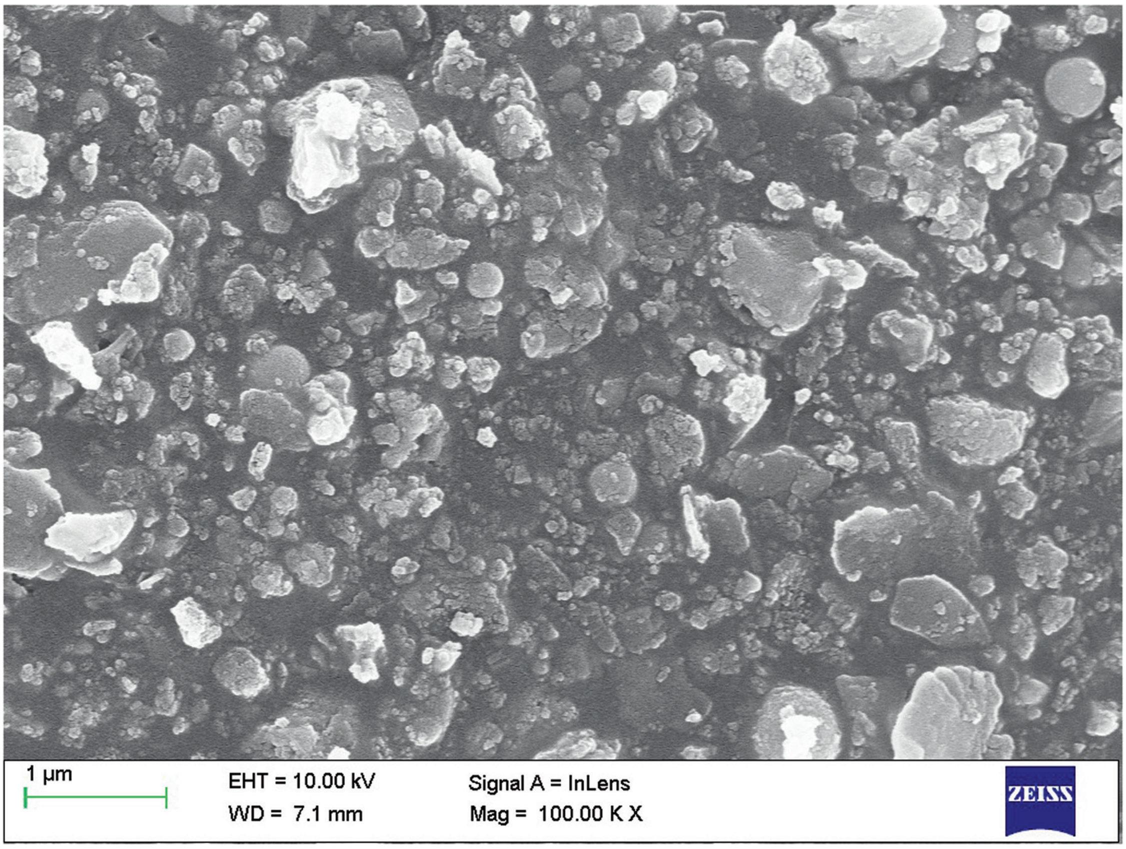

Figure 4: Morphology of raw fly ash @ 1 µm.

3.4. Acid attack test

The acid attack test has been conducted over the cube specimen for CC, RFAC-19, RFAC-23, and NFAC-23. After completion of normal water curing, the cubes were immersed in acid for 30, 60, 90, 120, 150 & 180 days curing then undergone for the loss of weight and compression strength test. Table 2 shows the test results of acid

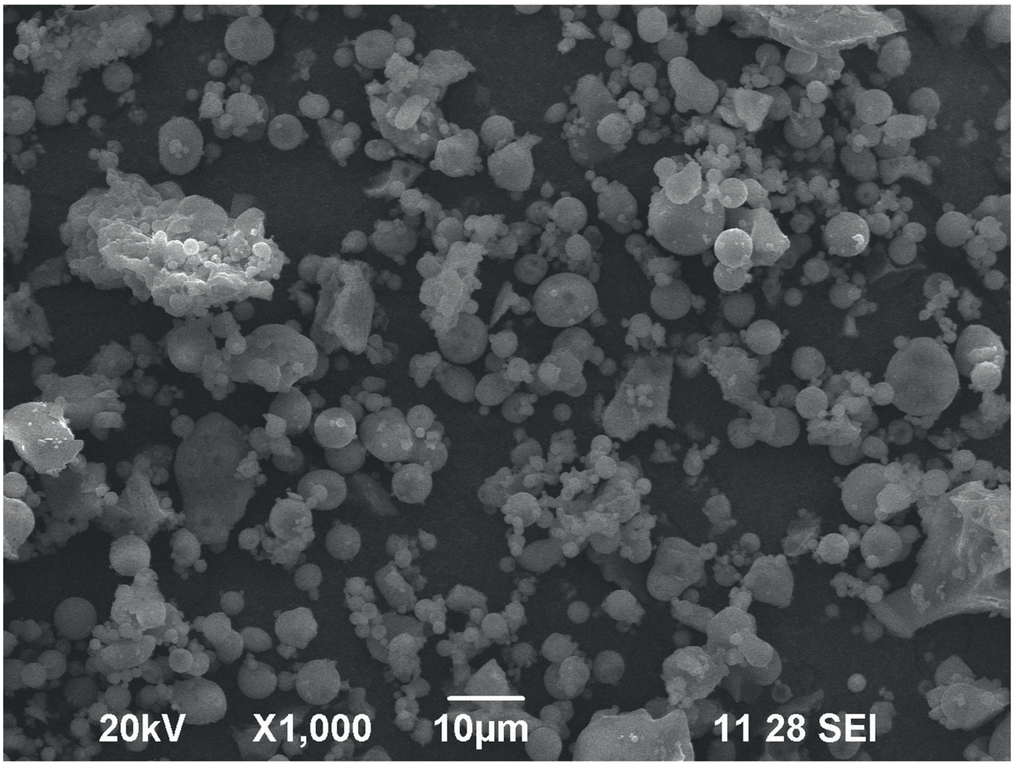

Figure 5: Morphology of nano fly ash @ 10 μm.

Table 2: Acid attack test results.

MIX ID / NO OF DAYS	COMPRESSIVE STRENGTH TEST (MPa)					
	30	**60**	**90**	**120**	**150**	**180**
CC	25.8	25.4	25	24.3	23.5	22.8
NFAC 23	34.5	34.3	34.2	33.8	33.5	33.2
RFAC 19	27.4	27.1	26.8	26.2	25.6	25.1
RFAC 23	26	25.7	25.5	24.9	24.3	23.8
MIX ID / NO OF DAYS	**PERCENTAGE OF WEIGHT LOSS (%)**					
	30	**60**	**90**	**120**	**150**	**180**
CC	1%	8%	16%	31%	45%	60%
NFAC 23	0.10%	6%	11%	23%	34%	45%
RFAC 19	2%	9%	16%	30%	44%	58%
RFAC 23	2.50%	11%	20%	33%	51%	60%

attack test. Figure 6 shows the surface profile of the cube specimen after acid attack of conventional, raw fly ash and nano fly ash specimens.

The CC has the compression strength test value of 25.80 N/mm^2 after normal curing. Then the specimens were immersed in acid medium and tested after 30, 60, 90, 120, 150 and 180 days. In that the corresponding compression strength test results were 25.80, 25.40, 25.00, 24.30, 23.50 and 22.8 N/mm^2. The loss in compression strength corresponding to its normal curing was 0.16%, 1.55%, 3.10%, 5.81%, 8.91% and 11.63%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 1%, 8%, 16%, 31%, 45% & 60% corresponding to its days of immersion in acid medium.

The NFAC-23 has the compression strength test value of 34.51 N/mm^2 after water curing. The compression strength of the specimen received from acid medium after 30, 60, 90, 120, 150, 180 days are 34.50, 34.30,

Figure 6: Typical surface profile and failure pattern of cube after acid attack.

34.20, 33.80, 33.50 and 33.20 N/mm^2. The loss in compression strength corresponding to its normal curing was, 0.03%, 0.61%, 0.90%, 2.06%, 2.93% & 3.80%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 0%, 6%, 11%, 23%, 34% & 45% corresponding to its days of immersion in acid medium.

The RFAC-19 has the compression strength test value of 27.40 N/mm^2 after 28 days of water curing. The compression strength of the specimens received from acid curing after 30, 60, 90, 120, 150, 180 days are 27.40, 27.10, 26.80, 26.20, 25.60 and 25.10 N/mm^2. The loss in compression strength corresponding to its normal curing was 0.07%, 1.09%, 2.19%, 4.38%, 6.57% and 8.39%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 2%, 9%, 16%, 30%, 44% and 58% corresponding to its days of acid curing.

The RFAC-23 has the compression strength test value of 26.90 N/mm^2 after 28 days of water curing. The compression strength of the specimens from acid curing after 30, 60, 90, 120, 150, 180 days are 26.00, 25.70, 25.50, 24.90, 24.30 and 23.80 N/mm^2. The loss in compression strength corresponding to its normal curing is 3.30%, 4.29%, 5.35%, 7.47%, 9.59% & 11.36%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 2.5%, 11%, 20%, 33%, 51% & 60% corresponding to its days of acid curing.

3.5. Sulphate attack test

The sulphate attack test has been conducted over the cube specimen for CC, RFAC-19, RFAC-23 and NFAC-23. After the completion of normal water curing, the cubes were immersed in sulphate curing for 30, 60, 90, 120, 150 & 180 days and then undergoes the loss of weight and compression strength test. The following Table 3 shows the test results of sulphate attack test. Figure 7 shows the typical surface of specimen after sulphate attack.

The CC has the compression strength test value of 25.80 N/mm^2 after normal curing. Then the specimens were immersed in sulphate curing and tested after 30, 60, 90, 120, 150, 180 days. The corresponding compression strength test results were 25.76, 25.30, 24.90, 24.10, 23.30 and 22.40 N/mm^2. The loss in compression strength corresponding to its normal curing is 0.16%, 1.94%, 3.49%, 6.59%, 9.69% and 13.18%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 1%, 8%, 30%, 40%, 44% and 54% corresponding to its days of sulphate attack.

The NFAC-23 has the compression strength test value of 34.51 N/mm^2 after water curing. The compression strength of the specimen received from sulphate curing after 30, 60, 90, 120, 150, 180 days are 34.50, 34.20, 34.00, 33.50, 33.00 and 31.40 N/mm^2. The loss in compression strength corresponding to its normal curing is, 0.03%, 0.90%, 1.48%, 2.93%, 4.38% and 9.01%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 3%, 12%, 28%, 33%, 38% and 44% corresponding to its days of sulphate curing.

The RFAC-19 has the compression strength test value of 27.40 N/mm^2 after 28 days of water curing. The compression strength of the specimens from sulphate curing after 30, 60, 90, 120, 150, 180 days are 27.38, 27.00, 26.70, 25.90, 25.20 and 24.50 N/mm^2. The loss in compression strength corresponding to its normal curing is 0.07%, 1.46%, 2.55%, 5.47%, 8.03% and 10.58%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 2%, 16%, 31%, 40%, 45% and 52% corresponding to its days of sulphate curing.

The RFAC-23 has the compression strength test value of 26.90 N/mm^2 after 28 days of water curing. The compression strength of the specimens from sulphate curing after 30, 60, 90, 120, 150, 180 days are 26.00, 25.40, 24.60, 23.60, 22.90 and 22.10 N/mm^2. The loss in compression strength corresponding to its normal curing is 3.30%, 5.65%, 8.68%, 12.38%, 14.75% and 18.03%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 8%, 20%, 34%, 46%, 51% and 56% corresponding to its days of sulphate curing.

Table 3: Sulphate attack test results.

MIX ID / NO OF DAYS	COMPRESSIVE STRENGTH TEST (MPa)					
	30	**60**	**90**	**120**	**150**	**180**
CC	25.76	25.3	24.9	24.1	23.3	22.4
NFAC 23	34.5	34.2	34	33.5	33	32.5
RFAC 19	27.38	27	26.7	25.9	25.2	24.5
RFAC 23	24.6	24.3	24	23.3	22.7	22.1
MIX ID / NO OF DAYS	**PERCENTAGE OF WEIGHT LOSS (%)**					
	30	**60**	**90**	**120**	**150**	**180**
CC	1%	23%	30%	40%	44%	54%
NFAC 23	3.00%	12%	28%	33%	28%	44%
RFAC 19	2%	16%	31%	40%	45%	52%
RFAC 23	8.00%	20%	34%	46%	51%	56%

Figure 7: Typical surface profile and failure pattern of cube after sulphate attack.

3.6. Chloride attack test

The chloride attack test has been conducted over the cube specimen for CC, RFAC-19, RFAC-23 and NFAC-23. After the completion of normal water curing, the cubes were immersed in chloride curing for 30, 60, 90, 120, 150 & 180 days and then undergoes the loss of weight and compression strength test. Table 4 shows the test results of chloride attack test. Figure 8 shows the typical surface profile of different specimens after chloride attack.

The CC has the compression strength test value of 25.80 N/mm^2 after normal curing. Then the specimens were immersed in chloride curing and tested after 30, 60, 90, 120, 150, 180 days. The corresponding compression strength test results were 25.70, 25.30, 24.80, 23.90, 22.90 and 22.00 N/mm^2. The loss in compression strength corresponding to its normal curing is 0.39%, 1.94%, 3.88%, 7.36%, 11.24% and 14.73%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 5%, 24%, 36%, 44%, 52% and 64% corresponding to its days of chloride attack.

The NFAC-23 has the compression strength test value of 34.51 N/mm^2 after water curing. The compression strength of the specimen received from chloride curing after 30, 60, 90, 120, 150, 180 days are 34.50, 34.00, 33.60, 32.60, 31.70 and 30.80 N/mm^2. The loss in compression strength corresponding to its normal curing is 0.03%, 1.48%, 2.64%, 5.53%, 8.14% & 10.75%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 2%, 19%, 32%, 44%, 46% & 53% corresponding to its days of chloride curing.

The RFAC-19 has the compression strength test value of 27.40 N/mm^2 after 28 days of water curing. The compression strength of the specimens from chloride curing after 30, 60, 90, 120, 150, 180 days are 27.38, 26.90, 26.50, 25.60, 24.80 and 23.90 N/mm^2. The loss in compression strength corresponding to its normal curing is 0.07%, 1.82%, 3.28%, 6.57%, 9.49% & 12.77%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 4%, 24%, 34%, 46%, 54% & 62% corresponding to its days of chloride curing.

Table 4: Chloride attack test results.

MIX ID / NO OF DAYS	COMPRESSIVE STRENGTH TEST (MPa)					
	30	**60**	**90**	**120**	**150**	**180**
CC	25.76	25.3	24.8	23.9	22.9	22
NFAC 23	34.5	34	33.6	32.6	31.7	30.8
RFAC 19	27.38	26.9	26.5	25.6	24.8	23.9
RFAC 23	25.2	24.7	24.4	23.6	22.8	22
MIX ID / NO OF DAYS	**PERCENTAGE OF WEIGHT LOSS (%)**					
	30	**60**	**90**	**120**	**150**	**180**
CC	5%	24%	36%	44%	52%	64%
NFAC 23	2.00%	19%	32%	44%	46%	53%
RFAC 19	4%	24%	34%	46%	54%	62%
RFAC 23	6.00%	28%	36%	49%	58%	69%

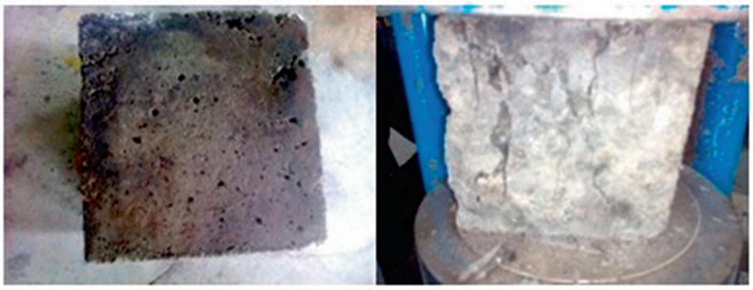

Figure 8: Typical surface profile and failure pattern of cube after chloride attack.

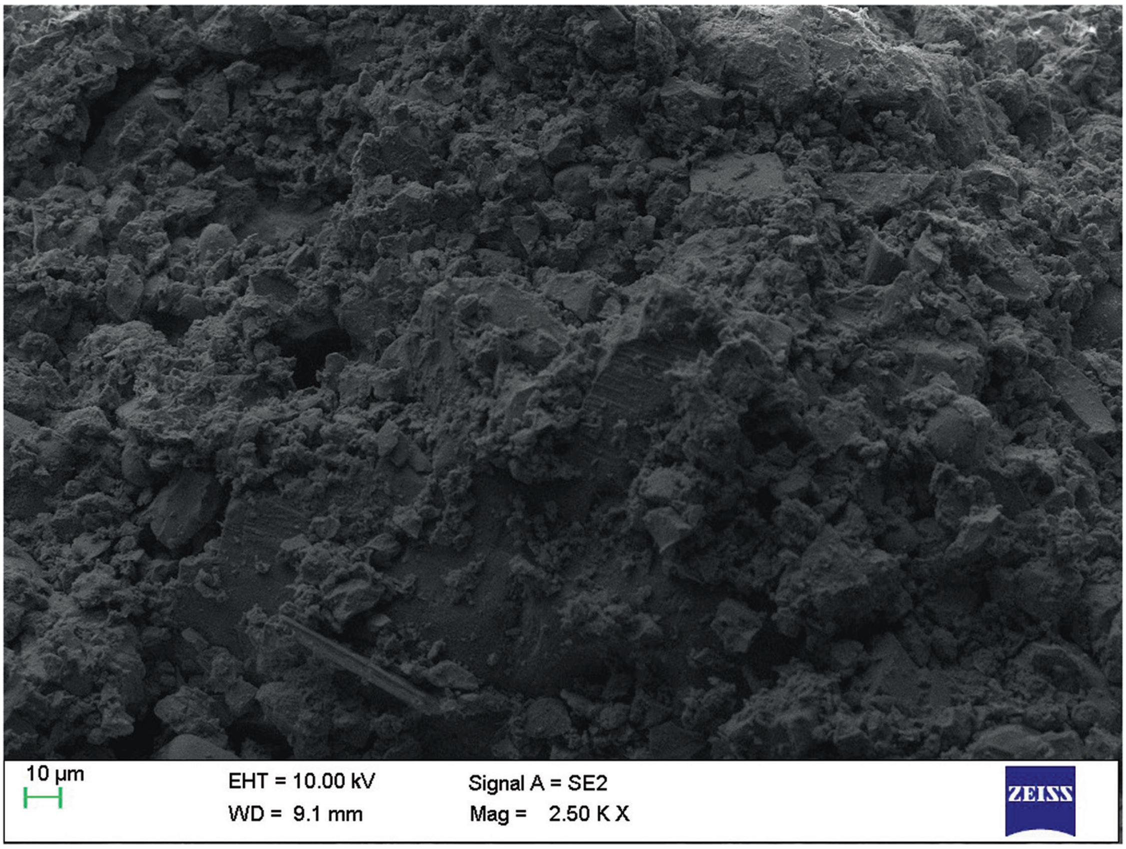

Figure 9: SEM – morphology of CC specimen.

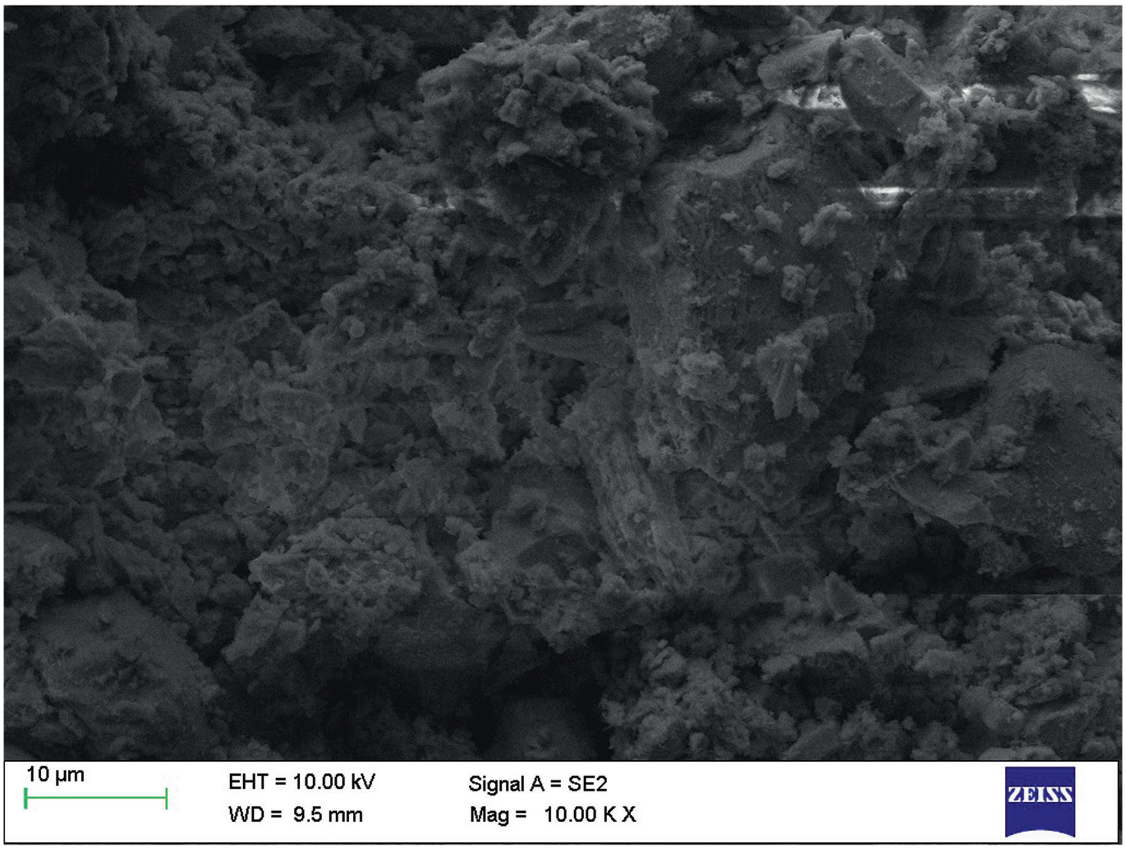

Figure 10: SEM – morphology of RFAC-19 specimen.

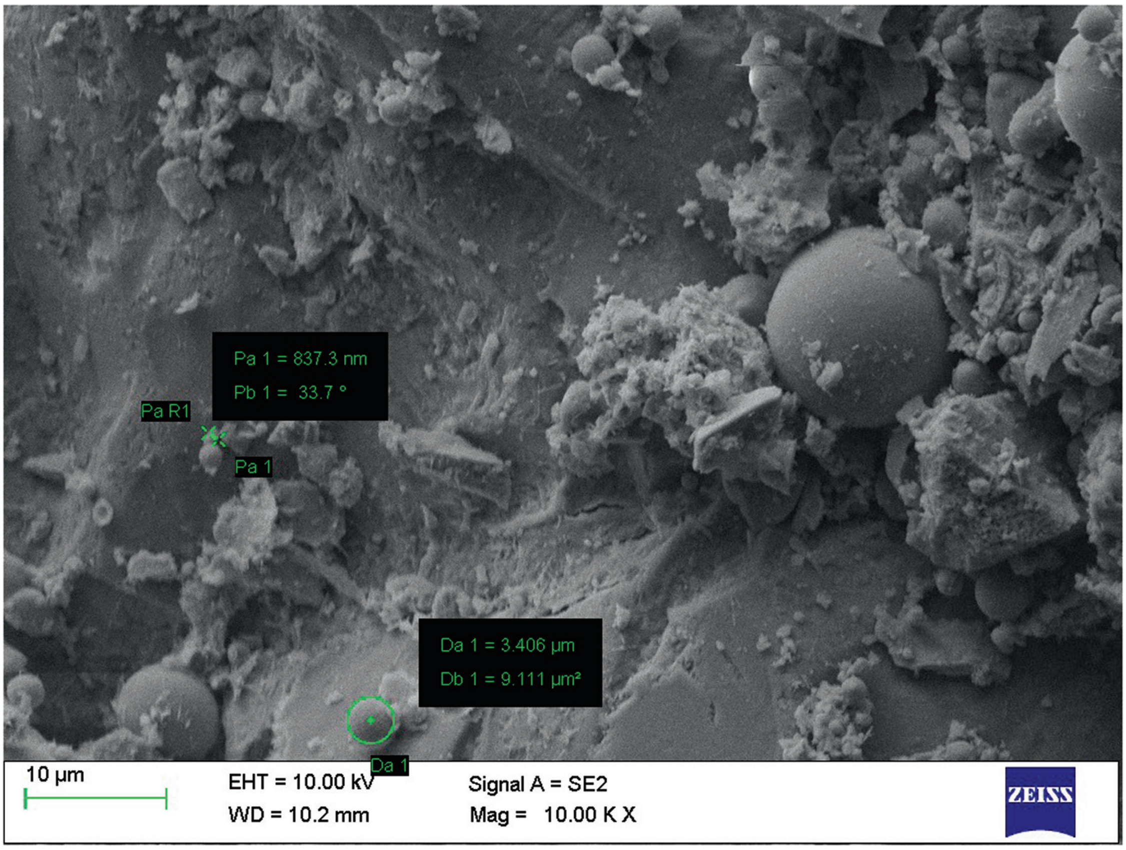

Figure 11: SEM – morphology of NFAC-23.

The RFAC-23 has the compression strength test value of 26.90 N/mm^2 after 28 days of water curing. The compression strength of the specimens from chloride curing after 30, 60, 90, 120, 150, 180 days are 26.30, 25.60, 24.90, 24.60, 23.10 and 22.20 N/mm^2. The loss in compression strength corresponding to its normal curing is 2.29%, 5.00%, 7.40%, 8.64%, 14.26% & 17.37%. Similarly, the weight loss of the cube specimen with respect to its normal cube weight is 6%, 28%, 36%, 49%, 58% & 69% corresponding to its days of chloride curing.

The observation found from the acid, sulphate and chloride attack test results after 180 days is that the addition of nano fly ash fills the pores accumulated with cement particles as it is rich in calcium and silicon. It makes concrete specimen much denser. So the voids present in the concrete are filled and is reduced much more rather than other concrete specimen and it more often offers good resistivity against this environmental condition.

3.7. Scanned electron microscopy results on concrete specimen

The scanned electron microscopy tests are used to find the morphology of the specimen. The SEM analysis has taken over the concrete specimen of size 10 mm × 10 mm × 10 mm. The above test results are derived from SEM analysis, in which the morphology of the CC, RFAC-19, and NFAC-23 has been displayed in Figures 9–11. The NFAC morphology ensures the presence of nano fly ash particles in the concrete. RFAC and CC did not have much variation in their morphology but the surface has the spreader particles of fly ash in RFAC.

4. CONCLUSIONS

Based on Durability Properties of test;

- The results achieved through acid attack test, alkaline attack test and sulphate attack test show that the NFAC has the value of loss of weight lesser when compared with CC, RFAC during the observation of 180 days duration.
- The compression strength values after 180 days of curing, the above test results did not indicate much difference. NFAC has the less value of loss in weight and loss in compression strength.
- The CC specimen has the loss in strength, varies from 0.02% to 14.7%. The NFAC-23 specimen has the loss in strength, varies from 0.03% to 10.75%. The RFAC-19 specimen has the loss in strength, varies from 0.03% to 12.8%. Also the RFAC-23 specimen has the loss in strength, varies from 3.35% to 18.22%.
- The CC specimen has the loss in weight, varies from 1% to 64%. The NFAC-23 specimen has the loss in weight, varies from 0.01% to 53%. The RFAC-19 specimen has the loss in weight, varies from 2% to 62%. Also the RFAC-23 specimen has the loss in weight, varies from 2.5% to 69%.
- According to the results of these studies, the nano particles of fly ash has been completely fill the pores which are available in the concrete, which makes the concrete to be much denser.

Based on SEM analysis test on concrete specimen;

- From the SEM analysis, the morphology of the NFAC specimen ensures that the presence of nano fly ash particles in the concrete is higher rather than the other specimen.
- Finally, the utilization of Nano sized fly ash in concrete leads to an effective alternate to produce concrete with good strength or high performance and also one of the effective minimization of wastage technique for the fly ash.

5. BIBLIOGRAPHY

[1] ANWAR MOHAMED, M., "Influence of nano materials on flexural behavior and compressive strength of concrete", *HBRC Journal*, v. 12, n. 2, pp. 212–225, 2016. doi: http://dx.doi.org/10.1016/j.hbrcj.2014.11.006.

[2] AMERICAN SOCIETY FOR TESTING AND MATERIALS, *ASTM C 1202: Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration*, West Conshohocken, ASTM international, 2009.

[3] RAO, J.B., NARAYANASWAMI, P., PRASAD, K.S., "Thermal stability of nano structured fly ash synthesized by high energy ball milling", *International Journal of Engineering Science and Technology*, v. 2, n. 5, pp. 284–299, 2010. doi: http://dx.doi.org/10.4314/ijest.v2i5.62577

[4] BHANUMATHIDAS, N., KALIDAS, N., "Fly ash: The resource for construction industry", *Indian Concrete Journal*, v. 77, n. 4, pp. 997–1004, 2003.

[5] HALL, C., "Water sorptivity of mortars and concretes: a review", *Magazine of Concrete Research*, v. 41, n. 147, pp. 51–61, 1989. doi: http://dx.doi.org/10.1680/macr.1989.41.147.51

[6] CHOUSIDIS, N., RAKANTA, E., IOANNOU, I., *et al*., "Mechanical properties and durability performance of reinforced concrete containing fly ash", *Construction & Building Materials*, v. 101, pp. 810–817, 2015. doi: http://dx.doi.org/10.1016/j.conbuildmat.2015.10.127

[7] SCHIAVON, J.Z., BORGES, P.M., SILVA, S.R.D., *et al*., "Analysis of mechanical and microstructural properties of high performance concretes containing nanosilica and silica fume", *Revista Materia*, v. 26, n. 4, pp. 304–319, 2021. doi: http://dx.doi.org/10.1590/s1517-707620210004.1304

[8] BEHFARNIA, K., SALEMI, N., "The effects of nano-silica and nano-alumina on frost resistance of normal concrete", *Construction & Building Materials*, v. 48, pp. 580–584, 2013. doi: http://dx.doi.org/10.1016/j.conbuildmat.2013.07.088

[9] RAFIEIZONOOZ, M., MIRZA, J., SALIM, M.R., *et al*., "Investigation of coal bottom ash and fly ash in concrete as replacement for sand and cement", *Construction & Building Materials*, v. 116, pp. 15–24, 2016. doi: http://dx.doi.org/10.1016/j.conbuildmat.2016.04.080

[10] NOGUEIRA, G.S.F., SCHWANTES-CEZARIO, N., SOUZA, I.C., *et al*., "Incorporation of nanossílica in cement composites", *Revista Matéria*, v. 23, n. 3, pp. 1–12, 2018. doi: http://dx.doi.org/10.1590/s1517-707620180003.0516

[11] PRINCE ARULRAJ, G., JEMIMAH CARMICHAEL, M., *et al*., "Effect of nanoflyash on strength of concrete", *International Journal of Civil and Structural Engineering*, v. 2, n. 2, pp. 475–482, 2011.

[12] MOHANA, R., BHARATHI, S.L., "Sustainable utilization of pre-treated and nano fly ash powder for the development of durable geopolymer mortars", *Advanced Powder Technology*, v. 33, n. 8, pp. 103696, 2022. doi: http://dx.doi.org/10.1016/j.apt.2022.103696

[13] SAID, A.M., ZEIDAN, M.S., BASSUONI, M.T., *et al*., "Properties of concrete incorporating nano-silica", *Construction & Building Materials*, v. 36, pp. 838–844, 2012. doi: http://dx.doi.org/10.1016/j.conbuildmat.2012.06.044

[14] SHEKARI, A.H., RAZZAGHI, M.S., "Influence of nano particles on durability and mechanical properties of high performance concrete", *Procedia Engineering*, v. 14, pp. 3036–3041, 2011. doi: http://dx.doi.org/10.1016/j.proeng.2011.07.382

[15] SRI, T., "Study the effect of adding nano fly ash and nano lime to compressive strength of mortar", *Procedia Engineering*, v. 95, pp. 426–432, 2014. doi: http://dx.doi.org/10.1016/j.proeng.2014.12.202

[16] LONGO, T., DE CAMARGO, G.F.N., ALBERTIM, M.F., *et al*., "Study of mechanical strength and strength to acid attack on cementitious composites with incorporation of silica nanoparticle", *Revista Materia*, v. 27, n. 4, pp. 346–355, 2022.

[17] KUMAR, V.P., DEY, S., "Study on strength and durability characteristics of nano-silica based blended concrete", *Hybrid Advances*, v. 2, pp. 1–15, 2023.

[18] INDIAN STANDARD, *IS8112: Ordinary Portland Cement, 43 Grade - Specification*, New Delhi, Bureau of Indian Standards, 2013.

[19] INDIAN STANDARD, *IS383: Specification For Coarse And Fine Aggregates From Natural Sources For Concrete*, New Delhi, Bureau of Indian Standards, 1970.

[20] INDIAN STANDARD. *IS2386 (Part I): Methods of Test For Aggregates For Concrete Part I Particle Size and Shape*, New Delhi, Bureau of Indian Standards, 1963.

[21] INDIAN STANDARD, *IS2386 (Part III): Methods of Test for Aggregates for Concrete Part III – Specific Gravity, Density, Voids, Absorption and Bulking*, New Delhi, Bureau of Indian Standards, 1963.

[22] INDIAN STANDARD, *IS456, Plain and Reinforced Concrete – Code of Practice*, New Delhi, Bureau of Indian Standards, 2000.

[23] INDIAN STANDARD, *IS10262: Concrete Mix Proportioning – Guidelines*, Bureau of Indian Standards, New Delhi, 2009.

[24] HARIHANANDH, M., VISWANATHAN, K.E., KRISHNARAJA, A.R., "Comparative study on chemical and morphology properties of nano fly ash in concrete", *Materials Today: Proceedings*, v. 45, pp. 3132–3136, 2021. doi: http://dx.doi.org/10.1016/j.matpr.2020.12.217

[25] HARIHANANDH, M., AMUDHAVALLI, N.K., "Micro structural study on nano fly ash concrete", *Ecology Environmental Conservation*, v. 25, n. 2, pp. 740–744, 2019.