

Apparent Influence of Anhydrite in High-Calcium Fly Ash on Compressive Strength of Concrete

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Abstract: This case study investigates five fly ashes with high CaO and SO₃ levels in their chemical composition and compares the apparent influence of the presence and absence of anhydrite on compressive strength. Another distinguishing feature of the above ashes is that they, more or less, naturally contain anhydrite. Two different series of mixed proportions were adopted. Series 1 is designed to understand the maximum possible replacement level of fly ash. Series 2 is designed to understand the effect of anhydrite on compressive strength development. The mineral composition and glass phase of fly ashes were determined by X-ray diffraction Rietveld analysis. As a result of this study, we have found that concrete containing anhydrite-rich fly ash exhibits a higher strength than concrete containing anhydrite-free fly ash at all ages. The compressive strength increases with an increasing fly ash replacement ratio when anhydrite-rich ash is used, but strength decreases when the replacement level exceeds a certain point. The optimal amount of anhydrite was 2 ± 0.5 kg/m³ of concrete, excluding the anhydrite contained in cement.

Keywords: anhydrite; Baganuur; compressive strength; insoluble residue; lignite; mineral composition; Mongolia; ready-mixed concrete; Shivee Ovoo



Citation: Pushpalal, D.; Kashima, H. Apparent Influence of Anhydrite in High-Calcium Fly Ash on Compressive Strength of Concrete. *Buildings* **2024**, *14*, 2899. <https://doi.org/10.3390/buildings14092899>

Academic Editors: Grzegorz Ludwik Golewski, Rajai Zuheir Al-Rousan, Ciro Del Vecchio and Daiyu Wang

Received: 8 July 2024

Revised: 7 September 2024

Accepted: 12 September 2024

Published: 13 September 2024



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1. Introduction

It is well known that fly ash has versatile capabilities to make durable, affordable, tough, and environmentally friendly (DATE) concrete. However, the chemical composition of fly ash is highly variable in countries other than Japan, and most of them contain less SiO₂ and more SO₃ and CaO than Japanese fly ash [1,2]. In this context, anhydrite became apparent as one of the key ingredients. Anhydrite-rich fly ash is an unavoidable byproduct of lignite-burning coal power plants, disregarding the method of burning coal. According to the World Energy Outlook 2018, there are 23.07 trillion tons of coal reserves in terms of resources, of which 20% is lignite [3]. Lignite is a very soft coal that contains water up to 70% by weight and it pollutes the air higher than other coals. With a carbon content of as low as 25 to 35%, lignite is the lowest rank of coal because of its low heat content compared to other coals such as anthracite [4]. Another unwelcome property of lignite coal is that it generates 10–50% of ash [4]. Burning lignite produces ash containing high amounts of Ca- and S-bearing minerals.

Several authors have reported the presence of anhydrite in ash resulting from burning lignite. McCarthy et al. [5] investigated 26 fly ash (FA) samples from North Dakota and argued that if lignite or sub-bituminous coal is burned, the formation of anhydrite is not astonishing, because of high SO₃ and CaO contained in the ash. They showed that free lime in the ash is acting as a built-in “scrubber” for SO₃. Examining Cretaceous lignite from the Hailar Basin, Inner Mongolia, China, Jia and coauthors [6] reported that burning temperature depends on the mineral phases of ash. Quartz and anhydrite with a high melting point dominated the high-temperature ashes burnt at 815 °C, because of naturally available sulfate minerals such as gypsum dehydrate at high temperatures, resulting in these changing into bassanite and anhydrite.

However, opinions on the effect of anhydrite in fly ash on the fresh and hardened properties of concrete are divided into those for and against. Ravina and Mehta [7] showed the impact of replacing up to 50% of cement with ASTM Class C fly ash and F fly ash on the compressive strength of lean concrete mixtures. They obtained a targeted compressive strength of 14 MPa at an earlier age with mixtures that incorporated CaO- and SO₃-rich Class C fly ash than with CaO- and SO₃-poor Class F fly ash. However, the delay of setting times was longer in the concrete mixtures with ASTM Class C fly ash than those with ASTM Class F fly ash [8]. This delay is attributed to the sulfate content of the fly ash that is found to be on the surface of the fly ash particles. Zhang and coauthors [9] reported that concrete made with fly ash containing a high content of CaO with SO₃ has shown high compressive strength, even at early ages.

Burning lignite is not the only way that anhydrite-rich ash can be produced. Fluidized Bed Combustion (FBC) generally produces ashes richer in anhydrite because FBC boilers use ground limestone as a sulfur-absorbing material, and several authors have studied the properties of concrete that incorporates anhydrite-rich ashes [10–13]. Comparing two Circulating Fluidized Bed Combustion (CFBC) ashes, Shen and coauthors [14] reported that the compressive strength of mortar specimens containing high-anhydrite CFBC ash is higher than low-anhydrite CFBC ash. Furthermore, they have insisted that CFBC ash can be efficiently used without harm to the volume stability of cement paste if the proportions are properly designed.

Poon and coauthors [15] have investigated the role of anhydrite in the activation of ASTM Class F fly ash mortar by adding anhydrite to mortar. Their study found that early and later age strengths were increased. A mortar system that incorporated fly ash up to 55% has been activated by adding 10% anhydrite, and the existence of an optimum quantity of anhydrite for obtaining the highest strength was discovered. However, the optimum amount of anhydrite, which should be contained in a unit volume of mortar or concrete, has not been quantified.

Fly ash replacement of up to 70% of the total cementitious content of concrete was investigated in this study. This research aimed to clarify the influence of the anhydrite phase contained naturally in fly ash on the compressive strength of concrete. Furthermore, an attempt has been made to quantify the optimum amount of anhydrite, which should be contained in a unit volume of concrete to obtain a high compressive strength.

2. Experimental Procedures

2.1. Materials

2.1.1. Fly Ash

Five types of fly ashes, which were collected from Ulaanbaatar City 4th coal power plant (PP4) in different seasons, were used. These ashes are a byproduct of burning lignite. FA-0, FA-1, and FA-2 derived from Shivee Ovoo coal [16] and FA-3 and FA-4 from Baganuur coal. Shivee Ovoo ashes contain a considerably high volume of anhydrite due to sulfur-bearing minerals naturally contained in coal. According to ASTM C 618 [17], these fly ashes are designated as Class C. The chemical composition of the fly ashes measured by an X-ray fluorescence spectrometer is given in Table 1. Ashes derived from Shivee Ovoo coal (FA-0, FA-1, and FA-2) show a higher SO₃ content than ashes from Baganuur coal, as also previously confirmed by several authors [18–22]. It should be noted that there are contradictory specifications on SO₃ content in fly ash. Chinese (GBT 1596:2018) and European (BS EN 450-1:2012) standards recommended that less than 3 wt%. ASTM C 618:2017 standards recommended less than 5 wt%, while there is no limit set by Japanese (JIS A6201:2015) and Korean (KSL 5405:2018) standards [3].

Table 1. Chemical composition of fly ashes.

Type of Fly Ash	Chemical Composition (wt%)									
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	SO ₃	MgO	Na ₂ O	K ₂ O	TiO ₂	MnO
FA-0	30.2	10.7	5.8	33.7	8.2	6.4	1.3	0.7	0.5	1.0
FA-1	43.4	12.4	7.1	23.8	4.2	4.6	0.9	1.1	0.8	0.5
FA-2	38.1	11.7	7.3	27.9	5.2	5.2	1.2	0.9	0.6	0.6
FA-3	47.2	14.2	12.0	18.6	1.2	2.2	0.6	1.1	0.6	0.3
FA-4	53.1	14.4	12.8	13.2	1.2	1.6	0.4	1.4	0.6	0.4

2.1.2. Other Materials

Class 42.5 Portland cement (specific gravity: 3.00, Blaine specific surface area: 3260 cm²/g) available in the market was used. The chemical composition and mineral composition are given in Tables 2 and 3, respectively. Sand (specific gravity: 2.60) sieved through 5 mm mesh was used. Gravel was 20 mm maximum in size, and the specific gravity was 2.65. A polycarboxylate ether-based high-range water-reducing admixture was used to achieve appropriate workability for the concrete mixtures.

Table 2. Chemical composition of Portland cement (wt%).

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	Insoluble Residue	Loss on Ignition	Total
19.69	5.63	3.88	59.13	1.2	1.15	0.2	2.19	3.74	3.19	100

Table 3. Mineral composition of Portland cement (wt%).

C ₃ S	C ₂ S	C ₃ A	C ₄ AF	Gypsum	Free-CaO	Total
41.75	25.20	8.36	11.81	4.00	1.45	92.75

2.2. Testing Procedures

2.2.1. X-ray Diffraction

An X-ray diffractometer (PHILIPS X'Pert MPD) was used. Fly ash was pulverized in an alumina mortar until grains could no longer be felt between the fingers and used as the sample. Powder X-ray diffraction was performed under the following conditions: target CuK α , tube voltage 45 kV, tube current 40 mA, scanning range $2\theta = 10\text{--}60^\circ$, and step width 0.05.

Rietveld analysis of fly ash was performed on fly ashes FA-1, FA-2, FA-3, and FA-4 with 20% corundum as an internal standard. High Score Plus was used as the Rietveld analysis software. The minerals quartz, hematite, magnetite, lime, akermanite, merwinite, and anhydrite were identified.

2.2.2. Insoluble Residues and Other Properties

The insoluble residue (IR) of fly ash and cement was determined following JIS R 5202 [23]. This method is a conventional method in which the insoluble residue in cement is obtained by treating the sample with a dilute hydrochloric acid solution so that the precipitation of soluble SiO₂ is minimal. The residue from this treatment is treated again with a boiling solution of Na₂CO₃ to re-dissolve traces of SiO₂ which may have precipitated. The residue is determined gravimetrically after ignition.

Loss on ignition was measured following JIS A 6201 [24]. Specific gravity was measured per JIS R 5201 [25]. A laser diffraction particle size distribution analyzer (SHIMADZU, SALD-7000) was used to measure the specific surface area and average particle size. Free CaO content in cement was measured according to procedures recommended by the Japan Cement Association [26].

2.2.3. Specimen Preparation and Testing

Two different series of mixed proportions were adopted, and they are tabulated in Tables 4 and 5. Series 1 is designed to understand the maximum possible replacement level of fly ash. Series 2 is designed to understand the effect of anhydrite on compressive strength development. All concrete mixtures were made to obtain a slump of 210 ± 20 mm by using the water-reducing admixture, considering easy pumpability in construction sites as required by RMC. Mixtures were mixed in a single-axis horizontal mixer according to the procedures explained below. First, the binder and aggregates were added to the mixer and mixed for 30 s. Next, water which was premixed with water-reducing admixture (SP) was added and mixed for 90 s. The slump was measured in accordance with Japanese Industrial Standard A 1101 [27], and $100 \varphi \times 200$ mm specimens were molded. The specimens were demolded after two days of curing in a moist room at $20 \pm 2^\circ$ and then cured in water at $20 \pm 2^\circ\text{C}$. All specimens were continuously water-cured until the time of strength testing. Compressive strength tests were carried out according to the Japanese Industrial Standard A 1108 [28] at 3, 7, 28, and 91 days.

Table 4. Mixed proportions of concrete (Series 1).

Mix Index	Type of FA	W/B (%)	FA/B (%)	Unit Weight (kg/m^3)					
				W	C	FA	S	G	SP
Control	-	35	0	136	389	0	994	961	6.8
FA-0-30	FA-0	35	30	133	266	114	971	938	6.8
FA-0-40	FA-0	35	40	132	226	151	964	931	6.8
FA-0-50	FA-0	35	50	131	187	187	956	924	6.8
FA-0-60	FA-0	35	60	130	148	223	949	917	6.8
FA-0-70	FA-0	35	70	129	111	258	942	910	6.8

W/B: water—binder (cement plus fly ash), FA/B: fly ash—binder (cement plus fly ash), W: water, C: cement, FA: fly ash, S: sand, G: gravel, SP: superplasticizer.

Table 5. Mixed proportions of concrete (Series 2).

Mix Index	Type of FA	W/B (%)	FA/B (%)	Unit Weight (kg/m^3)					
				W	C	FA	S	G	SP
Control	-	46	0	164	356	0	1001	878	5.0
FA-1-10	FA-1	46	10	164	320	36	999	878	5.0
FA-1-20	FA-1	46	20	163	284	71	996	875	4.9
FA-1-40	FA-1	46	40	162	212	141	991	871	4.9
FA-2-10	FA-2	46	10	164	320	36	999	878	5.5
FA-2-20	FA-2	46	20	164	284	71	997	876	5.2
FA-3-10	FA-3	46	10	164	320	36	999	878	5.5
FA-3-20	FA-3	46	20	163	284	71	996	876	5.2
FA-3-40	FA-3	46	40	163	212	141	992	871	5.1
FA-4-10	FA-4	46	10	164	320	36	999	878	5.7
FA-4-20	FA-4	46	20	163	284	71	997	876	5.7

W/B: water—binder (cement plus fly ash), FA/B: fly ash—binder (cement plus fly ash), W: water, C: cement, FA: fly ash, S: sand, G: gravel, SP: superplasticizer.

3. Results and Discussion

3.1. Mineral Composition and Other Properties of Fly Ashes

Figure 1A shows the X-ray diffraction pattern of FA-0. Figure 1B represents the X-ray diffraction patterns of fly ashes FA-1, FA-2, FA-3, and FA-4. These XRD patterns show that the main constituents of FA-0, FA-1, and FA-2 are quartz (SiO_2), hematite (Fe_2O_3), lime (CaO), and anhydrite (CaSO_4); however, akermanite ($\text{Ca}_2\text{Mg}(\text{Si}_2\text{O}_7)$) and merwinite ($\text{Ca}_3\text{Mg}(\text{SiO}_4)_2$) are also traced. On the other hand, quartz (SiO_2), hematite (Fe_2O_3), and magnetite (Fe_3O_4) are available in FA-3 and FA-4; however, anhydrite does not exist. The mineral composition and glass phase of fly ashes determined by XRD Rietveld analysis are shown in Table 6. Supporting the XRD patterns, Rietveld analysis proves the availability of anhydrite in FA-1 and FA-2.

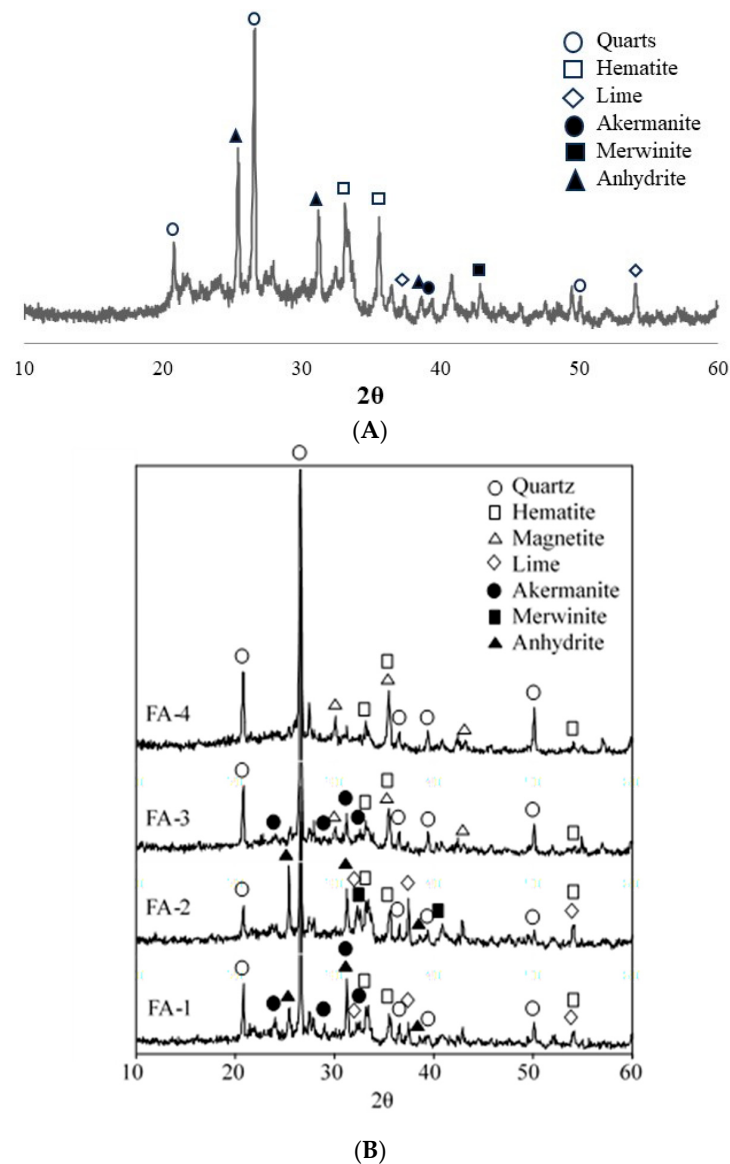


Figure 1. (A) X-ray diffraction pattern of fly ash FA-0. (B) X-ray diffraction patterns of fly ashes FA-1, FA-2, FA-3, and FA-4.

Table 6. Mineral composition and other properties of fly ashes FA-1, FA-2, FA-3, and FA-4.

Measured Item	Type of Fly Ash			
	FA-1	FA-2	FA-3	FA-4
Quartz (wt%)	13.4	8.4	18.4	21.2
Hematite (wt%)	4.5	3.6	1.7	1.6
Magnetite (wt%)	-	-	1.8	3.3
Lime (wt%)	0.7	1.9	-	-
Akermanite (wt%)	8.7	-	6.0	-
Merwinite (wt%)	-	6.9	-	-
Anhydrite (wt%)	2.8	5.7	-	-
Glass Phase (wt%)	67.1	72.7	70.6	73.4
Insoluble residue (wt%)	48.6	38.6	56.6	64.6
Loss on ignition (wt%)	0.8	0.8	1.5	0.5
Specific gravity	2.5	2.6	2.5	2.5
Blaine specific surface area (cm ² /g)	3746	5946	5770	7241
Average particle size (μm)	35.9	18.4	19.8	8.1

The percentage of the insoluble residue of fly ashes FA-1, FA-2, FA-3, and FA-4 is tabulated in Table 6. IR does not take part in the cementing action. Therefore, international standards limit IR to less than 1.5% in the case of cement. Kiattikomol remarked that IR is a measure of the adulteration of cement, largely coming from impurities [29]. IR in anhydrite-rich FA-2 is extremely low compared to other fly ashes expecting its high cementing action. Hanehara et al. [30] have shown that IR relates to the reaction ratio of fly ash, and this phenomenon has been confirmed by other authors too [31,32]. Figure 2 shows the relationship between CaO content (refer Table 1) and insoluble residue (refer Table 6), and it proves a strong correlation between the two. Though there is no similar correlation, we have found in the previous literature that studies made by the Greek Public Power Corporation remarked that “when insoluble residue is high the CaO content is low [33].” Furthermore, Goswami [34] has found that IR is higher in low-calcium FAs than high-calcium FAs, supporting our findings.

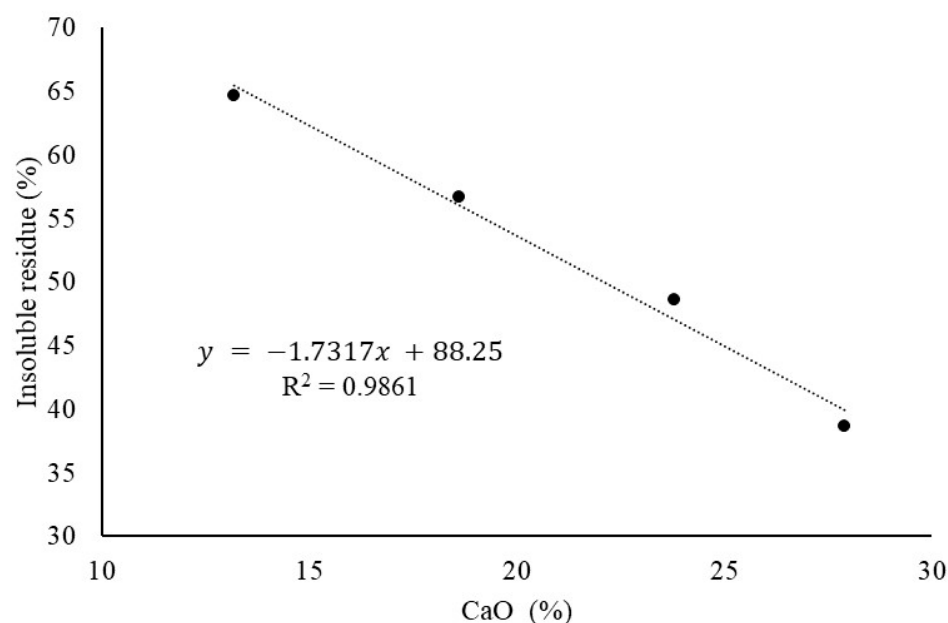


Figure 2. The relationship between CaO content and insoluble residue.

3.2. Compressive Strength

Figure 3 shows the time-dependent compressive strength of mixed proportions of Series 1. Fly ash replacement up to 60% of total cementitious content shows strength nearly equal to the control at later ages beyond 14 days. A large decrement in strength is seen when the fly ash content increases beyond 60%. While the early strength of all fly ash mixtures is lower than the control, 30 and 40 percent fly ash-incorporated mixtures have shown slightly higher strength development after 7 days, overpassing the control. A similar tendency has been confirmed by Zhang and coauthors [9], in which 50% replacement of high-calcium fly ash has shown higher later-age strength than the control mix proportion without fly ash. We attributed this tendency to being a result of the high CaO and SO₃ contents of FA-0. In order to confirm this hypothesis, we conducted the experiments in Series 2, using two clearly different sets of Class C fly ashes, in which FA-1 and FA-2 represent the set of ashes, which contains a considerable amount of anhydrite, as measured by XRD Rietveld analysis, while FA-3 and FA-4 have shown no anhydrite (see Table 6).

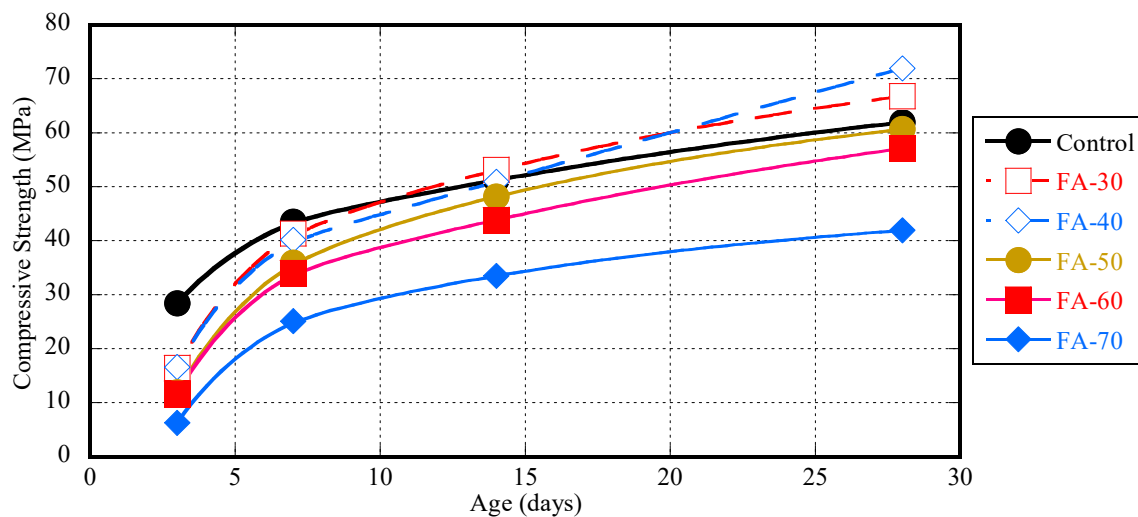


Figure 3. Compressive strength vs. age for control concrete and FA-0-incorporated fly ash concretes.

Figures 4–6 show the compressive strength of all eleven mixtures in Series 2. At all ages, FA-1 and FA-2 fly ash-incorporated concrete with replacement levels of 10% and 20% showed a higher strength compared to the control. The 40% replacement by FA-1 showed a lower strength up to the 28th day; however, it began to surpass the control after 28 days. On the contrary, FA-3 and FA-4 (i.e., both with no anhydrite) show a lower strength than the control, especially at early ages for all replacement ratios. Besides, FA-1- and FA-2-incorporated concrete exhibit higher strength than concretes with FA-3 and FA-4 at all ages. The existence of anhydrite in FA-1 and FA-2 might contribute to this achievement. Similar results have been obtained by Poon and coauthors [15] by adding 10% anhydrite to mortar with 35% fly ash replacement. When they compare gypsum and anhydrite in terms of an equivalent SO_3 content, the latter is more productive in increasing the early-age strength but less productive in increasing the later-age strength than gypsum. However, anhydrite is more beneficial in increasing the strength at all ages if a comparison is made in terms of an equal amount of addition. Therefore, anhydrite has shown itself to be advantageous over gypsum. Zhang and coauthors [9] used high-CaO and $-\text{SO}_3$ fly ash to make concrete and obtained a slightly higher early strength than low-CaO and $-\text{SO}_3$ fly ash and argued that the formation of ettringite might increase the early strength. Enders has suggested that anhydrite particles contaminated with aluminum are an easily accessible elemental source for the formation of the first ettringite during the hydration reaction of lignite fly ashes [35]. It is interesting in this context to note that these conclusions are in good agreement with what we found.

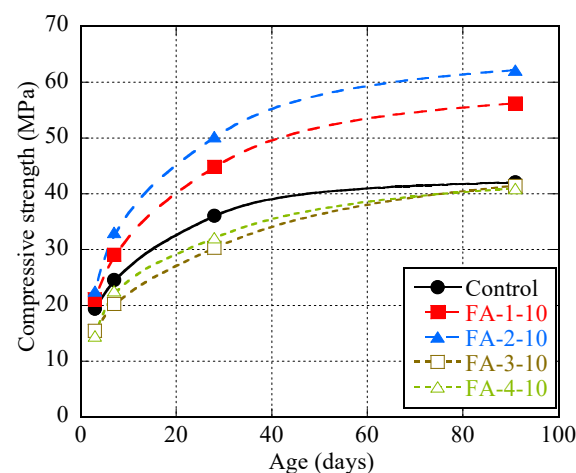


Figure 4. Compressive strength vs. age for control concrete and fly ash concretes at 10% replacement.

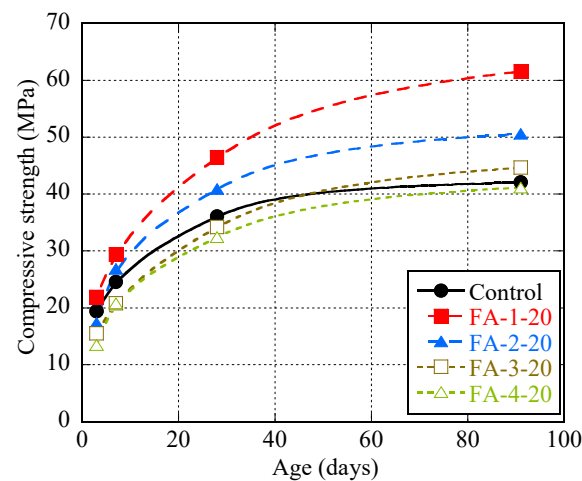


Figure 5. Compressive strength vs. age for control concrete and fly ash concretes at 20% replacement.

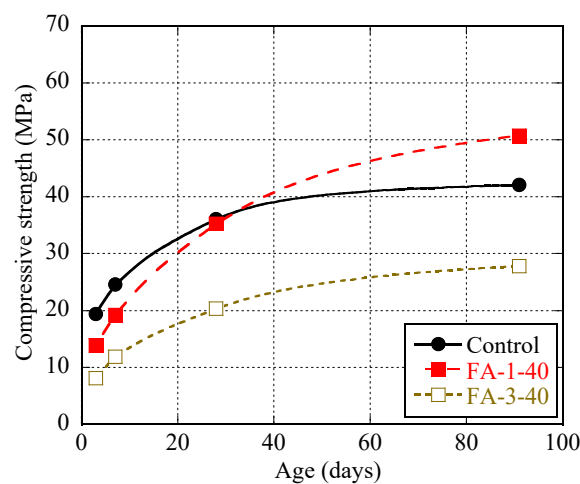


Figure 6. Compressive strength vs. age for control concrete and fly ash concretes at 40% replacement.

3.3. Optimum Amount of Anhydrite

Figures 7 and 8 show the relationship between anhydrite content, fly ash replacement ratio, and compressive strength of FA-1- and FA-2-incorporated concrete. Anhydrite quantity in fly ash concrete per 1 m³ was calculated according to the following Equation (1):

$$W_{\text{Anhydrite}} = FA \times (\% \text{Anhydrite}) \quad (1)$$

where $W_{\text{Anhydrite}}$: quantity of anhydrite in fly ash concrete (kg/m³), FA : quantity of fly ash in concrete (kg/m³), and $\% \text{Anhydrite}$: weight percent of anhydrite in fly ash (wt%). Figures 7 and 8 show the availability of an optimum level of anhydrite to be contained in concrete to obtain the highest strength. As exhibited by the curves, we arbitrate that the optimum amount lies within 1.5 to 2.5 kg/m³ of concrete. In support of this observation, Poon and coauthors [15] have also reported the existence of an optimum level of anhydrite, proving this fact by performing compressive strength tests of mortars cured at elevated temperatures. Zhou et al. [36] have found that the pozzolanic activity of anhydrite-rich CFBC is higher than that of pulverized coal combustion fly ash (PCA), which is poor in anhydrite. The activity index corresponding to the seventh-day compressive strength of CFBC can reach 103.76% when the mixing amount is 25%, while that of PCA is only 93.36%. However, the pozzolanic activity of CFBC decreases with increasing the mixing amount, the same as in our study.

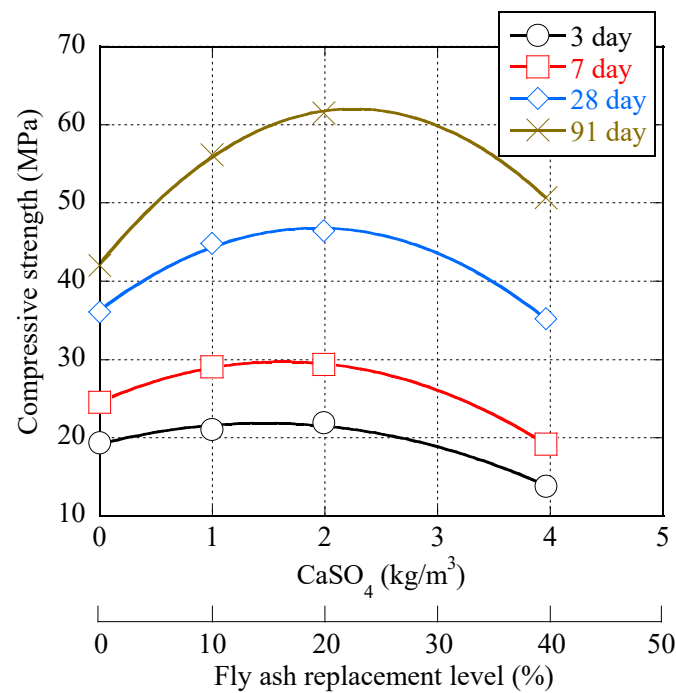


Figure 7. The relationship between anhydrite content, fly ash replacement ratio, and compressive strength for FA-1-incorporated concrete.

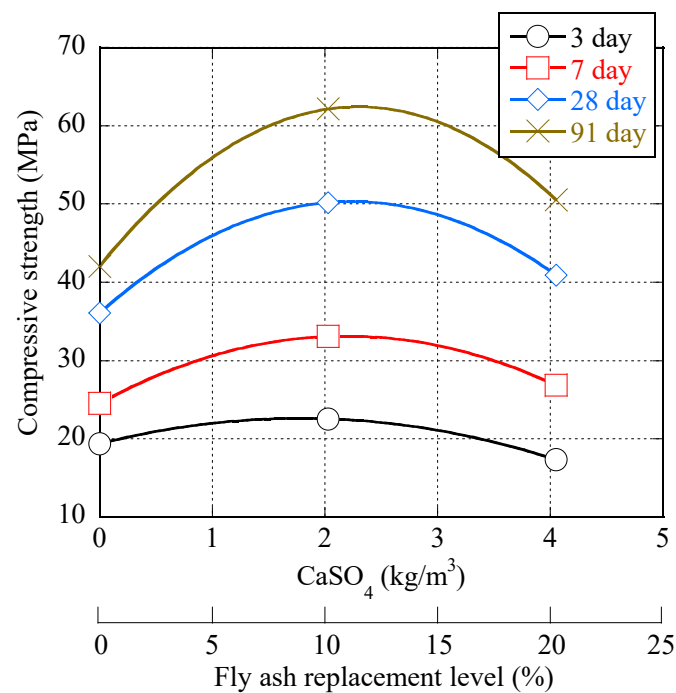


Figure 8. The relationship between anhydrite content, fly ash replacement ratio, and compressive strength for FA-2-incorporated concrete.

4. Conclusions

Two series of concrete incorporated with five high-calcium fly ashes were investigated to understand the influence of anhydrite in fly ash on the compressive strength of concrete. The main conclusions can be drawn as follows:

1. It is possible to achieve a compressive strength nearly equal to the control at a late age beyond 14 days if fly ash containing a high amount of CaO and SO₃ is used, up to a replacement level of 60%.
2. The replacement levels of up to 20% of fly ash give higher strength than control concrete at all ages except the third day when fly ash containing anhydrite is used.
3. When the CaO content is high, insoluble residue is low, showing high cementitious properties.
4. The compressive strength increases with an increasing fly ash replacement ratio when anhydrite-rich ash is used, but strength decreases when the replacement level exceeds a certain point. The optimal amount of anhydrite was 2 ± 0.5 kg/m³ of concrete.
5. This case study shows that anhydrite-rich fly ash is stronger than concrete-containing anhydrite-free fly ash even at early ages. However, this phenomenon is apparent. The factors behind this observation are still under investigation but may include anhydrite being an indirect indicator of other influential factors for producing high performances.

Author Contributions: D.P. and H.K. contributed equally to this work in all areas. All authors have read and agreed to the published version of the manuscript.

Funding: Part of this work was funded by JSPS KAKENHI Grant Number JP 25303004.

Data Availability Statement: Data are contained within the article.

Acknowledgments: The authors would like to thank Ochirbat Batmunkh (Premium Concrete LLC, Ulaanbaatar, Mongolia) for providing research support for this article.

Conflicts of Interest: Author Hiroo Kashima was employed by the company Shin-Etsu Industry Co., Ltd. The remaining author declares that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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