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Improvement of High-Volume Fly Ash Cementitious Material Using Single Alkali Activation

Wichian Chalee^{1*} , Reaksmey Soeurt², Pumipat Pachana^{3,4} and Smith Songpiriyakij⁵

Abstract

This research aimed to present the improvement of the cementitious material in high-volume fly ash using only one alkali activator. Fly ash was used as a partial replacement for Portland cement type I, varying from 0 to 60% by weight of the binder. Concentrations of NaOH varying from 0.00 to 1.25 molar were used as alkali activator. Paste properties and mortar compressive strength at the ages of 3, 7, 14, 28, 60, and 90 days of water curing were investigated. The results reveal that fly ash paste with an alkali activator provides shorter initial setting time when compared to control paste without alkali activator. The use of 0.50 molar NaOH concentration in mortar containing fly ash not exceeding 50% by weight of binder provides the highest compressive strength at any age of curing. At that concentration, there is a significant increase in the 28-day compressive strength of up to 45% over that of the control mortar. In addition, higher NaOH concentration (not exceeding 1.00 molar) has a significant positive effect on the compressive strength of mortar with higher fly ash content, especially over longer curing periods.

Keywords: paste, mortar, fly ash, compressive strength, NaOH concentration

1 Introduction

The use of industrial waste or by-products materials in concrete has become increasingly common in construction. Research has shown that some good-quality industrial waste not only reduces the cost of concrete production, but also improves concrete properties such as durability, alkali-silica reaction, heat reduction to prevent cracking in mass concrete, and cracking prevention due to shrinkage of concrete (Ke et al., 2018; Kim et al., 2016; Rashad, 2016; Yan et al., 2019). Pozzolanic material is one of by-products produced by industrial activities, commonly used in concrete. Previous studies have suggested that binder materials made with geopolymers using various pozzolanic materials without Portland

cement, and with high alkali concentration, produce an amorphous aluminosilicate gel with good binder properties, especially under high curing temperature (Kastiukas et al., 2020; Keke et al., 2019; Kotwal et al., 2015). More recent studies have investigated the use of cementitious materials using high volumes of pozzolanic materials activated by alkali (Li et al., 2019; Luukkonen et al., 2019; Manzi et al., 2020; Sun et al., 2020). The focus of such studies has been on using pozzolan-based binder materials to totally replace Portland cement in concrete work, or as much as possible.

Based on the previous research (Kastiukas et al., 2020; Keke et al., 2019; Luo et al., 2021), the pozzolan geopolymer is prepared by incorporating high alkali solution and sodium silicate. The binder from geopolymer was made without Portland cement. As for the alkali-activated materials, Portland cement and pozzolanic materials are mixed with alkali and sodium silicate or without sodium silicate to catalyze the reaction.

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Some pozzolan concrete has the disadvantage of low early compressive strength. Moreover, for some pozzolanic materials which have coarse particles, initiation of the pozzolanic reaction is slow and the compressive strength is clearly lower. Therefore, many pozzolanic materials that have good chemical properties cannot be used as a binder in concrete without being thoroughly ground (Chalee et al., 2013; Chindaprasirt et al., 2020; Mohammadhosseini et al., 2018). In addition, good-quality pozzolanic materials such as fly ash also contribute to low early compressive strength and, therefore, need to be used in limited quantities to prevent significant reduction in the compression strength of concrete (Herath et al., 2020; Yang et al., 2021). Thus, if an effective method can be found to accelerate the pozzolanic reaction and ensure it is more complete, it would result in higher compressive strengths, especially in the early stages, for concrete containing binder that includes pozzolanic materials. Additionally, a method enabling the use of pozzolanic materials with coarse particles without having to subject them to the grinding process would be an advancement in the concrete production process, as would methods that allow pozzolanic materials of already good quality to be used to replace Portland cement in higher volumes.

Pozzolanic reaction that occurs after the hydration reaction of cement and water by using calcium hydroxide ($\text{Ca}(\text{OH})_2$) produced from the hydration reaction as a reagent to react with silicon dioxide (SiO_2) and aluminum oxide (Al_2O_3) in the pozzolanic materials. The products obtained from the pozzolanic reaction, calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH), reduce the porosity of the paste, making the concrete denser and increasing its compressive strength (Mejdi et al., 2019; Neville, 1995). The use of alkali mixed with pozzolanic materials can elute silica and alumina from the pozzolanic materials (Chindaprasirt & Chalee, 2014; Rattanasak & Chindaprasirt, 2009). The silica and alumina that leach from the pozzolanic materials continually react with the calcium hydroxide that results from the hydration reaction to produce CSH and CAH, which are substances that provide strength to concrete.

High-volume fly ash binders have the disadvantage of low compressive strength especially at early age. The use of alkali to activate the pozzolanic reaction has a beneficial effect on compressive strength. This study will investigate the use of a single type of alkali at different concentrations to activate the pozzolanic reaction in high-volume fly ash mortar, to improve compressive strength. The results from this study may serve as a guide for the development of the mechanical properties of concrete made with other pozzolanic materials without having to improve their particle finesses.

2 Experimental Program

2.1 Materials

Binders used in this study consisted of Portland cement type I according to ASTM C150, sodium hydroxide solution (NaOH), water, and fly ash with a specific gravity of 2.23 and median particle size of 29.8 μm . For the chemical composition of fly ash, the sum of the main components, SiO_2 , Al_2O_3 , and Fe_2O_3 , was 72.2%, which is classified as class F fly ash in accordance with ASTM C618. The chemical composition of the binder is shown in Table 1.

Graded river sand passing a 4.75-mm sieve (No. 4) and retained on a 0.075-mm (No. 200) sieve with a specific gravity of 2.65, water absorption of 0.70%, and fineness modulus of 2.67, was used as a fine aggregate.

2.2 Specimens and Test Program

The mortar was prepared using Portland cement type I mixed with fly ash, with a binder-to-sand ratio of 1:2.75 by weight and mixed with water required for a standard flow of $110 \pm 5\%$. Fly ash was used to partially replace Portland cement type I at 30%, 40%, 50%, and 60% by weight of binder. Sodium hydroxide solution (NaOH) was used as an alkali activator with concentrations of 0.25, 0.50, 0.75, 1.00, and 1.25 molar. The mix proportions of the fly ash mortar are shown in Table 2. The fly ash was immersed in NaOH solution for 10 min as suggested by previous research (Rattanasak & Chindaprasirt, 2009) for good silica leaching efficiency from fly ash before being mixed with Portland cement and sand according to the specific mix proportions of each mortar specimen. For each mix proportion, the mortar cube specimens of 50 mm \times 50 mm \times 50 mm were cast in sufficient numbers for compression strength tests to be carried out at curing times in water of 3, 7, 14, 28, 60, and 90 days, and the value was taken as the average of the three samples.

Table 1 Chemical composition of Portland cements and fly ashes.

Chemical composition (%)	Portland cement Type I	Fly ash
Silicon dioxide, SiO_2	20.10	35.20
Aluminum oxide, Al_2O_3	5.20	19.20
Iron oxide, Fe_2O_3	3.15	17.81
Calcium oxide, CaO	60.24	16.65
Magnesium oxide, MgO	1.13	–
Sodium oxide, Na_2O	0.11	0.63
Potassium oxide, K_2O	0.43	2.44
Sulfur trioxide, SO_3	2.42	1.50
LOI	2.03	0.15

Table 2 Mixture proportions of alkaline activated fly ash mortar.

Mix	Mixture proportion (by weight)					NaOH solution				
	Cement Type I	Fly ash	Fine aggregate	Water						
					0.25 molar	0.50 molar	0.75 molar	1.00 molar	1.25 molar	
IF 30	0.70	0.30	2.75	0.63						
IF30-0.25 M	0.70	0.30	2.75		0.59					
IF30-0.50 M	0.70	0.30	2.75			0.58				
IF30-0.75 M	0.70	0.30	2.75				0.59			
IF30-1.00 M	0.70	0.30	2.75					0.60		
IF30-1.25 M	0.70	0.30	2.75						0.61	
IF 40	0.60	0.40	2.75	0.62						
IF40-0.25 M	0.60	0.40	2.75		0.58					
IF40-0.50 M	0.60	0.40	2.75			0.58				
IF40-0.75 M	0.60	0.40	2.75				0.58			
IF40-1.00 M	0.60	0.40	2.75					0.59		
IF40-1.25 M	0.60	0.40	2.75						0.60	
IF 50	0.50	0.50	2.75	0.61						
IF50-0.25 M	0.50	0.50	2.75		0.58					
IF50-0.50 M	0.50	0.50	2.75			0.57				
IF50-0.75 M	0.50	0.50	2.75				0.57			
IF50-1.00 M	0.50	0.50	2.75					0.58		
IF50-1.25 M	0.50	0.50	2.75						0.60	
IF 60	0.40	0.60	2.75	0.61						
IF60-0.25 M	0.40	0.60	2.75		0.57					
IF60-0.50 M	0.40	0.60	2.75			0.56				
IF60-0.75 M	0.40	0.60	2.75				0.56			
IF60-1.00 M	0.40	0.60	2.75					0.58		
IF60-1.25 M	0.40	0.60	2.75						0.59	

Testing of pastes to determine normal consistency was performed using the Vicat apparatus in accordance with ASTM C187. The method of mixing paste was in accordance with ASTM C305. Initial and final setting times were tested in accordance with ASTM C191. In addition, EDS analysis of mortar specimens based on the average of 9 grid point values was used to characterize the elemental composition of alkali-mortar.

3 Results and Discussion

3.1 Properties of Pastes

The water content required for normal consistency and setting time of paste is shown in Figs. (1) and (2), respectively. The results showed that higher sodium hydroxide concentration and higher fly ash replacement level influenced increased flow in the cement paste. The increase in flow can be attributed to the smooth, rounded surfaces of fly ash particles allowing the paste to mix more effectively. However, for paste with an alkali activator, increasing the proportion of fly ash replacement had less effect

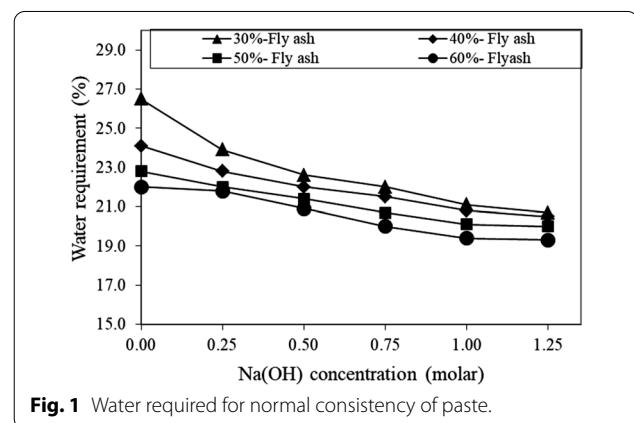
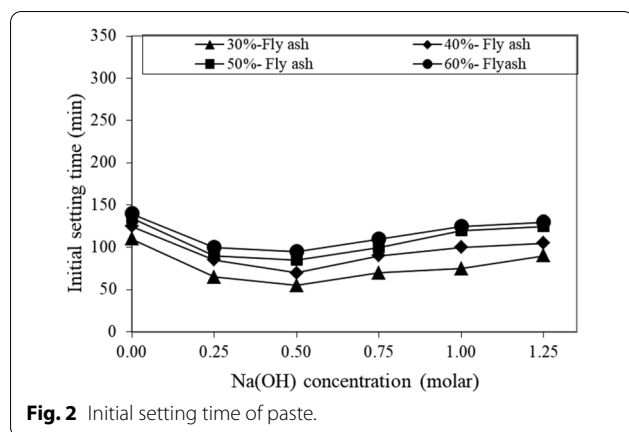


Fig. 1 Water required for normal consistency of paste.

on decreasing the water requirement than when compared with the control paste.

The effects of alkali activator on the initial setting time are shown in Fig. (2). It was found that using NaOH solution at concentrations up to 0.50 molar tends to decrease



the initial setting time, which then tends to increase as NaOH concentration increases up to 1.25 molar. However, all fly ash pastes with an alkali activator have shorter initial setting times compared to control pastes without alkali activator. The setting times for pastes with an alkali activator are shorter than those for control pastes. The short setting times may be caused by the silica and alumina compounds from the fly ash mixing with the NaOH solution. When more leaching occurs, it produces cavities on the surface, meaning the solution would be able to react more easily (Hefni et al., 2018; Moon et al., 2016). Therefore, the pozzolanic reaction between calcium hydroxide from the hydration reaction and the silica and alumina from the fly ash could produce more calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH), resulting in faster setting. In addition, higher NaOH concentrations resulted in slightly longer setting time, however, it was not significantly different from that of the conventional cement-based pastes. As expected, the initial setting time of the pastes in this study was longer when more fly ash was replaced in Portland cement. This is due to the decrease in the amount of Portland cement that affects the early setting of paste.

3.2 Effect of NaOH Concentration on Compressive Strength of Mortar

The compressive strength of fly ash mortar activated by NaOH solution is shown in Table 3. The results showed that mortars mixed with NaOH solutions with a concentration of not more than 1.00 molar had higher compressive strength than the control mortars, especially at the age of 28 days onwards. This is due to the NaOH solution dissolving silica from the fly ash surface to complete pozzolanic reactions, thus increasing the compressive strength of alkali-activated mortar. The SEM of fly ash is also compared before and after soaking the fly ash in the NaOH solution for 10 min, as shown in Fig. (3). It

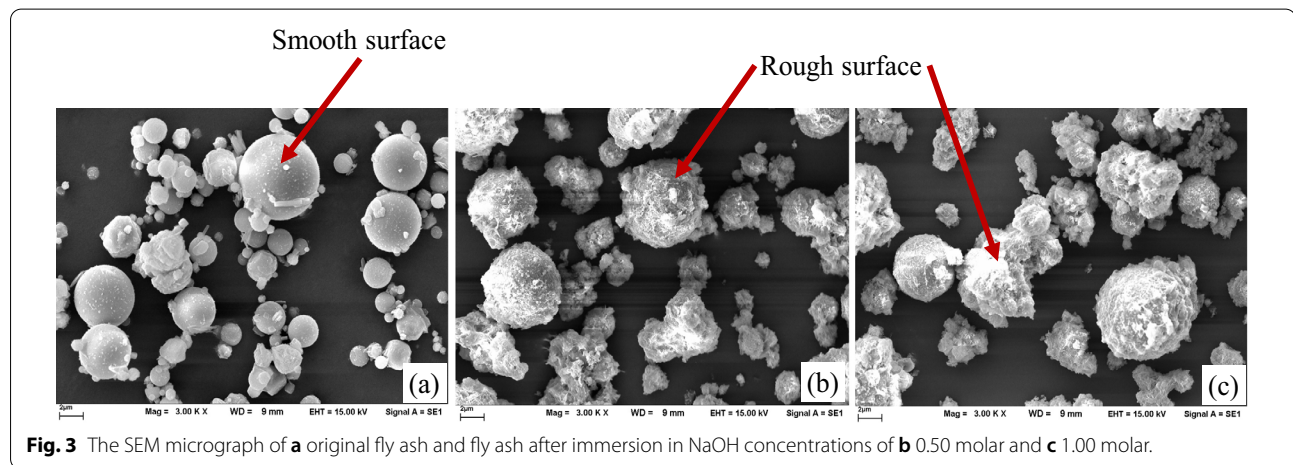
is shown that the silica on the surface of the fly ash has leached away, causing the rough surface of the fly ash particles. Previous studies show that high concentrations of NaOH can leach silica from the surface of fly ash (Chindaprasirt & Chalee, 2014; Rattanasak & Chindaprasirt, 2009). Such findings are consistent with this study, which shows that the use of low concentration NaOH solutions (i.e., pH value in the range 12–14) can leach silica from the surface of fly ash, as well. This finding, in turn, corresponds to a previous study showing that a pH of at least 13 is able to dissolve silica and alumina from the surface of fly ash (Komonweeraket et al., 2015).

As shown in Fig. (4)a, at the initial curing age of 3 days, it was found that using NaOH solution at a concentration of 0.50 molar provided a higher compressive strength of the mortar than the control mortar and the highest compressive strength for fly ash mortar with an alkali activator. Such a trend was more obvious when the age of mortar increased up to 28 and 90 days, as shown in Fig. (4)b, c, respectively. This result shows that NaOH concentration of 0.50 molar is suitable in eluting silica and alumina from fly ash to start the pozzolanic reaction with calcium hydroxide, which is a result of hydration reaction. This study found that the use of NaOH concentrations higher than 0.50 molar in the mortar containing fly ash not exceeding 50% by weight of binder resulted in lower compressive strength of the mortar. However, at the higher mortar ages of 60 and 90 days, the mortar with NaOH concentration not higher than 1.00 molar in 60% fly ash replacement provided a higher compressive strength of the mortar which was clearly more than that of the control mortar. It was noted that using higher concentrations of NaOH solution began to affect the activation of the pozzolanic reaction in mortars with higher volumes of fly ash and began to affect the increase in compressive strength, especially at long time periods. This is because the pozzolanic reaction occurs after the hydration reaction and produces higher compressive strength in the latter period. The use of NaOH solution with a concentration of up to 1.25 molar was found to produce lower compressive strength than the control mortar at any age of curing. Therefore, this NaOH concentration is not suitable to use as an alkali activator to accelerate the pozzolanic reaction in mortar or concrete. This is because concentrations of NaOH of more than 1.0 molar may give rise to the formation of calcium hydroxide from Portland cement and NaOH solution, and calcium hydroxide has a negative effect on the compressive strength of mortars.

The Ca/Si ratios obtained from EDS analysis were found in mortars without NaOH, and with concentrations of 0.50, 0.75, and 1.00 molar of NaOH at the age of 28 days, as shown in Fig. (5)a–d, respectively. Mortars

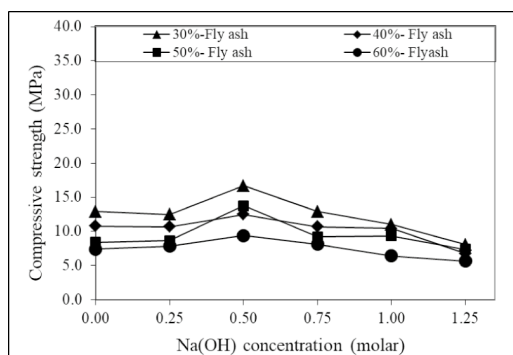
Table 3 Compressive strength of alkaline activated fly ash mortar.

Mix	Compressive strength (MPa)						Normalized compressive strength (%)	
	3 days	7 days	14 days	28 days	60 days	90 days	28 days	90 days
IF 30	12.9	14.6	18.1	21.9	26.8	30.2	100.0	100.0
IF30–0.25 M	12.5	13.6	17.4	23.2	27.4	30.7	106.0	101.9
IF30–0.50 M	16.7	18.2	21.6	27.1	30.6	33.9	123.7	112.2
IF30–0.75 M	12.9	15.1	18.6	22.5	25.9	28.4	102.7	94.0
IF30–1.00 M	11.0	12.1	14.3	19.3	22.4	24.7	88.3	81.8
IF30–1.25 M	8.1	8.3	11.7	16.0	18.0	19.3	72.9	63.9
IF 40	10.8	10.9	14.7	18.7	22.7	25.2	100.0	100.0
IF40–0.25 M	10.7	12.3	14.1	19.1	22.2	24.5	102.4	97.1
IF40–0.50 M	12.5	15.2	18.3	22.9	25.0	27.5	122.4	109.0
IF40–0.75 M	10.7	12.8	15.5	19.5	21.2	22.9	104.4	90.9
IF40–1.00 M	10.4	12.6	15.5	18.3	20.1	20.9	98.2	83.0
IF40–1.25 M	6.7	9.4	11.9	14.7	18.7	19.5	78.9	77.5
IF 50	8.4	10.2	13.0	16.6	19.3	21.3	100.0	100.0
IF50–0.25 M	8.6	10.8	15.9	19.0	22.4	25.1	114.3	117.8
IF50–0.50 M	13.8	15.8	20.2	24.2	26.8	28.2	145.8	132.2
IF50–0.75 M	9.2	11.9	15.4	19.0	23.4	27.8	114.3	130.5
IF50–1.00 M	9.4	11.9	15.9	19.7	21.7	22.8	118.8	106.9
IF50–1.25 M	7.4	9.1	12.6	15.0	18.0	20.5	90.7	95.9
IF 60	7.4	8.3	10.4	14.9	16.2	16.8	100.0	100.0
IF60–0.25 M	7.8	10.4	13.1	15.9	17.9	20.2	106.7	120.4
IF60–0.50 M	9.4	13.5	15.9	18.0	19.6	22.2	121.1	132.8
IF60–0.75 M	8.1	12.6	13.6	18.5	21.9	23.6	124.6	140.7
IF60–1.00 M	6.4	10.0	12.4	15.3	20.0	22.9	102.7	136.4
IF60–1.25 M	5.7	8.4	11.0	13.0	15.2	16.9	87.3	100.8

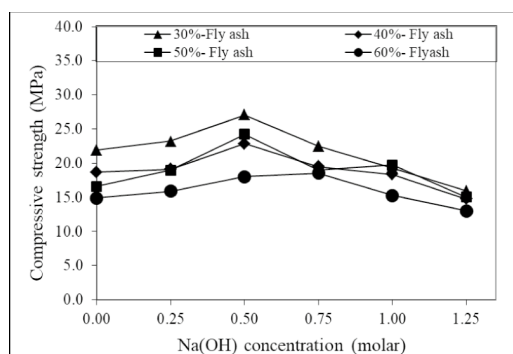
**Fig. 3** The SEM micrograph of **a** original fly ash and fly ash after immersion in NaOH concentrations of **b** 0.50 molar and **c** 1.00 molar.

mixed with a 0.50 molar NaOH solution had the lowest Ca/Si ratio of alkali-activated mortar and a lower ratio than those without NaOH solutions. Previous research (Kunther et al., 2017) has shown that a lower ratio of Ca/Si in CSH phases resulted in a significantly higher

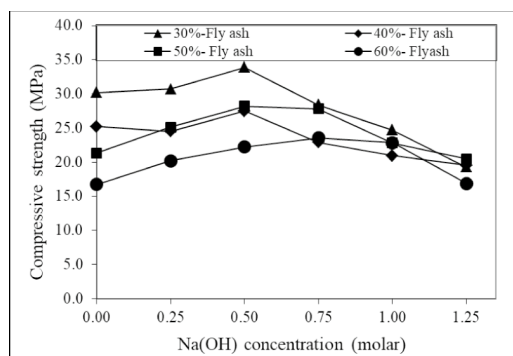
compressive strength of the mortar. Using NaOH solution to accelerate the pozzolanic reaction in this study, the increased compressive strength in the mortar using NaOH of 0.50 molar corresponds to the lower Ca/Si



(a) At the age of 3 days



(b) At the age of 28 days



(c) At the age of 90 days

Fig. 4 Effect of the NaOH concentration on the compressive strength of fly ash mortar at **a** 3 days, **b** 28 days and **c** 90 days.

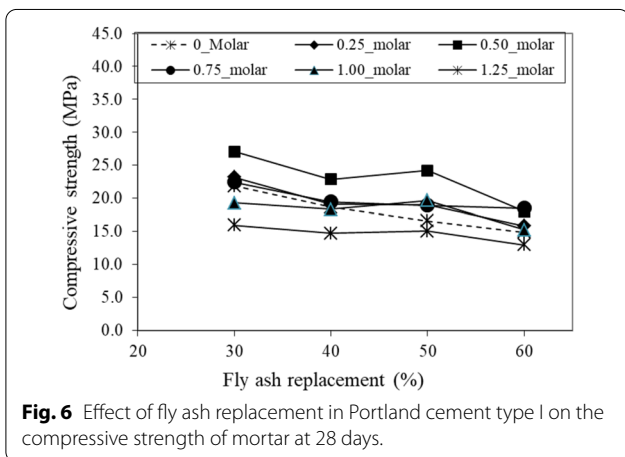
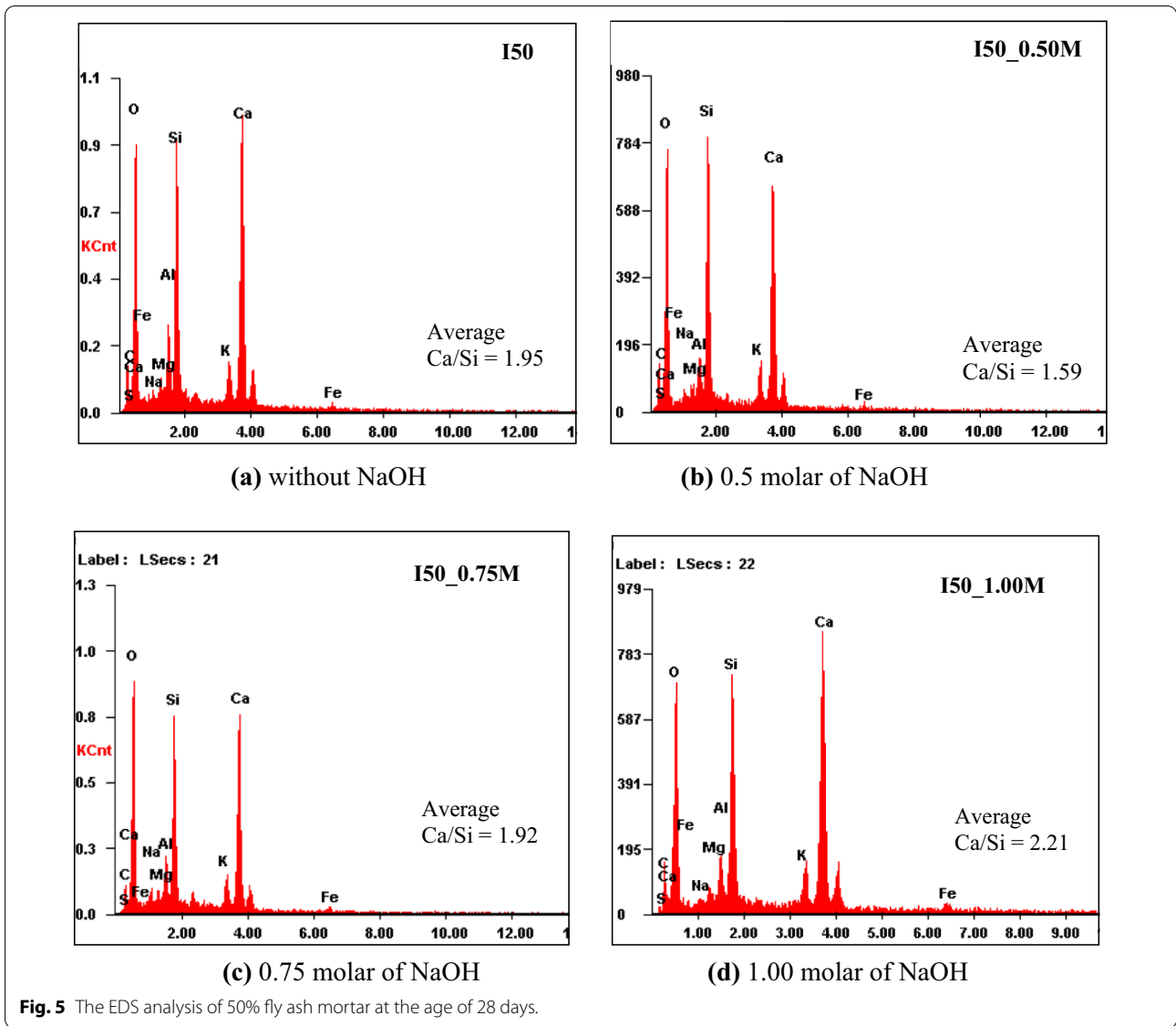
ratio from EDS analysis. It is therefore confirmed that NaOH solutions significantly improve CSH properties from the hydration reaction compared to those of mortars without NaOH. Previous studies (Richardson, 1999) have found that the Ca/Si ratio in CSH of cement-based binders is approximately 1.7 and has a variation in the range of 0.66–2.2 for supplementary

cementitious materials, which is consistent with the results of this study.

3.3 Effect of Fly Ash Content on Compressive Strength of Mortar

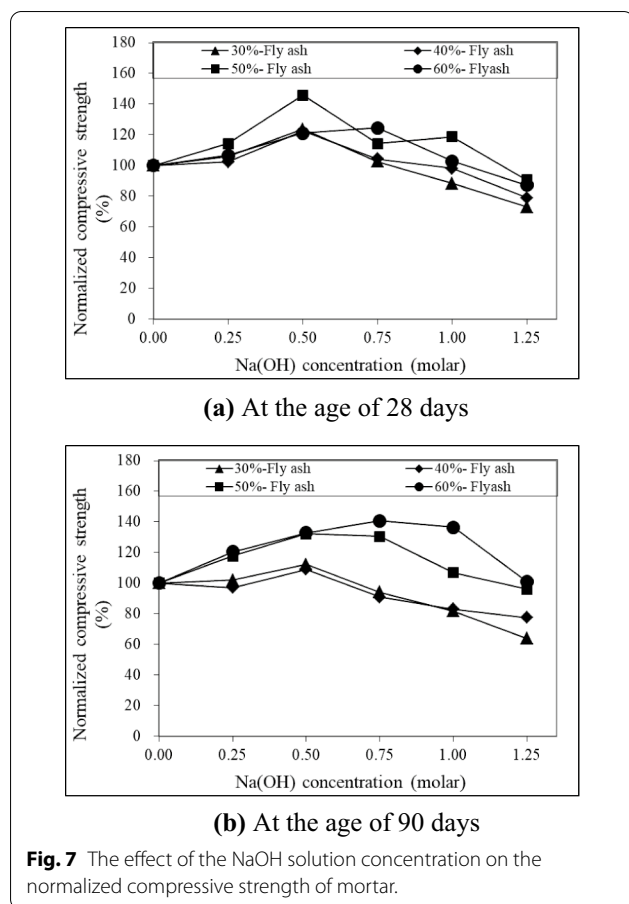
The effect of fly ash replacement in Portland cement type I on the compressive strength of mortar at 28 days of curing is shown in Fig. (6). As expected, the increase of fly ash content in mortar resulted in a decrease in the compressive strength of the mortar. The results were more obvious in the control mortar than in alkali-activated mortar. Such results may be caused by higher fly ash content, reducing the amount of Portland cement. As a result, the compressive strength caused by the hydration reaction of the cement with water was reduced as well (Herath et al., 2020; Yang et al., 2021). Using higher volumes of fly ash in mortar affected the reduction of the compressive strength of the mortar with alkali-activated mortar less than the control mortar. For instance, using more fly ash content from 30 to 50% by weight of binder resulted in a decrease in compressive strength of control mortars at 28 days of 5.3 MPa (down from 21.9 MPa in mortar IF30 to 16.6 Pa in mortar IF50), while for the same mortar that used NaOH concentration of 0.5 molar to activate the pozzolanic reaction, it was found that the compressive strength of the mortar at the age of 28 days decreased by only 2.9 MPa (down from 27.1 MPa in mortar IF30-0.50 M to 24.2 MPa in mortar IF50-0.5 M). In addition, it was found that the use of 0.5 molar of NaOH solution in mortar could produce an increase in compressive strength even when fly ash replacement level increased. This was observed from the increase of fly ash replacement of Portland cement type I from 40 to 50% by weight of binder, resulting in increased compressive strength of the mortar (up from 22.9 MPa of mortar IF40-0.50 M to 24.2 MPa of mortar IF50-0.50 M). This was because NaOH solution could elute silica and alumina from fly ash to undergo the pozzolanic reaction with calcium hydroxide and produce calcium silicate hydrate and calcium aluminate hydrate, which gave higher compressive strength to the mortar. Using NaOH solution with suitable concentration can accelerate the pozzolanic reaction in mortar or concrete mixed with pozzolanic materials, especially at the longer period.

The pozzolanic reaction was the main factor affecting the compressive strength of the single alkali-activated mortar. Since the compressive strength mechanism is the reaction between CaOH from the hydration products and silica, alumina which are leached from fly ash, therefore the expected compressive strength is due to the complete pozzolanic reaction according to the mortar curing age. The results are more evident when the mortar age is longer.



3.4 Normalized Compressive Strength of Mortar

The effect of the NaOH concentration on the normalized compressive strength of mortar (percentage compressive strength compared to the control mortar) at the age of 28 days is shown in Fig. (7)a. It was found that at any level of fly ash replacement, the use of NaOH concentration at 0.50 molar had the normalized compressive strength of mortar at 28 days more than 100% and tended to decrease when the NaOH solution concentration was higher than 0.50 molar. Interestingly, using NaOH solution concentration of 0.50 molar in mortar containing fly ash of 50% by weight of binder affected the increase in the compressive strength of the mortar the most, increasing the compressive strength at 28 days by almost 46% from the



control mortar (the normalized compressive strength at 28 days was 145.8%). The use of higher NaOH concentration not exceeding 1.00 molar has a significant effect on the increasing compressive strength in mortar with higher fly ash content, especially at long time periods, as shown in Fig. (7)b. For instance, the use of 0.75 molar NaOH solution in mortar containing fly ash of 60% by weight of the binder caused the highest increase in the compressive strength of mortar (the normalized compressive strength at 90 days was 140.7%), which was slightly higher than that for mortar with 0.5 molar NaOH solution. This may be because the use of higher concentration of alkali in the mortar with higher fly ash content can better elute silica and alumina to undergo the pozzolanic reaction. However, using 1.25 molar of NaOH concentration, the normalized compressive strength was found to be significantly decreased at any age of curing.

4 Conclusions

The conclusions of this study are summarized as follows:

1. The water requirement for normal consistency of the fly ash paste with an alkali activator was significantly reduced with increased NaOH concentration and fly ash content.
2. All fly ash pastes with an alkali activator exhibited initial setting times less than that of control paste without alkali activator.
3. The use of NaOH solution at a concentration of 0.50 molar provides higher compressive strength of the mortar than the control mortar and produces the highest compressive strength of fly ash mortar with an alkali activator. In addition, the mortar containing fly ash of 50% by weight of binder with NaOH concentration of 0.50 molar showed a significant increase of up to 45.8% in compressive strength at 28 days over the control mortar.
4. The increase in fly ash replacing Portland cement Type I in mortar resulted in decreases in the compressive strength of the mortar, which were more obvious in control mortar than in alkali-activated mortars.
5. The use of 0.50 molar of NaOH solution in mortar produced an increase in compressive strength even when the fly ash replacement level was increased.
6. Using higher NaOH concentration not exceeding 1.00 molar has a significant positive effect on the compressive strength of mortar with higher fly ash content, especially after long time periods.

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Authors' contributions

The authors investigated the test results and studied the mechanism of alkali-activated cementitious material in this study. All authors read and approved the final manuscript.

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Availability of data and materials

All data used during the study appear in the submitted article.

Declarations

Competing interests

The authors declare that they have no competing interests.

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