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Influence of calcium sulfate dihydrate as chemical activator on compressive strength of hardened fly ash-cement pastes

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KEYWORDS

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Cement paste
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Compressive strength
Fly ash

ABSTRACT

The present study focuses on evaluating influence of calcium sulfate dihydrate ($CaSO_4.2H_2O$) as a chemical activator on compressive strength of hardened pastes containing fly ash and cement as cementitious materials. A high amount of Class-F fly ash used to replace Portland cement ranged from 85 to 93 % by mass of cementitious materials. For all the pastes, a low water-to-cementitious materials ratio of 0.20 was prepared. The amounts of $CaSO_4.2H_2O$ added to the mixtures were 0.0, 1.5, 2.0, and 2.5 % by mass of cementitious materials. Experimental results showed that at the age of 3 days, the compressive strength of hardened paste specimens using $CaSO_4.2H_2O$ was higher 1.21–2.15 times than that of the control paste specimen without $CaSO_4.2H_2O$. At the age of 7 days, the compressive strength of hardened paste specimens using $CaSO_4.2H_2O$ still continued to be higher 1.03–2.06 times than that of the control paste specimen without $CaSO_4.2H_2O$. At the ages of 14 and 28 days, the compressive strength of hardened paste specimens using $CaSO_4.2H_2O$ was approximately equal to that of the control paste specimen without $CaSO_4.2H_2O$. Consequently, the addition of $CaSO_4.2H_2O$ improved the early-age compressive strengths of the fly ash-cement pastes, and the $CaSO_4.2H_2O$ content of 2.0 % by mass of the cementitious materials was effective in increasing the compressive strength of such pastes.

1. Introduction

Fly ash is a by-product from the thermal power plants where the combustion of coal emits fine particles collected at the exhaust gas settling unit. The potential use of fly ash became apparent until the middle of the twenty century [1]. It was estimated that the thermal ash amount in the world was 750 million in 2012, of which at least 70 % was fly ash [2]. In Vietnam, fly ash reserves are at an alarming level and if it were not reused, by 2025 – 2030, the backlog of fly ash in Vietnam would be about 249 – 423 million tons, respectively [3].

According to the Ministry of Industry and Trade of the Socialist Republic of Vietnam, the consumption rate of ash and slag reached 38.5 % in 2018 and was predicted to increase to 50 % by 2020 and about 80 % by 2025 [4]. The Ministry of Industry and Trade has also issued Decision No. 1818/QD-BCT dated 20/7/2021 approving the plan for the implementation of Directive No. 08/CT-TTg of the Government on promoting the treatment and use of ash and slag of thermal power plants, as raw materials for the production of construction materials and the use in construction works [4]. However, the reserves of fly ash in Vietnam are still large while the use is still limited. Studies of fly ash in concrete were carried out to clarify the role and reaction mechanism of fly ash in concrete [1] and found that one of the reasons for limiting the utilization of fly ash is that, the initial strength of fly ash concrete is low, especially very low when replacing Portland cement by fly ash greater than 20 % [5]. The low initial strength of the fly ash-cement system is due to the slow pozzolanic reaction of fly ash particles [5].

Recently, several methods in terms of chemical activation on pozzolanic reaction of fly ash have been proposed and implemented to increase the initial strength of the fly ash-cement system having a low water-to-cementitious materials (w/cm) ratio [6–9]. A previous study of Lee et al. [7] have shown that the addition of chemical activator such as Na_2SO_4, K_2SO_4, and triethanolamine to the mortar specimens containing fly ash accelerates the strength development of these specimens at early ages. This was explained that the activators not only decrease or maintain the amount of $Ca(OH)_2$ (CH), but also increase the production of ettringite (AFt) at early ages. An existing study of Bui et al. [8] indicated that the use of Na_2SO_4 with 4 % by mass of cementitious materials increases compressive strength up to aging for 28 days of the hardened pastes with a low w/cm ratio of 0.30, irrespective of fly ash replacement. However, the used maximum fly ash content in the existing studies [7–14] was in a range from 40 to 70 % by mass. In addition to these chemical activators, Poon et al. [9] added 10 % $CaSO_4$ to the cement-based mortars with up to 55 % fly ash replacement and found the strength increases up to 70 % when compared to the control mortars without $CaSO_4$ after 3 days of aging. A large amount of AFt was formed during the early phase of hydration

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of the $CaSO_4$-activated fly ash-cement systems. This might be the main cause of the high early strength of the systems, but less effective in increasing the later-age strength [9]. Briefly, studies in terms of effect of calcium sulfate dihydrate on the pastes with an extremely high amount of fly ash (i.e., higher than 85 %) have still been limited.

From that, the purpose of the present study was to investigate the effect of calcium sulfate dihydrate as a chemical activator on the compressive strength of the fly ash-cement systems having a w/cm ratio of 0.20 and amounts of fly ash higher than 85 % by mass to expand the application of fly ash in concrete production.

2. Experiment

2.1. Materials

Nghi Son Portland cement in compliance with TCVN 2682:2020 [15] and Class F fly ash in compliance with TCVN 10302:2014 [16] were used as cementitious materials in the present study. The physical properties and chemical compositions of cement and fly ash are listed in Table 1. Tap water prepared at the laboratory without impurities according to TCVN 4506:2012 [17] was used to mix cement pastes.

Calcium sulfate dihydrate ($CaSO_4.2H_2O$) having a density of 2.32 g/cm^3 was used as a sulfate activator and was expected to accelerate the pozzolanic reaction of fly ash in the pastes with an extremely high amount of Class F fly ash, resulting in increasing the compressive strength at the early ages. For all the pastes, a w/cm ratio was 0.20 and four fly ash replacement mass ratios were 85, 88, 90, and 93 % by mass of cementitious materials (i.e., 85FA, 88FA, 90FA, and 93FA, respectively). The contents of $CaSO_4.2H_2O$ activator added to the mixture were 0.0, 1.5, 2.0, and 2.5 % by mass of cementitious materials (i.e., 0.0C, 1.5C, 2.0C, and 2.5C, respectively). Table 2 presents sixteen mixture proportions of the pastes designed and prepared for the experiment.

Table 1. Physical properties and chemical compositions of the cementitious. Materials.

	Unit	Cement	Fly ash
Physical properties			
Density	g/cm^3	3.1	2.3
Fineness, retained content on 0.09-mm sieve	%	0.3	-
Fineness, retained content on 0.045-mm sieve	%	-	20
Chemical compositions			
SiO_2	%	23.8	56.4
Al_2O_3	%	4.3	24.9
Fe_2O_3	%	2.5	8.1
CaO	%	62.6	2.0
Na_2O	%	0.1	0.4
K_2O	%	0.7	5.2
MgO	%	1.5	0.8
SO_3	%	2.4	0.5
LOI	%	2.1	1.7

-: not measured

LOI: Loss on ignition

Table 2. Mixture proportions of all the pastes.

Mixture proportion	Portland cement (kg)	Fly ash (kg)	Water (L)	$CaSO_4.2H_2O$ (kg)
85FA0.0C	240	1358	319	0
88FA0.0C	191	1397	318	0
90FA0.0C	158	1424	316	0
93FA0.0C	110	1463	315	0
85FA1.5C	237	1344	316	24
88FA1.5C	189	1383	314	24
90FA1.5C	157	1409	313	24
93FA1.5C	109	1448	311	23
85FA2.0C	236	1339	315	32
88FA2.0C	188	1378	313	31
90FA2.0C	156	1404	312	31
93FA2.0C	109	1443	310	31
85FA2.5C	236	1335	314	39
88FA2.5C	187	1374	312	39
90FA2.5C	156	1400	311	39
93FA2.5C	108	1438	309	39

2.2. *Specimen preparation*

After quantitative weighing of the materials listed in **Table 2**, the paste was mixed in an automatic mortar mixer according to TCVN 6016:2011 [18] in the following sequence:

(1) Preparing an activator solution including water and $CaSO_4.2H_2O$ (if any).
(2) Mixing the dry mixture of cement and fly ash until the mixture was homogeneous.
(3) Adding the activator solution prepared in step 1 or only water to the mixer and mixing for 30 seconds at a slow speed.
(4) Stopping the mixer and dredging the sticky paste in the mixer.
(5) Mixing the mixture at a fast speed for 90 seconds to get the homogenous mixture.

After mixing, the specimens were cast in cube-shaped steel moulds having sizes of 50 × 50 × 50 mm and cured for 1 day. After that, the specimens were removed from the moulds and cured in water at room temperature 28 ± 2 ºC until compression test at designated ages.

2.3. *Compressive strength test*

Compressive strength of three cubic specimens having sizes of 50 × 50 × 50 mm of each mixture proportion was tested by a hydraulic compressor as per TCVN 6016:2011 [18] at each specified age, namely at ages of 3, 7, 14, and 28 days.

3. Results and discussion

3.1. *Effect of fly ash content on compressive strengths of fly ash-cement systems*

Effect of fly ash content on the compressive strengths at the ages of 3, 7, 14, and 28 days of the fly ash-cement systems with 0 % $CaSO_4.2H_2O$ addition, namely control specimens, is shown in Figure 1. Generally, compressive strength of all the pastes increased with increasing the curing age, regardless of fly ash content. It indicates the reactions in the paste continuously progressed with time. When replacing Portland cement by Class F fly ash in a range from 85 to 93 % at the age of 3 days, the compressive strength tended to decrease from 10.29 to 3.44 MPa. Similarly, at the age of 28 days, the compressive strength of the paste specimens decreased from 26.24 to 8.77 MPa. The compressive strengths of the 85FA specimens reached the highest values not only at early ages (i.e., at the ages of 3, 7, and 14 days) but also at later age (i.e., at the age of 28 days), while those of the 93FA specimens were the lowest one. It can be said that the greater the replacement of Portland cement by Class F fly ash, the less the compressive strength of the paste specimens, and vice versa. It indicates that fly ash reduced the compressive strength of the hardened pastes because of the slow pozzolanic reaction of fly ash. The pozzolanic reaction of fly ash only occurs when the fly ash-cement systems has a high alkali concentration generated from the hydration reaction of the cement [19]. Therefore, the paste systems with the less content of Portland cement and the higher amount of Class F fly ash in the present study resulted in the lower compressive strength, especially at early-age compressive strength which was consistent with the results of Durán-Herrera et al. [20].

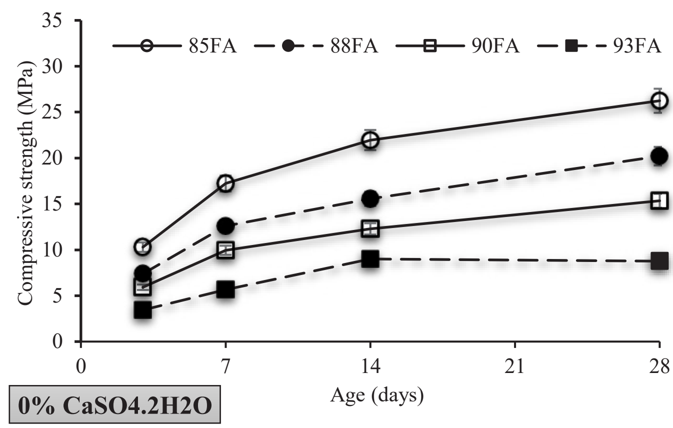


Figure 1. Compressive strength of the fly ash-cement systems with 0 % $CaSO_4.2H_2O$ up to age of 28 days.

3.2. *Effect of the $CaSO_4.2H_2O$ content on compressive strength of fly ash-cement systems*

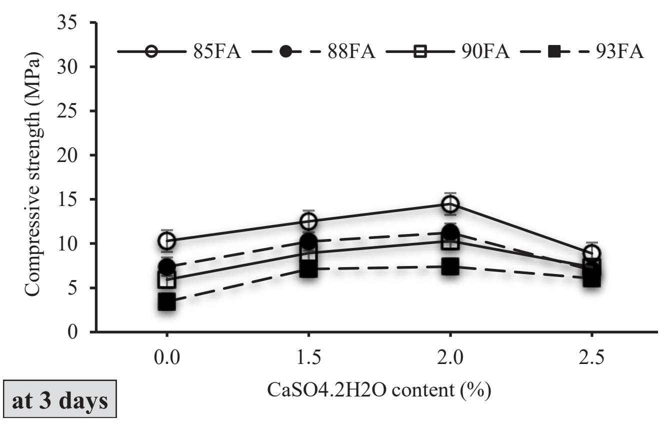
Effects of $CaSO_4.2H_2O$ content on the compressive strength of the fly ash-cement systems at the age of 3 days and normalized compressive strength of the pastes with $CaSO_4.2H_2O$ addition to the control specimen without $CaSO_4.2H_2O$ are shown in Figure 2 (a) and (b), respectively.

For the paste specimens with 1.5 % $CaSO_4.2H_2O$, the compressive strength tended to increase when compared with the specimens using 0 % $CaSO_4.2H_2O$. The compressive strength of 85FA specimens using 1.5 % $CaSO_4.2H_2O$ reached 12.50 MPa (Figure 2 (a)) which was higher 1.21 times than that of the 85FA specimen with 0 % $CaSO_4.2H_2O$ (Figure 2 (b)). The compressive strengths of specimens containing 88, 90, and 93 % replacements of fly ash activated by 1.5 % $CaSO_4.2H_2O$ were 10.24, 8.92, and 7.11 MPa (Figure 2 (a)), which were higher 1.39, 1.51, and 2.07 times than that of the specimens corresponding to same amount of fly ash activated by 0 % $CaSO_4.2H_2O$, respectively (Figure 2 (b)). Briefly, the paste specimens with fly ash activated by 1.5% $CaSO_4.2H_2O$ showed the higher compressive strength by 1.21–2.07 times than that of the control paste specimens with 0 % $CaSO_4.2H_2O$ (Figure 2 (b)).

For the paste specimens with 2.0 % $CaSO_4.2H_2O$, the compressive strength of 85FA specimens using 2 % $CaSO_4.2H_2O$ reached the highest value (i.e., 14.48 MPa) (Figure 2 (a)), which was higher 1.41 times than that of the 85FA specimen with 0 % $CaSO_4.2H_2O$ (Figure 2 (b)). The compressive strengths of specimens containing 88, 90, and 93 %

replacements of fly ash activated by 2.0 % $CaSO_4.2H_2O$ were 11.23, 10.29, and 7.40 MPa (Figure 2 (a)), which were higher 1.52, 1.74, and 2.15 times than that of the specimens corresponding to same amount of fly ash activated by 0 % $CaSO_4.2H_2O$, respectively (Figure 2 (b)). In brief, the paste specimens with fly ash activated by 2.0 % $CaSO_4.2H_2O$ showed the highest compressive strength (Figure 2 (a)), which was higher 1.41–2.15 times than that of the control specimens with 0 % $CaSO_4.2H_2O$ (Figure 2 (b)).

For the paste specimens with 2.5 % $CaSO_4.2H_2O$, the compressive strengths of specimens containing 90 and 93 % fly ash replacements were 7.34 and 6.09 MPa (Figure 2 (a)), which were higher 1.24 and 1.77 times than that of the specimens corresponding to same amount of fly ash activated by 0 % $CaSO_4.2H_2O$, respectively (Figure 2 (b)). However, the compressive strengths of specimens containing 85 and 88 % fly ash replacements were 8.88 and 7.00 MPa (Figure 2 (a)), which were 0.86 and 0.95 times of that of the specimens corresponding to same amount of fly ash activated by 0 % $CaSO_4.2H_2O$, respectively (Figure 2 (b)).



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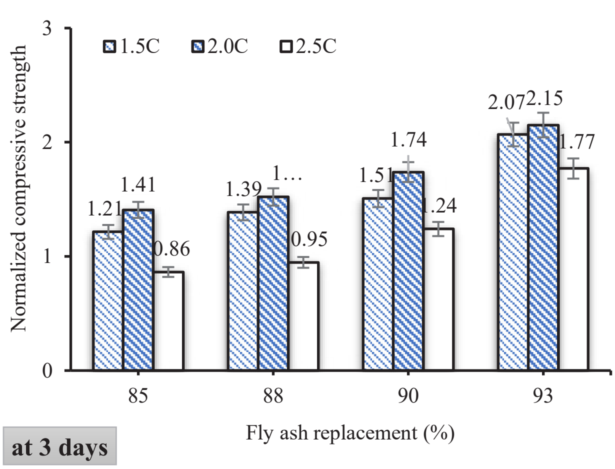
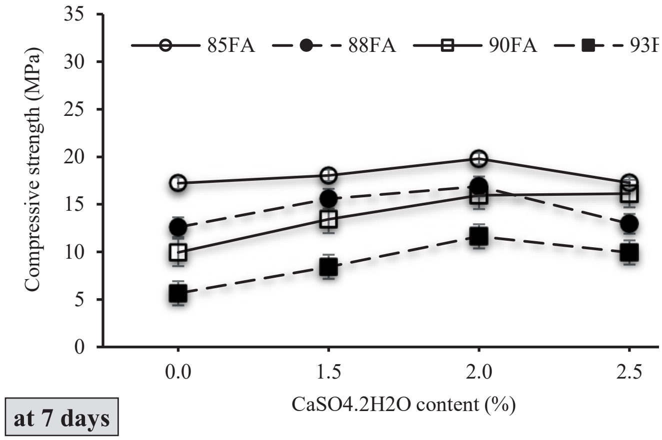


(b)

Figure 2. Effect of $CaSO_4.2H_2O$ content on compressive strength of the hardened fly ash-cement pastes at the ages of 3 days (a) and its normalized compressive strength (b).

Effects of $CaSO_4.2H_2O$ content on the compressive strength of the fly ash-cement systems at the age of 7 days and normalized compressive strength of the pastes with $CaSO_4.2H_2O$ addition to the control specimen without $CaSO_4.2H_2O$ are shown in Figure 3 (a) and (b), respectively. The compressive strengths at the age of 7 days of the specimens with 85, 88, 90, and 93 % replacements of fly ash activated by 1.5 % $CaSO_4.2H_2O$ were 18.05, 15.60, 13.42, and 8.43 MPa (Figure 3 (a)), which were higher 1.05–1.49 times than that of the control paste specimen with 0 % $CaSO_4.2H_2O$, respectively (Figure 3 (b)). For the specimens with 85, 88, 90, and 93 % replacements of fly ash activated by 2.0 % $CaSO_4.2H_2O$, the compressive strengths were 19.80, 16.89, 15.95, and 11.63 MPa (Figure 3 (a)), which were higher 1.15–2.06 times than that of the control paste specimen with 0 % $CaSO_4.2H_2O$, respectively (Figure 3 (b)). In addition, the compressive strengths of almost hardened paste specimens using 2.0 % $CaSO_4.2H_2O$ still continued to be the highest values. For the hardened paste specimens using 2.5 % $CaSO_4.2H_2O$, the compressive strength of specimens containing 85 % fly ash replacement was 17.27 MPa (Figure 3 (a)), which was equal to that of the specimens corresponding to same amount of fly ash activated by 0 % $CaSO_4.2H_2O$ (Figure 3 (b)). However, the compressive strengths of specimens containing 88, 90, and 93 % fly ash replacements were 12.95, 16.13, and 9.94 MPa (Figure 3 (a)), which were higher 1.03, 1.62, and 1.76 times than that of the specimens corresponding to same amount of fly ash activated by 0 % $CaSO_4.2H_2O$, respectively (Figure 3 (b)).

Effects of $CaSO_4.2H_2O$ content on the compressive strength of the fly ash-cement systems at the age of 14 days and normalized compressive strength of the pastes with $CaSO_4.2H_2O$ addition to the control specimen without $CaSO_4.2H_2O$ are shown in Figure 4 (a) and (b), respectively. At the age of 14 days, the compressive strength values of all the hardened paste specimens with 85 % replacement of fly ash activated by 1.5, 2.0, and 2.5 % $CaSO_4.2H_2O$ which were the highest one, corresponded to 22.42, 22.68, and 22.51 MPa (Figure 4 (a)), which were higher 1.02, 1.03, and 1.02 times than that of the specimens corresponding to same amount of fly ash activated by 0 % $CaSO_4.2H_2O$, respectively (Figure 4 (b)). For the paste specimens activated by 1.5 % $CaSO_4.2H_2O$, the compressive strengths at the age of 14 days of the specimens with 88, 90, and 93 % fly ash replacements were 17.75, 17.91, and 10.86 MPa (Figure 4 (a)), which were higher 1.14, 1.46, and 1.21 times than that of the control paste specimen with 0 % $CaSO_4.2H_2O$, respectively (Figure 4 (b)). For the paste specimens activated by 2.0 % $CaSO_4.2H_2O$, the compressive strengths at the age of 14 days of the specimens with 88, 90, and 93 % fly ash replacements were 20.67, 18.25, and 12.57 MPa (Figure 4 (a)), which were higher 1.33, 1.49, and 1.40 times than that of the control paste specimen with 0 % $CaSO_4.2H_2O$, respectively (Figure 4 (b)). For the paste specimens activated by 2.5 % $CaSO_4.2H_2O$, the compressive strengths at the age of 14 days of the specimens with 88, 90, and 93 % fly ash replacements were 19.51, 17.67, and 11.62 MPa (Figure 4 (a)), which were higher 1.25, 1.44, and 1.29 times than that of the control paste specimen with 0 % $CaSO_4.2H_2O$, respectively (Figure 4 (b)).



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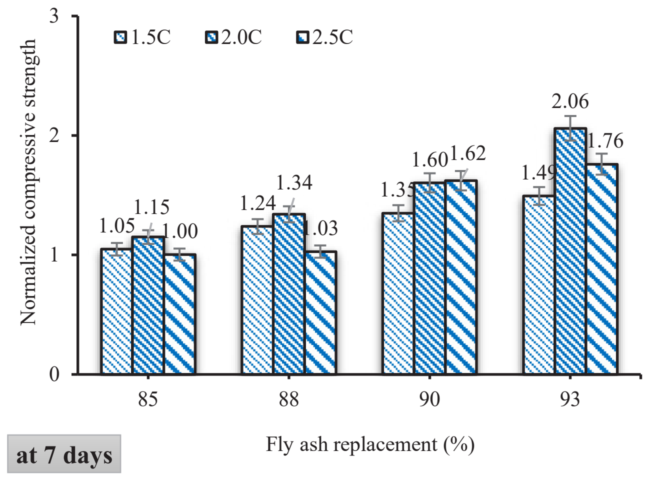
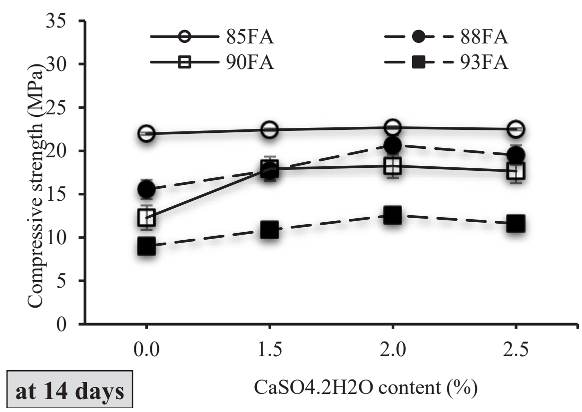


(b)

Figure 3. Effect of $CaSO_4.2H_2O$ content on compressive strength of the hardened fly ash-cement pastes at the age of 7 days (a) and its normalized compressive strength (b).



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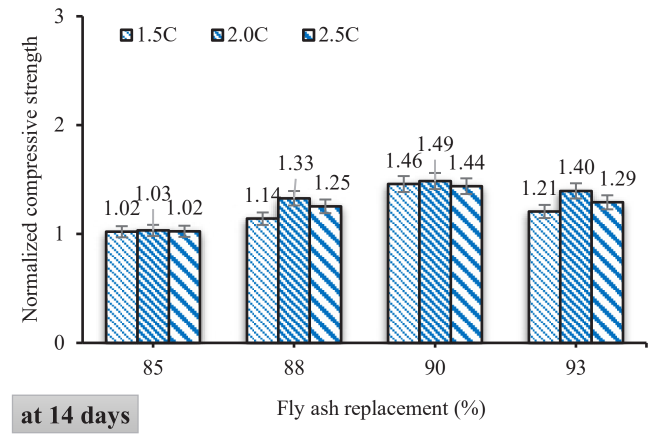


(b)

Figure 4. Effect of $CaSO_4.2H_2O$ content on compressive strength of the hardened fly ash-cement pastes at the age of 14 days (a) and its normalized compressive strength (b).

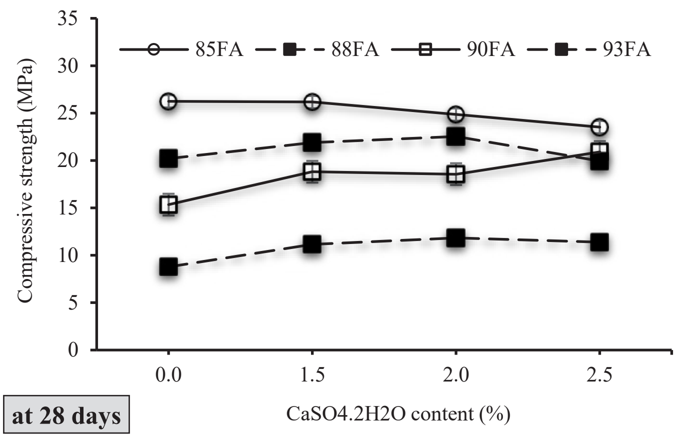
Effects of $CaSO_4.2H_2O$ content on the compressive strength of the fly ash-cement systems at the age of 28 days and normalized compressive strength of the pastes with $CaSO_4.2H_2O$ addition to the control specimen without $CaSO_4.2H_2O$ are shown in **Figure 5 (a)** and **(b)**, respectively.

At the age of 28 days, the compressive strength of hardened paste specimens with 85 % replacement of fly ash activated by 1.5 % $CaSO_4.2H_2O$ was equal to that of control specimen with 0 % $CaSO_4.2H_2O$ (**Figure 5 (a)**). Meanwhile, the compressive strengths at the age of 28 days of the specimens with 85, 88, 90, and 93 % replacement of activated by 1.5 % $CaSO_4.2H_2O$ were 26.16, 21.88, 18.81, and 11.15 MPa (**Figure 5 (a)**), which were higher 1.00, 1.08, 1.23, and 1.27 times than that of the control paste specimen with 0 % $CaSO_4.2H_2O$, respectively (**Figure 5 (b)**).

For the paste specimens activated by 2.0 % $CaSO_4.2H_2O$, the compressive strength of hardened paste specimens with 85 % fly ash replacement was lower than that of control specimen with 0 % $CaSO_4.2H_2O$ (**Figure 5 (a)**). Meanwhile, the compressive strengths at the age of 28 days of the specimens with 88, 90, and 93 % fly ash replacements were 22.53, 18.56, and 11.83 MPa (**Figure 5 (a)**), which were higher 1.12, 1.21, and 1.35 times than that of the control paste specimen with 0 % $CaSO_4.2H_2O$, respectively (**Figure 5 (b)**).

For the paste specimens activated by 2.5 % $CaSO_4.2H_2O$, the compressive strengths of hardened paste specimens with 85 and 88 % fly ash replacements were lower than that of control specimen with 0 % $CaSO_4.2H_2O$ (**Figure 5 (a)**). Meanwhile, the compressive strengths at the age of 28 days of the specimens with 90 and 93 % fly ash replacements were 20.90 and 11.38 MPa (**Figure 5 (a)**), which were higher 1.36, and 1.30 times than that of the control paste specimen with 0 % $CaSO_4.2H_2O$, respectively (**Figure 5 (b)**).

Generally, the increase in compressive strength of the paste specimens activated by chemical activators can be explained that the addition of chemical activators to the matrix accelerates the reactions including cement hydration in the cement paste and pozzolanic reaction of fly ash, leading to the increase in the early-age strength of the hardened fly-ash cement pastes [6, 7, 8, 9]. Therefore, $CaSO_4.2H_2O$ addition in the present study could promote the reactions in the fly ash-cement paste, leading to the increase in the early-age strength which would be confirmed via microstructure analysis in future works. In addition, the optimum $CaSO_4.2H_2O$ content of 2.0 % by mass of the cementitious materials was effective in increasing the compressive strength of the hardened cement pastes at early ages. However, the reason for reduction of compressive strength at later age of the fly ash-cement pastes should be explored in future works.



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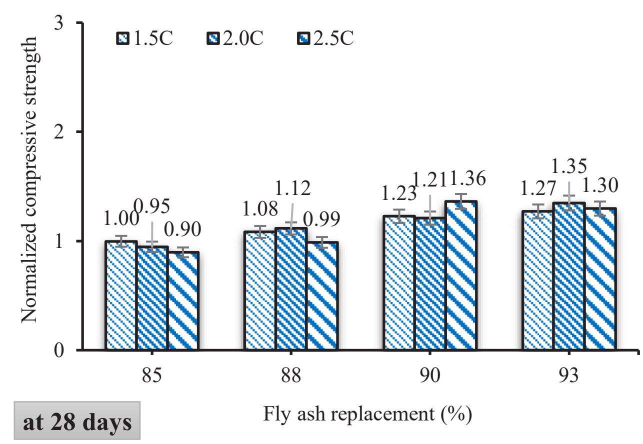

(b)

Figure 5. Effect of $CaSO_4.2H_2O$ content on compressive strength of the hardened fly ash-cement pastes at the age of 28 days (a) and its normalized compressive strength (b).

4. Conclusions

An activation by the addition of calcium sulfate dihydrate ($CaSO_4.2H_2O$) was investigated on the fly ash-cement system cured at 28 ± 2 °C. The following conclusions can be drawn within the limits of the present study:

The addition of $CaSO_4.2H_2O$ helped the matrix significantly improve the early-age compressive strengths (i.e., at the ages of 3, 7, and 14 days) of the fly ash-cement pastes using a large amount of Class F fly ash ranging from 85 to 93 % by mass of the cementitious materials.

The $CaSO_4.2H_2O$ content of 2.0 % by mass of the cementitious materials was effective in increasing the early-age compressive strength of the hardened cement pastes in which the optimal fly ash replacement was 85 % by mass of cementitious materials. Microstructure analysis of the paste using a large amount of Class F fly ash activated by $CaSO_4.2H_2O$ activator should be done in future works.

Acknowledgments

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