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Effect of High Loss on Ignition-Fly Ash on Properties of Concrete Fully Immersed in Sulfate Solution

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Abstract. This paper presents the experimental results of an investigation on the engineering properties and durability of high loss on ignition-fly ash concrete fully immersed in 5% sodium sulfate solution. The fly ash used in this study is a raw material with the loss on ignition of 15.8%, which is much higher than the upper limitation of 6% as stipulated by ASTM C618. Eight concrete mixtures were designed with different water-to-binder ratios of 0.35 and 0.45. For each water-to-binder ratio, the fly ash was used to replace 0%, 10%, 20%, and 30% amount of cement. Test results show that the workability of fresh concrete increased, while its unit weight decreased with increasing the fly ash replacement level. In addition, the concrete mixtures with 10% and 20% fly ash content showed the higher compressive strength, especially at long-term ages, than that of the fly ash-free concrete mixture. Further, all of the fly ash concrete mixtures exhibited the good quality with ultrasonic pulse velocity values of higher than 4100 m/s.

1. Introduction

The use of fly ash in concrete is not only environmental desirable and energy saving, but also improves the mechanical properties and durability of concrete. The 10-30% cement replacement by fly ash resulted in improving the compressive strength of concrete [1-4]. Some other studies have found that fly ash could be used to replace up to 60% cement in concrete with better or comparable compressive strength [5-8]. In addition, the workability of concrete increased with increasing the fly ash content [1, 2, 5]. Moreover, the combination of fly ash and ground granulated blast-furnace slag enhanced both short- and long-term properties of concrete [8].

Previous studies have demonstrated that the presence of fly ash improved the resistance of mortar and concrete to sulfate attack. Sahmaran et al. [9] showed that the utilization of about 13-32% fly ash increased the sulfate resistance of mortar, while Chindaprasirt et al. [10] reported that the use of fly ash incorporating rice husk ash increased the compressive strength and reduced the expansion of mortar. It is noted that mortar samples in both studies were immersed in 5% sodium sulfate solution. Moreover, Sumer [11] investigated the sulfate resistance of concrete containing class-F and class-C fly ash in 10% magnesium sulfate solution. Test results exhibited that, both class-F and class-C fly ash significantly improved the resistance to sulfate attack of concrete with 10-17% fly ash replacing cement. With the use of fly ash to replace 50% cement, Torii et al. [12] indicated that compressive strength of fly ash concrete was significantly greater than that of fly ash-free concrete exposed to the 10% sodium sulfate solution. Under haft-buried in sulfate soil, the 20% replacement of cement by fly ash improved the sulfate resistance of concrete [13]. The utilization of fly ash with other mineral admixtures as slag and silica fume was very effective in against sulfate attack [8, 14].

However, the properties of fly ash concrete have been strongly depended on the quality of fly ash used [15]. It is noted that most of the previous studies used the selected fly ash with the loss on ignition ranged from 0.45 to 3.2%, satisfying the requirement of below 6% as stipulated by ASTM C618 [16]. In order to fill more information to the gap in the literature, the main objective of this study is to investigate the performance of concrete made with a high loss on ignition-fly ash and fully immersed in a sulfate solution.

2. Materials and experimental works

2.1. Materials and mixture proportions

The fly ash used in this study was a raw material, which was sourced from Nghi Son thermal power plant in Vietnam, with a loss on ignition of 15.8%. The specific gravity of fly ash and cement were 2.16 and 3.12, respectively. Natural sand with fineness modulus of 2.97, density of 2.62 T/m^3, dry rodded weight of 1.50 T/m^3, moisture content of 4.35%, and water absorption capacity of 1.08% was used as the fine aggregate. The coarse aggregate used was crushed stone with the nominal maximum size of 12.5 mm, density of 2.69 T/m^3, dry rodded weight of 1.39 T/m^3, moisture content of 0.25%, and water absorption capacity of 0.08%. The superplasticizer with a specific gravity of 1.15 was used for all concrete mixtures with a dosage of 1% of the total amount of binder materials.

Table 1 shows the mix proportions for all concrete mixtures, which were designed with different water-to-binder ratios of 0.35 and 0.45. For each water-to-binder ratio, four concrete mixtures were designed with 0%, 10%, 20%, and 30% fly ash replacing cement. All of the concrete mixtures shown in Table 1 were designed in accordance with ACI 211.91 [17]. Nomenclature of the mixtures is described as follows: M35 and M45 denote the water-to-binder ratio of 0.35 and 0.45, respectively; the last numbers (0, 10, 20, and 30) are the percentages of fly ash replacing cement.

Table 1. Mix proportions for the preparation of concrete samples.

Sample ID.	w/b	Concrete ingredients proportion (kg/m^3)					
		Cement	FA	Sand	Stone	Water	SP
M35-0	0.35	514.1	0.0	985.2	751.1	175.5	5.1
M35-10		459.7	50.6	978.1	745.7	174.3	5.1
M35-20		405.1	101.5	970.9	740.3	173.0	5.0
M35-30		352.3	150.7	964.0	735.0	171.8	5.0
M45-0	0.45	400.4	0.0	1082.1	751.8	175.7	4.0
M45-10		358.3	39.8	1076.0	747.5	174.7	4.0
M45-20		316.7	79.2	1069.9	743.3	173.7	4.0
M45-30		275.6	118.1	1063.9	739.1	172.7	3.9

2.2. Samples preparation and test methods

The concrete ingredients were mixed in the laboratory using a mechanical mixer pan. Right after mixing, fresh concrete properties including slump and unit weight were measured. After that, the cylindrical concrete samples with a diameter of 100 mm and a height of 200 mm were prepared. These samples were de-molded 24 hours after casting and then fully immersed in 5% sodium sulfate solution until the testing days. The compressive strength and ultrasonic pulse velocity (UPV) tests were conducted at 3, 7, 14, 28, 56, and 91 days in accordance with ASTM C39 and ASTM C597, respectively. The reported values herein are the average value of three concrete samples.

3. Results and discussion

3.1. Characteristics of raw materials

Chemical compositions of both cement and fly ash used in this study are shown in Table 2. With a low amount of calcium and the sum of silicon dioxide (SiO_2), aluminum oxide (Al_2O_3), and iron oxide (Fe_2O_3) greater than 70%, this fly ash was classified as class-F according to ASTM C618 [16]. It is noted that the loss on ignition of this fly ash was 15.8%, which is significantly higher than that of fly ash used in previous studies as aforementioned in the literature review. On the other hand, Table 3 presents the toxicity characteristics of the fly ash. As shown, a number of heavy metals of this fly ash were significantly lower than the allowable level stipulated by Vietnamese Standard [18].

Table 2. Chemical compositions of cement and FA.

Compositions (wt. %)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	Others
Cement	22.4	5.3	4.0	55.9	2.8	2.1	0.8	0.3	4.5
FA	48.4	20.4	4.8	2.8	1.4	0.3	1.1	0.8	4.3

Table 3. Toxicity characteristics of the fly ash.

Items	Level of toxicity leached of heavy metals (mg/kg)					
	As	Cd	Pb	Hg	Zn	Cr^{6+}
Fly ash	< 0.8	< 0.6	16.2	< 1.0	35.1	< 1.0
Regulatory limit	40	10	300	4	5000	100

3.2. Properties of fresh concrete

The slump and unit weight of all concrete mixtures are given in Table 4. The slump values of M35 mixtures were lower than those of the corresponding M45 mixtures. This is because the workability of fresh concrete is closely associated with the water-to-binder ratio. Thus, the higher water-to-binder ratio resulted in higher workability. Moreover, for the same water-to-binder ratio, the slump of fresh concrete increased with increasing the replacement level of cement by fly ash. This is attributable to the spherical shape of fly ash particle and its dispersive ability. This finding is similar to the experimental results from previous studies [1, 2, 5]. It means that the effect of raw fly ash with a high loss on ignition on the workability of fresh concrete is similar to that of the selected fly ash with a low loss on ignition.
On the other hand, Table 4 also shows that the unit weight of fresh concrete decreased with increasing the fly ash content. This is due to the specific gravity of fly ash is much lower than that of cement. Additionally, when water-to-binder ratio increased, the unit weight of concrete decreased. Similarly, it is attributable to the highest specific gravity of cement as compared with other ingredients in concrete. Thus, more amount of cement resulted in the higher unit weight of concrete.

Table 4. Properties of fresh concrete mixtures.

Sample ID.	w/b	SP dosage (kg/m^3)	FA content (%)	Slump (mm)	Fresh unit weight (kg/m^3)
M35-0	0.35	5.1	0	0	2580
M35-10		5.1	10	6	2550
M35-20		5.0	20	12	2510
M35-30		5.0	30	35	2490
M45-0	0.45	4.0	0	36	2530
M45-10		4.0	10	45	2510
M45-20		4.0	20	57	2480
M45-30		3.9	30	70	2460

3.3. Compressive strength

Figures 1 and 2 show the compressive strength development of the M35 and M45 concrete mixtures that fully immersed in 5% sodium sulfate solution until 91 days. For the water-to-binder ratio of 0.35, the

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control concrete mixture without fly ash (M35-0) showed the highest compressive strength at early ages. After 14 days age, the concrete mixture with 10% fly ash (M35-10) showed the highest compressive strength. At 91 days age, compressive strengths of the M35-10 and M35-20 mixtures were higher than that of the control mixture (M35-0), while the compressive strength of M35-30 mixture was similar to that of the control mixture.

The performance of concrete against sulfate attack is associated with the permeability performance and the density of concrete structure. At the beginning, cement hydration dominantly happened to form a large amount of C-S-H gel (Equation 1), which provided the high compressive strength for M35-0 mixture at an early state. After several weeks, the pozzolanic reaction significantly happened. The higher resistance of the M35-10 and M35-20 mixtures against sulfate attack than that of the M35-0 mixture is due to the conversion of $Ca(OH)_2$ into a secondary C-S-H gel by pozzolanic reaction (Equation 2). The reduction in $Ca(OH)_2$ led to the high density of concrete structure. In general, the compressive strength of non-fly ash concrete mixture exhibited a lower increasing rate than that of fly ash concrete mixtures. It is attributable to the sulfate attack reaction (Equations 3-6) become more dominant after several weeks. The expansion of gypsum and ettringite formed during the reaction process led to a reduction in compressive strength of the concrete. The lower reduction rate in compressive strength of fly ash concrete is due to the denser structure that prevents the penetration of sulfate ion into concrete. In addition, the lower compressive strength of the concrete mixture with 30% fly ash is due to the presence of un-hydrated/ partially hydrated fly ash particle in concrete. Previous studies [1-8] have proved that the use of fly ash with optimal dosage improved the properties of concrete. However, the optimal dosage of fly ash was varying depended on its properties and concrete proportions. If the content of fly ash was over the optimal level, a part of it acted as fine aggregate instead of acting as a cementitious material.

For the water-to-binder ratio of 0.45, the concrete mixture with 10% fly ash (M45-10) showed the highest compressive strength value, while the concrete mixture with 30% fly ash (M45-30) exhibited the lowest compressive strength value. It is noted that the compressive strengths of concrete mixture with 20% fly ash (M45-20) and control mixture (M45-0) were similar before 56 days age. At 91 days age, however, M45-20 mixture showed a higher compressive strength than the M45-0 mixture, while M45-30 mixture showed a similar compressive strength to M45-0 mixture. Similar to M35 mixtures, the higher compressive strength of M45-10 and M45-20 mixtures than that of M45-0 mixture at long-term ages is also due to the denser structure of fly ash concrete compared with that of the control concrete without fly ash. This is associated with the pozzolanic reaction to form the secondary C-S-H gel in fly ash concrete. This finding proved that the sulfate attack resistance of concrete with a high loss on ignition-fly ash used in this study is similar to that of concrete with a low loss on ignition-fly ash used in previous studies [8-14]. It also means that the high loss on ignition-fly ash can be used in concrete under sulfate attack condition.

Cement hydration:

$$\left(\mathrm{C_3S,\ C_2S}\right) + \mathrm{H_2O} \rightarrow \mathrm{C\text{-}S\text{-}H} + \mathrm{Ca}\left(\mathrm{OH}\right)_2 \tag{1}$$

Pozzolanic reaction:

$$\mathrm{Ca}\left(\mathrm{OH}\right)_2 + \mathrm{SiO_2} \rightarrow \mathrm{C\text{-}S\text{-}H} \tag{2}$$

Sulfate attack reaction:

$$\mathrm{Na_2SO_4.2H_2O} + \mathrm{Ca}\left(\mathrm{OH}\right)_2 \rightarrow \mathrm{CaSO_4.2H_2O} + \mathrm{2NaOH} \tag{3}$$

$$\mathrm{Na_2SO_4.2H_2O} + \mathrm{C\text{-}S\text{-}H} \rightarrow \mathrm{CaSO_4.2H_2O} + \mathrm{N\text{-}S\text{-}H} \tag{4}$$

$$\mathrm{C_4AH_{13}} + \mathrm{3CaSO_4.2H_2O} + \mathrm{14H_2O} \rightarrow \mathrm{C_3A.3CaSO_4.32H_2O} + \mathrm{Ca}\left(\mathrm{OH}\right)_2 \tag{5}$$

$$\mathrm{C_3A.CaSO_4.12H_2O} + \mathrm{2CaSO_4.2H_2O} + \mathrm{16H_2O} \rightarrow \mathrm{C_3A.3CaSO_4.32H_2O} \tag{6}$$

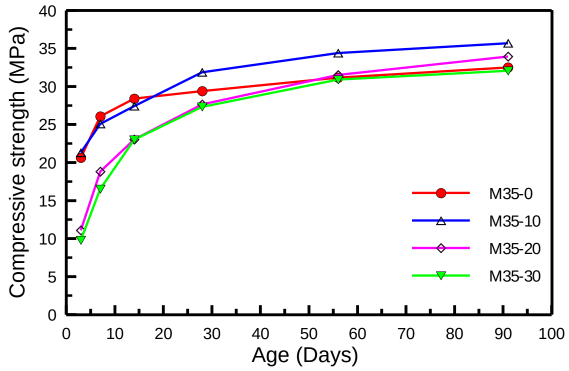

Figure 1. Compressive strength development of M35 mixtures.

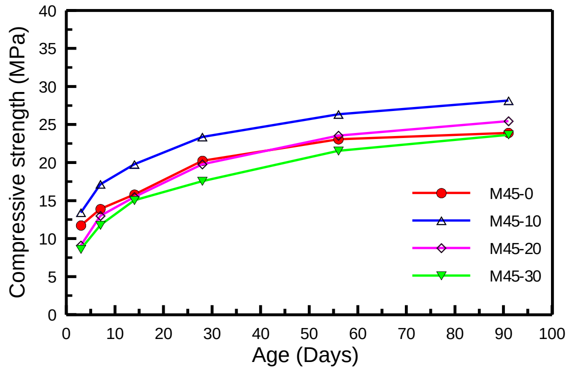

Figure 2. Compressive strength development of M45 mixtures.

3.4. Strength efficiency of cement

The strength efficiency of cement (SEC) of the M35 and M45 mixtures are presented in Figures 3 and 4, respectively. SEC is a yielded compressive strength provided by per kilogram of cement. It is related to the production cost, energy consumption, and carbon dioxide emission during the production of cement and concrete. In general trend, the concrete mixtures with 30% fly ash showed the highest SEC value, whereas the concrete mixtures without fly ash showed the lowest SEC value. In other words, high fly ash content provided high SEC value. It means that the utilization of fly ash is very effective in terms of production cost and environmental benefit.

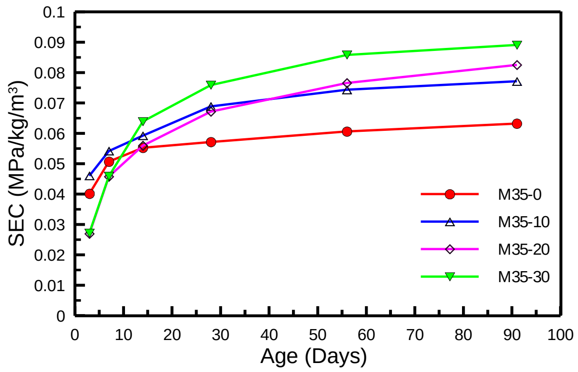

Figure 3. Strength efficiency of cement of M35 mixtures.

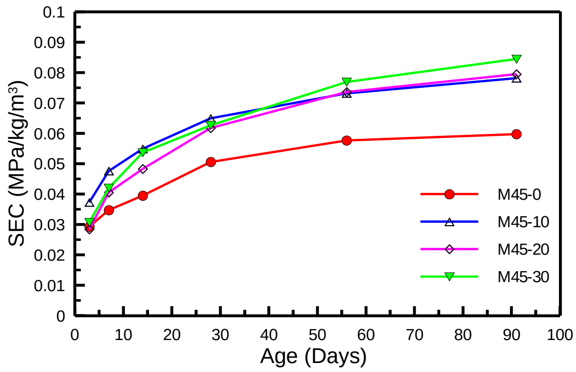

Figure 4. Strength efficiency of cement of M45 mixtures.

3.5. Ultrasonic pulse velocity

In order to evaluate the durable properties of concrete such as the presence of voids and cracks, porosity, and permeability, the ultrasonic pulse velocity (UPV) test was conducted. The UPV value is often associated with a concrete structure density and is an indirect indicator of concrete quality. Figures 5 and 6 present the UPV values of the M35 and M45 concrete mixtures over the time, respectively. For the water-to-binder ratio of 0.35, the fly ash-free concrete mixture (M35-0) showed the highest UPV value. However, at 91 days, the UPV value of the M35-10 concrete mixture was close to that of the M35-0 concrete mixture. For the water-to-binder ratio of 0.45, UPV values of concrete mixtures with no fly ash (M45-0) and 10% fly ash (M45-10) were similar. While the increasing rate of UPV values of two others with 20% and 30% fly ash content (M45-20 and M45-30) was higher than that of control concrete mixture without fly ash (M45-0). Due to the higher specific gravity of cement in comparison with that of fly ash, the control mixture showed a higher density and a faster cement hydration than the fly ash concrete at an early state. Thus, the UPV value of control concrete mixtures was high at early state. However, after 56 days, the inner structure of fly ash concrete was denser due to the contribution of dominant pozzolanic reaction to convert $Ca(OH)_2$ into a secondary C-S-H gel. This led to the higher increasing rate of UPV value of fly ash concrete than the control concrete mixtures. Moreover, all concrete mixtures had UPV values of higher than 4100 m/s. As suggested by Carcano and Moreno [19], the concrete sample with UPV value of above 4100 m/s can be classified as very good quality. This also means that all of the concrete mixtures prepared with a high loss on ignition-fly ash showed the good quality, which is comparable to that of the concrete made with selected fly ash.

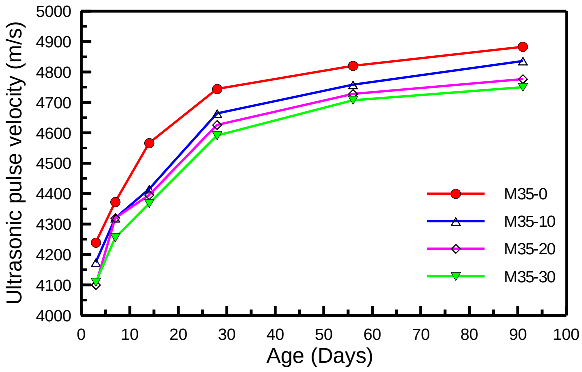

Figure 5. Ultrasonic pulse velocity values of M35 mixtures.

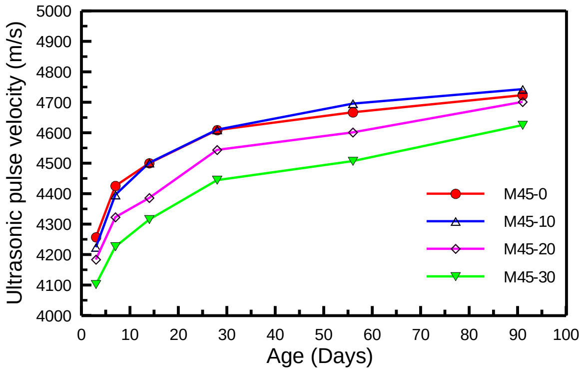

Figure 6. Ultrasonic pulse velocity values of M45 mixtures.

4. Conclusions
The present study presents the performance of concrete made with a high loss on ignition-fly ash that fully immersed in 5% sodium sulfate solution. Based on the experimental results, the following conclusions may be drawn:

- The workability of fresh concrete mixture increased and its unit weight reduced with increasing the fly ash content.
- Under 5% sodium sulfate solution, the concrete made with 10% and 20% fly ash replacing cement showed an improved compressive strength, while concrete made with 30% fly ash showed a comparable compressive strength value to the control concrete without fly ash in long-term ages.
- The strength efficiency of cement values of all concrete mixtures increased with increasing fly ash replacement level.
- All of the fly ash concrete samples showed the good quality with the ultrasonic pulse velocity values of greater than 4100 m/s.
- The experimental results of the present study demonstrated that the fly ash with a high loss on ignition can be used in concrete under sulfate condition with similar or even better concrete properties.

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