

Utilization of fly ash coming from a CFBC boiler co-firing coal and petroleum coke in Portland cement

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Abstract

Fly ash coming from a circulating fluidized bed combustion (CFBC) boiler co-firing coal and petroleum coke (CFBC fly ash) is very different from coal ash from traditional pulverized fuel firing due to many differences in their combustion processes, and thus they have different effects on the properties of Portland cement. The influences of CFBC fly ash on the strength, setting time, volume stability, water requirement for normal consistency, and hydration products of Portland cement were investigated. The results showed that CFBC fly ash had a little effect on the strength of the Portland cement when its content was below 20%, but the strength decreased significantly if the ash content was over 20%. The water requirement for normal consistency of cement increased from 1.8% to 3.2% (absolute increment value) with an addition of 10% CFBC fly ash; and the free lime (f-CaO) content of CFBC fly ash affected the value of increasing. The setting time decreased with an increase of CFBC fly ash content. The volume stability of the cement was qualified even when the content of SO₃ and f-CaO reached 4.48% and 3.0% in cement, respectively. The main hydration productions of cement with CFBC fly ash were C–S–H (hydrated calcium silicate), AFt (ettringite), and portlandite.

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1. Introduction

Circulating fluidized bed combustion (CFBC) is a clean technology for coal burning which has the advantages of adapting to a large variety of fuel, high combustion efficiency, lower NO_x emissions, and stable operation over a large range of load regulation. CFBC may be done either under atmospheric pressure (atmospheric fluidized bed combustion, AFBC) or at elevated pressure (pressurized fluidized bed combustion, PFBC) [1–3]. SO₂ can be captured by limestone added in situ during combustion. For increasing the SO₂ removal efficiency, the ratio of Ca/S is often increased to 2.0–2.5, so there are many unreacted CaO and desulfurized products CaSO₄ remaining in

AFBC ash [3], and many CaCO₃ and CaSO₄ remaining in PFBC ash [4].

CFBC ash is different from most typical coal combustion by-products for (1) high content of f-CaO and SO₃ (usually anhydrite) in AFBC ash, and high content of SO₃ (usually anhydrite) in PFBC ash; (2) few spherical particles can be seen in CFBC fly ash for the temperature in the CFBC boiler (often 800–900 °C) is lower than that in the pulverized coal-fired boiler (often 1300–1500 °C); (3) self-cementing; (4) vigorous reaction with water because of many f-CaO present in AFBC ash [4,5].

Fly ash is widely utilized in cement as admixture and its effects on the properties of Portland cement have been comprehensively investigated. Its addition in cement often decreases the strength of cement, increases the fluidity, and delays the setting time. However, there were only a few investigations of the use of CFBC ash in cement as an admixture [4,6–10], or as a setting retarder replacing gypsum [11,12]. There may be some harmful effects to cement

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or concrete, especially with regards to the property of volume stability [6,13], due to the high content of f-CaO and SO₃, but the report of Brnadštetr et al. [4] showed that f-CaO in CFBC ash caused no damage to cement because it is highly reactive. To avoid the harmful effects of expansion, many technologies were used, such as the technology of CERCHAR (Centre d'Études et de Recherches du Charbon) [14], and addition of additives [15]. Anhydrite present in the hydrating cement–CFBC fly ash mixture can be the source of “delayed ettringite formation” [16], and the SO₃ content in cement must match exactly with calcium aluminate or active alumina content [17]. Compressive strength of cement–CFBC ash depended on the mixing ratio of compounds and the composition of the binder, and the strength decreased with an increase of mixing water and increased with cement [10]. Cement with the ash had good strength development as it was cured. Its water requirement for normal consistency increased little, while the setting time increased with the increase of SO₃ content in cement [8]. In this research, the properties of Portland cement with fly ash coming from an industrial CFBC boiler co-firing coal and petroleum coke (CFBC fly ash) were investigated, with particular focus on the volume stability of the cement.

2. Materials and experimental methods

2.1. Materials

CFBC fly ashes were obtained from a 220 t/h Pyroflow CFBC boiler co-firing coal and high-sulphur petroleum coke, and using limestone as the SO₂ sorbent in Power Plant of Sinopec Jinling Petrochemical Corporation. The bituminous coal originated in Datong (Shanxi, China), and the petroleum coke was derived from the Mideast area. The ashes were collected from the ash-hoppers of the electrostatic precipitator of the CFBC boiler. There were two ashes used in this investigation, ash A and ash B, which were generated from co-combustion of coke with 30:70 (cal%) and 40:60 (cal%) blends of coal, respectively. The clinker was produced by Jiangnan-Onoda Cement Corporation. Table 1 showed the chemical compositions and physical properties of ash A, ash B and clinker. The mineralogical compositions of the two ashes were detected by X-ray diffraction (XRD).

2.2. Methods

2.2.1. Samples preparation

Clinker was cracked to less than 7 mm before grinding in a laboratory ball mill (Ø500 mm × 500 mm) for 25 min. The residue on a 0.08 mm sieve of the ground clinker was 3.4% and its Blaine special surface was 359 m²/kg. The contrast Portland cement was produced by grinding a mixture of 95.5% clinker and 4.5% gypsum. Its residue on a 0.08 mm sieve and the Blaine special surface were 4.0% and 366 m²/kg, respectively.

Table 1
Chemical compositions and physical properties of CFBC fly ashes and clinker

	Clinker	Ash A	Ash B	GB/T1596-2005 type C ash
<i>Chemical compositions (wt%)</i>				
L.O.I. ^a	1.40	18.64	15.78	≤8.0
SiO ₂	20.95	29.37	17.43	
Al ₂ O ₃	4.60	17.87	9.95	
Fe ₂ O ₃	3.75	2.32	1.19	
CaO	65.53	22.00	41.19	
MgO	1.07	1.51	1.13	
K ₂ O	0.81	0.70	0.45	
Na ₂ O	0.10	0.39	0.29	
f-CaO	0.21	7.55 ^b	14.67 ^b	≤4.0
		(4.1 ^c)	(7.2 ^c)	
SO ₃	0.73	4.21	10.13	≤3.5
Sum	98.94	97.01	97.54	
<i>Physical properties</i>				
Residue on 0.045 μm sieve (wt%)		16.4	7.2	
Blaine specific surface (m ² /kg)		467	378	
Specific gravity (g/cm ³)		2.38	2.56	
Strength activity index (%)		80.7	96.2	≥70

^a L.O.I: loss on ignition.

^b Determined by chemical method.

^c Subtract the content of Ca(OH)₂ from f-CaO content determined by chemical method.

Cement samples were prepared from CFBC fly ash, ground clinker and some gypsum mixed by hand for 2 min. The components of the cements were showed in Table 2. A little gypsum was used to increase the content of SO₃ to 2% in the cement if it was less than 2%.

2.2.2. Physical test

The strength test was done at the age of 3 days and 28 days according to Chinese standard GB/T17671-2001 (equivalent to ISO 679:1989) by using a 40 mm × 40 mm × 160 mm prism specimen with a sand to binder ratio of 3.0 and a water to binder ratio of 0.5. After the specimen was cured in the >90% R.H. (relative humidity) chamber at 20 ± 1 °C till 20–24 h, it was demolded and next cured in 20 ± 1 °C water for 3 days and 28 days.

The tests of water requirement for normal consistency and setting time were measured by a Vicat apparatus according to Chinese standard GB/T1346-2001 (equivalent to ISO9597:1989). Water requirement for normal consistency was defined as the water/cement–ash ratio when the metal bar penetrated the paste to a depth of 6 ± 1 mm from the bottom of the mold. The time required for the needle to penetrate the paste to a depth of 4 ± 1 mm from the bottom of the mold was defined as the initial setting time. After the initial setting time test, the mold was turned over 180° and continued to cure at 20 ± 1 °C, and the time at which the needle penetrated into the paste ≤0.5 mm was defined as the final setting time. Volume stability was

Table 2
Components of cements with CFBC fly ash admixture (wt%)

Sample	Symbol	Clinker	Ash	Gypsum	SO ₃ content	f-CaO content
Contrast Portland cement	C0	95.5	0	4.5	2.34	0.2
CA (cement with ash A)	CA10	87.5	10.0	2.5	2.06	0.6
	CA20	77.8	20.0	2.2	2.19	1.0
	CA30	68.4	30.0	1.6	2.33	1.4
	CA40	58.9	40.0	1.1	2.50	1.8
CB (cement with ash B)	CB10	88.8	10.0	1.2	2.15	0.9
	CB20	80.0	20.0	0	2.62	1.6
	CB30	70.0	30.0	0	3.55	2.3
	CB40	60.0	40.0	0	4.48	3.0

measured by the Le Chatelier apparatus according to GB/T1346-2001. After curing in the >90% R.H. chamber at 20 ± 1 °C for 24 ± 2 h, the paste with the Le Chatelier mold was boiled for 180 ± 5 min, and then cooled to room temperature. The expansive length of the Le Chatelier needle was measured. The volume stability of the cement–ash was judged as qualified when the expansive value was less than 5 mm.

2.2.3. Chemical test

2.2.3.1. Chemical composition. The chemical composition of each sample was analyzed 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 L.O.I.-determined fly ash was weighed into a platinum crucible and thoroughly mixed with 7.7 g of flux (mixture of Li₂B₄O₇ and LiBO₂); 1 ml of 40 g/l LiBr solution was then added to the mixture to enhance the fluxing process. The mixture was next heated at 1100 °C for 13.5 min and then poured into a platinum cup to form a glasslike penny-shaped disk that was ready for XRF analysis.

The f-CaO content was determined by a glycerine–alcohol solution extraction method according to GB/T176-1996. In actuality, the content of f-CaO determined by this method was the total content of f-CaO and Ca(OH)₂. The Ca(OH)₂ content was determined by thermal gravimetric analysis–differential scanning calorimetry (TGA–DSC). Then the f-CaO content was determined by subtracting the Ca(OH)₂ content from the total content of f-CaO and Ca(OH)₂.

2.2.3.2. XRD and TGA–DSC. The paste (component was shown in Table 2) was prepared with a water to binder ratio of 0.5, and then the paste was mixed by hand for 5 min and put into a PVC bag. The bag was tied and then immersed in 20 ± 1 °C water till the test day. At the test day, the specimen was split and ground in an agate mortar with acetone till the entire sample passed through a 0.045 mm sieve. Then it was stored in a chamber for 24 h. After that the sample was filtered and dried at 60 °C for 6 h in CO₂-free air. The dried sample was ready for XRD test and TGA–DSC test.

The XRD pattern of the sample was analyzed at an X'TRA X-ray diffractometer with Cu K α radiation. Step scan was performed over the range of 5–60° (2 θ) with stepping interval of 0.02° and a count time of 0.24 s (scanning rate 5.00 deg/min, 40 kV, 40 mA).

The TGA–DSC pattern was analyzed by an NETZSCH STA 409 calorimeter at a heating rate of 10 °C/min up to 1000 °C in a N₂ atmosphere.

3. Results and discussion

3.1. Characterization of CFBC fly ash

Table 1 shows that neither of the two CFBC fly ashes (ash A and ash B) can meet the Chinese standard GB/T1596-2005 (for admixtures in cement) because L.O.I. (loss of ignition), SO₃ and f-CaO are all beyond the limits defined in the standard. The high L.O.I. is not all caused by unburned coal as CaCO₃ and Ca(OH)₂ also contribute to it. But CaCO₃ and Ca(OH)₂ should be differentiated from unburned coal because they do not normally have harmful effects on the properties of cement or concrete. But the L.O.I. of CFBC fly ashes was beyond the limit even if the L.O.I. generated by CaCO₃ and Ca(OH)₂ was removed from the total.

Major mineralogical compositions of the two CFBC fly ashes (shown in Fig. 1) are lime (CaO), anhydrite (CaSO₄), α -quartz (SiO₂) and portlandite (Ca(OH)₂); also a little calcite (CaCO₃) can be found in the ash. The presence of Ca(OH)₂ is due to the reaction of f-CaO with water vapor in the air during the CFBC fly ash storage, which is advantageous for its use in cement or concrete. There is a diffuse halo peak at 21–29°, especially in the XRD pattern of ash A, which attributes to dehydrated clay minerals.

3.2. Strength of Portland cement with CFBC fly ash

The strength of cement with traditional pulverized fly ash (low in f-CaO and SO₃) usually decreases with the increase of ash content in the cement–ash mixture due to the lower pozzolanic activity of the ash [18], which is usually considered to be inert in cement paste in the early age of curing. The variations in the strength of CA and CB are

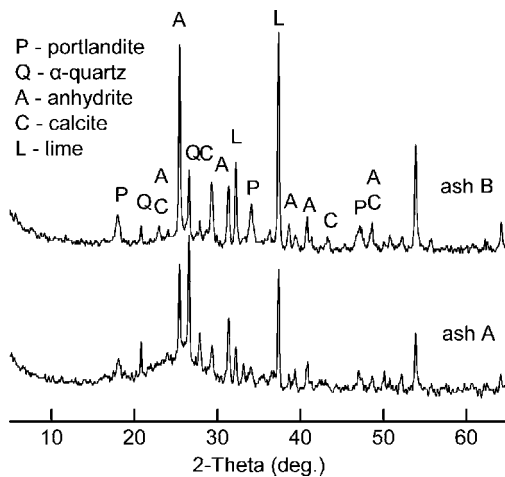


Fig. 1. XRD patterns of CFBC fly ashes.

shown in Table 3. The strength is found to decrease with the addition of ash A, but the strengths of mortars containing 10% and 20% ash A are comparable to that of C0 at 3 days. When ash B content is no more than 20%, the strength is higher than that of C0, and the strength decreases when it is over 20%. The comparable strength of cement with no more than 20% ash A or ash B is because the early hydration rate of the clinker is accelerated by the fly ash [19], and $\text{Ca}(\text{OH})_2$ [20]. Part of the water can be absorbed by the dehydrated clay minerals and f-CaO which decreases the water to binder ratio. A low water to binder ratio is advantageous for the strength of the cement. The self-cementitious properties of ash A and ash B are also beneficial for the strength development [21]. But at high ash content (over 20%), the decrease in strength is due to the reduction of the cement content in the mixture, and excessive $\text{Ca}(\text{OH})_2$ generated from the reaction of f-CaO with water which is disadvantageous for the mechanical properties of cement.

The strength of CB is higher than that of CA because ash B has higher content of f-CaO and CaSO_4 , both of which can activate the ash [22,23]. But the strength of B40 is lower than that of A40 (Table 3) because of the excess f-CaO and CaSO_4 in ash B (Table 2).

The SO_3 content of CA or CB is in the typical range except B30 and B40, but the retarder in this investigation is anhydrite or a mixture of anhydrite and gypsum. In the range of the optimum SO_3 addition (up to 3.5%), anhydrite shows behavior similar to that of gypsum in compressive strength development [24] and does not damage the cement.

According to GB/T1596-2005, the ratio of the compressive strength of Portland cement replaced by 30% ash to that of the contrast Portland cement is defined as the strength activity index of the ash. Here the strength activity indexes of ash A and ash B are 80.7% and 96.2%, respectively, which are all higher than 70%, the minimum limitation for use in cement as an active admixture regulated by the standard.

The strength of cement with CFBC fly ash can increase gradually with time of hardening [10]. The strength development of A30 and B20 are shown in Fig. 2. The compressive strengths of A30 and B20 increase gradually with the curing time up to 350 days. At 3 days, both of the compressive strengths of A30 and B20 are lower than that of C0, but the strength of B20 is higher than that of C0 at 28 days. The strength of A30 is lower due to more ash in it, but its strength increasing rate is higher than C0's, which is beneficial for its future strength development. This is due to the reaction of $\text{Ca}(\text{OH})_2$ with active silica and active alumina in CFBC fly ash which produces much C-S-H (hydrated calcium silicate; the main carrier of strength in hardened cement) and hydrated calcium aluminate. The strength development characteristics of cement with CFBC fly ash is very similar to those of cement with traditional pulverized fly ash [18,25], that is early age strength of cement with ash (especially before 28 days) is usually lower than that of contrast Portland cement, but its late age strength can increase significantly and even exceed that of the contrast Portland cement because of the pozzolanic reaction [25].

3.3. Water requirement for normal consistency

The variations in water requirements for normal consistency with a different percentage of ash A and ash B are presented in Fig. 3. The water requirement for normal

Table 3
Influence of CFBC fly ash on the strength of Portland cement

Symbol	Content of ash (%)	Flexible strength (MPa)		Compressive strength (MPa)		Volume stability
		3 days	28 days	3 days	28 days	
C0	0	6.1	9.0	27.6	49.1	Qualified
CA10	10	5.7	8.9	25.6	45.4	Qualified
CA20	20	5.6	8.2	25.3	42.6	Qualified
CA30	30	4.4	7.6	20.8	38.1	Qualified
CA40	40	3.4	6.4	16.6	30.8	Qualified
CB10	10	6.3	9.2	30.0	47.0	Qualified
CB20	20	5.7	8.8	30.9	49.5	Qualified
CB30	30	3.7	7.1	21.3	45.4	Qualified
CB40	40	2.8	4.6	15.2	26.7	Qualified

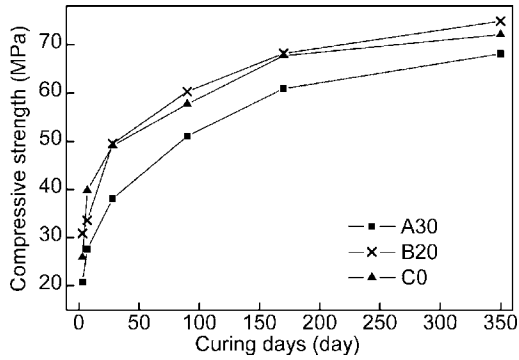


Fig. 2. Strength development of the controlled cement and cements with CFBC fly ash.

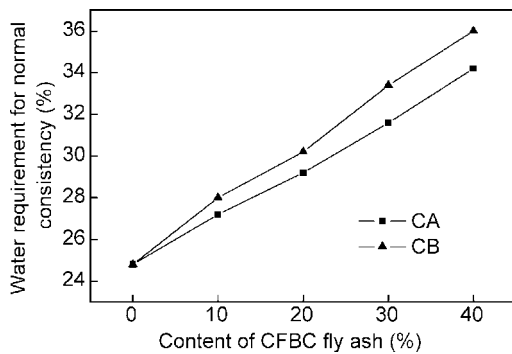


Fig. 3. Influence of CFBC fly ash on the water requirement for normal consistency of Portland cement.

consistency increases 2–2.6% with an increase of 10% ash A, or 1.8–3.2% for 10% ash B. f-CaO hydration demands about 32% water and releases much heat which enhances the temperature of the paste and quickens the hydration reaction rate of cement. As a result, the water requirement increased with an increase of f-CaO content during the early stage of hydration. This is the reason that CB has a higher water requirement for normal consistency. Specific gravity of ash A or ash B is less than that of cement which results in larger volume of ash A or ash B compared to the volume of cement replaced by mass. As a result, the overall volume is increased and more water is needed to form a paste of the same consistency. The dehydrated clay mineral in CFBC fly ash also results in a higher water requirement for normal consistency because of its large surface area generated during calcination.

3.4. Setting time

The setting time of cement with traditional pulverized fly ash often increases with an increase of ash content [26], and decreases with an increase of f-CaO content [20,27], or with the use of anhydrite, instead of gypsum, as the retarder [24]. The results are shown in Table 4. The trend shows a decrease in initial setting time and final setting time with the increase of ash A content from 0% to 40% or ash B con-

Table 4
Influence of CFBC fly ash on setting time of Portland cement

Content of ash (wt%)	Setting time of CA (min)		Setting time of CB (min)	
	Initial	Final	Initial	Final
0 ^a	132	200	132	200
10	165	242	112	189
20	151	226	95	172
30	128	190	90	158
40	116	191	98	163

^a The contrast Portland cement, C0.

tent from 10% to 40%. This trend is different from cement with traditional pulverized fly ash where f-CaO decreases the setting time [20]. CB has a shorter setting time than CA because of its higher f-CaO content. The setting time decreases slowly when ash A or ash B content is more than 30% because more ash reduces the cement content in the mixture. As a result, the hydration process slows down causing the setting time to increase. The setting time of CA10 and CA20 are longer than that of C0 for only 0.6% and 1.2% f-CaO in them, so the action of f-CaO in decreasing the setting time is weak. Cement replacement by ash also delays the setting time.

3.5. Volume stability

Volume stability of cements with ash A or ash B varied from 10% to 40% are qualified (Table 3). Two factors may damage the volume stability of cement with CFBC fly ash, which are the f-CaO and the excessive SO₃ (usually anhydrite), both of which may cause later expansion in the hardened mortar.

In this investigation, SO₃ content in all of the cements with CFBC fly ash are lower than 3.5% specified by GB/T1344-1999 except B30 and B40. Anhydrite used in the cement as a retarder is not harmful to the volume stability of cement. Even the volume stability of B40 is qualified whose SO₃ content is up to 4.48%.

Another method of detecting the instability caused by excessive CaSO₄ introduced in Ref. [25] was used. A paste biscuit, made to a normal consistency with water, having a diameter of 70–80 mm and a central height of about 10 mm above a glass flat, was cured in the >90% R.H. chamber at 20 ± 1 °C for 24 ± 2 h. After the paste biscuit was removed from the glass flat, it was immersed in 20 ± 1 °C water for 28 days. Volume stability was judged as qualified or unqualified according to the distortion of the paste biscuit. The results showed that the volume stability of all specimens were qualified.

f-CaO in the CFBC ash is usually harmless as it formed at low temperature (about 800–900 °C) is highly reactive and can hydrate to Ca(OH)₂ in several hours [4]. During the SO₂-removing process, the lime is encapsulated by CaSO₄. As a result, the hydration of lime is hindered because the water cannot go through the CaSO₄ layer

easily. However, in water, the CaSO_4 can be dissolved, and its solubility will increase about 50% in cement paste for the generation of AFt which is generated by the reaction of CaSO_4 , C_3A ($\text{Ca}_3\text{Al}_2\text{O}_6$) and $\text{Ca}(\text{OH})_2$ [28]. Under typical conditions, where the SO_3 content in cement is between 1.5% and 2.5%, the CaSO_4 usually can be consumed in 24 h [25]. Part of the CaSO_4 layer can be dissolved in several hours, and then the water can go through it and react with the lime. Solid phase volume will increase about 98% when the f-CaO reacts with water, which induces the CaSO_4 layer to break, and the reaction of f-CaO and water proceed again [29,30]. As shown in Fig. 5, no f-CaO peaks appear in the XRD patterns at the age of 1 day. The investigation of exothermic nature of AFBC ash by Brandsteter [4] showed that the maximal temperature appeared at 4–5 h after the addition of water. It meant that the hydration of the lime in AFBC ash was hindered but could quickly slake in several hours. Hydration of f-CaO in CFBC fly ash is more effective than that of high-calcium fly ash because the f-CaO in CFBC fly ash is not dead burned due to its lower formed temperature. Shi et al. [31] have reported that the volume stability of cement with high-calcium fly ash (generated in pulverized coal furnace) was unqualified if the content of f-CaO gotten from the ash exceeded 0.9%. In contrast, the volume stability in this investigation was qualified even when the f-CaO content of cement was up to 3% (Table 2). This is a result of the f-CaO in high-calcium fly ash which is generated at 1300–1500 °C and which is usually dead burned.

Even though the f-CaO in CFBC ash does not have deleterious effects on cement or concrete, the high content of f-CaO in cement or concrete is not good because its rapid heat release will increase the temperature of the mortar quickly and result in a cracked appearance.

3.6. Hydration products

The hydration products of hardened cement pastes with CFBC fly ash were detected by XRD and TGA–DSC (Figs. 4a,4b, and Fig. 5). The XRD patterns show that main hydration products in C0 and B20 are AFt, and portlandite (Fig. 4b). AFt can be found in C0 and B20 at the age of 28 days. In Fig. 5, the endothermic peak between 70 °C and 130 °C is due to the dehydration of AFt and C–S–H, the 400–430 °C peak is due to the dehydration of $\text{Ca}(\text{OH})_2$, and the 650–680 °C peak is due to decarbonation of CaCO_3 [32,33]. At the age of 1 day (Fig. 4a), all lime peaks disappear due to its quickly slaking. And strong $\text{Ca}(\text{OH})_2$ peaks can be found in C0 at 1 day for hydration of C_3S (tricalcium silicate), while lime slaking partly contribute to it in B20. C–S–H cannot be found in XRD patterns because of its poorly crystalline structure. C–S–H is produced mainly by the hydration reaction of C_3S and C_2S (dicalcium silicate) in C0, and partly by the reaction of $\text{Ca}(\text{OH})_2$ with the active silica in the CFBC fly ash in B20. The later reaction is the main reason for the activity of the CFBC fly ash. Addition of pulverized fly ash (low

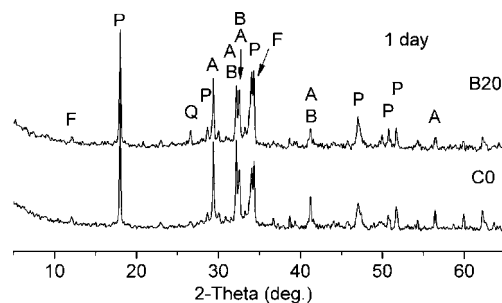


Fig. 4a. XRD patterns of hydrated cements.

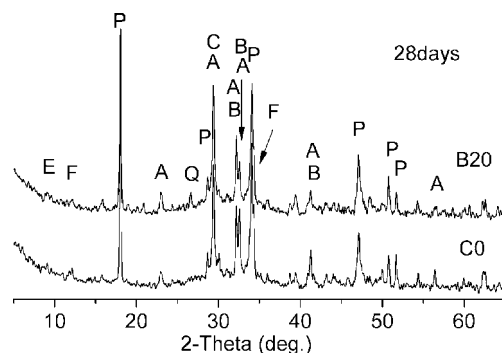


Fig. 4b. XRD patterns of hydrated cements. A – alite; B – belite; P – portlandite; C – calcite; Q – α -quartz; E – ettringite; F – ferrite.

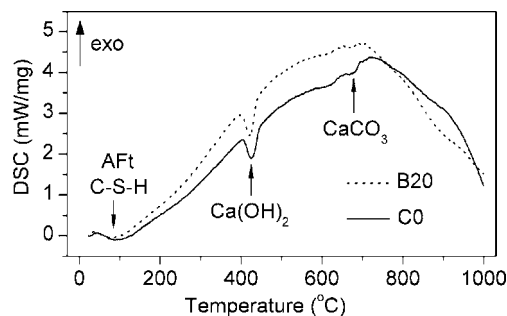


Fig. 5. DSC patterns of hydrated cements.

in CaO) to cement will decrease the $\text{Ca}(\text{OH})_2$ content in hardened Portland-ash cement mortar owing to the reaction [18]. This is different from CFBC fly ash because the excess f-CaO in CFBC fly ash will compensate for the consumption of $\text{Ca}(\text{OH})_2$.

4. Conclusion

The CFBC fly ash is suitable for cement as admixture as a result of its high activity in this investigation, and cement with the ash has a comparable strength with the contrast Portland cement when the ash is no more than 20% of the cement–ash mixture. The CFBC fly ash has no damaging effects on the volume stability of the cement for high slaking rate of f-CaO as demonstrated by the X-ray diffraction and volume stability test. The setting time of cement

decreases with an increase in CFBC fly ash content, and cement with high f-CaO content ash has a shorter setting time. Addition of 10% CFBC fly ash can increase the water requirement for normal consistency of Portland cement about 1.8–3.2%, and cement with a high content of f-CaO ash has a higher water requirement for normal consistency. How to reduce the water demand may be very important for its use in cement. The results of XRD and DSC show that the main hydration products of cement with CFBC fly ash are C–S–H, portlandite and AFt.

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