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Influence of activated fly ash on corrosion-resistance and strength of concrete

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Abstract

Various activation techniques, such as physical, thermal and chemical were adopted. By adopting these methods of activation, hydration of fly ash blended cement was accelerated and thereby improved the corrosion-resistance and strength of concrete. Concrete specimens prepared with 10%, 20%, 30% and 40% of activated fly ash replacement levels were evaluated for their compressive strength at 7, 14, 28 and 90 days and the results were compared with ordinary Portland cement concrete (without fly ash). Corrosion-resistance of fly ash cement concrete was studied by using anodic polarization technique. Electrical resistivity and ultrasonic pulse velocity measurements were also carried out to understand the quality of concrete. The final evaluation was done by qualitative and quantitative estimation of corrosion for different systems. All the studies confirmed that upto a critical level of 20–30% replacement; activated fly ash cement improved both the corrosion-resistance and strength of concrete. Chemical activation of fly ash yielded better results than the other methods of activation investigated in this study.

Keywords: Reinforcement corrosion; Fly ash cements; Activated fly ash; Electrical resistivity; Ultrasonic pulse velocity

1. Introduction

The development and use of blended cement is growing rapidly in the construction industry mainly due to considerations of cost saving, energy saving, environmental protection and conservation of resources. Fly ash, a siliceous material obtained from different thermal power stations is now being considered as a cementitious ingredient for concrete. The use of fly ash in mortar and concrete, as a partial replacement of Portland cement, appears to constitute a very satisfactory outlet for this industrial by-product. The use of fly ash to replace a portion of the cement has resulted in significant savings in the cost of production of concrete. It was found that, in order to get resemblance in properties with ordinary Portland cement (OPC), fly ash needs special treatments like mechanical grinding, thermal activation, alkali activation etc. [1,2].

A new method of fly ash activation with addition of $Ca(OH)_2$ and a small quantity of Na_2SiO_3 was reported [1]. Alkali activation showed improved accelerated setting and hardening [3]. Studies conducted on mortars containing 15–60% fly ash as a replacement of Portland cement showed good correlation between strength and particle mean diameters when fly ash substitution was 60% [4–6]. Addition of fly ash caused a decrease in chloride permeability of concrete upto 50% cement replacement [7]. Moreover, the threshold chloride level decreased with increasing fly ash content [8]. Values obtained were 0.70%, 0.65%, 0.50% and 0.20% (acid soluble chloride) by mass of cementitious material for concrete with 0%, 15%, 30% and 50% fly ash, respectively. Despite the lower threshold values, fly ash concrete was found to provide better corrosion protection to the steel due to its increased resistance to chloride ion penetration and increased electrical resistance.

Earlier work reported [9,10] that fly ashes accelerate reinforcement corrosion due to the presence of unburnt carbon and sulphur. In order to produce fly ashes with stable properties and adequate quality, many power

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plants have implemented their own sophisticated quality control measures. The property improvement of fly ash blended cements were extensively studied and reported that, the physical and mechanical properties were matched to that of OPC [11–14].

Although all the studies focused on the improvement of physical and mechanical properties of concrete, systematic and detailed studies on the corrosion performance of activated fly ash blended cement concrete are rare. Therefore, a realistic assessment of the corrosion-resistance of activated fly ash cement concrete is essentially needed in addition to assessing the important physical and mechanical properties of concrete.

The objective of the present investigation is to activate the as-received fly ash (AFA) by physical, chemical and thermal methods and to study the influence of activated fly ash on the corrosion-resistance and strength of concrete.

2. Experimental

2.1. Materials used

OPC	Conforming to IS: 8112-1989 (equivalent to ASTM C150-Type-I)
Fine aggregates	Local clean river sand (fineness modulus of medium sand equal to 2.6) conforming to grading zone III of IS: 383—1970.
Coarse aggregates	Locally available well graded aggregates of normal size greater than 4.75 mm and less than 16 mm having a fineness modulus of 2.72.

The specific gravity of fine and coarse aggregates are 2.41 and 2.78, respectively. Water absorption of fine and coarse aggregates are 0.5% and 0.1%, respectively.

The mix proportion used for casting is as follows: 1:1.71:3.1

OPC	415 kg/m^3
Fine aggregates	710 kg/m^3
Coarse aggregates	1287 kg/m^3
Water–cement ratio	0.50
Fly ash	10%, 20%, 30%, 40% and 50% replacement level of OPC by weight of cement

2.2. Fly ash used

The fly ash collected from Neyveli Lignite Corporation, Neyveli, Tamilnadu, India was utilized for analysis. The physical properties, such as specific gravity and specific surface area, of fly ash used were 1.9 and 127 m^2/g, respectively. The chemical composition of cement and fly ash used are reported in Table 1. Fly ash is used in the unprocessed form and as well as in the processed form produced by physical, chemical and thermal methods.

Table 1
Composition of OPC and fly ash used in this study

Constituents	OPC (%)	FA (%)
SiO_2	22.14	64.03
Fe_2O_3	3.35	6.50
Al_2O_3	9.93	15.50
CaO	60.68	4.62
MgO	1.30	3.00
Loss on ignition	2.6	4.35
Insoluble residue	–	2.00

2.3. Physical activation

The fly ash as-received was sieved to remove any coarser and foreign particles and then mechanically ground in a ball mill to a fine powder. The particle size distribution measured was found to be between 40 and 90 μm.

2.4. Thermal activation

Finely ground fly ash was kept at a temperature of about 900–1000 °C in a graphite pot for 1 h. After cooling at room temperature, the finely ground fly ash was used for investigation. The carbon, sulphur and other impurities are removed by thermal activation.

2.5. Chemical activation

Finely ground fly ash was subjected to chemical activation by treatment with sodium hydroxide solution, filtered and dried. Further, calcium oxide (5% by weight of cement) was added to the concrete during mixing.

The various systems used and the abbreviations are as follows:

As-received fly ash	AFA
Physically activated fly ash	PFA
Thermally activated fly ash	TFA
Chemically activated fly ash	CFA

2.6. Techniques used

2.6.1. Weight loss measurements

Cylindrical mortar specimen of size 55 mm diameter and 60 mm height were cast using OPC and OPC containing various activated fly ashes at 10%, 20%, 30% and

40% replacement levels. Mild steel rod of 6 mm diameter and 45 mm long was embedded centrally. Initially the mild steel rebar samples were cleaned in hydrochloric acid, degreased with acetone and washed with double distilled water and dried. The initial weight of the rebar sample was taken before casting for gravimetric weight loss measurements.

Mortar specimens were prepared using 1:3 mix with a w/c ratio of 0.45. The specimens were mechanically vibrated. After 24 h, the specimens were demoulded and cured for 28 days in distilled water in order to avoid any contamination. After the curing period was over, all the specimens were completely immersed in 3% NaCl solution. The specimens were maintained in the same condition for 15 days and then subjected to drying for another 15 days. One alternate wetting and drying cycle consists of 15 days immersion in 3% NaCl solution and 15 days drying in open air at room temperature. In order to induce the accelerated corrosion 3% NaCl solution was used.

All the mortar specimens were subjected to 18 complete cycles of test period. Tests were conducted on a minimum of six replicate specimens and the average values are reported.

2.6.2. Anodic polarization technique (potentiostatic method)

Mild steel rods were embedded in cylindrical mortar (1:3) specimens of size 58 mm diameter and 60 mm height using w/c ratio of 0.45. The mortar specimens only with OPC (control) and OPC replaced by fly ash at 10%, 20%, 30%, 40% and 50% replacement levels were subjected to anodic polarization studies using a three electrode system which consists of embedded steel in mortar as anode, stainless steel ring electrode as cathode and saturated calomel electrode as reference electrode [9]. Anodic polarization studies have been carried out in 3% NaCl solution. The current flowing at +300 and +600 mV were recorded for all the specimens.

2.7. Compressive strength test

100 mm $\times$ 100 mm $\times$ 100 mm concrete cubes were cast using 1:1.71:3.10 mix proportion with w/c ratio of 0.50. Specimens with OPC (control) and OPC replaced by fly ash (as-received as well as activated) at 10%, 20%, 30% and 40% replacement levels were cast. During moulding, the cubes were mechanically vibrated. After 24 h, the specimens were removed from the mould and subjected to water curing for 7, 14, 28 and 90 days. After curing, the specimens were tested for compressive strength using a compression testing machine of 2000 kN capacity. The tests were carried out on six specimens and average compressive strength values were obtained.

2.8. Electrical resistivity and ultrasonic pulse velocity measurements

Electrical resistivity of concrete was carried out on 100 mm $\times$ 100 mm $\times$ 100 mm concrete cubes using 1:1.71:3.10 mix with w/c ratio of 0.5 for the curing period of 90 days. After the curing period was over, the specimens were kept in a hot air oven at a temperature of 100 °C for an hour for the removal of moisture content. Then the specimens were cooled at room temperature. Resistivity measurements were made on triplicate specimens using four-probe resistivity instrument.

The same specimens were subjected to another quality control test using ultrasonic pulse velocity (UPV) tester.

2.9. Qualitative and quantitative estimation of corrosion

At the end of the exposure period, the specimens were split open and visual observation data on corrosion were made and expressed in terms of percentage of area rusted. The corrosion rate of mild steel anodes embedded in control (OPC) and various systems studied were determined by gravimetric weight loss method and expressed in mmpy as reported earlier [15,16].

2.10. Chemical analysis for alkalinity and free chloride contents

The concrete samples collected near the anode were crushed mechanically and powdered. Then 100 gm of powdered sample was shaken with 100 ml of double distilled water in a conical flask using Microid flask shaker for 1 h. The extract was then filtered through a Whatmann filter paper no. 42. The extract prepared from the powdered sample was then analyzed for alkalinity and the free chloride contents as per the procedures outlined in Ref. [17].

Fifty cc of filtered solution was taken in 100 ml beaker and the alkalinity of the sample was measured in terms of pH using a standard calibrated pH meter. Before taking measurements, pH meter was standardized using a buffer solution of 7 and 9.5.

Twenty cc of filtered solution was taken and the free chloride content was estimated by standard silver nitrate solution using potassium chromate as an indicator. The amount of free chloride content present was expressed in terms of parts per million (ppm) on the basis of weight of sample taken for analysis.

3. Results and discussion

3.1. Weight loss measurements

The corrosion rates calculated in mmpy for mild steel rebars embedded in various systems studied are reported in Table 2. From the Table 2 it was found that, the corrosion rate for OPC system was found to be 0.0739 mmpy at the end of 18 alternate wetting and drying cycles. In the case of AFA system except at 10% replacement level, all other replacement levels namely 20%, 30% and 40% were showing higher corrosion rates when compared to OPC, which necessitates the requirement of activation. All the activated systems indicated corrosion rate of less than 0.0739 mmpy upto 30% replacement levels at the end of exposure period of 18 complete cycles, which represent the tolerable limit of replacement with better corrosion-resistance properties. At 40% replacement level, all the activated systems showed a corrosion rate of greater than 0.0739 mmpy. Among the systems studied, CFA showed lowest corrosion rate for all replacement levels at the end of the test period. The better performance of CFA system is as follows. In the case of CFA system, the synergistic effects of more CSH gel formation due to presence of CaO and the filler effect reduced the corrosion rate. But in PFA and TFA systems only filler effect reduced the corrosion rate.

On the basis of gravimetric weight loss measurements, the reduction in corrosion rate for various systems follows the order: CFA > TFA > PFA > OPC > AFA.

These data illustrated the necessity of activation of fly ash to enhance the corrosion-resistance of concrete. PFA and TFA improved the corrosion-resistance of concrete by pore filling effect whereas CFA improved the corrosion-resistance both by filler and buffer effects and thereby maintaining the perfect alkalinity near the steel anode.

3.2. Anodic polarization technique

Anodic polarization studies has been carried out in 3% NaCl solution. The current flowing at +300 and +600 mV were recorded for mild steel embedded in OPC and OPC replaced by various fly ash systems at 10%, 20%, 30%, 40% and 50% replacement levels and the corresponding magnitude of current for a fixed duration of 12 h are reported in Table 3. From the table it was found that for OPC, the current measured was found to be 0.43 and 1.04 mA. For AFA the current measured at 30% replacement level was found to be 0.34 and 0.99 mA. On the other hand TFA and CFA systems showed superior properties even upto 50% replacement level. For example in the case of CFA system, the current measured was found to be 0.40 and 0.56 mA at 50% replacement level. These data clearly illustrated that activated fly ashes improved the corrosion-resistance properties even upto 50% replacement level.

3.3. Compressive strength

The average compressive strength vs. curing time for OPC and OPC replaced by various fly ash systems at 10%, 20%, 30% and 40% replacement levels were shown in Figs. 1–4. From these figures it was noted that, curing time increased the compressive strength of concrete. The same trend was observed in these studies also irrespective of amount of fly ash replaced. In the case of OPC, a twofold increase in compressive strength was observed at the end of 28 days. At 7 and 28 days the compressive strength were 20.2 and 43.8 MPa, respectively. On the other hand, in fly ash (as-received as well as activated)

Table 2
Qualitative and quantitative estimation of corrosion for OPC and various activated fly ash blended reinforced mortar

System	Replacement level (%)	Area rusted (%)	Corrosion rate (mmpy)	pH	Free chloride (ppm)
OPC	Control	50	0.0739	12.1	3240
AFA	10	75	0.0788	11.9	4000
	20	80	0.1267	11.9	4720
	30	85	0.1955	11.7	4800
	40	95	0.2102	11.7	5000
PFA	10	25	0.0362	12.2	1320
	20	30	0.0534	12.2	1920
	30	35	0.0681	12.2	1960
	40	40	0.1567	12.2	2360
TFA	10	15	0.0245	12.2	920
	20	20	0.0431	12.2	1200
	30	30	0.0543	12.2	1625
	40	35	0.1235	12.2	1850
CFA	10	10	0.0152	12.5	700
	20	10	0.0362	12.5	760
	30	10	0.0494	12.5	780
	40	20	0.0852	12.5	920

Table 3
Anodic polarization test parameters for OPC and various activated fly ash blended cement concrete cylinders in 3% NaCl solution

System	AFA		PFA		TFA		CFA	
	+300 mV shift current (mA)	+600 mV shift current (mA)	+300 mV shift current (mA)	+600 mV shift current (mA)	+300 mV shift current (mA)	+600 mV shift current (mA)	+300 mV shift current (mA)	+600 mV shift current (mA)
OPC (100%)	0.43	1.04	0.43	1.04	0.43	1.04	0.43	1.04
OPC + 10% FA	0.24	0.55	0.16	0.40	0.06	0.08	0.05	0.07
OPC + 20% FA	0.27	0.75	0.21	0.62	0.09	0.14	0.07	0.10
OPC + 30% FA	0.34	0.99	0.30	0.83	0.18	0.26	0.11	0.15
OPC + 40% FA	0.46	1.08	0.38	0.99	0.24	0.40	0.19	0.20
OPC + 50% FA	1.26	2.50	1.00	1.50	0.40	0.66	0.40	0.56

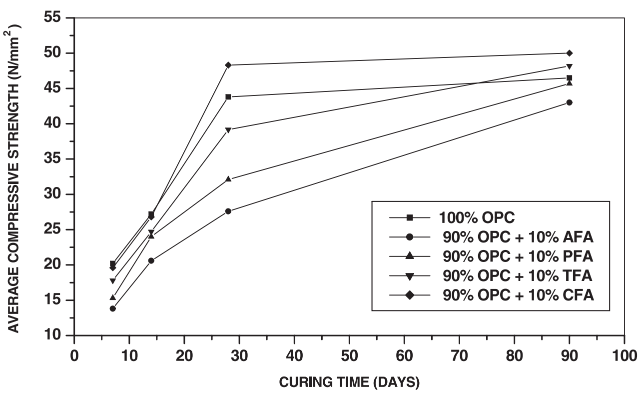

Fig. 1. Average compressive strength vs. curing time for OPC and 10% fly ash cement concretes.

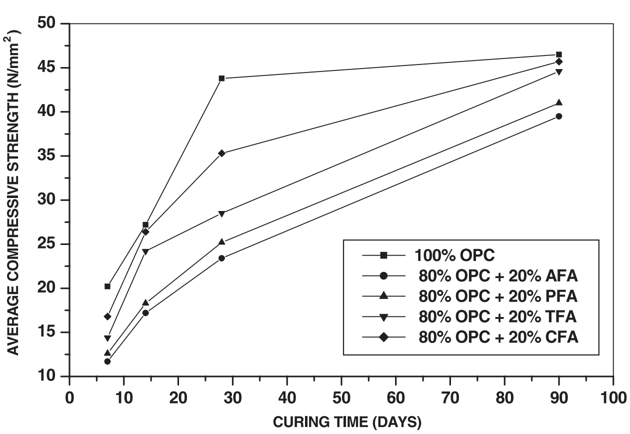

Fig. 2. Average compressive strength vs. curing time for OPC and 20% fly ash cement concretes.

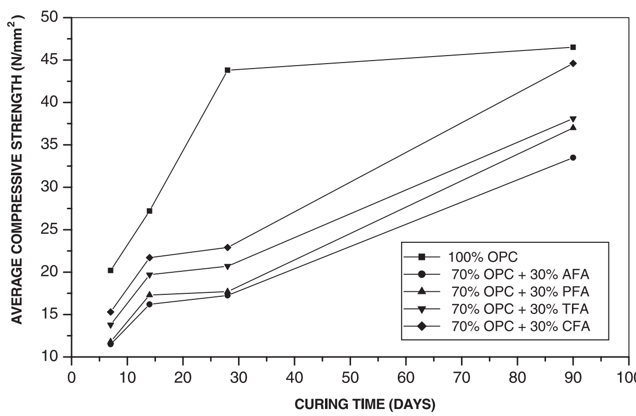

Fig. 3. Average compressive strength vs. curing time for OPC and 30% fly ash cement concretes.

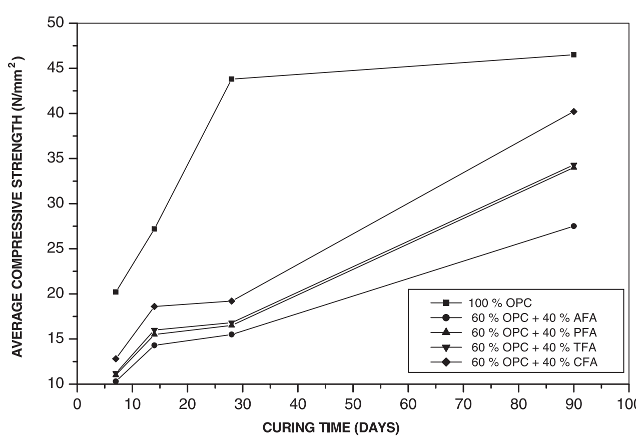

Fig. 4. Average compressive strength vs. curing time for OPC and 40% fly ash cement concretes.

systems, 1.5 times increase in the compressive strength was observed. At 40% replacement level for AFA system, the compressive strength was 10.3 MPa and 15.5 MPa, respectively, at 7 and 28 days. A decrease in compressive strength was observed as the percentage of fly ash replacement increased irrespective of type of activated fly ash used. For example, in the case of TFA, at 7 days of curing the compressive strength at 10% and 40% replacement levels were 17.8 and 11.2 MPa, respectively. A decrease in compressive strength i.e. 1.5 times was observed as the percentage of fly ash replacement increased from 10% to 40% replacement levels irrespective of the method of activation. In the case of AFA system, compressive strengths comparable to OPC were observed only at 10% replacement level. At 7 and 14 days the compressive strengths were not affected at 20% and 30% replacement levels. But at 40% replacement level inferior properties were observed. On the other hand, in the case of CFA system comparable results were obtained for 10% and 20% only at 90 days of curing. In the case of TFA and PFA systems, same results were obtained upto 28 days of curing even at 40% replacement level. These data clearly illustrated that activation of fly ash improved the strength of concrete. However, the compressive strength of fly ash concrete was less than that of OPC even after 90 days of curing.

Among the activation systems studied, CFA system improved the compressive strength to a certain extent, only 10% and 20% replacements. Since the CFA surface layer is etched by a strong alkali to facilitate more cement particles to join together and also the addition of CaO which is further promoting the growth of CSH gel and $Ca(OH)_2$ which is more advantageous to enhance the strength development.

The important pre-requisite for good activated system are: (i) to increase the corrosion-resistance of steel embedded in concrete (ii) should not significantly affect the strength properties of the concrete. In this aspect, CFA system was found to be more effective in controlling the corrosion of steel in concrete and did not reduce the compressive strength, particularly when used upto 20% replacement level.

3.4. Electrical resistivity and ultrasonic pulse velocity

The average electrical resistivity and UPV values measured for various replacement levels for the different systems are shown in Table 4. From this table it was found that the electrical resistance of AFA system showed less resistivity values than OPC at replacement levels of 20% and above. This behaviour was observed due to the fact that, in AFA the compatibility with hydrated cement phases and the remaining mineral phases act as more porous in the concrete formulation. With the result, the decrease in resistivity was observed. In addition, AFA contains impurities like unburnt carbon, sulphur etc., increases the electrical conductivity and consequently decreases the resistivity values. On the other hand, all the activated fly ash blended cement concrete showed higher resistivity values than OPC which indicates the activated fly ash blended cement concretes are more closely packed and perhaps the connected pores are fewer and smaller. This is possible because the pozzolona reacts with the free lime liberated during setting to form CSH gel. Therefore, the quality of the concrete was not much affected for all the activated fly ash blended cement concrete.

UPV values observed for OPC is found to be 4.35 km/s, whereas that for AFA system ranged between 4.36 and 4.00 km/s, for PFA system the values ranged between 4.50 and 4.28 km/s, for TFA system the values ranged between 4.34 and 4.15 km/s and for CFA system the values lies between 4.46 and 4.00 km/s. All these values indicates the better performance of activated systems.

Table 4
Average resistivity and UPV values for OPC and various activated fly ash cement concrete

System	Replacement level (%)	Average resistivity (kΩ cm)	Average UPV (km/s)
OPC	Control	79	4.35
AFA	10	79	4.36
	20	77	4.35
	30	72	4.10
	40	69	4.00
PFA	10	90	4.50
	20	88	4.34
	30	85	4.34
	40	84	4.28
TFA	10	131	4.34
	20	110	4.16
	30	86	4.16
	40	86	4.15
CFA	10	139	4.46
	20	118	4.26
	30	101	4.16
	40	86	4.00

3.5. Alkalinity and free chloride contents

The results of concrete sample analysis are reported in Table 2. From this table it can be observed that both OPC and OPC containing AFA showed a slight decrease in alkalinity (pH = 11.9) whereas all the activated systems showed preservation of concrete alkalinity (pH = 12.2–12.5) even after 18 cycles of alternative wetting and drying. This observation clearly proved that the alkalinity of concrete was not at all affected by different percentages of activated fly ash replacement levels. The preservation of alkalinity indicated that the activated systems maintained the perfect passive condition of steel throughout the test period.

The free chloride contents estimated are also reported in Table 2. Among all, CFA system was found to have very low penetration of chloride in the ranges from 700 to 900 ppm. But TFA and PFA systems showed in the ranges from 900 to 2300 ppm of chloride as compared to 3240 ppm in OPC. The chloride concentration in AFA was in the range of 4000–5000 ppm.

The visual observation data for different systems studied are reported in Table 2. From the table it was shown that in the case of OPC the rusted area was 50%. But in the case of activated fly ash systems as the percentage of replacement level increases the percentage of area rusted also increased. AFA showed 75–95% area rusted, when the replacement level increased from 10% to 40%. But activated fly ash systems showed the least percentage of area rusted. Among the activated systems CFA showed only 10–20% of rusted area even upto 40% replacement level.

The reason for better performance of various activated fly ashes are as follows. It is a fact that the presence of unburnt carbon and sulphur in the unprocessed fly ash may enhance the corrosion of reinforcement [18]. Unburnt carbon content is an undesirable constituent of fly ash for use in reinforced concrete constructions. Besides its various harmful effects, it increases the electrical conductivity. Because of the oxidizing atmosphere at power stations, the sulphur present in the fly ash is usually in the form of sulphates and has an effect (expansion and disruption of concrete) similar to that of sulphates present in the normal Portland cement. The need of chemical activation of fly ash mainly involves the breaking of bonds and dissolution of three-dimensional network structure of glass. It has also been reported that, when $Ca(OH)_2$ is present, the solubility of SiO_2 in fly ash markedly increases [19]. Thermal activation affects both fly ash reactivity and the kinetics of dissolution. Pietersen et al. [20] and Ma et al. [21] also reported that a significantly faster glass breakdown occurs at elevated temperatures by thermal activation.

The observed corrosion-resistance performance of activated fly ash cement concrete may be due to the combined interactive effect of the chemical and physical characteristics of fly ash blended cement. The better corrosion-resistance performance of fly ash blended cement concrete compared to plain cement concrete due to the longer corrosion initiation time and lower corrosion rate is attributable to the improved physical structure of the cement matrix due to the fly ash blending.

4. Conclusions

The following broad conclusions can be drawn from the above results:

1. Weight loss measurements, visual observations and anodic polarization tests confirmed that upto a critical level of 20–30% replacement, activated fly ash improved the corrosion-resistance of concrete.
2. Compressive strength data showed that, upto 30% replacement level, the activated fly ash systems improved the strength of concrete.
3. Among the activated systems, CFA improved both the corrosion-resistance and strength of concrete to a greater extent.
4. The chemical and thermal activated fly ash concretes performed well when compared to OPC.
5. Chemical activation of fly ash yielded better results than the other methods of activation investigated in this study.

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