Carbonation of Circulating Fluidized Bed Boiler Fly Ash Using Carbonate Liquids

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ABSTRACT

In this study, unstable CaO was converted into a stable Ca compound by using carbonation in a circulating fluidized bed boiler of fly ash to confirm material usability as cement admixture; also undertaken was carbonation test and mortar to examine chemical and physical change by measuring absorption rate and compressive strength. To investigate the chemical properties of circulating fluidized bed boiler fly ash, XRD and TG-DTA were used to determine how the properties of the reaction product change quantitatively during carbonation. In order to stabilize CaO, carbonation of CaO is considered to be the most desirable process. This is because CaCO₃, which is a Ca compound, was produced by carbonate reaction of unstable CaO, and decrease of the absorption rate and improvement of the compressive strength were observed when the carbonated fly ash was replaced with cement.

Key words : Fluidized bed boiler fly ash, Calcium oxide, Free-CaO, Carbonation, Calcium carbonate

1. Introduction

Currently, about 8 million tons of coal ash waste is discharged from thermoelectric power plants in Korea.¹⁾ The most frequently used methods of combusting coal to produce energy at thermoelectric power plants are pulverized coal firing and circulating fluidized bed combustion. Due to the advantages of circulating fluidized bed combustion in comparison with pulverized coal firing, including a relatively low combustion temperature (800 to 900°C) to suppress the generation of nitrogen oxides (NO_x) and the immediate desulfurization of limestone in the furnace, the number of thermoelectric power plants employing circulating fluidized bed combustion is gradually increasing.²⁾ As a result, fluidized bed fly ash generated from circulating fluidized bed combustion boilers is also increasing. The general fly ash generated from pulverized coal firing is often used as a mixing material for cement and concrete, because general fly ash mixed with cement and concrete by Pozzolanic reaction provides advantages such as increased long-term strength, decreased drying contraction, and excellent chemical resistance.³⁾ However, the application of fly ash from a fluidized bed as a mixing material for cement and concrete is limited because such fly ash may cause an early drastic

heat release, cracks, and reduced fluidity due to free CaO resulting from the CaO and SO₃ component remaining after the desulfurization. Therefore, to mix fly ash from a fluidized bed boiler with ordinary Portland cement (OPC), pure CaO needs to be stabilized. Generally, the carbonation of pure CaO involves the reactions of Equation (1) and (3) occurring consecutively along with mixing with an aqueous carbonate solution or the production of carbonate by the reaction of Equation (3):

When Na₂CO₃ or K₂CO₃, used in the present study, is involved, 1 M of CaO requires 1 M of CO₂, as shown in Equation (4). When Na₂CO₃ or K₂CO₃ is dissolved in water, as in Equation (5), as one mol of CO₂ is dissociated (I), NaOH and NaHCO₃ are dissolved in water. When the reaction further progresses, CaO reacts with CO₂ or NaHCO₃ dissolved in water to produce CaCO₃, as shown in Equation (6). According to the theoretical stoichiometry, complete carbonation of one mol CaO requires one mol of Na₂CO₃ or K₂CO₃.

Therefore, in the present study, the optimal conditions of

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carbonation (the kinds and concentrations of aqueous carbonate solution, the reaction time, and the free CaO content) were established for easy carbonation through simple chemical reactions to convert CaO existing in large amounts in fly ash from circulating fluidized bed boilers to chemically stable Ca compounds, such as CaCO_3 and Ca(OH)_2 , through the carbonation of an aqueous carbonate solution. In addition, to measure the properties of the cement mortar, the circulating fluidized bed boiler fly ash to which the optimized carbonation was applied was mixed with cement mortar.

2. Experimental Procedure

2.1. Materials

Fly ash used in the present study was fluidized bed boiler fly ash (FBA), a side product from a circulating fluidized bed boiler at the “Y” thermoelectric power plant in Korea; ash has a specific surface area of $4070 \text{ cm}^2/\text{g}$ and a specific weight of 2.60. Table 1 shows the chemical composition of the FBA containing about 19% of free CaO. Fig. 1 shows the results of X-ray diffraction (XRD) analysis of the FBA. The XRD results show that the main components of the FBA were lime (CaO), anhydrite (CaSO_4), and quartz (SiO_2); the FBA also contained other compounds such as periclase (MgO), portlandite (Ca(OH)_2), and calcite (CaCO_3). Fig. 2 shows the thermogravimetric-differential thermal analysis (TG-DTA) results for the FBA, indicating that the endothermal peaks and the weight decreased at 100, 450 and 750°C . The decrease of the weight may have been due to the evaporation of free water at 100°C , the degradation of Ca(OH)_2 to CaO and H_2O at 450°C , and the degradation of CaCO_3 to CaO and CO_2 at 750°C . An aqueous carbonate solution needed for oxidation was prepared using reagent grade Na_2CO_3 and K_2CO_3 . The cement for the analysis of the properties used in the present study was an OPC produced by

Fig. 1. XRD pattern of FBA.

Table 1. Chemical Composition of a General FBA

CaO (free-CaO)	SiO_2	Al_2O_3	Fe_2O_3	K_2O	TiO_2	MgO	SO_3	Ig. loss
30.7 (19.4)	36.2	16.8	5.2	1.5	0.7	4.1	0.9	3.87

Fig. 2. TG-DTA results of FBA.

the “S” company. The fine aggregate used in the present study was Jumunjin standard sand.

2.2. Methods

2.2.1. Experiment for determining reaction mixing water ratio

FBA, containing a large amount of CaO and CaSO_4 , is known to show a high initial reactivity when mixed with water.⁴ The high reactivity stems from free CaO contained in the FBA; the free CaO is the main target of carbonation. By considering the reaction time, the rate of reaction of the reactants FBA and purified distilled water was investigated to determine the mixing water ratio. In the experiment, mixing was performed for three minutes using a vibration mortar mixer; times of mixing were 1 min and 30 sec at Rate 1 and 1 min and 30 sec at Rate 2. The mixture was kept in air for 1 h to verify the initial reactivity, and then dried in a dryer at 105°C for 24 h. After drying, the weight was measured again to verify the maximum ratio of the mixing water that may react with FBA. The rate of reaction of the reactants in the experiment was obtained by calculating the difference between the dried sample weight (W_{105}) and the initial sample weight (W_s), which was then divided by the initial sample weight (W_s) and multiplied by 100, as shown in the following Equation (7):

$$\text{Rate of reactants (\%)} = \frac{W_{105} - W_s}{W_s} \times 100 \quad (7)$$

where W_s = the initial sample weight and W_{105} = the dried sample weight (105°C , 24 h).

Table 2 shows the reaction mixing water ratio used in the experiment.

2.2.2. Carbonation of FBA

The effective components from which CO_2 may be

Table 2. Mixing Proportions of Reaction Mixed Water

Type	Mixing ratio (wt.%)		Reaction time (h)
	FBA	Water (D.I)	
FBA-0.1	100	10	1
FBA-0.2		20	
FBA-0.3		30	
FBA-0.4		40	
FBA-0.5		50	

absorbed and captured chemically were alkaline salts such as CaO, MgO, Na₂O, and K₂O; CO₂ can be captured from these reactants after they are converted to hydrate salts in an aqueous solution.⁵⁾ The unstable CaO contained in FBA at a high ratio was converted to stable Ca compounds (CaCO₃ or Ca(OH)₂) through the binding with CO₂ that had been dissociated from an aqueous carbonate solution. In the experiment, the production of Ca compounds and their yields were investigated by varying the experimental variables, including the kind of aqueous carbonate solution (Na₂CO₃, K₂CO₃ solution), the molar concentration (0, 0.5, 1.0, 2.0, and 3.0 mol/L), and the reaction time (0, 1, 2, and 4 hr). Table 3 shows the carbonation conditions and the vari-

ables. Table 4 shows the mixing ratios for the carbonation.

2.2.3. Analysis of properties

Drying and sieving were performed to analyze the products of the carbonation. In the analysis of the properties, for a qualitative analysis of the crystal structure of the minerals produced by the carbonation, XRD analysis was performed at 2θ values in a range of 5° to 80° at a heating rate of 4°/min. The XRD – Rietveld method was applied to qualitatively identify the produced Ca compounds. In addition, to quantify the Ca compounds (Ca(OH)₂, CaCO₃) produced by the reaction of FBA with an aqueous solution, and to measure the thermal properties, TG-DTA was performed by increasing the temperature at a rate of 10°C/min up to 1100°C while injecting N₂ gas, an inert gas, at a constant rate of 50 ml/min.

2.2.4. Measurement of water absorption of FBA-mixed mortar

Generally, free CaO stabilized using water is converted to Ca(OH)₂. Application of Ca(OH)₂ to OPC mortar increases the water absorption of the mortar and decreases the compressive strength. Therefore, to investigate the effect of carbonated FBA (C-FBA) on the OPC mortar, the water

Table 3. Experimental Method

Type		Evaluation items	Leveis
Carbonate reaction of Fly ash	Carbonate aqueous solution	Na ₂ CO ₃ , K ₂ CO ₃	2
	Mixed water ratio (%)	30	1
	Molarity (mol/L)	0.5, 1.0, 2.0, 3.0	4
	Reaction time (hr)	1, 2, 4	3
	Characterization	XRD-(Rietveld), TG-DTA	2
Mortar test of Fly ash	W/B (%)	0.5	
	Binder	Cement	OPC
		Fly Ash	FBA, *C-FBA
	FBA (%)	0, 10	
	Check items	Compressive strength	Water absorption

*C-FBA : Carbonated FBA

Table 4. Mixing Proportions of Carbonation

Type	Mixing ratio (wt.%)								
	FBA	Na ₂ CO ₃ solution				K ₂ CO ₃ solution			
		0.5 M	1.0 M	2.0 M	3.0 M	0.5 M	1.0 M	2.0 M	3.0 M
FBA-N0.5	100	0.3							
FBA-N1.0			0.3						
FBA-N2.0				0.3					
FBA-N3.0					0.3				
FBA-K0.5						0.3			
FBA-K1.0							0.3		
FBA-K2.0								0.3	
FBA-K3.0									0.3

Table 5. Mixing Proportions of Mortar

Type	Binder (wt.%)			W/B	S/B
	OPC	FBA	C-FBA		
OPC	100				
FBA 10%	90	10		0.5	3.0
C-FBA 10%	90		10		

absorption of the mortar was measured in the experiment according to the KS F 4419 method. Rectangular column-shaped specimens ($40 \times 40 \times 160$ mm) were prepared according to KS L ISO 679 and dried at 105°C for 24 h; specimens were then cooled at room temperature for 4 h. The cooled specimens were weighed and then immersed in water for 24 h. Then, after removing the water on the surface, the specimens were weighed to measure the water absorption depending on the mixing ratio. The water absorption was calculated by the following equation:

$$\text{Water Absorption (\%)} = \frac{W_{sd} - W_d}{W_d} \quad (8)$$

where W_d denotes the weight of a cooled specimen and W_{sd} the weight of a specimen after removing the water on the surface.

2.2.5. Measurement of compressive strength of FBA-mixed mortar

In order to examine the feasibility of using carbonated FBA (CFBA) as an OPC substitution material, part of the OPC was replaced by CFBA. Specimens were prepared according to the cement compressive strength test method of KS L ISO 679. The specimens were de-molded after 24 h. The prepared specimens underwent water curing at $20 \pm 2^\circ\text{C}$, and the compressive strength of the specimens was measured at the material ages of 3, 7, and 28 days. Table 5 shows the mixing ratios of the specimens prepared to measure the physical properties.

3. Results and Discussion

3.1. Determination of reaction mixing water ratio

An experiment was performed to determine the reaction mixing water ratio for FBA. The experiment was performed to measure the quantity of water lost by the evaporation of the mixing water due to the drastic exothermic reaction during the mixing of the free CaO contained in the FBA with water, as well as the quantity of water needed for stabilization of the free CaO. Fig. 3(a) is a plot showing the rate of reaction of the reactants depending on the quantity of the reaction mixing water. As the ratio of the reaction mixing water increased, the rate of reactants increased and then remained at a constant value after the ratio of the reaction mixing water was increased to over 30%. Since the heat of reaction of the CaO decreased as the ratio of the reaction mixing water increased, the rate of the hydration reactants remained in a range of 4.5% to 4.7% without large variation. Fig. 3(b) is a plot showing the XRD results obtained by performing the carbonation of FBA by varying the reaction mixing water ratio between 30% and 50% using only water. Even when the ratio of the reaction mixing water increased, the intensity of the CaCO_3 peak at a 2θ value of 29.3° did not change. Thus, in the present study, the optimal ratio of the reaction mixing water was determined to be 30% for the carbonation of FBA. However, since the ratio was determined on the basis of the amount of free CaO contained in the FBA used in the present study, the optimal ratio of the reaction mixing water may be different if the amount of the free CaO is different in other samples.

3.2. Effect of reaction time on carbonation

Figure 4 shows the XRD results depending on the reaction time after the initiation of the carbonation of FBA at a ratio of reaction mixing water of 30% when 0.5 M of Na_2CO_3 and K_2CO_3 aqueous solutions were used. The XRD plot for the FBA that underwent a reaction with the 0.5 M Na_2CO_3 aqueous solution shows that the intensity of the CaCO_3 peak increased but that of the $\text{Ca}(\text{OH})_2$ peak decreased as

Fig. 3. Semi reactant rate of water according to the reaction of a mixed water and XRD patterns of FBA showing W/F ratio.

Fig. 4. Change of XRD peak height at 2 theta 29.3° and XRD patterns of carbonated FBA with different reaction times.

the reaction time increased. The XRD plot for the FBA that underwent a reaction with the 0.5 M K_2CO_3 aqueous solution also shows that the intensity of the CaCO_3 peak increased as the reaction time increased, with the highest intensity found at the reaction time of 4 h. Fig. 4 shows the intensity of the CaCO_3 peak at a 2θ value of 29.3° depending on the aqueous carbonate solution. The intensity of the peak increased as the reaction time increased. The effect of the reaction time on the carbonation was not significant after 1 h. Thus, the optimal reaction time was considered to be 1 h. On the other hand, in the FAB-N0.5 specimen with the lowest K_2CO_3 concentration, calcite was the most produced material, indicating that more calcite may be produced at a low concentration than at a high concentration because the reaction with free CaO may sufficiently occur at low concentration. Additional kinetic analysis may be required to determine the concentration and the reaction time.

3.3. Effect of molar concentration on carbonation

Figure 5 shows the results of XRD at a 2θ value of 28°, where the main peaks of the product pattern are identified. For the carbonation of FBA performed with the aqueous carbonate solutions, the intensity of the CaO and Ca(OH)_2

peaks decreased but that of the CaCO_3 increased as the molar concentration of the aqueous solution increased. Fig. 6 shows the yields of the Ca compounds depending on the kind of aqueous carbonate solution; results were determined by XRD – Rietveld method; the patterns of the yields were similar. In the case of the aqueous solution of Na_2CO_3 , the CaCO_3 yield was highest at 27.1 wt% when the molar concentration of the aqueous Na_2CO_3 solution was 0.5 M; the yield decreased as the molar concentration increased. In the case of the aqueous solution of K_2CO_3 , the CaCO_3 yield increased as the molar concentration increased, and the highest yield of 27.8 wt% was found at 3 M. Fig. 7 shows the qualitative verification of the production of CaCO_3 on the basis of the weight decrease of the carbonated FBA, as analyzed by TG-DTA. The total ratio of the weight decrease was 18.8% when the aqueous K_2CO_3 solution was used; this decrease was about 0.4% higher than the case in which the aqueous Na_2CO_3 solution was used (18.4%). In the decarbonation of CaCO_3 , the weight changes due to thermal degradation occurring in a temperature range of 700 to 800°C, as the CaCO_3 is degraded to CaO and CO_2 . Thus, a high weight decrease ratio means the production of a large amount of CaCO_3 . The ratio of the weight decrease was

Fig. 5. XRD patterns according to molarity of carbonate aqueous solution.

Fig. 6. Comparison of amount of Ca compounds in each carbonate aqueous solution.

Fig. 7. TG curves of carbonated FBA with different solution and molarity.

8.65% when a 3 M aqueous K_2CO_3 solution was used and 7.27% when a 3 M aqueous Na_2CO_3 solution was used, indicating that the ratio of the weight decrease was 1.38% higher when a 3 M aqueous K_2CO_3 solution was used. This indicates that more CaCO_3 was produced by the carbonation performed using a 3 M aqueous K_2CO_3 solution than by the carbonation using a 3 M aqueous Na_2CO_3 solution. This was because K^+ ions, having a weaker electrostatic attractive force and a higher ionization energy, were dissociated more in the aqueous solution and provided a condition that was more advantageous for the production of CaCO_3 , because free CaO may undergo a reaction more easily in the presence of more dissociated carbonate ions or at a higher molar concentration of a CO_2 carbonate solution.

3.4. Effect of Na^+ and K^+ ions on Ca compounds

Figure 8 shows the XRD results of the C-FBA produced for 1 h of the reaction time and at a W/B ratio of 0.3 by using 3 M aqueous Na_2CO_3 and K_2CO_3 solutions. The overall peak patterns are similar, but the intensity of the CaCO_3 peak was higher when the carbonation was performed using a 3 M aqueous K_2CO_3 solution. The peak intensity of the C-FBA

at a 2θ value of 29.3° was higher when the carbonation was performed using an aqueous K_2CO_3 solution than it was when using an aqueous Na_2CO_3 solution. In addition, the peaks of mayenite, a calcium aluminate-based compound, and dihydrate gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), which is gypsum containing water molecules, were found around the 2θ values of 29.8° and 30.7° . These compounds may have been produced by the reaction with free CaO that did not participate in the CaCO_3 reaction. With regard to the effect of ions on the production of CaCO_3 , more CO_3^{2-} ions may have been separated from the aqueous K_2CO_3 solution, because K^+ ions, having a longer nucleus-electron distance and thus a weaker nucleus-electron attraction in comparison with Na^+ ions, may lose electrons more easily and may undergo reaction with CaO (or free CaO) more easily. Fig. 9 shows the TG results, indicating that more FBA weight was lost by the carbonation performed using a 3 M aqueous K_2CO_3 solution when CaCO_3 was degraded in a temperature range of 700 to 800°C . Fig. 8 compares the intensity of the CaCO_3 peaks at a 2θ value of 29.3° . The intensity of the CaCO_3 peak was higher in the carbonation performed using a 3 M aqueous K_2CO_3 solution. Therefore, for increasing the yield of CaCO_3 , a Ca-based

Fig. 8. XRD patterns of reactants determined by FBA-N3.0 and FBA-K3.0.

Fig. 9. TG curves of carbonated FBA of Na_2CO_3 and K_2CO_3 3 M solution.

compound, by the carbonation of FBA, K^+ ion-based carbonate compounds have an advantage over Na^+ -based ions. To produce CaCO_3 , the optimal conditions for the carbonation may be the use of a K^+ ion-based carbonate compound, a reaction time of 1 h, and a reaction mixing water ratio of 30%.

3.5. Properties of C-FBA mixed mortar

Figure 10 compares the water absorption and the compressive strength of common OPC mortar and the mortars mixed with FBA or C-FBA. The water absorption was higher in FBA-mixed mortar than in the OPC mortar. The water absorption of the C-FBA-mixed mortar was lower than that of the conventional FBA-mixed mortar. This may be because the free CaO contained at a high ratio in the conventional FBA may have reacted with water to produce $\text{Ca}(\text{OH})_2$. This may have resulted in volumetric expansion of the FBA, which may have changed the internal pores and increased the water absorption. On the contrary, since a large amount of free CaO is converted in the carbonation through the reaction with carbonate to CaCO_3 , which is a stable compound, the C-FBA mixed with OPC to prepare mortar does not cause a significant change in the internal pores. Therefore, the water absorption of the C-FBA-mixed mortar was found to be about 0.04% lower than that of the conventional FBA-mixed mortar. The compressive strength values of the OPC mortar and the mortars prepared by mixing 10 wt% of FBA and C-FBA were measured at material ages of 3, 7, and 28 days. Generally, the compressive strength of the mortars prepared by mixing FBA and C-FBA was lower than that of the OPC mortar. However, the compressive strength of the C-FBA-mixed mortar was higher than that of the FBA-mixed mortar. As the free CaO contained in the FBA underwent a rapid exothermic reaction and expansion, the internal pores may have increased, which may have resulted in cracks and reduced strength. On the contrary, as the unstable free CaO contained in the FBA was converted by carbonation to a stable Ca compound (CaCO_3) existing in C-FBA, the degree of the decrease of the compressive strength may have been reduced. In addition, the produced CaCO_3 may have filled the internal pores of the OPC hydrates so that the compressive strength of the C-FBA-mixed mortar is higher than that of the FBA-mixed mortar.⁶⁾ With reference to the material age of 28 days, the compressive strength values of the FBA-mixed mortar and the C-FBA-mixed mortar were respectively approximately

Fig. 10. Water absorption and compressive strength of mortar.

88% and approximately 95% of that value of the OPC mortar. On the other hand, the long-term compressive strength of the C-FBA-mixed mortar may recover as the OPC hydrate ($\text{Ca}(\text{OH})_2$) and the small amount of pozzolan materials (silicate or aluminate) existing in the C-FBA undergo a reaction to produce a hydration product (C-S-H gel).

4. Conclusions

In the present study, to increase the applicability of FBA, free CaO contained in FBA was converted to a Ca compound, CaCO_3 , under appropriate carbonation conditions; the carbonated FBA was mixed with OPC and the properties of the resulting FBA-mixed OPC were measured. The following conclusions can be drawn from the present study.

1) The rate of reactants, which was measured to verify the reactivity of FBA with an aqueous carbonate solution, remained in a range from 4.5% to 4.7% at ratios of the aqueous carbonate solution over 30%, indicating that 30% may be an appropriate ratio of the aqueous carbonate solution.

2) Regardless of the kind of carbonate involved in the carbonation of FBA, the intensity of the CaCO_3 peak increased for 1 h and then remain constant, indicating that the appropriate reaction time for the FBA carbonation may be 1 h.

3) As the molar concentration of the aqueous carbonate solution increased, the yield of CaCO_3 increased. The highest CaCO_3 yield was found to be 27.8 wt% when the carbonation was performed using a 3 M aqueous K_2CO_3 solution.

4) Measurement of the properties of the mortars prepared by mixing 10% of the conventional FBA and the C-FBA with OPC showed that the water absorption of the C-FBA-mixed mortar was lower by 0.045 than that value of the FBA-

mixed mortar. At the material age of 28 days, the compressive strength of the C-FBA-mixed mortar was about 95% of that of the OPC mortar.

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