

Enhancing environmental sustainability through utilization of CFBC ash

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Circulating fluidized bed combustion (CFBC) technology is an efficient combustion technique for combustion of high sulfur containing fuels like petcoke which results in generation of CFBC ash as by-product. This poses environmental risks and presents challenges in utilization due to significantly higher levels of CaO, SO₃ etc. CFBC ash has different chemical composition, mineralogy and morphology in comparison to conventional coal based fly ash due to different combustion process with combustion temperature of about 900°C. Characterization of CFBC ash is done using XRF, XRD, SEM, Particle Size Distribution etc. In this study, CFBC ash based mortar cube, concrete cube and pavers block have been made. It is seen that the strength of CFBC ash based concrete gets enhanced due to interfacial bonding of CFBC ash and other raw materials. The mineralogical and morphological studies of concrete confirm the formation of hydration products like Calcium Silicate Hydrate, Tricalcium Silicate, Gypsum, Portlandite and Ettringite which are extremely important for strength development and durability. The leachability results have shown that developed product is non-toxic and safe from environmental and leaching aspects. The study recommends use of CFBC ash as a sustainable building material for broad application spectrum.

Keywords: Engineering properties, Leachability, Mineralogy, Morphology, Pavers block

1 Introduction

Circulating fluidized bed combustion technology is efficient for burning fuel consisting of sulfur in higher concentration, with the potential to capture emitted SO₂ during combustion by addition of limestone to minimize the environmental impact¹⁻². Presently, petcoke having higher sulfur content is used as fuel along with coal in CFBC boiler due to its high calorific value. To enhance the efficiency of SO₂ removal during combustion, the ratio of CaO and SiO₂ is raised to the range of 2.0- 2.5⁽³⁾. The combustion temperature of CFBC boiler lies between 800-900°C which is relatively lower as compared to conventional coal fired technology due to which there is unreacted lime and desulfurized products and traces of CaCO₃ in CFBC ash. Hence, it may have lesser pozzolanic property than the conventional coal fly ash generated at higher temperatures^{4,5}. Many researchers have reported utilization of CFBC ash in Portland Cement or as mineral admixture. According to the literature, addition of Pulverised Coal Ash (PCA) along with CFBC ash leads to prolonged initial setting time while showing slight decrease in compressive strength when compared to OPC⁶. It is reported that

OPC, ground blast furnace slag and CFBC fly ash when mixed together, results in reduced shrinkage, delayed cracking time and increase in compressive strength⁷. The literature also indicates that the combination of CFBC ash and OPC encourages cement hydration, leading to the production of Ca(OH)₂ thereby influencing cement hydration and enhancing early strength development⁸. It is also reported that CFBC ashes that adhere to physical and chemical criteria of ASTM C618 can be utilized on their own or mixed with other pozzolans for concrete applications⁴. However, after extensive literature review, limited work has been reported on utilization of CFBC ash as cement substitute in making building materials like pavers block.

This research article is an effort to evaluate effect of CFBC ash as partial substitute for Portland Pozzolana Cement (PPC). The study aims to investigate potential of utilizing pozzolanic materials derived from industrial by-products and waste utilization as means of addressing global concern. The research focuses on utilizing CFBC ash for production of concrete materials. The most recent environmental approach aims to attain sustainable development by conserving natural resources, reducing the dumping of CFBC ash and reduction of carbon footprint for

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sustainable application. Efforts have been made to effectively utilize CFBC ash in developing environment friendly and high strength pavers block for commercial application. Physico-chemical characterization of CFBC ash was carried out by determining pH, bulk density, particle density, porosity, conductivity, Blaine fineness, lime reactivity, mineralogical and morphological analysis was carried out using XRD and SEM techniques available at the Institute.

2 Materials and Methods

2.1 Raw materials

2.1.1 CFBC Ash

The sample of CFBC ash generated in CFBC boiler from combustion of petroleum coke and Indian coal with a blending ratio of 80:20 (wt%) respectively were collected from silos of Oil Refinery located in Madhya Pradesh, India. The collected sample was oven dried at 105°C for 24 hours followed by detailed characterization including metal oxides which were determined through X- Ray Fluorescence (XRF) and given in Table 1.

2.1.2 Portland pozzolana cement

In this study, Type-I Portland Pozzolana Cement (PPC) conforming to IS1489 (Part 1&2)1991⁹, which has Blaine fineness of 3220 cm²/kg and specific gravity of 3.15 was used.

2.1.3 Aggregates

The coarse aggregate used in the study is crushed stone aggregates of size 20 mm and 10 mm. The aggregates are retained on suitable sieve conforming to IS383¹⁰. River sand with specific gravity of 2.60 was used in this study.

2.1.4 Water

The water used in the study for preparing and curing the concrete sample is normal tap water having pH value of 7.5. The water is free from oil, acid, alkali, clay and organic impurities. Other experimental work and

preparation of chemicals were done in demineralized double distilled water using Milli-Q.

2.2 Casting of different samples

Different specimen of mortar cube, concrete cube and pavers block were prepared using CFBC ash, PPC and river sand in varying proportions as shown in Fig. 1.

2.2.1 Mortar cubes

Four mixes for mortar cubes of size (7×7×7) cm³ as combination of CFBC ash and PPC were made in different proportions viz.50:50, 60:40, 70:30, 80:20 along with river sand as shown in Fig. 1(a). The mixes were coded as m50, m60, m70 and m80 respectively where m represents mortar specimen and numeric value represents CFBC ash proportion. The details of mix composition for four proportions are shown in Table 2. The raw material was put in Pan Type Mixer for

Table 1 — Physico- chemical properties of CFBC ash.

Parameters	Values
pH	13.10
Color	Grey
Texture	Powder
Bulk Density (g/cc)	1.23
Particle Density (g/cc)	2.61
Porosity (%)	52.9
Conductivity (mS/cm)	11.6
Blaine Fineness (cm ² /g)	3050.0
Lime Reactivity (MPa)	8.60
Metal Oxides (%)	
Sulphur Trioxide (SO ₃)	22.8
Calcium Oxide (CaO)	37.9
Silicon Oxide (SiO ₂)	17.6
Magnesium Oxide (MgO)	4.07
Aluminium Oxide (Al ₂ O ₃)	3.62
Iron Oxide (Fe ₂ O ₃)	2.78
Potassium Oxide (K ₂ O)	0.54
Titanium Oxide (TiO ₂)	0.12
Vanadium Oxide (V ₂ O ₅)	0.09
Loss on Ignition (LoI)	9.90

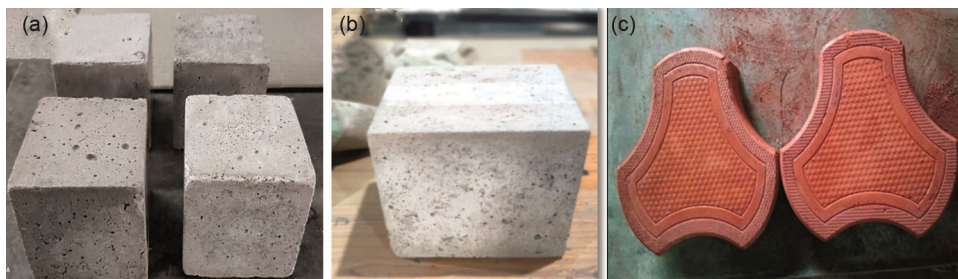


Fig. 1 — CFBC ash based (a) mortar cube (b) concrete cube and (c) cosmic shape pavers block.

Table 2 — Mix composition for CFBC ash based specimens.

Sample Code	CFBC Ash (kg)	PPC (kg)	Sand (kg)	Aggregate (20 mm) (kg)	Aggregate (10 mm) (kg)	Water: Cement Ratio
Mortar Cubes						
m50	1.0	1.0	6.0	NA	NA	0.45
m60	1.2	0.8	6.0	NA	NA	0.45
m70	1.4	0.6	6.0	NA	NA	0.45
m80	1.6	0.4	6.0	NA	NA	0.45
Concrete Cube						
C50	0.89	1.04	2.31	4.76	NA	0.45
C60	1.06	0.83	2.31	4.76	NA	0.45
C70	1.24	0.62	2.31	4.76	NA	0.45
C80	1.42	0.42	2.31	4.76	NA	0.45
Pavers Block						
P50	0.751	0.879	1.95	1.41	2.62	0.45
P60	0.902	0.704	1.95	1.41	2.62	0.45
P70	1.052	0.527	1.95	1.41	2.62	0.45
P80	1.202	0.352	1.95	1.41	2.62	0.45

specified time and calculated amount of water was added into mixer. The casted samples were water cured for 3days, 7days, 14days and 28 days respectively followed by determination of compressive strength as per IS 4031 (Part 6): 1988 (Reaffirmed 2005)¹¹.

2.2.2 Concrete Cubes

Concrete cubes of size (15×15×15) cm³ were casted with M-25grade [1 part is CFBC+ PPC, 1 part is sand and 2 part is coarse aggregate] as shown in Fig. 1(b). The samples were coded as C50, C60, C70 and C80 respectively. The composition followed for the concrete using PCFA, PPC and other components is shown in Table 2. Standard test procedure was followed as per IS: 516-1959¹² using Compression Testing Machine.

2.2.3 Pavers Block

Cosmic shape paver blocks were casted as shown in Fig. 1(c) using PCFA, PPC, sand and coarse aggregates (10mm and 20mm) and their composition is shown in Table 2. M25 mix was adopted while casting these samples coded as P50, P60, P70 and P80 respectively where P represents pavers block specimen and numeric value represents the proportion of CFBC ash. The samples were tested as per IS 15658- 2006¹³ using Compression Testing Machine.

2.3 Engineering properties of developed products

2.3.1 Strength Test

For each parameter, three specimen were tested and average value was observed. The water absorption of the concrete sample was tested using oven drying

method as per IS 1124–1974 (Reaffirmed 1993)¹⁴ and calculated as per Eq.(1). The compressive strength was tested using touch screen display based Compression Testing Machine (AIM 320E-DG-1-T) Make AIMIL with loading rate of 1000N/s and was calculated using Eq.(2). The flexural strength of pavers block was determined using fully automatic touchscreen display based Flexural Testing Machine (AIM 332E-DG-1) Make AIMIL and was calculated using Eq.(3).

$$\text{Water Absorption (\%)} = \frac{W_1 - W_2}{W_2} \dots(1)$$

where w₁ and w₂ are wet and dry weights of samples respectively.

$$\text{Compressive Strength (F), MPa} = P/A \dots(2)$$

where A is the cross section of the area of the material resisting the load and P is the maximum load applied to the material (or load until failure)

$$\text{Flexural Strength}(\sigma_f), \text{MPa} = 3PL/2bd^2 \dots(3)$$

Where P stands for the load, L for the support span, b for the sample width and d for the sample depth at a given point.

2.3.2 Abrasion test

Concrete pavements have ability to withstand abrasion which is primarily dependent on the quality of top 3-5 mm of the surface layer. A sample measuring (70.6×70.6) mm² was put on a revolving disc moving at 30 rpm and subjected to a constant load of 300N in order to assess the abrasion. After a predetermined constant revolution of 22 rotations, the disc is uniformly covered with 20g of abrasive powder (sand). This process is continued until 220 revolutions are completed. Wear is determined by comparing the values from a measuring device taken before and after abrasion. This wear value was compared with the specimen's average thickness loss, which was determined using the Eq.(4).

$$T = (W_1 - W_2) \times V_1 / (W_1 \times A) \dots(4)$$

Where, T = Average loss in thickness (mm), W₁= Initial mass of the specimen (g), W₂ = Final mass of the abraded specimen (g), V₁= Initial volume of the specimen (mm³), A = Surface area of the specimen (mm²).

2.3.3 Hydration products

The crushed specimen was finely grinded into powder using mortar & pestle and passed through

sieve no. 200 (75 μ m) for hydration phase analysis by X- Ray Diffractometer Make Rigaku with CuK α radiation ($\lambda = 0.15419$ nm) over a 2θ range from 0 to 80°. The resultant intensity peaks were confirmed through JCPDS file.

2.3.4 Scanning electron microscopy

Morphological studies were done by Scanning Electron Microscope Model, JEOL JCM 6000 Plus. The grinded sample was sonicated for 15 minutes and the sample was mounted over the carbon tape on a stud using micro pipette followed by making its surface conductive in high vacuum mode with deep gold coating. Backscattered electron (BSE) images of CFBC ash were obtained at magnification of 2000X and 4300X with 10 μ m and 5 μ m resolutions respectively.

2.3.5 Leachability test

The leachability of toxic/heavy metals from CFBC ash and samples were determined using Toxicity Characteristics Leaching Procedure (TCLP) according to EPA 1311 Method- 1992¹⁵. The mortar and concrete samples were crushed and brought to size ≤ 10 mm. The collected leachate from TCLP was analyzed in Atomic Absorption Spectrophotometer Make Thermo Fisher Scientific iCE 3000 Series for detection of toxic /heavy metals.

3 Results and Discussion

3.1 Characterization of raw materials

3.1.1 Heavy metals analysis

The CFBC ash sample was digested in Nitric Acid using hot plate at temperature of 95°C. The sample was filtered and analyzed for heavy metal analysis for elements like Iron, Lead, Zinc, Nickel, Copper, Cobalt, Cadmium, Manganese, Aluminum, Chromium and Vanadium as shown in Table 3. The results showed higher concentration of aluminum iron & vanadium followed by Nickel, Manganese, Zinc and Chromium. Other metals like Lead, Copper, Cadmium and Cobalt were found to be Below Detection Limit (BDL). The presence of heavy metals in CFBC ash is mainly contributed from the primary fuel which is Petcoke blended with Indian Coal combusted in the CFBC boiler.

3.1.2 Particle size analysis

The particle size of CFBC ash was analyzed through Laser Scattering Particle Size Distribution Analyzer, Make Partica LA- 950, HORIBA Scientific

Northampton, UK. The dispersion medium used was water having refractive index 1.33 and CFBC ash with refractive index 1.60. The average mean particle size of CFBC ash was found to be in the range of 5.74 μ m- 6.46 μ m. The results of CFBC ash (D10- 1.86 μ m, D50- 2.98 μ m, D90- 16.35 μ m) indicate that 10% of the total volume has diameter ≤ 1.86 μ m, 50% of the total volume has diameter ≤ 2.98 μ m and 90% of the total volume has diameter ≤ 16.35 μ m respectively. Increased fineness results in enhanced hydration of lime and anhydrite as well as increased dissolution of alumina and silica. As a result, finer particles exhibit higher concentration of SO₃ and CaO which enhances the overall efficiency of hydration.

3.1.2 Mineralogical study

The X-ray diffraction pattern of CFBC ash is shown in Fig. 2 which was identified with JCPDS file. The result shows presence of various mineralogical phases like Anhydrous Gypsum (CaSO₄) (JCPDS No. 6-226), Vanadium Oxide (V₂O₅) (JCPDS No. 19-1398), Calcite (CaCO₃) (JCPDS No. 24-27), Lime

Table 3 — Heavy metals concentration in CFBC ash.

Elements	Concentration (mg/L)
Aluminium	9508.0
Iron	6024.4
Vanadium	848.0
Nickel	201.3
Manganese	145.7
Zinc	29.8
Chromium	17.5
Lead	BDL
Copper	BDL
Cadmium	BDL
Cobalt	BDL

BDL signifies Below Detection Limit

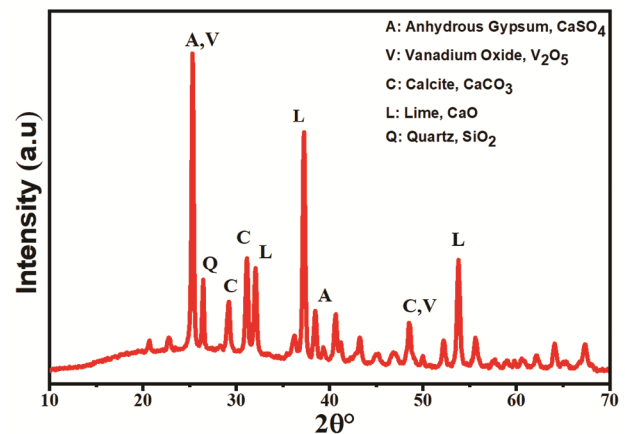


Fig. 2 — X- ray diffraction pattern of CFBC ash.

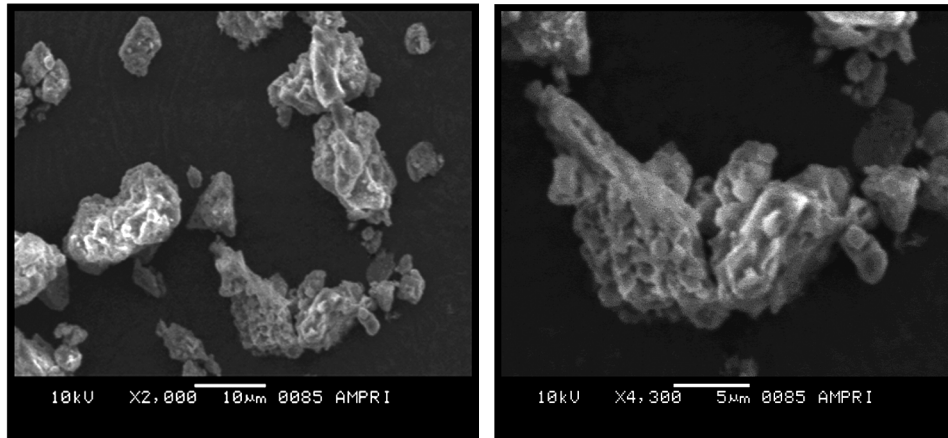


Fig. 3 — SEM images of CFBC ash.

(CaO) and Quartz (SiO₂) (JCPDS No. 6-226). The main mineralogical phase of Anhydrous Gypsum is due to reaction of limestone added to capture emitted Sulfur Dioxide from high Sulfur content Petcoke blended with Coal during combustion process in CFBC boiler. The presence of Lime and Calcite is due to addition of desulfurizing agent limestone (CaCO₃) whereas Vanadium Oxide is contributed from primary fuel Petcoke¹⁶.

3.1.3 Morphological studies

The SEM micrograph of CFBC ash was recorded at 10µm and 5µm at magnification of 2000X and 4300X respectively as shown in Fig. 3. The findings indicate that agglomeration occurs through the sintering of CaSO₄ crystals creating a framework that extends uniformly in three dimensions resulting in a stronger cohesion. The loose, amorphous masses observed between these structures exhibit a more complex and varied composition potentially attributed to the presence of unburnt particles. CFBC ash particles exhibit a subangular shape and possess internal porosity because they remain unburnt during the combustion process¹⁷.

The Energy Dispersive X-ray Spectroscopy (EDS) results show that CFBC ash is predominantly composed of Aluminium, Calcium, Silicon, Vanadium and minor amount of other elements like Sulfur. Mass% represents the weight percentage of each element in the CFBC ash while Atom% represents the atomic percentage of each element. Calcium is a dominant element in CFBC ash with a significant contribution of 43.08% in both mass% and atom%. It is followed by vanadium (25.55%), aluminium (15.22%), Silicon (10.57%) and Sulphur (5.58%). These findings are consistent with previous studies confirming the composition and elemental

Table 4 — Engineering properties of CFBC ash based specimens.

Sample Composition	Density (g/cc)				Compressive Strength (MPa)			
	3day	7day	14day	28day	3day	7day	14day	28day
Mortar Cube								
m50	2.22	2.24	2.24	2.25	8.2	14.2	20.0	22.7
m60	2.21	2.20	2.22	2.23	6.9	10.2	17.2	21.0
m70	2.18	2.21	2.23	2.25	5.1	7.9	11.0	20.5
m80	2.2	2.21	2.21	2.23	3.8	5.8	10.8	13.1
Concrete Cube								
C50	2.51	2.51	2.52	2.53	12.0	18.3	23.3	27.0
C60	2.50	2.50	2.51	2.52	11.5	15.3	20.7	26.5
C70	2.50	2.50	2.51	2.53	10.0	16.7	21.1	27.0
C80	2.49	2.49	2.50	2.50	9.4	14.1	20.1	24.3
Pavers Block								
P50	2.34	2.35	2.36	2.38	15.0	19.5	22.0	27.1
P60	2.35	2.35	2.37	2.37	12.8	16.5	19.7	26.5
P70	2.34	2.34	2.35	2.37	13.10	18.6	21.8	27.1
P80	2.33	2.34	2.35	2.35	12.01	15.2	19.0	24.7

distribution in CFBC ash¹⁸. Presence of calcium containing minerals in the fuel plays crucial role in ash properties such as pozzolanic activity and reactivity with water.

3.2 Properties of mortar and concrete cubes

3.2.1 Mortar cube

The engineering properties of mortar cubes coded as m50, m60, m70, m80 having different ratios of CFBC ash and PPC are shown in Table 4. The density of different composition of CFBC ash based mortar are shown in Fig. 4(a). The difference in density of the mortar cube over 3days, 7days, 14days and 28days is due to variation in hydration, changes in pore structure and curing conditions. The initial hydration may lead to denser microstructure as

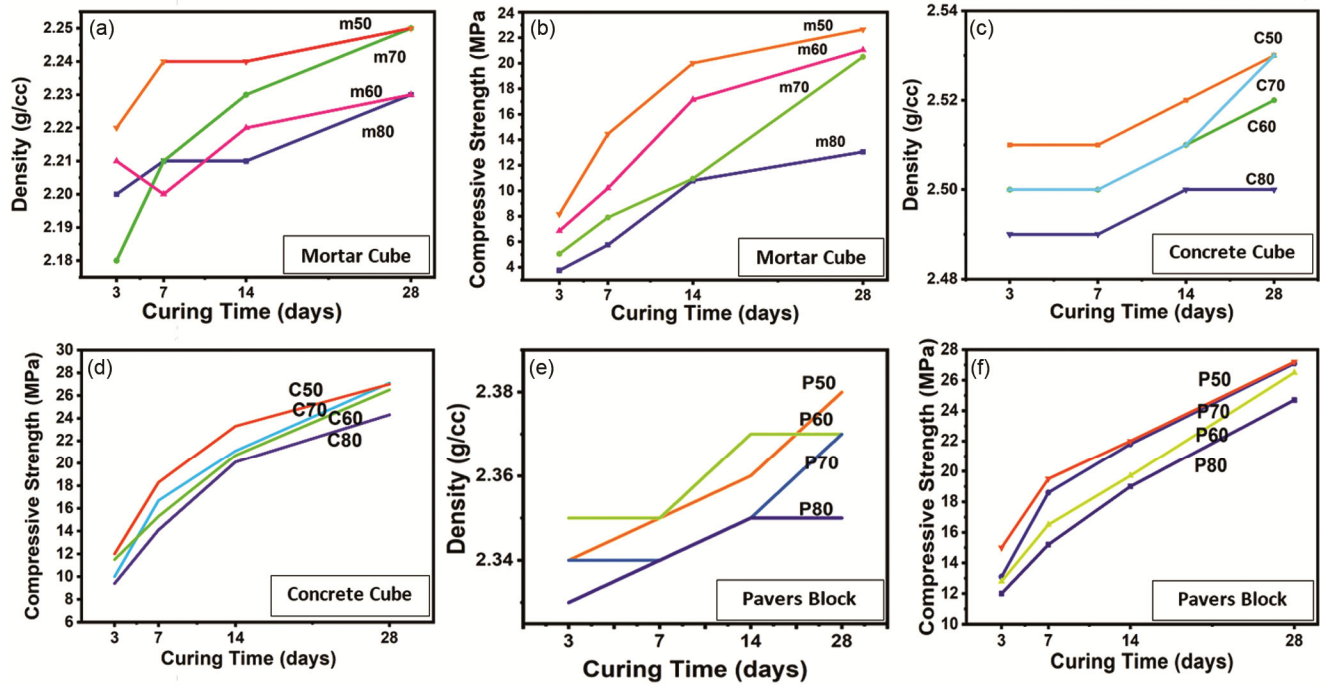


Fig. 4 — Variation in properties with curing time for different CFBC ash based specimen (a, b) mortar cube, (c, d) concrete cube and (e, f) pavers block.

hydration products start forming and filling the void spaces within the material¹⁹. Fig.4(b) shows the compressive strength of the mortar cube with different proportion of CFBC ash. It can be observed from the results that the compressive strength decreases with increase in composition of CFBC ash which may be due to finer particles of CFBC ash and its higher porosity. The water-to-cement (W/C) ratio required to achieve a paste of normal consistency remained uniform in all the samples i.e. 0.45 which may be due to fineness of CFBC ash. Higher ash content requires more water for hydration and workability potentially leading to increased porosity or reduced compaction which reduces the compressive strength²⁰. It can also be observed that the properties of m50, m60 and m70 are closely related to one another but varies in m80 as steep reduction is observed in its compressive strength.

3.2.2 Concrete cube

The use of blended Portland Pozzolana Cement using CFBC ash was first reported in 1994²¹ and further research was conducted to study the properties of blended Portland cement including CFBC fly ash for potential construction application²²⁻²⁴. In the present study, concrete samples were prepared using CFBC ash blended with PPC in different proportion like 50% CFBC ash+ 50% PPC (50:50), 60% CFBC

ash + 40% PPC (60:40), 70% CFBC ash + 30% PPC(70:30) and 80% CFBC ash + 20% (80:20) PPC coded as C50, C60, C70, C80 respectively and engineering properties were evaluated as given in Table 4.

M-25 grade was adopted in this study and it was observed from the results that it followed similar trend as observed for mortar specimen. The variation in the density of concrete with varying proportions of CFBC ash, is depicted in Fig. 4(c). Furthermore, it is observed that 70:30 concrete exhibits higher density, potentially attributable to difference in pore structure and initial hydration. The compressive strength shows inverse relationship with the CFBC ash content as shown in Fig. 4(d). It was found that as the CFBC ash content increases, comprehensive strength decreases. From above results, it can be seen that for getting desired cementitious properties, the content of CFBC ash should not exceed 70% as further increase in CFBC ash content results in reduction in strength of the specimen.

3.2.3 Pavers block

With increasing complexity of environmental challenges, industrial waste management and development of sustainable and eco-friendly materials has become paramount. This has led to development of pavers block for non-traffic load conditions. As

given in Table 4, the cosmic shape pavers block samples were prepared from M-25 grade using CFBC ash blended with PPC in different proportion like 50% CFBC ash + 50% PPC (50:50), 60% CFBC ash + 40% PPC (60:40), 70% CFBC ash + 30% PPC (70:30) and 80% CFBC ash + 20% PPC (80:20) coded as P50, P60, P70, P80 respectively. The pavers block with thickness of 60 mm are prepared with fine aggregate, coarse aggregate and water. The engineering properties are as shown in Fig. 4(e) and Fig. 4(f). Based on the results, it was found that best results were obtained in mix ratio of 70:30.

The properties of optimized pavers block with mix ratio 70:30 were evaluated as per IS 15658 (2006) and compared with conventional pavers block. Result shown in Table 5 indicate that compressive strength of paver blocks made with CFBC ash is either superior to or at par with conventional pavers block. The enhanced performance of CFBC ash based pavers block can be attributed to the hydration process of anhydrous gypsum present in the CFBC ash. Upon hydration, anhydrous gypsum transforms into gypsum crystals within the concrete matrix. This transformation is critical as it contributes significantly to the development of strength in the paver blocks. Specifically, the primary binder in concrete Calcium Silicate Hydrate (C-S-H) precipitates on the gypsum crystals which act as nucleation sites. This precipitation process further enhances the strength of

the concrete. It is reported in previous studies that gypsum is known to accelerate hydration of C_3S ^{25,26} which helps to improve the engineering properties of concrete.

3.3 Hydration products of CFBC Ash

The XRD graphs of different specimen are shown in Fig. 5 which signifies that there is slight difference in the mineralogical phases for CFBC ash mortar and concrete specimen but the main hydration products responsible for quick strength and setting of paste are Tricalcium Silicate, Calcium Silicate Hydrate, Gypsum and Ettringite. The intensity of Tricalcium Silicate (Ca_3SiO_5) peak is more prominent in mortar specimen as compared to concrete specimen whereas additional peaks of Portlandite ($Ca(OH)_2$) and Anorthite ($CaAl_2Si_2O_8$) were observed in concrete and pavers block samples.

The hydration products found in hardened mixture of CFBC ash i.e. mortar, concrete cube and pavers block were Tricalcium silicate (C_3S), Calcium Silicate Hydrate (C-S-H) and Gypsum. The main peaks of C_3S were found at (2θ : 32.3°, 33.9°, 28.9°, 50.6° and 41.3°) and peaks of C-S-H were found at (2θ : 32.0°, 39.2° and 27.3°)²⁷. The dissolution of Anhydrite ($CaSO_4$) in hydrated pastes is indicated by the presence of Gypsum phase at (2θ : 30.9°, 20.5° and 33.2°) which may be due to reaction of Anhydrite of CFBC ash and water²⁸. Formation of Ettringite is attributed to high SO_3 in CFBC ash¹⁷. The presence of reactive crystalline phases such as free lime (f-CaO) and Anhydrite ($CaSO_4$) in CFBC ash results in improving the hydration reaction by the addition of CFBC ash in Portland Cement²⁹. The crystalline meta stable

Table 5 — Properties of pavers block.

Properties	CFBC Pavers Block (70:30)	Conventional Pavers Block
Shape	Cosmic	Cosmic
Thickness(mm)	60	60
Weight (Kg)	7.10	6.60
Density (g/cc)	2.37	2.20
Water Absorption (%)	5.90	4.81
Compressive Strength (MPa)		
3 days	13.1	12.0
7days	18.6	17.1
14 days	21.8	21.2
28 days	27.1	25.3
Flexural Strength (N/mm ²)		
3 days	1.02	1.15
7days	1.50	1.60
14 days	2.07	2.06
28 days	2.23	2.28
Abrasion Resistance (mm)	1.06	1.50

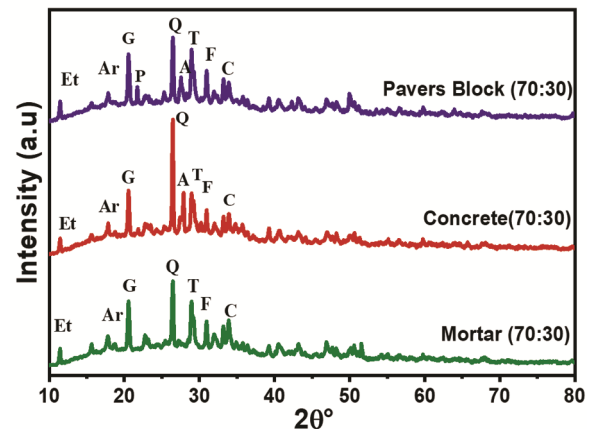


Fig. 5 — X-ray diffraction pattern of different specimen at optimized ratio (Et: Ettringite; T:Tricalcium Silicate; P:Portlandite; A: Anorthite; Ar :Aragonite; F: Iron Vanadium Oxide; G: Gypsum; C:Calcium Silicate Hydrate; Q: Quartz).

polymorphs of Calcium Carbonate i.e. Aragonite phases are also present in specimen and the peaks were found at (2θ : 17.0° , 17.8° , 11.4° and 35.8°) which is in accordance with reported literature^{30,31}. The Portlandite $\text{Ca}(\text{OH})_2$ is formed due to hydration of free lime in CFBC ash and PPC at room temperature. It is also reported in previous studies that Portlandite originated from both CFBC ash and also due to the hydration of lime which is present in it³². Similar results of XRD pattern were also reported in the literature^{33,34}.

3.4 Microstructure characteristics of developed products

Scanning Electron Microscopy is used to examine the microstructure characteristics of CFBC concrete which provides valuable insights into surface morphology and properties. The surfaces of mortar cubes, concrete cubes and pavers blocks were polished and gold coated before observation under SEM. The SEM images presented in Fig. 6 depict the microstructure of these specimen after being hydrated for 28 days with an optimized ratio of 70% CFBC ash, 30% PPC (70:30) and other materials. The images captured at $50\mu\text{m}$ at lower magnification ($1000\times$) show smooth surface of the concrete and pavers blocks Fig. 6(a), Fig. 6(c), Fig. 6(e) indicating good particle packing and interfacial bonding. This smooth surface suggests that the CFBC ash along with other components contributed to formation of dense microstructure with well-compacted particles. Such structure is beneficial for enhancing the engineering properties of the concrete.

The SEM images at $5\mu\text{m}$ at higher magnification of $10000\times$ as depicted in Fig. 6(b), Fig. 6(d), Fig. 6(f) reveal the microstructure of hydration products and pore size distribution within the PCFA and PPC based concrete matrix. The addition of CFBC ash tends to fill voids leading to denser microstructure. The study shows that CaO present in CFBC ash undergoes hydration to form $\text{Ca}(\text{OH})_2$ contributing to the formation of Ettringite and Calcium Silicate Hydrate³⁵. Higher lime content in CFBC ash influences the production of Ettringite and Calcium Silicate Hydrate. The formation of Calcium Silicate Hydrate and Ettringite during the hydration process contributes to pore filling and improves the compactness of the specimen as reported in literature³⁴. This densification is crucial for improving the permeability and mechanical strength of the concrete which is confirmed through XRD results showing main hydration products like Tricalcium Silicate, Gypsum and Portlandite.

3.5 Leachability studies of developed products

TCLP test was performed to determine the leaching of toxic/heavy metals from CFBC ash based specimen like mortar, concrete cube and pavers block using Toxicity Characteristic Leaching Procedure (TCLP) Apparatus under acidic condition ($\text{pH } 4.0 \pm 0.05$). TCLP tests were carried out for the crushed samples solidified at hydration of 28 days. The concentration of heavy metals in collected leachate were detected through Atomic Absorption Spectrophotometer, Model Thermo Fisher Scientific iCE 3500. The

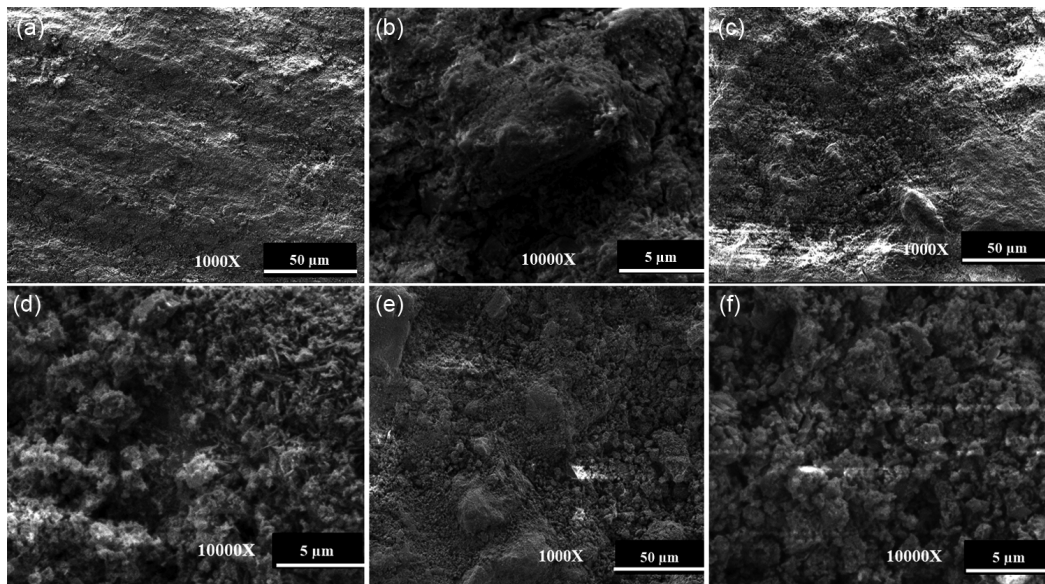


Fig. 6 — SEM images of specimen at 70:30 ratio after 28 days hydration (a, b) mortar cube, (c, d) concrete cube and (e, f) pavers block.

experiments were conducted in triplicate and the results were calculated as the mean of three sets of measurements.

The results showed presence of Iron, Manganese, Zinc, Aluminium and Nickel in the range of 1.285-3.052mg/L, 0.239-0.353mg/L, 0.088-0.355mg/L, 3.075-9.856mg/L and 0.044-0.079mg/L respectively whereas Copper, Lead, Chromium, Cobalt, Cadmium and Vanadium are found to be Below Detection Limit (BDL). However, all heavy metals concentration are found to be well within the permissible limits as per USEPA Method 1311. As reported, the denser structure showed higher compressive strength and contributed to reduction in the leaching of heavy metals³⁶. From the results, it can be noted that the mortar sample had slightly higher leachability of heavy metals compared to the concrete samples, indicating that heavy metals were well encapsulated in a matrix of concrete samples with higher density. Hence, CFBC ash based products can be considered as non-hazardous and environment friendly for wider application spectrum in development of various building material components.

4 Conclusion

Based on the experimental results, following conclusions can be drawn:

- a X-ray Diffraction (XRD) pattern of CFBC ash shows presence of Anhydrite, Lime, Silicon Oxide, Calcite and Vanadium Oxide. These findings suggest that the hydrate components of concrete include Calcium Silicate and Portlandite which promote the formation of C-S-H phase when incorporated into the cementitious matrix.
- b The study recommends use of CFBC ash for suitable application as partial replacement for PPC. CFBC ash can be used in combination with PPC in an optimized ratio of 70 (CFBC ash):30 (PPC) for making mortar, concrete cubes and pavers block which qualify the desired results as per relevant IS codes.
- c The strength of CFBC ash concrete got enhanced by the interfacial bonding of CFBC ash and PPC at an optimized ratio of 70:30. The main hydration products responsible for quick strength development and setting of paste in both mortar and concrete are Tricalcium Silicate (Ca_3SiO_5), Calcium Silicate Hydrate (C-S-H), Gypsum and Ettringite.
- d The SEM images depict smooth surface of the concrete and pavers block indicating strong particle packing and interfacial bonding. This densification enhances mechanical strength and durability of the concrete.
- e The TCLP studies of mortar, concrete cube and pavers block suggest that CFBC ash products are non-hazardous, environmentally safe and free from leaching aspects.
- f Use of CFBC fly ash as partial replacement of PPC facilitates environment friendly approach towards disposal of high sulfur content CFBC ash.
- g The developed products enable consumption and disposal of CFBC ash on a large scale and also help in development of various building components which have benefits of industrial waste utilization and environment protection.

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References

- 1 Lupiáñez C, Guedea I, Bolea I, Diez LI & Romeo LM, *Fuel Process Technol*, 106 (2013) 587.
- 2 Go ES, Kim BS, Ling JL, Oh SS, Park HJ & Lee SH, *Environ Res*, 225 (2023) 115582.
- 3 Mohapatra BN, Narayan R, Joshi SM, Gupta RM, Gautam SP & Jain RK, *J Solid Waste Technol Manag*, 36(1) (2010) 698.
- 4 Rajabipour F, Zahedi M & Kaladharan G, *Evaluating the performance and feasibility of using recovered fly ash and fluidized bed combustion (FBC) fly ash as concrete Pozzolan*, ACI Concrete Research Council, Final Report (2020).
- 5 Li XG, Chen QB, Huang KZ, Ma BG & Wu B, *Constr Build Mater*, 36 (2012) 182.
- 6 Wu T, Chi M & Huang R, *Constr Build Mater*, 66 (2014) 172.
- 7 Jia G, Wang, Y & Yang F, *Adv Mater Sci Eng*, 2022 (1), 7099430.
- 8 Lee BY, Jeon SM, Cho CG & Kim HK, *Constr Build Mater*, 257 (2020) 119507.
- 9 Indian standard of specification of portland pozzolana cement, IS 1489 (Part I) Bureau of Indian Standards, New Delhi, India, 1991 (Reaffirmed 2002).
- 10 Indian Standard on Specification for coarse and fine aggregates from natural sources for concrete, Bureau of Indian Standards, New Delhi, India, IS:383 – 1970 (2002).
- 11 Indian standard methods of physical tests for hydraulic cement, Part 6: Determination of compressive strength of hydraulic cement (other than masonry cement) IS 4031 (Part 6), Bureau of Indian Standards, New Delhi, India, 1988 (Reaffirmed 2005).
- 12 Indian standard methods of tests for strength of concrete, Bureau of Indian Standards, New Delhi, India, IS:516-1959.
- 13 Indian standard method of Precast concrete blocks for paving Specification, IS 15658: 2006.
- 14 Indian standard method of test for determination of water absorption, apparent specific gravity and porosity of natural

- building stones [CED 6: Stones], IS:1124 – 1974 (Reaffirmed 1993).
- 15 EPA Method 1311 (SW-846): Toxicity Characteristic Leaching Procedure, July 1992.
- 16 Petr B & Hlincik T, *Adsorp Sci Technol*, (2021) 8604778.
- 17 Zahedi M & Rajabipour F, *ACI Mater J*, 116(4) (2019) 163.
- 18 Lee SH & Kim GS, *J Korean Ceram Soc*, 54(2) (2017) 128.
- 19 Jing P, Song X, Zhang J & Nowamooz H, *Constr Build Mater*, 322 (2022) 126446.
- 20 Luan C, Wu Z, Han Z, Gao X, Zhou Z, Du P, Wu F, Du S & Huang Y, *J Clean Prod*, 415 (2023) 1377352023.
- 21 Behr-Andres CB & Hutzler NJ, *J Environ Eng*, 120(6) (1994) 1488.
- 22 Lin WT, Lin KL, Chen K, Korniejenko K, Hebda M & Lach M, *Mater*, 12(24) (2019) 4204.
- 23 Wu R, Dai S, Jian S, Huang J, Lv Y, Li B & Azizbek N, *Constr Build Mater*, 237 (2020) 117644.
- 24 Sebestova P, Cerny V & Drochytka R, *Mater Technol*, 54 (2020) 157.
- 25 Shen Y, Qian J, & Zhang Z, *Constr Build Mater*, 40 (2013) 672.
- 26 Siler P, Bayer P, Sehnal T, Kolářová I, Opravil T & Šoukal F, *Constr Build Mater*, 78 (2015) 181.
- 27 Parmar V, Padmakaran P & Khan MA, *CNS&E*, 1(1) (2024), 36.
- 28 Hanisková D, Bartoníčková E, Koplí J & Opravil T, *Procedia Eng*, 151 (2016) 394.
- 29 Yue Y, Wang JJ, Basheer PM, Boland J & Bai Y, *Constr Build Mater*, 135 (2017) 369.
- 30 Zhou M, Chen P, Chen X, Ge X & Wang Y, *Constr Build Mater*, 251 (2020) 118993.
- 31 Silva LA, Nahime BO, Lima EC, Akasaki JL & Reis IC, *Ceram*, 66 (2020) 373.
- 32 Kim HJ, Tafesse M, Lee HK & Kim HK, *Cem Concr Res*, 123(2019) 105771.
- 33 Kim H J & Lee HK, *Miner*, 7(12) (2017) 237.
- 34 Mehta PK & Monterio PJM, *Concrete-Microstructure, Properties and Materials*, 3rd ed.; McGraw-Hill Companies, Inc.: New York, NY, USA, 2006.
- 35 Baek C, Seo J, Choi M, Cho J, Ahn J & Cho K, *Sustain*, 10(12) (2018) 4854.
- 36 Liu W, Hou H, Zhang C, Zhang D, *Waste Manag Res*, 27(3) (2009) 258.