

Self-cementitious properties of fly ashes from CFBC boilers co-firing coal and high-sulphur petroleum coke

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Abstract

Self-cementitious properties of fly ash from circulating fluidized bed combustion boiler co-firing coal and high-sulphur petroleum coke (CPFA) were investigated. CPFA was self-cementitious which was affected by its fineness and chemical compositions, especially the contents of SO_3 and free lime ($f\text{-CaO}$). Higher contents of SO_3 and $f\text{-CaO}$ were beneficial to self-cementitious strength; the self-cementitious strength increases with a decrease of its 45 μm sieve residue. The expansive ratio of CPFA hardened paste was high because of generation of ettringite (AFt), which was influenced by its water to binder ratio (W/A), curing style and grinding of the ash. The paste cured in water had the highest expansive ratio, and grinding of CPFA was beneficial to its volume stability. The hydration products of CPFA detected by X-ray diffraction (XRD) and scanning electron microscopy (SEM) were portlandite, gypsum, AFt and hydrated calcium silicate (C–S–H).

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Keywords: CFBC fly ash; Self-cementing; Expansion; Hydration product

1. Introduction

Circulating fluidized bed combustion (CFBC) is an effective technology for high-sulphur coal burning, and SO_2 can be captured by limestone added in situ during combustion [1]. High-sulphur petroleum coke is a by-product of high-sulphur oil refining industry. For its high calorific value, it is often fired in CFBC as fuel with coal [2]. For increasing the SO_2 -removal efficiency, the ratio of Ca/S is often increased to 2.0–2.5, and then there are many unreacted CaO and desulphurized products CaSO_4 , and a little CaCO_3 remaining in the fly ash and bed ash of CFBC [1]. X. R. Fu [3] and F. H. Li et al. [4] have comprehensively investigated the properties of fly ash from CFBC boilers co-firing coal and high-sulphur petroleum coke (CPFA), and the results show that the properties and micrograph of the CPFA are different from the pulverized ash due to many differences in their combustion processes.

High-calcium fly ash often has the property of self-cementing, namely it can set and harden with addition of water [5–8]. The

reasons for the self-cementing property are the C_3A , $\beta\text{-C}_2\text{S}$ and other cementitious minerals existing in high-calcium fly ash which can produce AFt and C–S–H etc. [6]. CFBC fly ash has the same property of self-cementing [9], and several papers [10–14] have reported that CFBC fly ash can be used to make no-cement concrete with pressured fluidized bed combustion ash (PFBCA) or pulverized fuel fly ash (PFA). Zhang and Qiu [15] have manufactured a cement with CFBC fly ash and PFA which can attain about 32.5 to 42.5 MPa of compressive strength at the age of 28 days. The investigation of J. Iribarne et al. [11] has showed that the no-cement mortar made with CFBC fly ash and PFBCA has a higher strength than that of CFBC fly ash and PFA, and the curing style has a large influence on the strength. The no-cement mortar shrinks when it is cured in a moist atmosphere and it expands when it is cured in water [11]. Freidin [16] has reported that a high $f\text{-CaO}$ and high SO_3 content fly ash has good self-cementitious properties, the compressive strength can develop gradually, and the curing style affects the strength; curing in the open air has the lowest compressive strength. CPFA is different from high-calcium fly ash because of a different mineral phase which results in different self-cementing, and only a few papers report on it. This paper is dedicated to investigating the self-cementing properties of CPFA.

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2. Materials and experimental methods

2.1. Materials

The CPFA used in this investigation came from a 220 t/h Pyroflow CFBC boiler firing coal and high-sulphur petroleum coke and using limestone as the SO₂ sorbent in Power Plant at the Sinopec Jinling Petrochemical Corporation. The ratios of coal to coke were from 50:50 (cal %) to 70:30 (cal %). Four fly ashes were used which were collected from the ash-hoppers of the electrostatic precipitator of the CFBC boiler and identified as DF1, DF2, DF3, and DF4. Their chemical compositions and finenesses were shown in Table 1. XRD patterns were shown in Fig. 1.

We know from Table 1 that all the CPFAs have high content of LOI, SO₃ and *f*-CaO. Major mineralogical compositions of CPFA detected by XRD are lime (CaO), anhydrite (CaSO₄), calcite (CaCO₃), portlandite (Ca(OH)₂) and α -quartz. The presence of Ca(OH)₂ in CPFAs is due to the reaction of *f*-CaO with water vapor in the air during the CFBC ash storage. There is a diffuse halo peak at 21° to 29°, especially in the XRD pattern of DF1, which is attributed to dehydrated clay minerals [17,18], or maybe glass phase.

2.2. Methods

2.2.1. Samples preparation

The DF1 through DF4 samples were used directly in the self-cementitious strength test. Portions of DF4 were ground in a laboratory ball mill (\varnothing 500 × 500 mm) for 20 min, 40 min and 60 min and designated as DF4-2, DF4-4 and DF4-6, respectively. Their residues on a 45 μ m sieve are 10.3%, 5.0% and 2.4%, respectively; while Blaine specific surfaces are 352 m²/kg, 428 m²/kg and 475 m²/kg, respectively.

2.2.2. Physical test

The strength test was done at the age of 7, 28 and 60 days according to Chinese standard GB/T 17671-2001 (equivalent

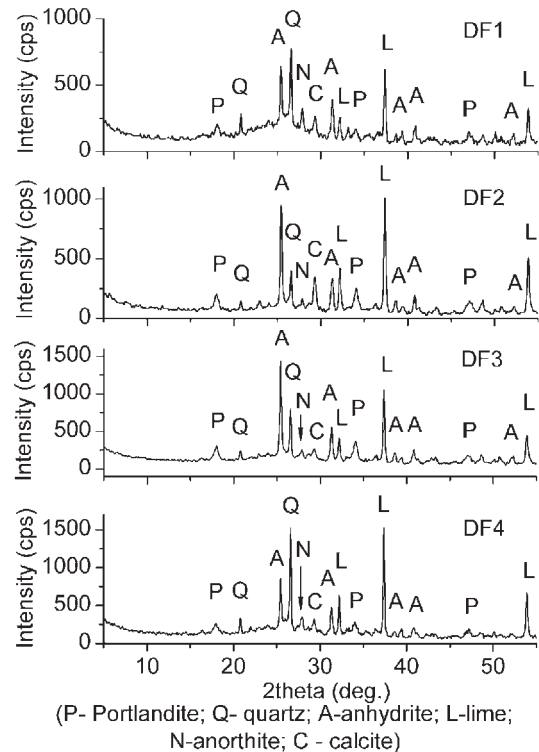


Fig. 1. XRD patterns of materials.

to ISO 679:1989) by using a 40 mm × 40 mm × 160 mm prism specimen with a water to ash ratio (W/A) of 0.6, but the W/A of the strength tests of ground ashes were 0.4. 0.8% JM-F naphthalene superplasticizer was added for reducing the water requirement. After curing at 20 ± 1 °C and in ≥ 90% R.H. (relative humidity) chamber for 20 to 24 h, samples were demolded and cured in 20 ± 1 °C air at ≥ 90% R.H. till the strength test day. If the sample can't be demolded after 24 h, it would be demolded after 48 h for another 24 h of curing. DF1 and DF2 were sprinkled until all surfaces of the prisms were wet every 3 days in the curing process. The test of setting time was done in accord with Chinese standard GB/T1346-2001 by using the Vicat method.

The expansion prism was made with dimensions of 40 mm × 40 mm × 160 mm and cured at 20 ± 1 °C and in a ≥ 90% R.H. chamber for 24 ± 1 h. Afterwards the specimen was demolded and cured at 20 ± 1 °C and in ≥ 90% R.H. air (with the exception that DF4-2B was cured in 20 ± 1 °C water). The original length was tested after 24 h.

2.2.3. Chemical test

2.2.3.1. Chemical composition. The chemical compositions of the sample were measured on an ARL-9800 X-ray fluorescence (XRF) spectrometer by the fused disk method. For the fused disk method procedure, approximately 0.7 g of the LOI-determined (LOI, loss on ignition) fly ash was weighed into a platinum crucible and thoroughly mixed with 7.7 g of flux. The mixture was heated at 1100 °C for 13.5 min and poured into a platinum cup to form a glass-like penny-shaped

Table 1
Chemical compositions and finenesses of ashes

	DF1	DF2	DF3	DF4
<i>Chemical compositions (wt.%)</i>				
LOI ^a	18.64	15.78	16.01	16.33
SiO ₂	29.37	17.43	19.58	25.19
Al ₂ O ₃	17.87	9.95	10.37	15.34
Fe ₂ O ₃	2.32	1.19	1.22	1.68
CaO	22.00	41.19	37.46	30.25
K ₂ O	0.70	0.45	0.49	0.57
Na ₂ O	0.39	0.29	0.30	0.31
MnO	0.07	0.06	0.03	0.03
MgO	1.91	1.83	3.37	1.25
TiO ₂	0.85	0.67	0.68	0.74
<i>f</i> -CaO	7.55	14.67	15.97	13.23
SO ₃	4.21	10.13	9.89	7.30
Total	98.33	98.97	99.40	98.99
<i>Physical properties</i>				
Residue on 45 μ m sieve (wt.%)	16.4	7.2	14.1	35.9
Blaine specific surface (m ² /kg)	467	378	321	249

^a LOI: loss on ignition at 950–1000 °C.

disk that was ready for analysis by XRF. The *f*-CaO content was determined by a glycerine–alcohol solution extraction method according to Chinese standard GB/T 176-1992.

2.2.3.2. XRD. Portion of the sample left after the strength test was ground in an agate mortar with acetone till all passed through the 45 μm sieve. After that the sample was filtered to remove the acetone and dried at 60 °C for 6 h in CO₂-free air. The dried sample was prepared for XRD analysis by an X'TRA X-ray diffractometer with Cu Kα radiation. Step scan was performed over the range of 5° to 55° (2θ) with stepping interval of 0.02° and a scanning rate of 5.00°/min. Diffraction peaks on the XRD spectrum were detected by software package MDI Jade 5.0 with automatically peak seek and also manually.

2.2.3.3. SEM. The sample for the SEM test was obtained from the compressive strength test, immersed in acetone for 24 h, and then dried at 60 °C for 6 h in CO₂-free air. The instrument used for SEM was a LEO-1530VP scanning electron microscope equipped with an OXFORD INCAX-300 energy-dispersive X-ray spectrometer.

3. Results and discussion

3.1. Self-cementitious strength

Self-cementitious strengths of CPFAs are shown in Table 2. The results show that DF1 through DF4 are self-cementitious. The self-cementitious strength is affected by the chemical composition and fineness of CPFA, in particular the content of *f*-CaO and SO₃. The self-cementitious strength of DF1 and DF4 are lower because of their lower *f*-CaO and SO₃ content, while the coarse particle size of DF4 results in the lowest self-cementitious strength. This is expected as the coarse particles of DF4 have a lower reactivity. DF2 and DF3 have the greater strength as a result of the higher content of *f*-CaO and SO₃. CPFA can self-cement due to the formation of C–S–H and AFt as it is mixed with water [18]; and gypsum generated from hydration of anhydrite redounds to the early strength development. C–S–H and AFt come from the reaction of *f*-CaO and active silica in the ash, and the reaction of *f*-CaO, CaSO₄ and active alumina in the ash [18]. Higher content of *f*-CaO and SO₃ is beneficial to the generation of more C–S–H and AFt. This is the reason why DF2 and DF3 have the higher self-cementitious strength. The self-cementitious properties of CPFA are different from those of high-calcium fly ash (often class C ash) because

Table 2
Self-cementitious strength and setting time of CPFAs

Materials	W/A	Setting time (min)		Flexible strength (MPa)		Compressive strength (MPa)	
		Initial	Final	7 days	28 days	7 days	28 days
DF1	0.6	1545	1767	0.1	0.8	0.4	2.2 ^a
DF2	0.6	20	68	0.6	0.7	1.5	2.1 ^a
DF3	0.6	305	409	0.5	2.7	1.1	9.8
DF4	0.6	371	519	0.2	0.4	0.4	0.7

^a Cracks appeared after 10 days' curing.

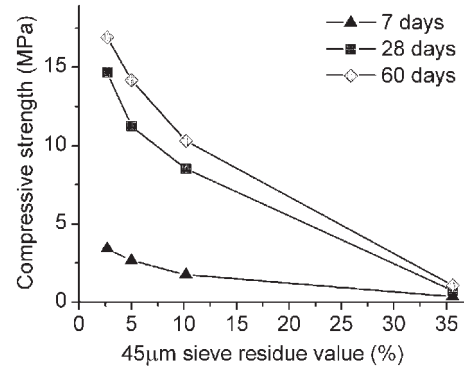


Fig. 2. Effects of fineness on the self-cementitious strength.

high-calcium fly ash includes cementitious materials, such as C₃A, β-C₂S and others [5–7]. The low-calcium fly ash (often class F ash) is often not self-cementitious if it is only mixed with water.

The setting time of CPFA is short because of the quick hydration of *f*-CaO, but setting time of DF1 is very long because of its lower *f*-CaO content.

There are some cracks on the surface of DF1 and DF2 at about 10 days' curing, but no cracks on the surface of DF3 and DF4. Our later experimental results show that cracks appear after 28 days' curing on the surface of DF3 and DF4. Cracks appear as a result of the expansion of AFt. The extent of AFt-related expansion depends on the curing style. The expansion increases with increasing amounts of water absorbed from the environment while AFt is formed [19]. The curing style of sprinkling can supply more water than moist curing. As a result, DF1 and DF2 have the greater extent of expansion which results in the appearance of cracks.

3.2. Fineness of CPFA on the self-cementitious strength

The compressive strengths of ground DF4 are shown in Fig. 2. The self-cementitious strength increases with the decrease of 45 μm sieve residue value, which is consistent with pulverized fly ash whose pozzolanic reactivity often increases with a decrease of particle size [20]. The strength increases a little if the 45 μm sieve residue value is over 10%,

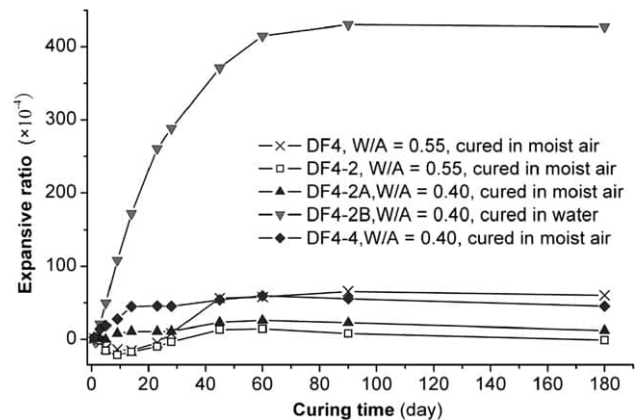


Fig. 3. Expansive ratio.

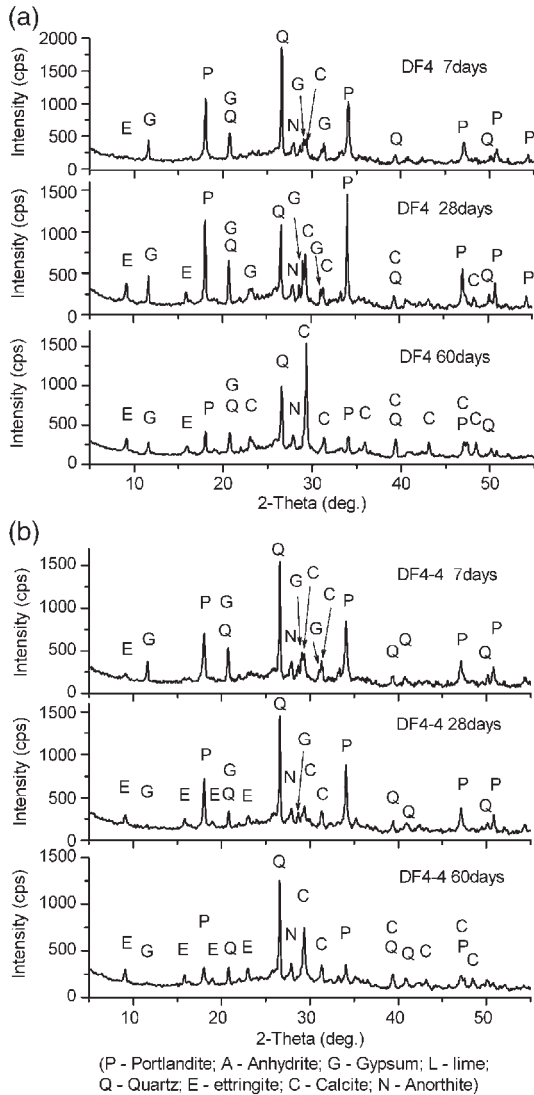


Fig. 4. (a) XRD patterns of hydrated DF4 at 60 days. (b) XRD patterns of hydrated DF4-4 at 60 days.

while it increases significantly when it is below 10%. For example, when the 45 μm sieve residue value is 2.7%, the compressive strengths at 7 days, 28 days and 60 days are 3.4 MPa, 14.7 MPa and 16.9 MPa respectively, which are 9.8, 19.9 and 15.5 times, respectively, as great as that of DF4. The increase of fineness is beneficial to the hydration of anhydrite [21] and lime, and the dissolution of the active silica and active alumina in the CPFA, which quickens the generation of AFt, C–S–H, and gypsum. As a result, the self-cementitious strength increases.

3.3. Expansion

Expansion often happens with the curing of CFBC fly ash–water system, but it is effected by the curing style [10,11,22]. The expansive ratios of CPFA are shown in Fig. 3, some samples are shrinking in the early curing days but all are expansive in later days. Firstly, DF4, DF4-2 and DF4-2A are expansive, and then shrinking, but expansive in the end, while

DF4-2B and DF4-4 are expansive all along. During early curing, the hydration of *f*-CaO and anhydrite, and absorption of water by calcined clay minerals will reduce the water content between particles in the hardened paste; as a result, the capillary force increases [23], then the shrinkage happens, that is the reason of shrinkage of DF4, DF4-2 and DF4-2A. But it is not shown in all the samples because expansion happens simultaneously due to the generation of AFt and gypsum.

DF4 has a comparable shrinkage ratio with DF4-2, but it quickly expands after 14 days, especially between 14 days and 45 days. DF4 has the lowest specific surface, as a result, the AFt generates slowly for slow dissolution of active alumina in DF4. In CFBC ash, lime particles are often covered by anhydrite shell [9] and its slaking rate is lower than that of industrial lime. The lower hydration rate of lime in CFBC fly ash is due to the slow transport of H₂O molecules through the sulphate shell in an earlier stage and through Ca(OH)₂ layers in a later stage [24]. Delayed hydration of lime often damages the hardened paste.

Grinding of ash can accelerate the generation of AFt due to the dissolution of active alumina in ash that is quickened. As a result, DF4-4 has a higher expansive ratio than DF4-2. We found that some cracks can be found on the surface of DF4 and

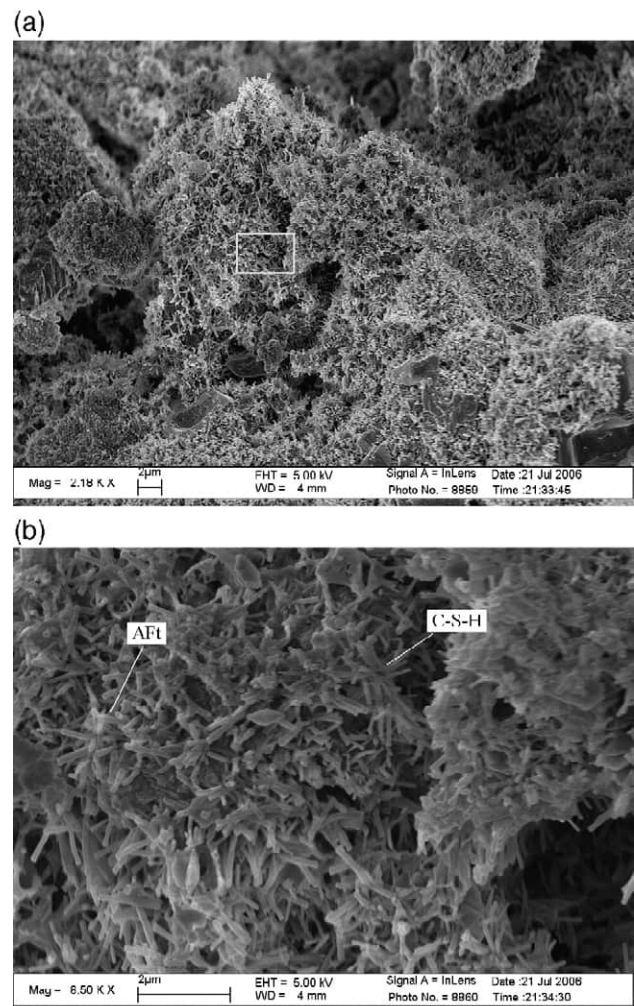


Fig. 5. (a) SEM micrograph of hydrated DF4 at 60 days. (b) Magnification of (a).

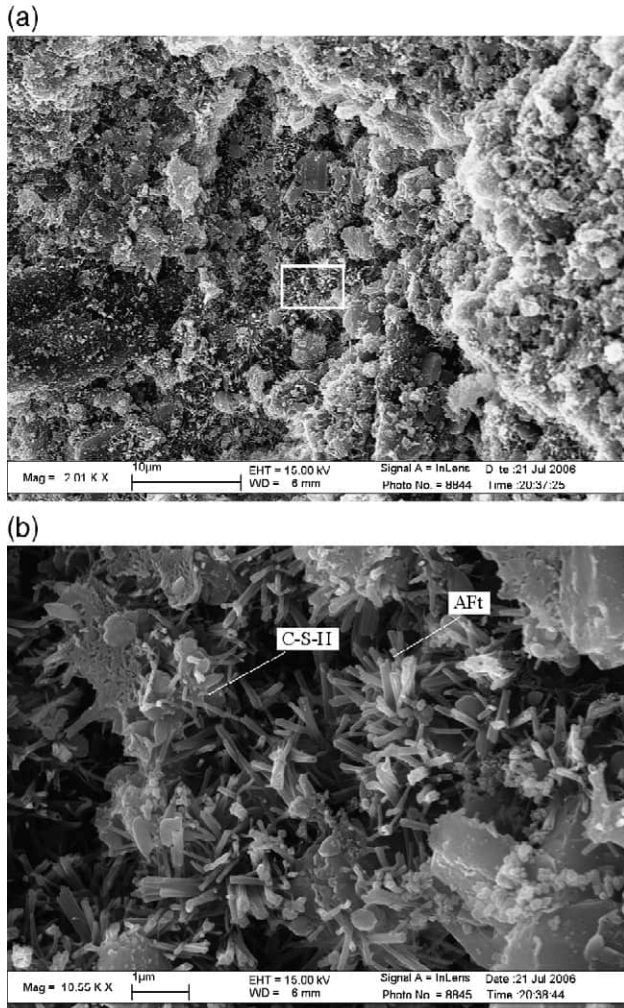


Fig. 6. (a) SEM micrograph of hydrated DF4-4 at 60 days. (b) Magnification of (a).

distortion happens after 28 days while other ground samples cured in moist air are undeformed. This is due to its fast increase of expansion between 14 days and 45 days and its lower mechanical strength. From this result, we know that grinding is beneficial to improve the volume stability property of hardened CPFA paste. This is because that grinding of the CFBC fly ash can destroy the sulphate shell which is generated during SO_2 removal [9], and decrease the particle size, which quickens the hydration of anhydrite and lime.

DF4-2 has a lower expansive ratio than that of DF4-2A because of its higher W/A (shown in Fig. 3). A higher W/A provides a higher porosity which will supply more space for AFt growth, and, as a result, the expansion is reduced [25].

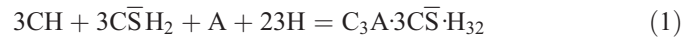
DF4-2B cured in water has the highest expansive ratio. Some cracks appear on its surface after 5 days of curing due to AFt

expansion, and the surfaces become loosened after 28 days for easy dissolution of gypsum [21]. Curing in water is beneficial to the expansion of AFt because it can absorb water easily from the environment [19]. Research of J. Iribarne et al. [11] also showed that the expansive ratio of CFBC fly ash paste cured in water was higher than that cured in a moist atmosphere.

At the age of 90 days, all samples' expansive ratios decrease except DF4 and DF4-2B. This may be the result of the carbonation of $\text{Ca}(\text{OH})_2$ since the intensity of calcite increases significantly and the intensity of portlandite decreases significantly at 60 days compared to that at 28 days (shown in Fig. 4).

3.4. Hydration products

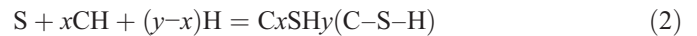
The hydration productions of DF4 and DF4-4 are investigated by XRD and SEM, and the results of XRD and SEM are shown in Figs. 4, 5 and 6, respectively. The summary of XRD is shown in Table 3. The results of XRD (Fig. 4) show that the main hydration products are portlandite, gypsum and AFt, which are consistent with the results of J. Iribarne et al. [11] and E.J. Anthony et al. [10]. The portlandite is attributed to the hydration of lime, while gypsum is attributed to the hydration of anhydrite, and AFt is due to the reaction (1).



Here A is the active alumina in CPFA. AFt is very important for the hardening and compressive strength development of CPFA–water system [11,18,25,26]. The expansive characteristics of the hardened CPFA paste also correlate with it [11].

The strong lime and anhydrite peaks can be found in DF4 (shown in Fig. 1), and all disappear before 7 days, while strong gypsum and portlandite peaks appear at the age of 7 days. A weak peak of AFt appears in both DF4 and DF4-4. However, portlandite, gypsum and AFt remain in hydrated DF4 and DF4-4 at the age of 28 days and 60 days. But the peaks of portlandite ($d=0.2628$ nm) and gypsum ($d=0.7601$ nm) become weak in both DF4 and DF4-4, in particular the peak of gypsum in DF4-4 becomes very feeble due to the generation of AFt.

At the age of 60 days, the ($d=0.2628$ nm) and ($d=0.4911$ nm) peaks of portlandite are feeble for the reactions (1), (2) and (3).

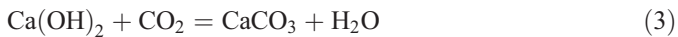


Here S is the active silica of CPFA. C_xSH_y is hydrated calcium silicate which is very important for the later strength development of CPFA–water system. The reaction (2) often happens in fly ash–lime–water system or pozzolanic material–lime system. But the results of J. Iribarne et al. [11] showed that the compositions of

Table 3
Hydrated phases identified in DF4 and DF4-4 (cps)

Materials	Portlandite			Gypsum			Ettringite		
	7 days	28 days	60 days	7 days	28 days	60 days	7 days	28 days	60 days
DF4	1245	1671	244	335	406	211	56	242	198
DF4-4	739	793	251	257	43	37	66	127	166

C–S–H generated from hydration of CFBC fly ash were various and part of Si was replaced by Al and S. The generation of AFt and C–S–H is the main reason for self-cementing of CPFA [18].



Reaction (3) explains the carbonization of portlandite, which results in the decrease of portlandite. The carbonization phenomenon is often found in CFBC fly ash–water system [10]. It can be confirmed by the stronger intensity of the peak ($d=0.3039$ nm) of calcite at 60 days than at 28 days, while the intensity of the peak ($d=0.2628$ nm) of portlandite decrease significantly.

The SEM micrographs are shown in Figs. 5 and 6. Many AFt crystals grow mixed with C–S–H; they are about 1 to 2 μm long and 0.1 to 0.2 μm wide, and can be found in DF4 and DF4-4 at the age of 60 days. It is difficult to identify C–S–H in the XRD patterns because of its poor crystal structure.

The hydration products of high-calcium fly ash, which often includes strätlingite (C_2ASH_8), ettringite ($\text{C}_3\text{A}\cdot 3\text{CS}\cdot \text{H}_{32}$), monosulphoaluminate or “monosulphate” ($\text{C}_3\text{A}\cdot 3\text{CS}\cdot \text{H}_{12}$), gypsum ($\text{CS}\cdot \text{H}_2$), and hydrogarnet (C_3AH_6) [7,8], are different from those of CPFA. It is due to their different mineral compositions.

4. Conclusion

- (1) CPFA is self-cementitious; its strength is affected by its fineness and chemical compositions, in particular its f -CaO and SO_3 content. The high f -CaO and SO_3 contents are beneficial to self-cementitious strength development. The self-cementitious strength increases with the decrease of its residue on 45 μm sieve.
- (2) CPFA hardened paste is expansive for the generation of AFt. A high W/A ratio results in a low expansive ratio, and grinding the ash is beneficial to the paste's expansive stability. The paste cured in water has the highest expansive ratio which has a detrimental effect on the hardened paste.
- (3) The main hydration products of CPFA are portlandite, gypsum, AFt and C–S–H; portlandite and gypsum are consumed gradually in the curing. Carbonation results in more calcite remaining in the hardened CPFA paste.

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