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**Technical Paper**

# Fresh, hardened and durability properties of concrete made with flyash and limestone based Portland composite cement

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**Abstract:** Clinker substitution with alternative cementitious materials is one of the way to develop low clinker cement. In this work, three compositions of Portland composite cement (PCC) were prepared by inter-grinding of clinker, gypsum, flyash and limestone where limestone content were kept as 5 %, 7 % and 10 % and flyash content kept as 25% & 35 %. In the present work, study was conducted at 0.60 w/c and in total 4 concrete mixes were designed including one control mix (made with PPC). Fresh properties like air content & fresh density, hardened properties like compressive strength, flexural strength & drying shrinkage and durability behaviour of concrete made with PCC was compared with control concrete. Various durability tests like Rapid chloride permeability test (RCPT), electrical resistivity, accelerated carbonation and sulphate resistance test were carried out to evaluate the performance of concrete made with PCC. The study indicates that with limited addition of limestone i.e., up to 10%, early strength as well as later age strength increases marginally. This may be due to the synergistic effect of flyash and limestone. However, durability aspect was influenced by limestone addition. Test results of RCPT, electrical resistivity as well as sulphate attack indicates the dilution effect of limestone dominates over other physical effects that leads to an overall increase in effective w/c ratio whereas carbonation depth is predominantly affected by the fly as content.

**Keywords:** Portland composite cement (PCC), flyash, limestone stone, RCPT, Carbonation, Sulphate.

## 1. Introduction

Cement is an essential constituent in the concrete and considered to be main problem when it comes to carbon footprint, as production of 1 tonne of Portland cement releases approximately 1 tonne of $CO_2$ [1]. According to UN Environment and International Energy Agency, greenhouse gas intensity of concrete depends upon the manufacturing process of cement which is responsible for 8-9 % of overall $CO_2$ emissions [1,2]. One of the best alternative to reduce $CO_2$ emission coming through cement and concrete industry involves partial replacement of Portland cement with suitable proportions of supplementary cementitious materials (SCMs) [3]. SCMs such as flyash, ground granulated blast furnace slag, silica fume etc. are the most commonly used cementing materials obtained as by product from industrial processes [4,5]. The production as well as availability of SCMs such as flyash, ground granulated blast furnace slag, silica fume etc. is already being used in full swing in different countries around the world [6]. However, still there is need to explore other sustainable cements with new alternative SCMs to have wider option and application. In view of this, use of limestone as an alternative material is advantageous due to its wide availability [7]. Use of limestone as one of the main constituents in the manufacturing of cement has already been permitted in many standards such as European standard EN 197-1[8], Canadian standard CSA A3000 [9], American standards (ASTM C595) [10]. Indian standard IS 269 [11] allows the use of limestone up to 5 % in the manufacturing of ordinary Portland cement as a performance improver. As reported in various literature, interaction mechanism of limestone depends upon physical as well as chemical composition of the constituents in the cementitious system [12–15]. Ojha et al. [15] has broadly categorised the contribution of limestone

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in terms of physical and chemical effects. The physical effects primarily correspond towards its filler action that includes effects like dilution, shearing action and particle packing effect. The chemical effects mainly include suppression of $C_3A$ hydration along with acceleration of $C_3S$ hydration by providing nucleation site [16]. SCMs like flyash is pozzolanic in nature and requires alkaline medium for the reaction. Ability of limestone to serve as a nucleation site for pozzolanic reaction of flyash is one of the beneficial effect of cementitious system comprising of limestone and flyash. Some of the reported studies shows an increased in degree of clinker hydration as well as SCMs reaction in the presence of limestone [17,18].

Yilmaz et al. [19] had studied the effect of ternary blended composite system of OPC, flyash (FA), limestone (LS) and dolomitic limestone (DLS) on mechanical and microstructural behaviour of cement mortar. Volumetric expansion in ternary blended composite was found to be less than OPC and OPC+FA. An increase in compressive strength was observed in ternary composite system in comparison to flyash mortar. Hydration studies indicates that amount of calcium hydroxide formed in OPC + (LS/DLS) + FA composite system was higher in comparison to OPC + FA. Strong peak of $CaCO_3$ was observed in all cement blends containing limestone. Sirisawat et al. [20] had studied the effect of interground flyash mortar and limestone mortar in sulphate solutions. Their study indicates that limestone mortar performs better in $Mg_2SO_4$ environment whereas flyash mortar shows less expansion in $Na_2SO_4$. Similar, study was conducted by Hossack et al. [21] on cement mortars samples made with SCMs like flyash and slag in Portland limestone cement. It was observed that addition of SCMs significantly improves the resistance of Portland limestone mortars samples against external sulphate attack either due to dilution effect that not only reduces the $C_3A$ content but also lowers the $Ca(OH)_2$ content or due to formation of secondary C-S-H that reduces the permeability. Sui et al. [22] works described about durability performance of mortar samples made with different combinations of SCMs like limestone, flyash, slag, calcined clay with respect to chloride ingress. The study indicates that 15 % limestone addition does not adversely impact the performance of ternary systems w.r.t chloride resistance. The apparent chloride diffusion coefficient of mortar made with combination of flyash (35%) and limestone (15 %) has a lower diffusion coefficient corresponding to binary system as well as conventional mortar made with OPC.

Limestone and siliceous fly ash give a synergistic effect only with early strength (two days) and when 10% addition of limestone, which matches the results obtained by De Weerdt et al. [23]. Study had indicated that 5% limestone substitution in OPC cement resulted in a 2% strength increase whereas the use of flyash based cement (35% fly ash versus 30% flyash and 5% limestone) resulted in a strength increase of 13%. This synergetic effect between flyash and limestone powder is mainly because of additional aluminates provided by the fly ash during its pozzolanic reaction, which acts as catalyst for the chemical interaction between limestone powder and aluminate phases [14,18]. Apart from this reaction with calcium aluminate, the substitution of limestone to the cement accelerates the C3S phase reaction which is due the nucleation effect, in which $CaCO_3$ grains act as additional crystallisation germs for cement hydration products. The presence of limestone powder in cementitious system to the tune of 10 percent seems to be more effective for the fly ash cements than for the ordinary Portland cement. When the flyash reacts, aluminates are liberated by dissolution of fly ash; subsequently decreasing the sulphate to aluminate ratio. This causes more ettringite to decompose after sulphate depletion starts and react with the aluminates to form calcium monosulphate hydrate. The presence of limestone has a larger effect as it stabilises the ettringite by reacting with the additional aluminates provided by the fly ash to form calcium carboaluminate hydrates. The net output is more ettringite, more chemically bound water and a larger volume of hydrates leading to lower porosity resulting into higher strength and durability which highlights true synergistic effect of limestone and flyash. P N Ojha et al. [3] had studied the effect of flyash and limestone powder (fineness in the range of 768-785 $kg/m^3$) on the fresh and hardened properties of roller compacted concrete it was observed that due to synergistic effect of flyash and limestone, compressive strength as well as setting behaviour gets affected. The setting time of the concrete made with OPC, flyash and limestone was found to be somewhat in between setting time of concrete made with OPC and flyash as well as concrete made with OPC and limestone. In terms of compressive strength, concrete mixes made with OPC, flyash and limestone had shown high early as well as later age compressive strength. Most of the study related to durability aspect of flyash and limestone combinations are conducted on either mortar sample or cement paste. Limited work has been carried out on the concrete produced through the utilisation of flyash and limestone combination.

In the present work, performance assessment of flyash and limestone-based Portland composite cement (PCC) has been studied. This study may help in the manufacturing of limestone and flyash based composite cements with reduce $CO_2$ impact on the environment and also enables to produce cement with lower clinker factor.

## 2. Experimental Plan

A total of 3 compositions of PCC blends has been prepared in the laboratory by keeping flyash content as 25 % and 35 % whereas limestone content has been kept in the range of 5% to 10 %. Four number of concrete mixes were designed at w/c ratio of 0.60 including control mix made from PPC, flyash content as 35 %. Fresh properties like air content and fresh density of PCC concrete have been compared with control concrete. Mechanical properties of concrete such as compressive strength, flexural strength and drying shrinkage were evaluated at different ages and comparative assessment with respect to control concrete has been carried out. Durability aspect of the concrete made with PCC blends in terms of resistance against chloride ingress has been determined through tests like rapid chloride penetration test (RCPT), resistance against flow of ions has been studied through electrical resistivity test based on four-point wenner probe technique whereas resistance against ingress of $CO_2$ has been evaluated through accelerated carbonation test. The performance of PCC blends against external sulphate attack has been carried out on mortar bar.

### 2.1 Portland composite cement (PCC) based on flyash and limestone was designed for the following three compositions as mentioned in table 1.

The proportions of the gypsum, clinker, flyash and limestone was decided based on the preliminary trials conducted on cement mortar samples such that 28 days compressive strength of prepared blends met the requirement of PPC given in IS: 1489 Part-I. Study by Weerdt et al. [23] also highlighted that to obtain the synergistic effect between flyash and limestone in PCC blends ,limestone replacement with flyash needs to be kept up to 10%.

Table 1 – Portland composite cement (PCC) mix compositions

| Sr. No. | Sample ID | Clinker + Gypsum (%) | Flyash (%) | Limestone (%) |
|---|---|---|---|---|
| 1. | PCC/25/5 | 70 | 25 | 5 |
| 2. | PCC/25/10 | 65 | 25 | 10 |
| 3. | PCC/35/7 | 58 | 35 | 7 |

Table 2 – Physical and chemical characteristics of fly ash

| Sl. No. | Properties | Results |
|---|---|---|
| | Physical | |
| 1 | Specific gravity | 2.14 |
| 2 | Fineness by Blaine ($m^2/kg$) | 336 |
| 3 | Soundness by Auto Clave Exp. (%) | 0.03 |
| 4 | Retention on 45μ IS Sieve by Wet Sieving (%) | 22.3 |
| 5 | Lime Reactivity ($N/mm^2$) | 4.7 |
| 6 | Compressive strength at 28 days as % of the strength of mortar cubes | 86.2% |
| | Chemical | |
| 1 | Loss on Ignition (% by mass) | 0.14 |
| 2 | Magnesium Oxide (% by mass) | 0.89 |
| 3 | Total Sulphur ($SO_3$) (% by mass) | 0.19 |
| 5 | Chloride (% by mass) | 0.002 |
| 6 | Alkali (% by mass)<br>Sodium Oxide<br>Potassium Oxide<br>Eq. as $Na_2O$ | <br>0.03<br>0.74<br>0.52 |
| 7 | Silica (% by mass) | 59.95 |
| 8 | Iron Oxide (% by mass) | 7.69 |
| 9 | Alumina (% by mass) | 27.23 |
| 10 | Calcium Oxide (% by mass) | 1.97 |

These PCC blends were prepared by intergrinding all constituents in laboratory ball mill. The Portland clinker used in the manufacturing of PCC blends conforms to the requirement of IS 16353, gypsum used was mineral gypsum with more than 95 % purity and flyash conforms to the requirement of IS 3812 Part 1 [24] with reactive silica content as 23.40%. The nature of flyash used in PCC blends is Class F (siliceous) and physical & chemical characteristics of flyash are given in Table-2. The physical & chemical characteristics limestone used in the study has been given in Table-3. According to Panda et al. [25], limestone used in the PCC blends falls in marginal grade category whereas Ramaiah et al. [26] classify such type of limestone as high calcium limestone. However, as per end use grade classification given in Indian minerals year book (2020) [27], such type of limestone should be placed under beneficiable/blendable cement. The physical and chemical characteristics of the PCC and PPC with 35 % flyash is given in table 4.

Table 3 – Test results of limestone powder sample

| S. No. | Properties | Results |
|---|---|---|
| | Physical Analysis | |
| 1 | BET fineness, $m^2/kg$ | 785 |
| 2 | Setting time, minutes<br>Initial<br>Final | <br>140<br>150 |
| 3 | Specific Gravity | 2.65 |
| 4 | % Passing on 75 microns | 99.5 |
| 5 | % Passing on 150 microns | 100 |
| 6 | Water absorption, % | 13.29 |
| 7 | Lime reactivity, $N/mm^2$ | 0.4 |
| | Chemical Analysis | |
| 1 | Loss on Ignition (% by mass) | 42.32 |
| 2 | Magnesium Oxide (% by mass) | 1.25 |
| 3 | Sulphuric Anhydride (% by mass) | 0.10 |
| 4 | Free silica | 4.74 |
| 5 | Chloride (% by mass) | 0.007 |
| 6 | Alkali (% by mass)<br>Sodium Oxide<br>Potassium Oxide<br>Eq. as $Na_2O$ | <br>0.10<br>0.19<br>0.23 |
| 7 | Silica (% by mass) | 5.74 |
| 8 | Iron Oxide (% by mass) | 0.73 |
| 9 | Alumina (% by mass) | 1.50 |
| 10 | Calcium Oxide (% by mass) | 43.20 |

Table 4 – Physical and Chemical characteristics of cements

| Sl No. | Properties | PCC/25/5 | PCC/25/10 | PCC/35/7 | PPC |
|---|---|---|---|---|---|
| | Physical characteristics | | | | |
| 1 | Fineness (Blaine), $m^2/kg$ | 357 | 335 | 324 | 416 |
| 2 | Compressive strength, $N/mm^2$ | | | | |
| | 3 days | 30.5 | 25.5 | 17.0 | 26.5 |
| | 7 days | 38.0 | 36.5 | 26.5 | 35.0 |
| | 28 days | 47.5 | 46.0 | 43.0 | 41.5 |
| 3 | Setting time, minutes | | | | |
| | Initial | 185 | 195 | 215 | 155 |
| | Final | 250 | 270 | 275 | 210 |
| 4 | Drying shrinkage, % | 0.05 | 0.04 | 0.02 | 0.01 |
| | Chemical characteristics (% by mass) | | | | |
| 1 | Loss of Ignition (LOI) | 3.66 | 4.60 | 3.98 | 1.55 |
| 2 | Silica ($SiO_2$) | 30.74 | 30.12 | 33.35 | 35.02 |
| 3 | Iron oxide ($Fe_2O_3$) | 4.31 | 3.78 | 3.39 | 3.52 |
| 4 | Aluminium oxide ($Al_2O_3$) | 9.33 | 10.07 | 12.49 | 12.57 |
| 5 | Calcium oxide (CaO) | 43.54 | 45.22 | 39.36 | 41.48 |
| 6 | Total Alkalies as $Na_2O$ Equivalent | 0.69 | 0.78 | 0.99 | 0.96 |
| 7 | Chlorides | 0.008 | 0.011 | 0.006 | 0.016 |
| 8 | Insoluble Residue | 23.30 | 26.26 | 33.27 | 33.50 |

### 2.2 Aggregates

The coarse aggregates (20 mm and 10 mm) and crushed fine aggregate (Zone II), conforming to IS 383-2016 [28] were used. Material finer than 75 micron in fine aggregate was found to be 5.42 % which is within the permissible limit of 15 % and fineness modulus was 2.65. The gradation curve for fine aggregate is given in figure-1 and it meets the requirement of Zone II category fine aggregate as per IS 383-2016. Physical characteristics of coarse aggregates are given in table 5.

Table 5 – Test results of aggregates

| Property | | Coarse aggregate | |
|---|---|---|---|
| | | 20 mm | 10 mm |
| Specific gravity | | 2.81 | 2.77 |
| Water absorption (%) | | 0.32 | 0.28 |
| Sieve Analysis Cumulative Percentage Passing (%) | 40 mm | 100 | 100 |
| | 20mm | 87 | 100 |
| | 10 mm | 1 | 92 |
| | 4.75 mm | - | 11 |
| | 2.36 mm | - | 1 |

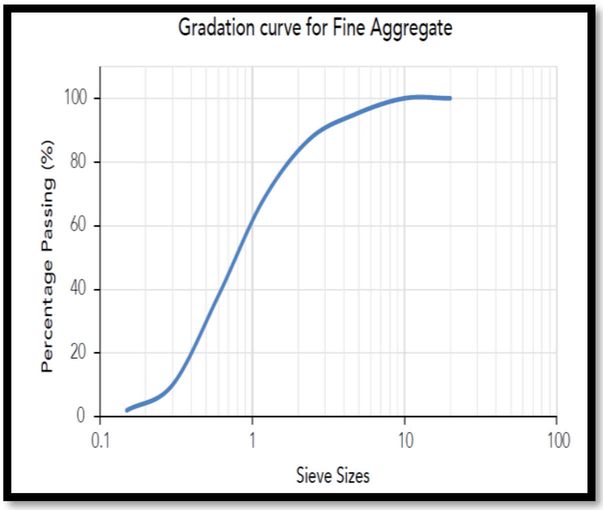


Fig. 1 – Gradation curve for fine aggregate

### 2.3 Admixture

Naphthalene based super plasticizer conforming to IS 9103 [29] was used in concrete mix.

### 2.4 Concrete mix design

The present investigation was carried out at 0.60 w/c, the cement content was kept as 300 kg/m$^3$. The concrete mixes were designed to achieve a slump value of 50-75 mm and accordingly dosage of superplasticiser was adjusted. Fresh properties namely percentage air content and fresh density was measured, refer to table 6. Table 6, also provides details of concrete mix proportions.

Table 6 – Concrete mix proportions and Fresh concrete properties

| Sl. No. | Sample identification | Type of cement (Mix composition) | Mix constituents | | | | | Air content (%) | Slump achieved (mm) | Fresh density kg/m$^3$ |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Cement kg/m$^3$ | Sand kg/m$^3$ | Coarse aggregate kg/m$^3$ | Water kg/m$^3$ | Dose of admixture % by Wt. of Cement | | | |
| 1. | WAUA | PCC/25/5 | 300 | 699 | 1243 | 180 | 0.40 | 1.3 | 65 | 2392 |
| 2. | WAUB | PCC/25/10 | 300 | 698 | 1241 | 180 | 0.40 | 1.3 | 50 | 2389 |
| 3. | WAUC | PCC/35/7 | 300 | 693 | 1231 | 180 | 0.40 | 2.0 | 70 | 2357 |
| 4. | WAUD | PPC | 300 | 697 | 1233 | 180 | 0.30 | 2.8 | 70 | 2375 |

### 2.5 Test conducted

2.5.1 Fresh properties

a) Air content - The test was carried out in accordance to IS 1199 Part 4 [30] by water column method.
b) Fresh density – Fresh density of all the concrete mixes were determined as per IS 1199 Part 3 [30] using cylindrical container with inside diameter of 250 mm and inside height of 280 mm.

2.5.2 Hardened properties

a) Compressive strength -Resistance of concrete against compressive forces was determined as per IS 516 Part1 / Sec.1 [31]. Concrete cube specimens (150x150x150 mm) were subjected to a loading rate of approximately 14 $N/mm^2/min$. The testing was continued until the specimen was not able to withstand any sustained load. At the end of the experiment, load was recorded and is defined as crushing load. The cube compressive strength is calculated as crushing load per unit area. The reported compressive strength is an average of three cube specimens. The compressive strength was conducted at an age of 3, 7, 28, 56 and 90 days.

b) Flexural strength -Flexural strength is an indirect measure of direct tensile strength of concrete. Flexural strength generally indicates ability of unreinforced concrete beam to withstand failure under bending stresses. This test was conducted on concrete beam of size 100mm x 100mm x 500 mm as per IS: 516 Part1 / Sec1 [31] at 7, 28 and 56 days.

c) Drying Shrinkage - This test was conducted on concrete beam of size 75mm x 75mm x 300 mm as per IS 516 Part 6 [32]. After demoulding the specimens were kept under laboratory environment of relative humidity 65±5 % and temperature of 27±2℃ for 7 days and thereafter water cured up to an age of 28 days. The testing was carried out as per the procedure laid in IS 516 Part6. According to IS 516 Part 6, initial length was taken up after removal of specimens from the water tank and subsequently specimens were kept for drying in an oven at 50±1° C and 17±2% humidity for 44 hours followed by 4 hrs for cooling and reduction in length was measured. The cycle of drying, cooling and measurement was repeated until constant length is attained i.e. diff btw two consecutive readings is less than 0.01 mm.

2.5.3 Durability tests

a) Rapid chloride Penetration test (RCPT)- This test is used to evaluate the basic concrete making materials against the chloride ion penetration under external potential and was conducted as per ASTM C 1202 [33]. The test results are influenced by various factors such as w/c, type of mineral admixture/ SCMs, grade of concrete, curing regime, age of testing, concrete mix proportioning etc. In this test method, a steady external electrical potential of 60 volts D.C potential was applied to the concrete specimen of 50 mm thick and 100 mm diameter for period of 6 hours as shown in figure 2. The anode and cathode were filled with 0.30 N sodium hydroxide and 3.0% sodium chloride solutions respectively. The total charge passed during the 6-hour test was recorded and used as a measure to chloride ion penetration in concrete. The test was conducted at 28 days and 56 days' water cured concrete.

b) Electrical Resistivity using four point wenner probe technique -Electrical resistivity is an intrinsic property of the concrete and indicates resistance of concrete against flow of ions [34]. The electrical resistivity test was conducted on unreinforced saturated concrete slabs (300x300x100 mm) at 28 days and 56 days. During the testing, four equally spaced probes were applied to the concrete slabs (300x300x100 mm) in a line as shown in figure 3. The two outer probe induces the current to the specimen and the two inner electrodes measure the resulting potential drop.

c) Accelerated carbonation resistance test - The test was conducted on concrete beam specimen dimension 100mmx100mmx500mm (No's=2) as per ISO 1920 Part 12 [35]. After 28 days of water curing, the concrete specimens were shifted to controlled laboratory environment of temperature = 27+ 2℃ and Relative humidity = 65+5% for 28 days. After 28 days of laboratory conditioning, top and bottom longitudinal faces and two end faces of the beam were sealed using paraffin wax and carbonation was allowed only on the two cast longitudinal faces. This is done to prevent multi-directional carbonation. After the sealing the faces, the concrete beam specimens were shifted to the carbonation chamber, $CO_2$ level was kept as 4±0.5%, temperature as 27± 2 ℃ and relative humidity of 65±5% as shown in figure 4. The carbonation depth was measured by cutting a slice of 50 mm thick from the concrete beam specimen and exposing the cut surface to 1% phenolphthalein solution and measuring the colourless portion. The concrete beam specimens were exposed to $CO_2$ for an exposure periods of 70 days.

d) Sulphate expansion test - This test was conducted on cement mortar samples (25mmx25mmx282mm) made with PCC as mentioned in table1 and compared with cement mortar samples made with PPC as per ASTM C1012 [36]. The cement mortar mix proportioning consists of 1-part cement to 2.75 parts of standard sand by mass. The mortar samples were cast at water -cement ratio of 0.485.A total of 6 mortar bars (25mmx25 mmx280mm) and 12 cubes (50 mmx50mmx50mm) were cast. After 24 hours of warm water curing, moulds from the tank were removed and de-moulded. After de-moulding, all mortar bars and cube specimens, except the two cubes to be broken were stored in curing tank maintained at 27±2°C. The two cube specimens were tested for compressive strength. The mortars bars and cube specimens were kept in curing tank until a minimum cube compressive of 20 MPa was achieved. After achievement of minimum cube compressive strength of 20 MPa, initial length readings of mortar bars were taken and thereafter mortars bars were shifted to container carrying test solution of 5% sodium sulphate concentration as shown in figure 5. The readings were taken up to 6 months.

Fig. 2 – Rapid chloride Penetration test (RCPT)


Fig. 3 – Electrical Resistivity using four point wenner probe technique

Fig. 4 – Accelerated carbonation resistance test

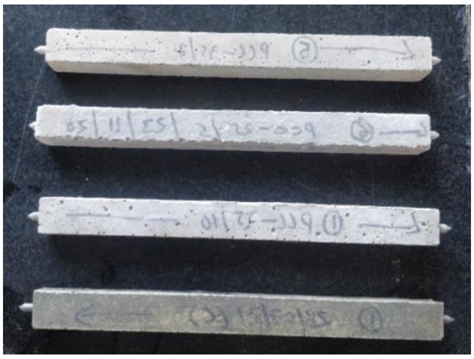

Fig. 5 – Sulphate expansion test

## 3. Results and Discussion

### 3.1 Fresh properties of PCC concrete

From the table 4., it can be observed that for the same slump value, admixture requirement in case of concrete made with PCC i.e. composite blends of Portland cement clinker -flyash- limestone is slightly higher in comparison to that of control mix i.e. concrete made with PPC. The shape as well as particle size distribution of limestone present in Portland composite blends may have affected the workability of concrete [7,12]. Air content of concrete mixes made with Portland composite cement-based on flyash and limestone was found in the range of 1.3 % to 2.0 % which is significantly lower in comparison to air content of control mix i.e. 2.8%. Air content of concrete mixes made with PCC was found to be 30% to 50 % less than that of control mix made with PPC. This may be probably due to filling effect of limestone particles that has reduces the void between the particles of cement and flyash, thus resulting into improved particle packing density [30]. The fresh density of the concrete mixes varies from 2357 kg/m$^3$ to 2392 kg/m$^3$.

### 3.2 Hardened properties of PCC concrete

3.2.1 Compressive strength

It is one of the most important mechanical property of the concrete. Concrete mixes designed with PCC cement has been tested at various ages as described in 2.5.2 a. On analysing the compressive strength results at 3 days from the fig.6 it can be observed that the addition of limestone attributes towards higher early age compressive strength in concrete mixes designed with PCC in comparison to control mix. This is probably due to dilution as well as nucleation effect created by the addition of limestone that may had accelerated the degree of hydration of Portland clinker [14,15,30]. Also, aluminates in the flyash may have chemically interacted with $CaCO_3$ that will leads to the formation of hydration products like hemi and mono-carboaluminates. As reported by various researches development of hemi and mono-carboaluminates results into better strength characteristics [3,14]. Similar trend has been observed at 7 days, 28 days and 56 days where the compressive strength of concrete mixes made with PCC was found to be higher to comparable w.r.t.

control mix. However, at later age i.e. 90 days, the compressive strength of the all concrete mixes made with PCC is higher in comparison to control mix. Due to pozzolanic activity of flyash, formation of additional hydration products like C-S-H and C-A-S-H may have significantly improves the microstructure, thus resulting into better strength. The synergistic effect of flyash and limestone is noticeable at later age [3,37–38].

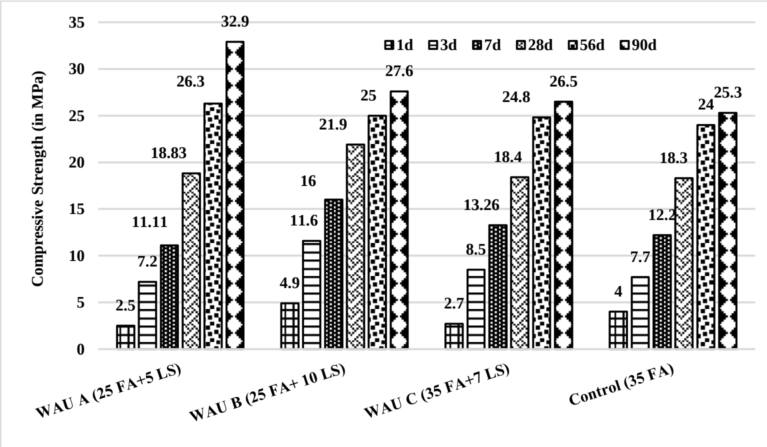

Fig. 6 – Compressive strength at various ages

Results indicate that addition of limestone is beneficial in achieving high early age strength. WAUA concrete made with PCC comprising of 25 % flyash and 5 % limestone has the highest compressive strength when compared to all other concrete mixes including the control mix. Although at 28 days' compressive strength of concrete mix made with PCC blends comprising of 35 % flyash and 7 % limestone was found to be comparable w.r.t control mix. However, while designing concrete mixes with PCC, overall substitution level of Portland cement clinker with flyash and limestone needs attention. When additionof limestone is done beyond 5 percent in the PCC blends, decrease in 28 days strength is noticed both in case of 7 percent and 10 percent limestone substitution. As reported earlier also [23], this trend can be attribute to the reduction in quantity of aluminates provided by the fly ash or Portland clinker during its hydration reaction, which amplify the chemical interaction between the limestone powder and the aluminate phases (AFm and AFt). The chemical interaction between the limestone powder and the aluminates phases are critical in the fly ash-containing cements as fly ash will liberate additional aluminates during its pozzolanic reaction.

3.2.2 Flexural strength

Improvement in flexural strength value with age has been observed for all concrete mixes (figure-7). At 7 days, flexural strength of all concrete mixes made with PCC except WAUC concrete is higher to comparable w.r.t control mix whereas at 28 days and 56 days, concrete made with PCC i.e. WAUA and WAUB has flexural strength higher to comparable w.r.t control mix. The flexural strength results of the concrete mixes made with PCC blends were found in line with compressive strength results. The test results also indicate that concrete made with PCC blend with a composition of 35 % flyash, 7 % limestone and remaining portion as clinker has the lowest flexural value in comparison to all other concrete mixes. This is may be due to lower clinker factor and the reduction in quantity of aluminates provided by the fly ash or Portland clinker during its hydration reaction.

3.2.3 Drying shrinkage

The drying shrinkage values for most of the concrete mixes were found to be comparable irrespective of cement composition with a slight reduction in shrinkage value has been observed in concrete mixes made with PCC blends. However, WAUA concrete (concrete made with PCC with a mix composition of 25 % flyash, 5 % limestone and remaining quantity as Portland cement clinker) has the lowest drying shrinkage value among all other concrete mixes. WAUC concrete has the highest drying shrinkage value in comparison to all other concrete mixes. Various researchers had reported dual behaviour of limestone addition on shrinkage value. When limestone is ground to a fineness more than that of Portland cement, it chemically reacts with the aluminates and from carboaluminates, which leads to an overall increase in the total volume of hydrates products. They will lead to reduced chemical shrinkage. However, at same or lower fineness level than cement grain, addition of addition mainly shows dilution effect i.e. it serves as a filling material which increases the relative water to cement ratio, thereby may results into higher drying shrinkage [12,39–42]. From the test results (figure-8) of WAU A and WAU B concrete, an increase in shrinkage strain was observed as the limestone quantity increases. Similar, trend was noticed in case of WAU C and Control concrete, as limestone is substituted in place of Portland clinker, an increase in drying shrinkage value was observed. The drying shrinkage value of all the concrete mixes are found to be less than limited value of 0.03% as given in clause 6.2.4.1 of IS 456 [43]. The difference in shrinkage behaviour caused by flyash and limestone addition can be attributed to pore structures and fractals which includes the air voids, as well as the morphological intermixing constituents of powders, commonly present extremely complex and irregular features, which are difficult to describe in terms of geometry [18,23].

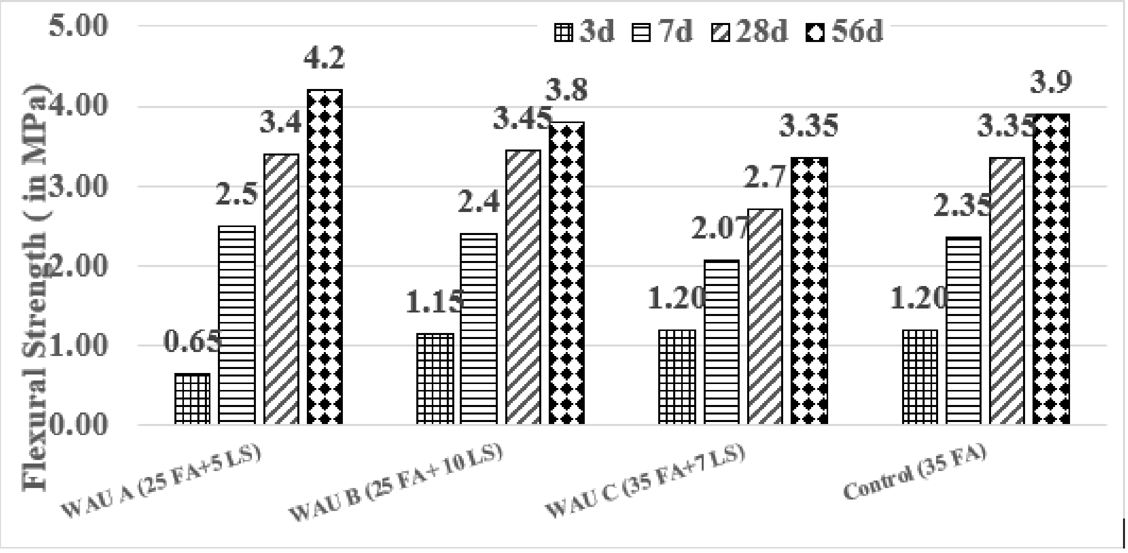


Fig. 7 – Flexural strength at various ages

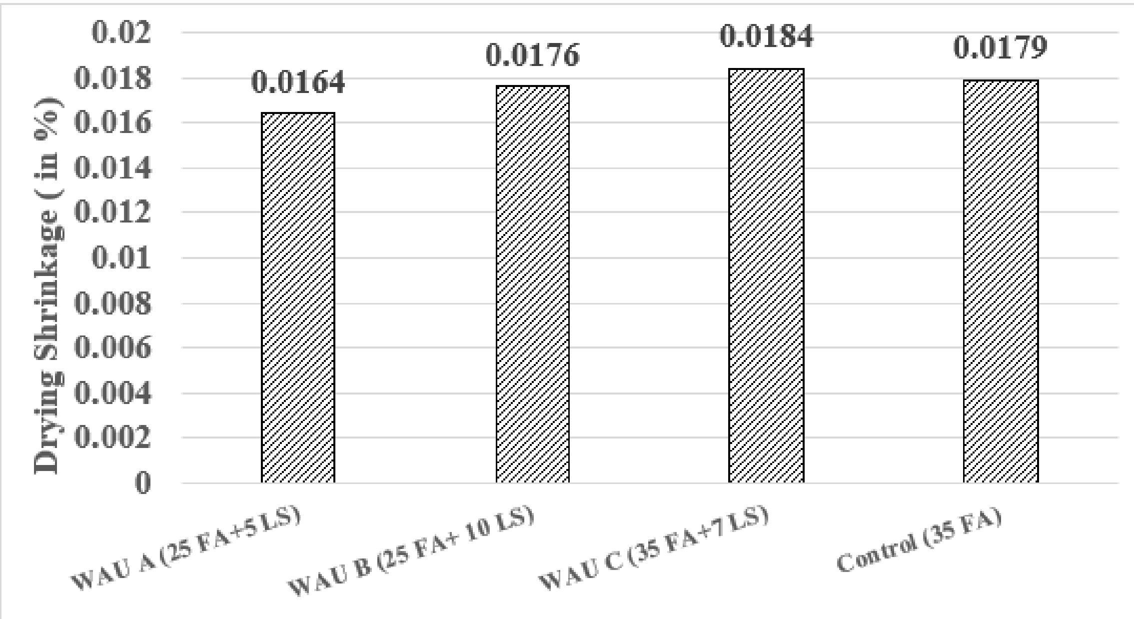


Fig. 8 – Drying shrinkage

### 3.3 Durability aspect of PCC concrete

3.3.1 Rapid chloride penetrability test (RCPT)

Rapid chloride penetrability test as per ASTM C 1202 is one of the most convenient test method used by the researchers to determine the chloride resisting ability of any concrete making constituents. The test is carried at 28 days and 56 days; results are given in table 7.

Table 7 – Rapid chloride penetrability test (RCPT) results

| Age of testing / Specimen ID | 28d | 56d | Change in chloride ion penetrability class with age |
|---|---|---|---|
| WAU A (25 FA+5 LS) | 1304 coulombs | 664 coulombs | Low to very low category |
| WAU B (25 FA+ 10 LS) | 1807 coulombs | 922 coulombs | Low to very low category |
| WAU C (35 FA+7 FS) | 873 coulombs | 477 coulombs | No change i.e. very low category |
| Control (35 FA) | 677 coulombs | 412 coulombs | No change i.e. very low category |

In general, amount of charge passed through the concrete specimens found to be decreasing with the ageing of the concrete i.e. when tested at 56 days' charge passed had been reduced in comparison to 28 days' test results. According to the classification proposed in ASTM C 1202, a drastic shift in the chloride ion penetrability class from moderate to low and low to very low category has also been noticed at 56 days as indicated by the decreased RCPT values. However, concrete mixes made with 35 % flyash i.e. WAUC and control mix, there has been no change in the chloride ion penetrability class although WAUC had slightly higher RCPT value in comparison to control mix. This is may be due to the presence of limestone as a replacement to Portland cement clinker in WAUC concrete. Similar, observation had been noticed in the WAUA concrete (made with PCC/25/5) and WAUB concrete (made with PCC/25/10) where the amount of flyash is same i.e. 25 % but as the limestone content increases from 5 % to 10 %, an increase in the coulombs i.e. charge passed has been significant. On comparing WAUB concrete and control concrete both are designed at same Portland clinker level, a significant difference in the RCPT value between the two concrete mixes was observed that indicates limestone contribution as a replacement to Portland clinker in WAUB concrete is less whereas its effect as a filler is more dominant. As discussed earlier also, when limestone act as a filler, dilution effect dominates and lead to a relative increase in water-cement ratio [12–14]. As reported by P N Ojha et al. [3], limestone in the fineness range of768-785 $kg/m^3$ provides a better packing density and helps in reducing overall permeability whereas at lower fineness, limestone primarily serves as diluting agent. Various literatures show linear dependency of RCPT value on w/c ratio [44–46]. Among all the PCC concrete mixes, WAUB concrete had shown least performance against the chloride ingress. Overall, addition of limestone leads to a slight increase in RCPT value whereas addition of flyash decreases the RCPT value. This can be attributed to the hydration of cement fills the volume initially occupied by water thereby decreasing the overall porosity of systems. The pozzolanic activity of fly ash consumes portlandite and precipitates secondary C-S-H, without altering the porosity, but reducing the interconnectivity of the pore structure.

3.3.2 Electrical resistivity

Electrical resistivity using four point wenner probe technique had been determined in accordance to "Test methods for on-site measurement of resistivity of concrete—a RILEM TC-154 technical recommendation" (35). Generally, electrical conductance of the concrete is influenced by the factors such as water binder ratio, strength, porosity as well as pore size distribution, chemistry of the pore fluid, type and extend of SCMs [46-48]. On the perusal of test results as shown in fig.9, it can be observed that electrical resistivity of the concrete at 28 days found in the range of 13.40 to 22.00 Kohm-cm. At 56 days, electrical resistivity of the concrete was found in the range of 25.83 to 39.00 Kohm-cm that indicates concrete resistance against flow of ions improves with the age of the concrete. This is probably due to pozzolanic reaction taking place at later ages. The trend in electrical resistivity value of concrete mixes at 56 days was same as that at 28 days. Concrete made with 35% flyash i.e. WAUE (control concrete) had shown highest resistivity in comparison to concrete made with combination of flyash and limestone at both 28 days and 56 days. It should be noted that, as the limestone content increases, decrease in electrical resistivity value has been significant. The trend of electrical resistivity results was found in line with the results of RCPT. Performance of WAUB concrete i.e. concrete made PCC comprising of 65 % Portland cement clinker +25 % flyash +10 % flyash against flow of ions is least among all other concrete mixes. From the electrical resistivity test results also, it is quite evident that addition of limestone primarily resulting into filler action. Being a non-reactive ingredient, limestone addition leads to a decrease in reactive components of the cementitious system that affects microstructure behaviour of the concrete.

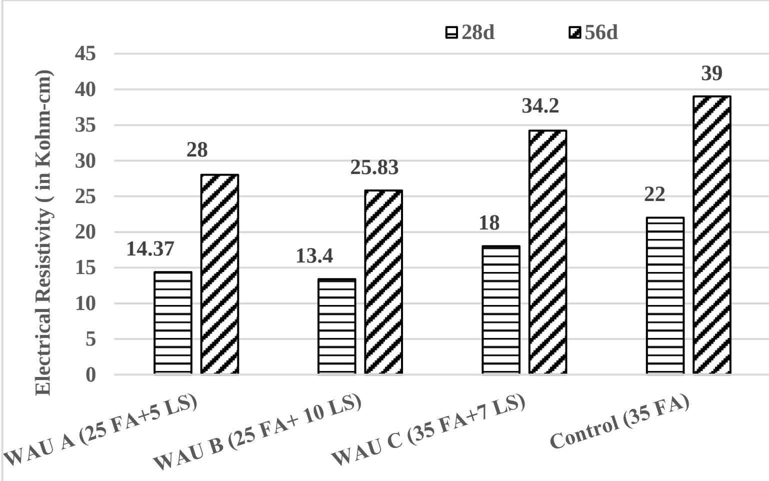


Fig. 9 – Electrical Resistivity with age

3.3.3 Accelerated carbonation test

The term carbonation is a complex physicochemical phenomenon that involves reaction of atmospheric $CO_2$ with calcium bearing phases in the concrete such as portlandite (CH), C-S-H and results in the formation of $CaCO_3$. This process leads to cause reduction in the pH value of the pore solution in the range of 12.5 to 13.5 to a pH less than 9 [48]. When carbonation front reaches the surface of reinforcing bars, the reduced pH destabilizes the passive layer resulting in the loss of passivity. Under such circumstances, if moisture and oxygen are available in sufficient quantity, corrosion initiates and results into loss in load bearing capacity of the structure. However, conversion of CH into calcium carbonate also results an overall increase in the solid volume by 11 percent, thus resulting into decease in porosity [38,42,48–51]. The test results are graphically represented in fig.10. The test results as shown in fig.9 indicates that concrete mix WAUC designed with PCC blend of 58 % Portland cement clinker+35% flyash +7%limestone has lowest resistance against $CO_2$ ingress in comparison to other composite blends and carbonation depth was found to be 40 % higher in comparison to control concrete. The concrete ability to resist carbonation as observed in WAUB concrete made with 65 % Portland cement clinker+ 25% flyash +10 %limestone is 25 % higher than that of control mix that comprises of 65 % Portland cement clinker+ 35% flyash. Even though clinker content in WAUA concrete is slightly higher than WAUB as well as control concrete, still it is more carbonating. It is a matter of research which requires further investigation. However, concrete mix WAUA and WAUB had carbonation depth less than that of control mix

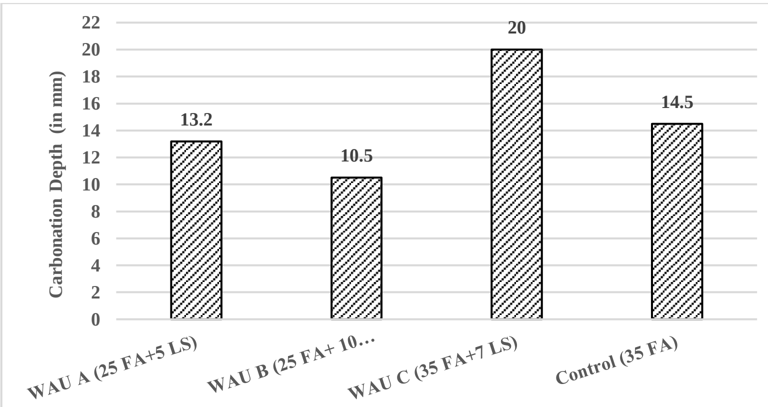


Fig. 10 – Carbonation depth after an exposure period of 70 days

Carbonation resisting ability of any cementitious system depends upon amount of Portland cement clinker, type of SCMs and its substitution rate [51,52]. In the present study, Portland cement clinker has been replaced with flyash and limestone, PCC blends have been prepared and concrete study was carried out at fix water-cement ratio i.e. 0.60. Flyash being pozzolanic in nature have tendency to consume $Ca(OH)_2$ and results into lower alkalinity. Literature also reports that as the flyash content in the cementitious system increases, its ability to resist $CO_2$ ingress reduces [53–57]. The present results also show that with the increase in flyash along with reduction in Portland cement clinker, carbonation depth increases. For example, on comparing WAUB and control concrete at same Portland cement clinker content, WAUB has less carbonation depth than control concrete due to less amount of flyash whereas in case of WAUC and control concrete designed at same flyash content, WAUC concrete has higher depth of carbonation than control due to less amount of Portland cement clinker content. The decrease in the depth of carbonation in WAUB as compared to other PCC blends can be linked with the additional amounts of products formed later (70 days of carbonation period) from the reaction between cement hydration products and active mineral additives which settle in the pores of hardening cement slurry and hinder the permeation and penetration of aggressive ions.

3.3.4 Sulphate resistance

For high sulphate environment, the limit prescribed in ASTM C595 corresponding to 6 months sulphate expansion is 0.05 % [58]. The test results of sulphate expansion as shown in fig. 11 up to an age of 6 months for all the composite blends of Portland cement clinker -flyash –limestone when tested in accordance to ASTM C1012 were found within the limit of ASTM C595. However, PCC blend of 58 % Portland cement clinker+35% flyash +7% limestone i.e. PPC/35/7 had shown highest expansion value in comparison to other composite blends and also found to be more than that of control cement sample i.e. PPC 35.

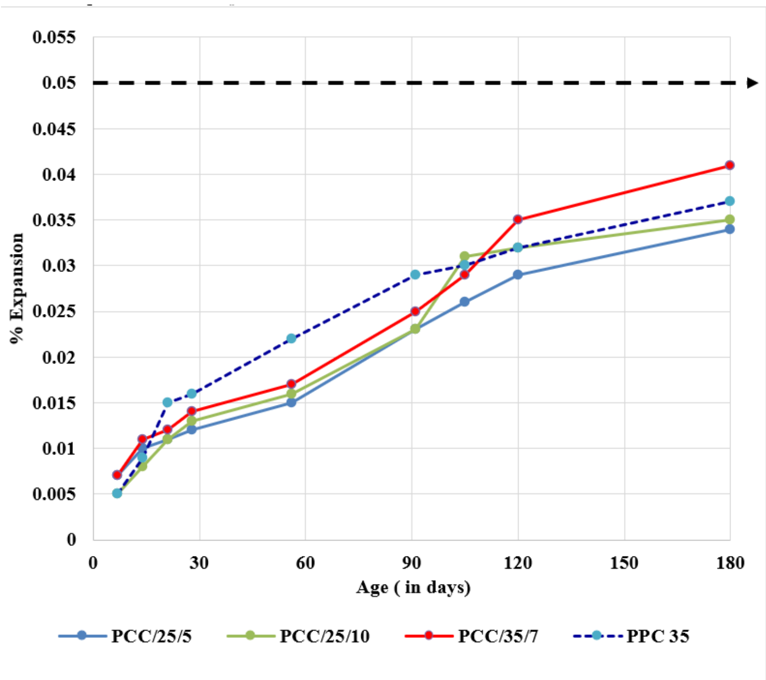


Fig. 11 – Sulphate Expansion up to 180 days

In case of PCC/25/5 and PCC/25/10 mortar samples, as the limestone content increases from 5 % to 10 %, an increase in sulphate expansion values is noticed. Similarly, in case of PCC/35/7 and PPC35 mortar samples, as soon as limestone is added, a surge in the sulphate expansion value had been observed. As reported by other researchers also, limestone addition reduces the ability of cement paste to resist sulphate attack because of increase in effective water-cement ratio due to filler effect [14,58]. Therefore, it can be concluded that limestone addition has a negative impact on sulphate resisting ability of mortar samples. However, sulphate expansion readings of PCC/25/5 and PCC/25/10 was found to be lower in comparison to control mortar i.e. PPC 35. This is mainly due to synergistic effect of limestone and flyash.

## 4. Conclusion

- In the present study, PCC blends made by intergrinding of clinker, gypsum, flyash, limestone had a fineness in the range of 324-357 $m^2$/kg. The study indicates that with limited addition of limestone i.e. up to 10%, early strength as well as later age compressive strength increases marginally. This may be due to the synergistic effect of flyash and limestone. The aluminates present in the flyash may have chemically interacted with $CaCO_3$ that will leads to the formation of hydration products like hemi and mono-carboaluminates that provides better strength characteristics. The test results of flexural strength were also found in line with the compressive strength results. However, while designing concrete mixes with PCC, overall substitution level of Portland cement clinker with flyash and limestone needs attention.

Drying shrinkages of concrete made with PCC blends were found comparable to that of control mix.

- Durability aspect of concrete made with Portland composite cements based on flyash and limestone are greatly influenced by the addition of limestone and overall replacement of Portland cement clinker. At 28 days, RCPT value of concrete made with PCC blends varies in the range 873 to 1807 coulombs whereas RCPT value of control mix was 677 coulombs. At 56 days, a reduction in the RCPT value has been observed in all the concrete mixes. Electrical Resistivity value of concrete made with PCC blends varies in the range of 13.4 to 18.0 Kohm-cm whereas resistivity value of control mix was 22 Kohm-cm. At 56 days, an increased in the electrical resistivity value has been observed in all the concrete mixes. From the test results of RCPT and electrical resistivity, it is quite evident that addition of limestone is primarily resulting into filler action and thereby resulting into an increase in effective water-cement ratio.
- The carbonation depth result shows that with the increase in flyash content along with reduction in Portland cement clinker, carbonation depth increases. The carbonation depth of concrete made with PCC blends was found to be less than control mix other than PCC blend having clinker substitution of 42 percent.
- The sulphate resistance of mortar made with PCC blends was found to be higher than control mix other than PCC blend having clinker substitution of 42 percent.

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