

Effect of Nano Iron Oxide on Strength and Durability Characteristics of High Volume Fly Ash Concrete for Pavement Construction



Bimal Kumar, Sanjeev Sinha, Hillol Chakravarty

Abstract: Cement is the principal component of cement concrete used for construction of rigid pavements and is produced by an energy intensive process. Large scale production and its subsequent utilization detrimentally contributes towards global warming. In order to cater for sustainable development, there is a need to utilize waste materials having cementitious properties as a partial substitute for cement. Fly ash is one of such waste which is being extensively used for the production of cement concrete. Concrete produced by utilizing fly ash more than fifty percent of cement is termed as high volume fly ash concrete (HVFAC). Although HVFAC facilitates utilization of large volume of fly ash, it however has the disadvantage of delayed gain in strength which limits its usage as pavement quality concrete (PQC). Contemporary literatures show the usage of various types of nanomaterials to overcome this disadvantage. The present study was carried out to investigate the influence of nano iron oxide on strength and durability properties of HVFAC. The HVFAC used in the study was prepared by replacement of fifty five percent ordinary Portland cement with F-type fly ash obtained from thermal power plant. Nano iron oxide was utilized in different percentages to improve the strength and durability characteristics of HVFAC. The strength properties of the concrete was evaluated by flexural, compressive and split tensile strength tests, whereas the durability characteristics were evaluated by density, permeability, sorptivity, ultrasonic pulse velocity and rapid chloride penetration tests. The tests were carried out at 28, 56 and 90 days age of concrete. The test result showed that HVFAC modified with 0.75% nano iron oxide by weight gave the optimal strength and durability results which were comparable with that of normal cement concrete used for construction of rigid pavements.

Index Terms: Durability, HVFAC, Nano Iron oxide, Rigid Pavements, Strength.

I. INTRODUCTION

Rigid pavements have high flexural strength and are made of cement concrete. With its inherent high flexural strength,

long service life and lesser susceptibility to moisture damage, the rigid pavements have gained much popularity in recent years. According to estimate of EAPA & NAPA [1], about one-tenth of all the paved roads in Europe, USA and Canada are of this type respectively. The increase demand for rigid pavements has necessitated the availability of cement and other cementing materials for construction of such pavements. The cement concrete is obtained by mixing cementing material, water, aggregates and sometimes admixtures in required proportions which hardens into rock-like mass [2]. Cement production is an energy intensive process. It involves producing clinkers by heating limestone at 1450°C which is achieved by burning fossil fuel leading to emission of CO₂. 0.8 tons of CO₂ is estimated to be produced for each ton of production of Portland clinkers [3]. The emission of CO₂ by cement industry is 5 -7 % of total CO₂ emission [4, 5]. CO₂ is one of the significant greenhouse gases leading to global warming. This emission continues to increase with time leading to environmental degradation [6]. This had led to development of green concrete which uses sustainable waste cementitious material thereby leading to reduced mining, manufacturing emissions and air pollutants [7]. Fly ash which is a waste product of thermal power plants, has cementitious properties and is now being utilized globally in cement and cement concrete production. In thermal power station pulverized coal is fed into boiler furnace. Lighter and finer particles fly along with flue gases which are passed through electrostatic precipitator (ESP), mechanical separators or bag fillers are collected and is called fly ash [8, 9]. Worldwide large quantities of fly ash are being produced. China, USA and Germany produce about 100, 75 and 40 million tons of fly ash every year. In India too, fly ash production is huge at 196.44 MT in the year 2017-18 [10]. Therefore, its usage becomes important as it otherwise results discharging into land-reservoirs as ash-ponds. When it ends up as land-fill, fly ash not only occupy very significant amount of agricultural land but also is detrimental as toxic elements present in it contaminates the environment [11]. Disposing the fly ash is cost consuming too, which is borne by the company and in turn, by the customers. Thus, utilizing fly ash for other purposes is crucial. Fly ash has utilities in various activities such as supplementary cementitious material (SCM) in cement and concrete, backfills, embankment and base course construction in pavements, subgrade stabilization, soil improvement, control of water pollution, light weight aggregates, bricks, blocks and paving stones etc. [7,9].

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The fly ash which is being produced in coal based thermal power plants are differentiated into two kinds depending on the Calcium oxide content. Mostly on combusting anthracite or bituminous coal, results in production of fly ash with amount of reactive Calcium oxide less than 10% and is known as class F fly ash. Class C fly ash contains more than 10% and upto 40% of reactive Calcium oxide is produced by combusting sub-bituminous or lignite coal [12].

However, it has been observed that the quantity of fly ash utilized by various countries is rather limited. The utilization of fly ash in China and India which are the two most fly ash producing nations, were at about 45% and 38% respectively in 2014 [13]. Ahmaruzzaman [11] pointed out numerous economical, institutional, technical and economic barriers which hinders excessive utilization of fly ash. Reduced amount of utilization of fly ash is subjected to the reason that they usually do not comply to standard set forward for its utilization by various agencies [14]. Results have shown that when large volume of fly ash was used as tertiary SCM in concrete production, the resulting concrete was observed to have delayed gain for strength because of late hydration of fly ash [15]. However, recent policies of sustainable development by various governments all over the globe has emphasized on its utilization at larger quantities by various developed nations such as Denmark, Italy, Netherlands, France, Germany etc. [13].

As per the definition given by Mehta [16], a concrete having a minimum cement replacement level of 50% by fly ash is termed as high volume fly ash concrete (HVFAC). Although fly ash in concrete enhances various properties of concrete such as lesser water demand, reduced heat of hydration applicable for mass concreting, reduced bleeding, durability against sulphate resistance is increased etc. [12] but due to lesser early strength as mentioned above, the use of such high volume has been limited. These drawbacks of fly ash concrete could be mitigated by promoting faster pace of C-S-H gel formation by addition of some external agents. Hence modification is sine-qua-non for achievement of high early strength.

Taking into account the above problem, large number of relevant literatures were explored and it have been observed that nano-materials are effective in overcoming the delayed strength as well as durability requirements of HVFAC with its potential application in its use as concrete for rigid pavements. Nanotechnology can be defined as the design, characterization, production and application of structures, devices and systems by controlling shape and size at the nano scale. Whereas, nano materials can be defined as physical substances with at least one dimension between 1 to 100 nm.

Nanotechnology, thus in concrete, is considered as an innovative way of improving concrete and cementitious properties at an atomic scale [17]. Nano-concrete is prepared by Portland cement and some other particles enhancing cementitious properties with dimensions less than 500 nms. [18, 19].

II. LITERATURE REVIEW

Nazari *et al.* [20] investigated compressive strength and workability of concrete prepared by partial replacement of

cement using nano Fe_2O_3 particles of 0.5%, 1.0%, 1.5% and 2% by weight. The size of nanoparticles was determined to be 15 nm. It was observed that the strength of concrete increased with respect to control samples with increased replacement. However, the maximum strength was attained at 1.0% replacement. However, the workability decreased continuously with increase in replacement. In another study, Nazari & Riahi [21] investigated split tensile strength, flexural strength and setting time of concrete with same replacement as above. The split tensile strength and flexural strength showed an increasing trend with increase in replacement, however, the setting time decreased. It was thereafter suggested that the split tensile strength could improve by using needle type reinforcement.

Oltulu & Sahin [22] studied the compressive strength and capillary water absorption of cement mortars containing fly ash using nano Fe_2O_3 (NF), nano Al_2O_3 (NA) and nano SiO_2 (NS) powders and their ternary and binary combinations at ratios corresponding to 0.5%, 1.25% and 2.5% of binder. An increase in 7-32% of compressive strength and 14% decrease in capillary absorption were determined to be the best result.

Yazdi *et al.* [23] conducted research to determine the compressive and tensile strength of cement mortar containing Fe_2O_3 nanoparticles of 1, 3 and 5% by weight of cement. An increase in mechanical properties was observed for 1& 3% use of nano Fe_2O_3 which decreased for 5%. SEM images confirmed that the nanoparticles fill up the pores. Reduction in large crystals of $\text{Ca}(\text{OH})_2$ nanoparticles were observed, and thus, hydrated products were observed to be denser and compact by filling C-S-H gel nanostructures. The decrease for 5% addition of nano Fe_2O_3 was subjected to the fact that the nanoparticles at higher concentration starts to agglomerate.

Mechanical strength properties such as compressive, flexure and split tensile strength as well as co-efficient of water absorption on high performance self-compacting concrete was measured. 4% addition of nano Fe_2O_3 was found to provide best results for strength and water permeability. This was explained by the hypothesis that nano Fe_2O_3 acts as foreign nucleation sites thereby accelerating C-S-H gel formation due to increase in $\text{Ca}(\text{OH})_2$ crystals at initial stage of hydration. Moreover, the nanoparticles acts as filler and improve the pore structure of specimen by containing the pores harmful as per water permeability is concerned. This is supported by the accelerated peak in conduction calorimetry tests, significant increased weight loss during thermo-gravimetric analysis and more rapid hydrated product related peaks in XRD [24].

III. METHODOLOGY

As per above literatures, this research was conducted to assess the effect of nano iron oxide on the strength and durability characteristics of high volume fly ash concrete.

The fig. 1 shows the step by step process applied for carrying out the research.

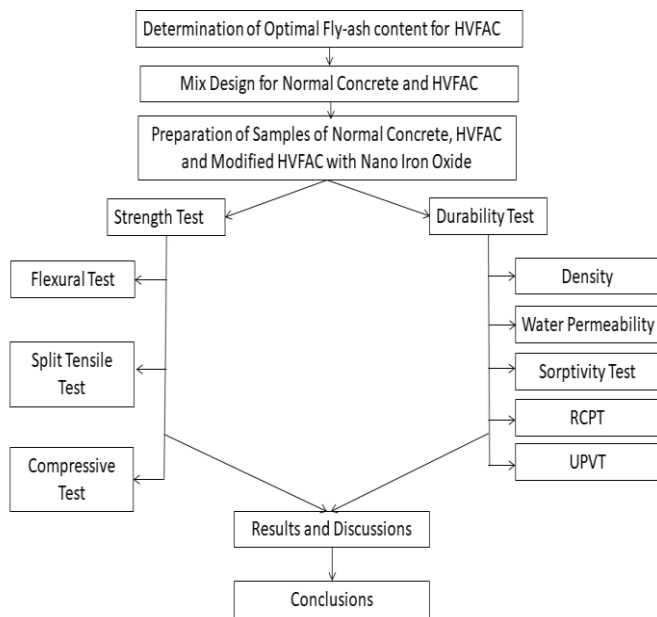


Fig. 1: Methodology for carrying out the research.

For determining the amount of Ordinary Portland Cement (OPC) to be replaced with fly ash for preparing high volume fly ash concrete (HVFAC), compressive strength test were performed. For this, five different types of samples of concrete were prepared by replacement of OPC, with F type fly ash by 0%, 50%, 60% and 65% respectively. Fly ash was obtained from National Thermal Power Corporation (NTPC), Kahalgaon, Bihar, India. Samples were water cured for 28 days and 90 days. Fig. 2 indicates that the 28 days and 90 days compressive strength of the HVFAC having 55% replacement by F type fly ash comes to 22.05 MPa, 42.6 MPa respectively and that of for 50% replacement are 22.51 MPa, 43.03 MPa respectively. It was observed that the 28 days and 90 days compressive strength of both samples are nearly equal.

Therefore 55% replacement of cement by the fly ash has been taken up in the present study as it utilizes more quantity of fly ash and thus is more sustainable.

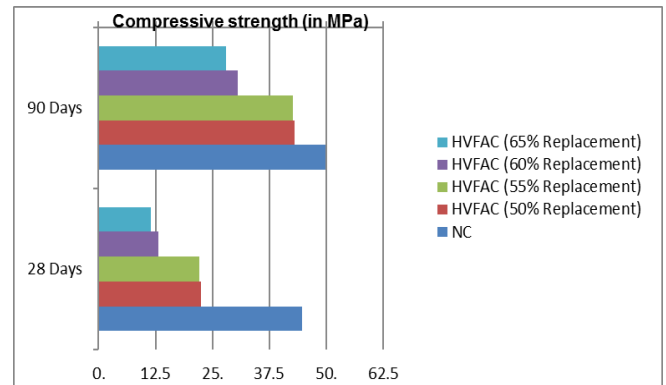


Fig. 2 Compressive strength of different types of concrete with varying percentage replacement of OPC by fly ash for HVFAC.

Mix proportioning for concrete was carried as per IS 10262: 2016 and IRC: 44-2008. The mix proportions for different types of concrete samples are given in the table 1. After the fly ash content was fixed at 55%, the subsequent samples were modified by adding 0.5%, 0.75% and 1% nano Fe₂O₃ respectively by weight of cementitious materials. Thus, samples were named as normal concrete (NC) having 0% fly ash, concrete prepared with 55% cement replaced by fly ash were named as high volume fly ash concrete (HVFAC); Concrete prepared with 55% cement replacement by fly ash and by adding 0.5% nano Fe₂O₃ of cementitious material as HVFAC + 0.5% NF; similarly HVFAC + 0.75% NF and HVFAC+1%NF. Nano iron oxide powder and superplasticizer were thoroughly mixed in 200 ml of water by a high speed rotor operated at 2500 rpm for 4 minutes. This sample is fed into rotating concrete mixture machine containing cement, fly ash and aggregates with rest amount of water.

Table 1 Designed mix proportions for different types of concrete sample in Kg/m³

Sample	OPC-43 Grade	Fly ash (Class F)	Coarse Aggregate	Fine Aggregate Zone II	Chemical Admixture (SP)	Water	Nano Fe ₂ O ₃
NC	383.13	Nil	1102.75	727.67	0.76	153.25	0
HVFAC	189.6	231.8	1085.8	716.5	0.76	153.25	0
HVFAC+ 0.5% NF	189.6	231.8	1085.8	716.5	0.76	153.25	1.92
HVFAC+ 0.75% NF	189.6	231.8	1085.8	716.5	0.76	153.25	2.87
HVFAC+ 1% NF	189.6	231.8	1085.8	716.5	0.76	153.25	3.83

The nano Fe₂O₃ used was obtained from Nano Lab Pvt. Ltd. which also provided specifications of the material as given under:

Table 2 Specifications of nano Fe₂O₃ obtained from Nano Lab

Average particle size (APS)	Specific Surface Area (SSA)	Colour	Morphology	True Density	Melting Point	Molar Mass
30-50 nm	20-60 m ² /g	Red Brown	Spherical	5.24 g/cc	1566°C	159.69g/ mol

The percentage of components present in nano Fe₂O₃ were provided by Nano Lab along with SEM images to predict the size of the nanoparticles. The respective table and figure are given below:

Table 3 Percentage analysis of different components of Nano Fe₂O₃ provided by Nano Lab.

Fe ₂ O ₃	Si	K	Mn	Zn
99.5%	0.07%	0.03%	0.1%	0.09%

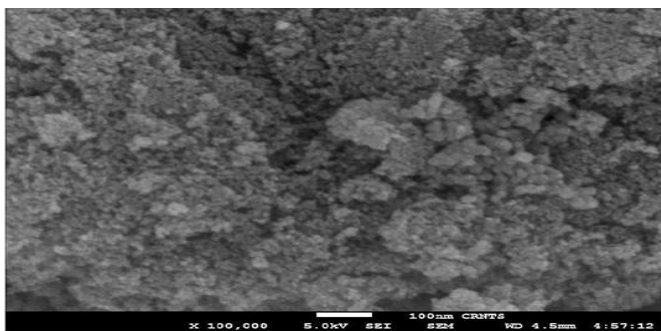


Fig. 3 SEM images of nano Fe₂O₃ (Source: Nano Lab Pvt. Ltd)

IV. RESULTS AND DISCUSSIONS

This study was an attempt to understand the effect of nano iron-oxide on strength and durability properties of high volume fly ash concrete having 55% cement replaced with fly ash.

A. Strength Tests

Strength tests were conducted on the samples made up of NC, HVFAC and modified HVFAC. The results obtained for the strength tests are depicted below:

Flexural strength test

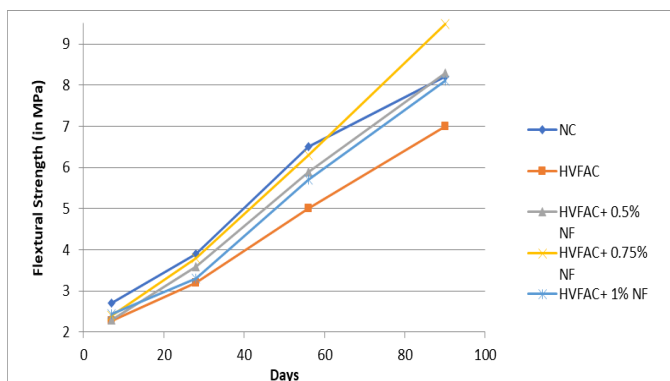


Fig. 4 Variation of flexural strength of NC, HVFAC and modified HVFAC with different percentages of nano Fe₂O₃ at different ages.

Flexural strength is the guiding parameter for the concrete used for rigid pavement construction as these pavements are designed on the basis of the ability of the concrete to resist flexural loads. The flexural strength test were conducted as per ASTM C78, third point loading method. The flexural strength of all samples of concrete were observed to increase with age. The values of HVFAC with 0.75 percent nano Fe₂O₃ have identical flexural strength with NC after 28 days that is about 4.0 MPa. The strength of HVFAC with 0.75 percent nano Fe₂O₃ becomes even more than that of NC after 56 days. The values of flexural strength of HVFAC with 0.5 percent and 1.0 percent nano Fe₂O₃ is lesser than that with 0.75 percent nano Fe₂O₃, however, it becomes almost equal to that of NC at about 90 days.

Compressive strength

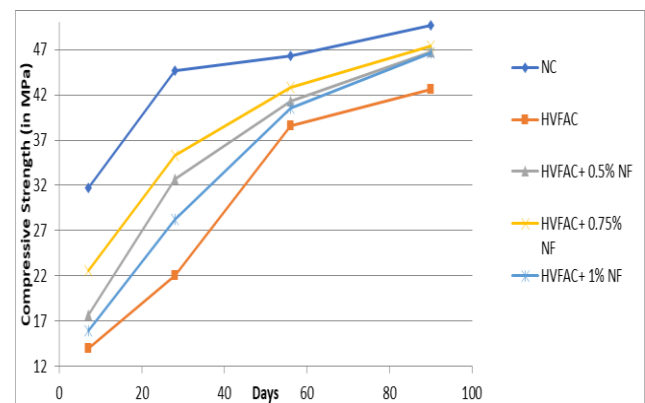


Fig. 5 Variation of compressive strength of NC, HVFAC and modified HVFAC with nano Fe₂O₃ at different ages.

The specimen were tested for compressive strength as per ASTM C39. The results of compressive strength were found for each mix at test age of 7, 28, 56 and 90 days respectively. A trend similar to that for flexural strength was observed. However the compressive strength for NC was observed to be higher than that of HVFAC with 0.75 percent nano Fe₂O₃. However, the gap between NC and HVFAC with nano Fe₂O₃ decreases with increase in age of the modified HVFAC. When compared to HVFAC it can be concluded that the addition of nano Fe₂O₃ increases the rate of formation of C-S-H gel leading to early increase in strength.

Split tensile strength

The split tensile strength gives the idea about the resistance of concrete to indirect tension. The test was performed as per ASTM C 496. The tensile strength of HVFAC with 0.75 percent nano Fe₂O₃ was observed to have the highest tensile strength even higher than NC. The HVFAC with 0.5 and 1 percent nano Fe₂O₃ similar indirect tensile strength as compared to that of NC after 28 days upto 56 days.

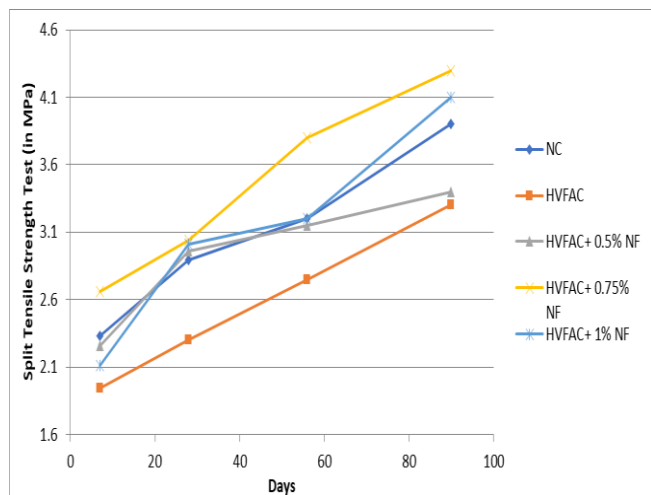


Fig. 6 Variation of indirect tensile strength of NC, HVFAC and modified HVFAC with nano Fe₂O₃ at different ages.

B. Durability Tests

A durable concrete is one that performs satisfactorily on the working environment during its anticipated exposure conditions during service. One of the main characteristics influencing durability of concrete is its permeability to the ingress of water, oxygen, carbon dioxide, chloride, sulphate and other potentially deleterious substances (IS 456-2014). Durability tests were also conducted on the samples made up of NC, HVFAC and modified HVFAC and the results obtained are given below:

Density

It is an indirect measure of durability as it gives the compactness of concrete. Higher is the compactness of concrete, more durable is the concrete.[2]

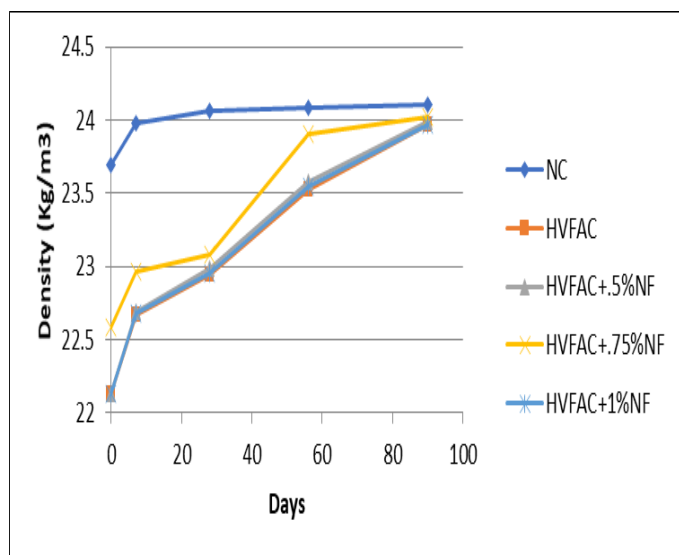


Fig. 7 Variation of density of NC, HVFAC and modified HVFAC with nano Fe₂O₃ at different ages.

Higher density is achieved rapidly for NC. However, with the age, density of modified HVFAC with 0.75 percent nano Fe₂O₃ increases rapidly and becomes similar as that of NC after 56 days. There is an increase in density of HVFAC and modified HVFAC with 0.5, and 1 percent nano Fe₂O₃ and this

trend continues and similar values were observed for all specimens after 90 days.

Water permeability

This test is carried out as per guidelines of ASTM C 577-07 in which the amount of water percolated in 100 hours through concrete cube under 15 kg/cm² pressure was measured to ascertain the permeability of concrete. Water permeability of the concrete were observed to decrease with age. Permeability of HVFAC was found to be more than that of NC. However, the permeability of modified HVFAC with nano Fe₂O₃ is reduced. This shows that durability of concrete increases with the use of fly ash and nano iron oxide.

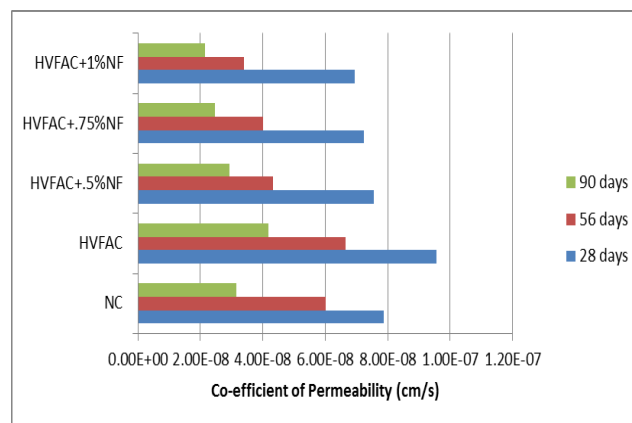


Fig. 9 Variation of coefficient of permeability for NC, HVFAC and modified HVFAC with 0.5%, 0.75% and 1% nano Fe₂O₃ at different ages.

Sorptivity

Sorptivity test is a measure of durability of concrete where the test was carried out as per ASTM C1585-13. In this method the gain in mass of specimen by capillary rise was determined with respect to square root of time when only one surface was exposed to sorption of water. Initial mass of specimen was noted and change in mass due to sorption were measured for first six hours, which determines initial sorptivity. Subsequently, measurement of mass of the specimen were taken at 24 hrs, 2 days, 5 days and 7 days. Higher is the value of sorption, poorer is the concrete.

Absorption of water was highest for HVFAC and it increases with square root of time for 28 days and 90 days ages. The values of initial absorption were least for NC and that of HVFAC had the highest value. The addition of nano Fe₂O₃ reduces the sorptivity. The dosage of 0.75 percent nano Fe₂O₃ with HVFAC gave almost similar results as that of NC.

Effect of Nano Iron Oxide on Strength and Durability Characteristics of High Volume Fly Ash Concrete for Pavement Construction

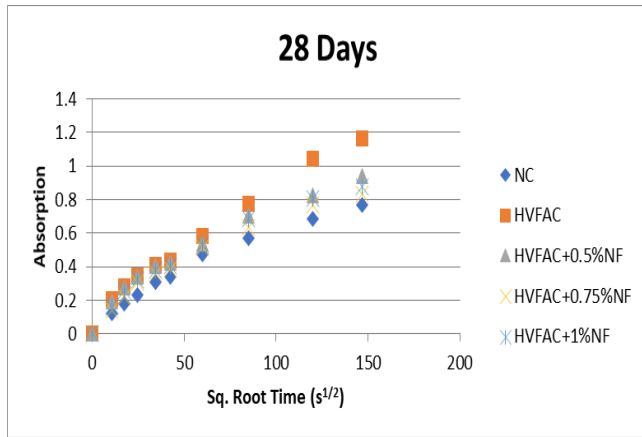


Fig. 10 Variation of sorptivity of NC, HVFAC and modified HVFAC with nano Fe₂O₃ (28 days)

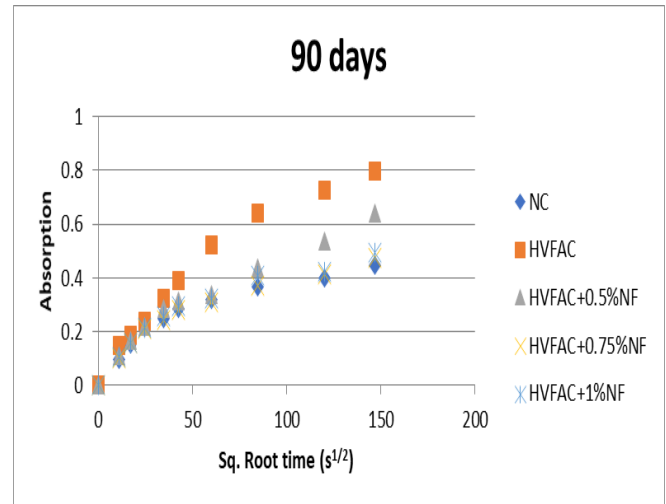


Fig. 11 Variation of sorptivity of NC, HVFAC and modified HVFAC with nano Fe₂O₃ (90 days)

Table 4 Variation of initial sorptivity of NC, HVFAC and modified HVFAC with nano Fe₂O₃

Sample	28 Days		90 Days			
	R ²	Equation (upto 6 hrs)	Initial Sorptivity (*10 ⁻⁴ mm/s ^{1/2})	R ²	Equation (upto 6 hrs)	Initial Sorptivity (*10 ⁻⁴ mm/s ^{1/2})
NC	0.9547	0.005x+0.096	50	0.8393	0.0026x+0.1112	26
HVFAC	0.9817	0.0075x+0.117	75	0.9337	0.0053x+0.1132	53
HVFAC+0.5%NF	0.9477	0.0058x+0.15	58	0.9483	0.0039x+0.945	39
HVFAC+0.75%NF	0.9434	0.0054x+0.124	54	0.8786	0.0028x+0.1051	28
HVFAC+1%NF	0.9432	0.0056x+0.138	56	0.8679	0.0029x+0.1097	29

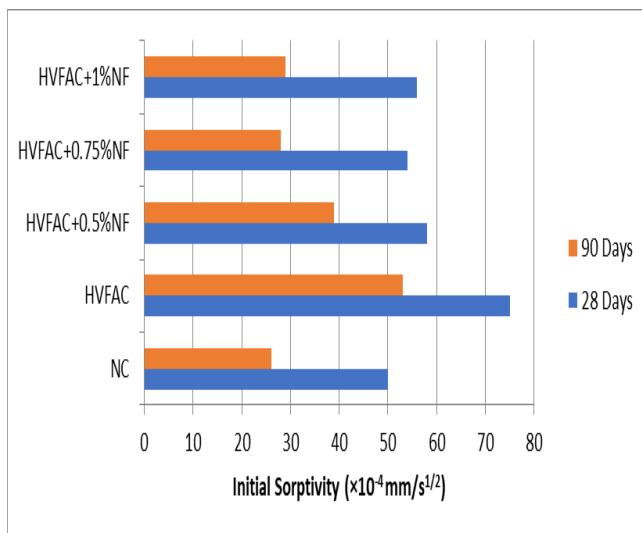


Fig. 12 Initial sorptivity for NC, HVFAC and modified HVFAC with 0.5%, 0.75% and 1% Nano Fe₂O₃ at different ages.

Ultrasonic pulse velocity test (UPVT)

This test was carried out as per ASTM C 597-16. This determines the material homogeneity, density, internal flaws and defects. Pulse velocity is measured through the concrete which gives an assessment of durability of concrete.

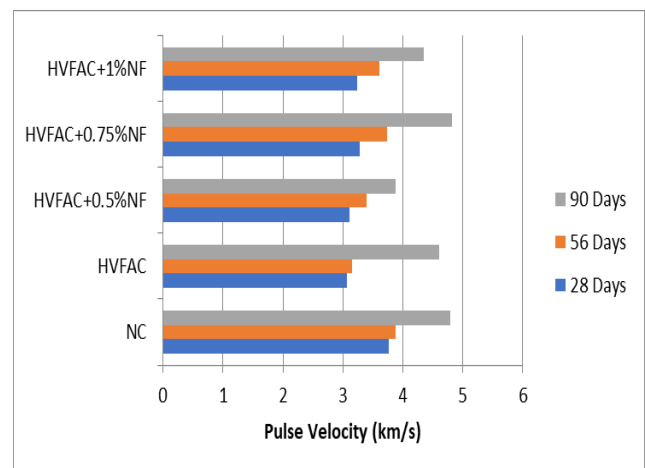


Figure 13 Variation of UPV of NC, HVFAC and modified HVFAC nano Fe₂O₃ at different ages.

At 28 days age of concrete, the UPV value was the highest for NC and thereafter similar values were observed for modified HVFAC with 0.75 percent nano Fe₂O₃. HVFAC, NC and modified HVFAC with 0.75 percent nano Fe₂O₃ showed similar results at 90 days.

Rapid Chloride Penetration Test (RCPT)

It is a durability test where the measurement of charge is done when 60 V DC voltage is applied for 6 hours to determine the chloride ions that have penetrated into the concrete. The test was performed as per ASTM C 1202 in which different samples 50 mm thick and 100 diameter were taken for carrying out the test. A higher value of current measurement indicates larger penetration of chloride ions leading to the decrease in durability.

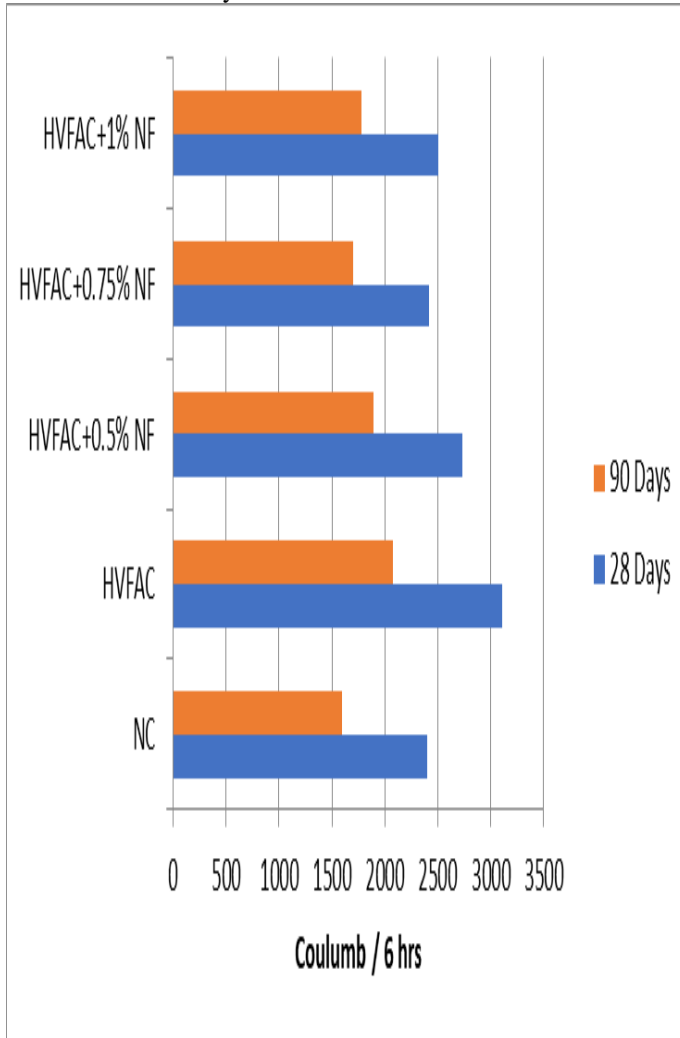


Figure 14 Variation of RCPT of NC, HVFAC and modified HVFAC with nano Fe₂O₃ at different ages.

Table 5 RCPT values for NC, HVFAC and modified HVFAC with 0.5%, 0.75% and 1% Nano Fe₂O₃ at 28 days and 90 days.

RCPT (Coulombs/ 6 hours)

	NC	HVFAC	HVFAC +0.5% NF	HVFAC +0.75% NF	HVFAC +1% NF
28 Days	2396	3108	2733	2416	2506
90 Days	1598	2078	1892	1702	1782

The RCPT test result confirmed the result of sorptivity test and showed that HVFAC had the highest value of chloride ion penetration whereas NC had the least value. The modified HVFAC with 0.75 percent nano Fe₂O₃ gave similar values as that of NC where other modified HVFAC with 0.5 and 1.0

percent nano Fe₂O₃ gave better results than that of HVFAC but gave inferior result as compared to NC.

V. CONCLUSIONS

In this study, it was observed that by the addition of small quantities of nano iron oxide, both the strength and durability characteristics of modified HVFAC get significantly affected. The flexural strength of modified concrete with 0.75 percent nano iron oxide had identical strength as that of normal concrete (NC) after 28 days and had even more strength than NC after 56 days. The rate of gain of flexural strength was highest in modified HVFAC with 0.75 % nano iron oxide after 56 days. It was about 20% higher than that of HVFAC. It might have occurred due to higher quantity of fly ash present in HVFAC, as the pozzolanic reactivity associated with fly ash increases with the time. Increase in flexural strength was also observed with modified HVFAC with 0.5% and 1% nano iron oxide but it was lesser than 0.75% nano iron oxide.

The compressive strength of NC was observed to be highest followed by modified HVFAC with 0.75 % nano iron oxide. The gap between the two was observed to narrow down with age. The rate of gain of compressive strength was highest in HVFAC after 28 days.

The tensile strength of the concrete was assessed by indirect tensile strength test. The result showed that modified HVFAC with 0.75 percent nano iron oxide, was the highest and was even higher than that of NC. The tensile strength of concrete was found to increase with the addition of 0.5 percent and 1 percent nano iron oxide.

The durability of the concrete was assessed through the density, permeability test, sorptivity test, UPVT and RCPT. The results showed that the maximum density was observed for NC. However, at about 56 days, for the modified HVFAC with 0.75 percent nano iron oxide the density increases rapidly to that of NC and similar densities were achieved by HVFAC and other modified HVFAC after 90 days.

The HVFAC was observed to have more permeability than that of NC, whereas the addition of nano iron oxide to the HVFAC causes decrease in permeability. The absorption of water was highest for HVFAC in the sorptivity test whereas that was least for NC. A dosage of 0.75 percent nano iron oxide decreased the absorption up to the level of NC as observed by the sorptivity data. The sorptivity test also gave similar results as obtained from permeability results.

The chloride penetration obtained from RCPT results showed that NC performed the best with least chloride penetration, whereas the HVFAC had highest penetration. Amongst all the modified HVFAC with nano iron oxide, 0.75% addition of nano iron oxide gave the best results. The UPVT showed similar results.

It was observed that the most desirable properties were obtained corresponding to modified HVFAC with 0.75% nano iron oxide. The above figures show that the improvement starts declining with further addition of nano iron oxide. This may be due to agglomeration of nano iron oxide particles in concrete specimens.

Effect of Nano Iron Oxide on Strength and Durability Characteristics of High Volume Fly Ash Concrete for Pavement Construction

Thus, it could be concluded that modified HVFAC with 0.75% addition of nano iron oxide is suitable for pavement quality concrete (PQC) as it fulfils the strength and durability requirements. However, tests with varying percentage of fly ash from different sources and water-cement ratio needs to be conducted for coming up with conclusive evidence and setting up norms for design of PQC for use of fly ash for road construction.

CONFLICT OF INTEREST:

The authors declare that they have no conflict of interest. No financial assistance was provided to carry out this work.

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