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by Antoni Antoni

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Enhancing Compressive Strength of Very-High-Volume Fly Ash (VHVFA) Mortar using Low-Molarity Alkali Solution and Thermal Activation

Samuel Santosa, Antoni Antoni, Djwantoro Hardjito

(samuel@petra.ac.id)

Department of Civil Engineering - Petra Christian University, Surabaya, Indonesia

Abstract

The utilization of fly ash as a waste product of coal combustion is currently limited to being a supplementary cementitious material (SCM). Fly ash integrates well with cement, demonstrating favorable qualities in concrete such as good workability, high ultimate strength, and durability. However, the use of fly ash in very high proportions has not been extensively explored due to its weakness in early strength development in concrete. Methods have been investigated to enhance the early compressive strength and compressive strength of very-high-volume fly ash mortar. This research explores the incorporation of fly ash at a very high percentage (80%), also known as very-high-volume fly ash (VHVFA), into mortar using low-molarity alkali solution and thermal activation. The activation of fly ash is examined through alkali activation, specifically utilizing sodium hydroxide (NaOH) solution, and thermal activation involving temperature and activation time. Pre-activating Class F fly ash for 2 hours using a 0.3 M NaOH solution at a temperature of 60°C increased the early compressive strength (at 7 days) by 35% and the compressive strength at 28 days by 12%. Pre-activation for 30 minutes at room temperature with a 1 M NaOH activator was able to increase the early compressive strength of VHVFA mortar (at 7 days) by 79% for Class C fly ash and by 43% at 28 days. In this study, it is shown that through the use of low-molarity alkali solution and thermal activation, the compressive strength of VHVFA mortar can be increased whether using Class F fly ash or Class C fly ash.

Keywords: class F fly ash, class C fly ash, very-high-volume fly ash (VHVFA) mortar, low molarity alkali activation, thermal activation, sodium hydroxide activator

1. Introduction

An effective approach to reducing carbon emissions in the cement industry is to decrease the consumption of clinker by utilizing supplementary cementitious materials (SCMs) (Limbachiya & Bostanci, 2014). The utilization of fly ash, a waste product from coal combustion in power plants, in combination with ground granulated slag, a waste product from the steel smelting industry, as mixtures in Portland cement, exemplifies a comprehensive strategy for mitigating the negative environmental impacts of this industry. The utilization of fly ash in concrete from sources other than coal combustion, such as sugarcane bagasse ash and even industrial and agricultural waste, has also been studied (Tanu & Unnikrishnan, 2022, 2023a, 2023b). The benefits of fly ash have long been known due to its pozzolanic properties (McCarthy & Dyer, 2019) as it can enhance the microstructure properties of concrete (Filho et al., 2013). Research shows that the bond performance of steel bars embedded in concrete with fly ash content also yields satisfaction (Padavala et al., 2023). Numerous studies have explored the utilization of fly ash as an SCM, especially at lower percentages (Mehta, 2004). While research on fly ash utilization typically involves no more than 35% fly ash content, the common range is 15–25% (Mehta, 2004; Donatello et al., 2013; Garcia-Lodeiro et al., 2013; Bondar D, 2017; Aydin & Hasan, 2017). However, there has been increased interest in more ambitious utilization, such as 50–60%, which is known as High-Volume Fly Ash (HVFA) concrete (Malhotra & Cheyrezy, 1986). Utilization exceeding 80% of fly ash as the binder material is referred to as Very-High-Volume Fly Ash (VHVFA) (Donatello, Fernández-Jimenez, et al., 2013). Research is underway to investigate the application and attributes of utilizing 100% fly ash in concrete by leveraging the self-cementing properties inherent in fly ash (Wattimena et al., 2022). Both HVFA and VHVFA are environmentally friendly concrete solutions, with VHVFA use aimed at maximizing the reduction of cement content to the maximum limit.

As a result of hydration reactions, fly ash in concrete forms Calcium Silicate Hydrate (CSH) and a portion of Portlandite $\text{Ca}(\text{OH})_2$ or (CH). Each of these components plays a distinct role, with CSH contributing to the strength of the mixture while CH increases the alkalinity necessary for subsequent reactions involving fly ash.

The pozzolanic reaction between fly ash and CH contributes to strength development in later stages. Maintaining a certain level of alkalinity is crucial for this pozzolanic reaction, underscoring alkalinity as an initial indicator of fly ash reactivity.

Alkali activation entails the use of diluted alkali solutions instead of regular water or pre-mixing (activating) the fly ash, leading to a significant enhancement in compressive strength (Chalee et al., 2021). Thermal activation involves elevating the temperature of the fly ash mixture or conducting the curing process at higher temperatures. Concrete with a high fly ash content faces a primary drawback in terms of early strength development and setting time (Thomas, 2010; Bentz, 2014). To address these critical limitations, various studies have explored methods such as thermal activation and alkali activators (Wilińska & Pacewska, 2018). Numerous efforts have been undertaken to boost the reactivity of fly ash by introducing alkali activators. Research studies have investigated the efficacy of Na_2SO_4 , CaSO_4 , $\text{Ca}(\text{OH})_2$, and NaOH as alkali activators. However, their exploration has primarily concentrated on polymerization reactions rather than on the hydration reaction stream (Arjunan et al., 2001; Donatello et al., 2013; García-Lodeiro et al., 2013; Abdollahnejad et al., 2014; Martauz et al., 2016). Dilute NaOH solutions with low molarities (0.01–0.09 M) have been shown to enhance the reactivity of fly ash (Pratiwi et al., 2020). Another approach to enhance fly ash reactivity involves pre-mixing the fly ash with alkali activators before incorporating them with other materials.

The addition of alkali solutions increases the pH, thus augmenting the reactivity of the mixture (Donatello, Maltseva, et al., 2013; Donatello, Fernández-Jimenez, et al., 2013; Garcia-Lodeiro et al., 2016). The Arrhenius equation elucidates that numerous factors influence the pace of a chemical reaction, including temperature and concentration. Activating fly ash with a dilute NaOH solution for 2 hours at 90°C results in an augmented compressive strength (Arjunan et al., 2001). Curing by raising the temperature is known to increase the early compressive strength but it reduces the compressive strength at later ages, a phenomenon known as the “cross-over effect” (Malhotra & Carino, 2003).

The pozzolanic reaction between cement and fly ash has been observed to improve when utilizing 0.14 M NaOH at 60°C , which leads to enhanced initial compressive strength by a numerical model (Wang & Park, 2015). Curing for 48 hours at 90°C has a positive impact on Class F HVFA by increasing both its initial and final compressive strength (Zhang et al., 2000; Dong et al., 2020). The influence of high-calcium fly ash (Class C fly ash) activation using 0.023 M Li_2CO_3 and 0.005 M $\text{Al}_2(\text{SO}_4)_3$ activators, along with fly ash activation and curing at 20°C for 1 day, has been shown to enhance the compressive strength of the mortar and shorten its setting time (He et al., 2019). The pozzolanic reaction of low-calcium fly ash in mixtures activated at 20°C and 60°C using 0.14 M NaOH leads to a significant increase in compressive strength at an early age, particularly with activation at 20°C (Wang & Ishida, 2019). The use of fly ash with a very high volume has the main disadvantage of a slow increase in compressive strength and the increase in the fly ash content will lead to a decrease in compressive strength. The experiments in this research were aimed at improving the compressive strength (28 days) and early strength (7 days) of very-high-volume fly ash mortar containing 80% fly ash.

The significance and novelty of this study lie in the utilization of both Class F and Class C fly ash in HVFA concrete, coupled with the use of a low-molarity alkali solution and thermal activation methods to enhance its compressive strength, which has not been studied yet. To ensure that the reaction occurring is a hydration reaction, an alkaline chemical activator (NaOH) was used at different low molarities (0.01, 0.1, 0.3, 0.5, and 1 M). Factors such as activation time (direct activation/0 minutes, pre-activation 30 minutes, and pre-activation 2 hours), activation temperature (at room temperature 28°C , 60°C , 80°C), and post-casting curing temperature (at room temperature 28°C , 60°C , 80°C) were manipulated.

2. Materials

This study presents the oxide composition of cement type PPC (Portland Pozzolan Cement) (Dynamix brand) as determined through XRF analysis, along with the composition of fly ash as detailed in Table 1. The sand obtained from a quarry in Lumajang, east Java, Indonesia, yielded a Fineness Modulus (FM) of 2.82 as per ASTM C 136. The sand was prepared in a Saturated Surface Dry (SSD) condition. Water was obtained from the Surabaya water tap. Sodium hydroxide (NaOH) as the alkali activator was obtained in flake form and prepared as 0.01, 0.1, 0.3, 0.5, and 1 M solutions. The solutions were prepared 24 hours before use. Fly ash was sourced from PT. PLN Nusantara Power Up Pacitan, a power plant located in Pacitan (later designated as Class F), as shown in Figure 1(a), and PT. Paiton Operations & Maintenance Indonesia (POMI) Unit 3 (later designated as Class C) in East Java, Indonesia, as illustrated in Figure 1(b). Superplasticizer was Viscocrete 1003 from Sika that comprised an

aqueous solution of modified polycarboxylate copolymers, used at a dosage of 0.176% by mass of the binder material.

The fly ash and materials used in this study underwent pH testing according to ASTM D 5239, as shown in Table 2. The pH testing involved mixing 20 grams of fly ash with 80 mL of distilled water and then allowing the solution to stand for 2 hours. The water–binder ratio was determined after conducting the normal consistency test according to ASTM C 305 for each type of fly ash in order to determine the required amount of water for achieving sufficient workability. The setting time for each type of fly ash was measured according to ASTM C 191 in order to determine the duration of the initial set and final set, as shown in Table 2. A longer setting time indicates a lower reactivity of the fly ash (Wijaya et al., 2017; Liu et al., 2020; Akmalaiuly et al., 2023; Overmann et al., 2024). The Specific Gravity (SG) values of the materials used in this study can be found in Table 2.

Table 1. The XRF Results of Fly Ash and Cement

Oxides	Cement (%)	Fly Ash (%)	
		Class F	Class C
CaO	59.97	10.49	19.12
SiO ₂	13.00	43.17	35.28
MgO	4.30	6.03	7.26
Al ₂ O ₃	3.18	17.14	14.00
Fe ₂ O ₃	3.03	15.25	16.95
SO ₃	2.13	1.55	1.43
K ₂ O	0.54	0.92	1.11
TiO ₂	0.24	0.78	0.84
Na ₂ O	0.23	1.63	2.19
P ₂ O ₅	0.12	0.23	0.26
MnO ₂	0.04	0.29	0.20
Cr ₂ O ₃	< 0.01	-	-
LOI	-	0.41	0.63



(a)



(b)

Figure 1. Physical condition of (a) Class F Fly Ash, (b) Class C Fly Ash

Table 2. Material Properties

Material	pH	Setting Time (minute)		Specific Gravity
		Initial	Final	
Cement	12.4	100	150	2.88
Fly Ash Class F	11.2	180	>360	2.33
Fly Ash Class C	12.2	45	75	2.75
0.3M NaOH	13.5	-	-	-
Sand	-	-	-	2.47

According to ASTM C-618 standards, the fly ash obtained from Pacitan falls under Class F, whereas the fly ash from Paiton belongs to Class C. Based on the Canadian standards whereas the fly ash from Paiton belongs

to Class C. Based on the Canadian standards (Carette & Malhotra, 2011), where fly ash classification is primarily based on the percentage of CaO, both are classified as Class C1 (8–20% CaO). Scanning Electron Microscopy (SEM) imaging was conducted to visualize the morphology of the fly ash used, as seen in Figure 2. The Particle Size Analysis (PSA) was performed to determine the particle size distribution of the fly ash and cement, as shown in Figure 3, which indicated that the fly ash obtained from Pacitan is less fine than that from Paiton.

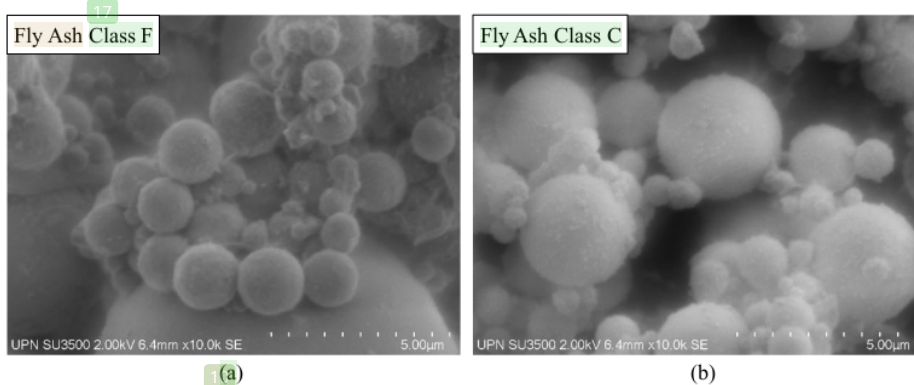


Figure 2. The SEM Images of Fly Ash (a) Class F Fly Ash, (b) Class C Fly Ash

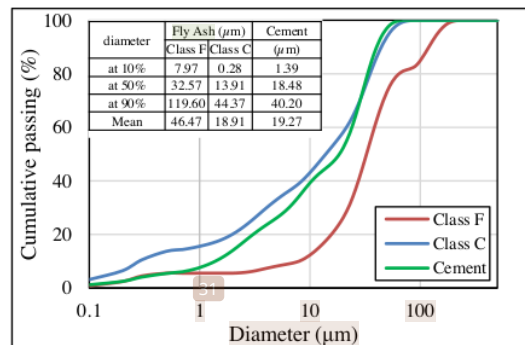


Figure 3. Particle Size Analysis

3. Mix Design

The composition of the mixture (by mass) used was as follows: cement:sand = 1:2; fly ash and cement = 4:1 or 80%:20%. The experimental method employed in this study involved the following steps. The cement, sand, and fly ash were mixed in the desired proportions. The alkali activator (NaOH solution) was slowly added to the mixture while continuously stirring. Water was gradually added to the mixture to achieve the desired consistency. The superplasticizer (Viscocrete 1003) was added to the mixture at a dosage of 0.176% (by mass) of binder material. A higher normal consistency value indicates a higher need for water in the mixture. In this study, a water–binder (w/b) ratio of 0.2 was used for mixtures with 0%, 40%, and 60% fly ash. For the mixture containing 80% fly ash, a w/b ratio of 0.15 was selected in order to maintain the desired viscosity without becoming overly fluid (Qafleshi et al., 2013) because as the percentage of fly ash in the mixture increases, so does its workability (Antoni et al., 2015). The water–binder ratio was determined after conducting the normal consistency test according to ASTM C 305 for each type of fly ash in order to determine the amount of water required to achieve sufficient workability, as shown in Table 3 and according to ASTM C 1437.

The mixture was thoroughly mixed until a homogeneous mortar mixture was obtained. The mortar mixture was placed into molds to form test specimens of the desired size. The specimens were cured under controlled temperature and humidity conditions for a specified period. After the curing period, the specimens were subjected to compressive strength tests. The mixing of fly ash with water or an activator is known as “activation.” In this study, the activation time involved direct mixing (0 minutes) with other materials or allowing it to sit for a

pre-activation 30 minutes or pre-activation 2 hours. The activation/mixing process is differentiated as mixing/activation at room temperature (28°C), with increased temperatures of either 60°C or 80°C. The mixing process to form mortar was continued by combining cement, sand, and superplasticizer. The mixture was stirred using an electric mixer and trowel for 2 minutes. The alkalinity of the mixture was measured using a pH meter and the obtained results were recorded after a 2-minute waiting period. To determine its workability, the mixture underwent workability testing according to ASTM C 1437.

Table 3. Mix Design

Binder		Water : Binder, or Alkali Solution : Binder
Cement	Fly Ash	(by mass)
100	0	0.2
60	40	0.2
40	60	0.2
20	80	0.15

Binder : sand (by mass) = 1 : 2
Superplasticizer = 0.176% (by mass binder)

4. Testing

The mixture was poured into a 5x5x5 cm cube mold while being vibrated using an electric motor-driven vibrator for 30 seconds. After 24 hours, the test specimens were removed from the mold and wrapped in plastic to prevent excessive evaporation. The samples were stored at room temperature (RT) (28°C, humidity 65%). Specimens that received high-temperature post-casting curing treatment were placed in an oven at 60°C or 80°C for 24 hours, then stored at room temperature (RT) (28°C, humidity 65%) until the next compressive strength test. Each mixture comprised 9 test specimens. Compressive strength was tested for each specimen (3 samples) after 7, 14, and 28 days, following ASTM C 109 standards. A summary of this methodology and testing can be viewed in Figure 4.

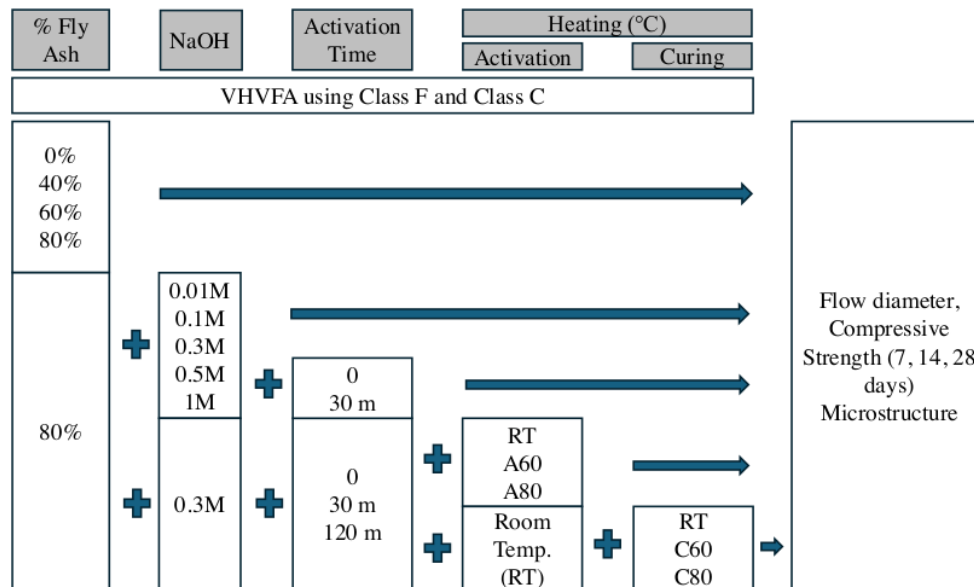


Figure 4. Experimental Design

5. Results and Discussion

From the fly ash used in this study, the first fly ash was identified as Class F, while based on the percentage of CaO content (10.49%), it falls into Class CI, according to the pH testing the alkalinity is 11.2. The other fly ash in this study was classified as Class C (19.12%), and Class CI is the same as the previous fly ash based on the CaO content (19.12%), and from the pH testing, a higher pH of 12.2 was obtained. From the two properties data above, it can be seen that the first fly ash (Class F) is less reactive than the second fly ash (Class C), which is consistent and correlates with its compressive strength test results (Shirkhanloo et al., 2021; Nassar & Room, 2023; Nazar et al., 2023; Wadhawan et al., 2023).

5.1. The workability of VHVFA Mortar Using Low-Molarity Alkali Solution and Thermal Activation

According to the test procedure for determining mortar flow (ASTM C230), D1 represents the diameter of the mortar immediately after the mold is removed, while D2 represents the diameter of the mortar after it has been struck. To determine the workability of mortar using low molarity alkali solution and thermal activation, testing of the mortar flow was conducted as shown in Table 4. Generally, the presence of fly ash in the mortar improves workability and reduces water demand. This occurs because the spherical shape of fly ash particles is smaller than that of cement particles, allowing them to fill the voids between larger particles more efficiently, thus creating denser packing. Such a microstructure results in a ball bearing effect, reducing friction between particles and potentially decreasing water demand. Therefore, overall, it enhances the microstructure in hardened mortar, these findings are consistent with previous research result (Babalu et al., 2023). Class F fly ash has a higher silica content and lower calcium oxide content, thus possessing better pozzolanic properties and fewer cementitious properties compared to Class C fly ash.

Using low molarity NaOH in VHVFA mortar increases workability by accelerating the pozzolanic reaction of fly ash. In the process of fly ash activation in high-volume fly ash (VHVFA) mortar, using low molarity alkali at higher temperatures, there is an increase in pozzolanic reaction. This increase enhances workability due to efficient leaching and the release of silica from fly ash for the formation of an amorphous aluminosilicate gel, which occurs more effectively. In summary, the use of low molarity alkali at elevated temperatures promotes better workability by optimizing the pozzolanic reaction and gel formation.

Table 4. Flow of Mortar

Fly Ash (%)	Water / NaOH	Activation		Class F			Class C		
		(min)	(°C)	D1	D2	pH	D1	D2	pH
				(cm)			(cm)		
0	Water	0	RT	11	17	12.5	-	-	-
40	Water	0	RT	11	18	11.5	11.5	18	12.5
60	Water	0	RT	13	20	11	13	19	12
80	Water	0	RT	13	20	10	12	20	11.5
80	0.01M	0	RT	13	20	10	12	16	11.5
80	0.1M	0	RT	13.5	20	10	13	18	12
80	0.3M	0	RT	14.5	22	10	14	20	12.5
80	0.5M	0	RT	14.5	23	10.5	14.5	22	13
80	1M	0	RT	15	24	10.5	14.5	22	13
80	0.01M	30	RT	13	20	10	12	16	11.5
80	0.1M	30	RT	13.5	20	10	13	18	12
80	0.3M	30	RT	14.5	22	11	14	20	12.5
80	0.5M	30	RT	14.5	22	11	14.5	22	13
80	1M	30	RT	15	23	11	14.5	22	13.5
80	0.3M	120	RT	15	22	11	15	21	12.5
80	0.3M	30	60	14	19	11	12	18	12.5
80	0.3M	30	80	14	18	11	12	18	12.5
80	0.3M	120	60	12	18	11.5	11	17	12.5
80	0.3M	120	80	12	18	11	11	17	12

5.2. The Compressive Strength of VHVFA Mortar without Activation

The effect of partially substituting cement with fly ash in the mortar was tested by replacing the cement content with Class F or Class C fly ash. Compressive strength testing was conducted on mortars using 100% cement and 40%, 60%, and 80% fly ash content at days 7, 14, and 28. In Figure 5, the compressive strength of mortars made with 100% cement and with different percentages of Class F and Class C fly ash can be observed. Generally, mortars using fly ash at different percentages showed an increase in compressive strength with age. At 7 days, the compressive strength of the mortar using 100% cement (as the control mortar) was 43.23 MPa, whereas the mortar using 80% Class F fly ash had a compressive strength of 21.49 MPa, almost half of the control mortar's strength. Class C fly ash contributes more to an increase in compressive strength compared to Class F fly ash due to its higher CaO content, promoting the formation of quartz and CSH, and its faster hydration reaction, resulting in more hydration products in a shorter time (Herath et al., 2020).

The tested samples with Class C fly ash contributed more to compressive strength than Class F fly ash. This can be seen in the compressive strength results for mortars at 7 and 28 days using 80% Class C fly ash compared to the control mortar using 100% cement. At the early age of 7 days, the compressive strength percentage of the mortar using 80% Class C fly ash was 75% (32.64 MPa) compared to the compressive strength of mortar using 100% cement (43.23 MPa). Like Class C fly ash, VHVFA mortar with Class F fly ash also increases for later ages (28 days). The percentage of compressive strength for mortars using 80% fly ash (43.83 MPa) at 28 days is 91% compared to the mortar using 100% cement (47.94 MPa). These findings are consistent in that Class C fly ash will develop compressive strength much better than Class F fly ash (McCarthy & Dyer, 2019) because the reactivity of fly ash is influenced by its constituent minerals and characteristics (Bapat, 2001). Previous studies indicated that a high percentage of fly ash inclusion will initially decrease compressive strength (Liu & Presuel-Moreno, 2014). Still, it has a beneficial effect on improving it at later ages due to the pozzolanic activity of the fly ash (Chandra et al., 2011; Saha, 2018; Lustosa & Magalhães, 2019; Amran et al., 2020).

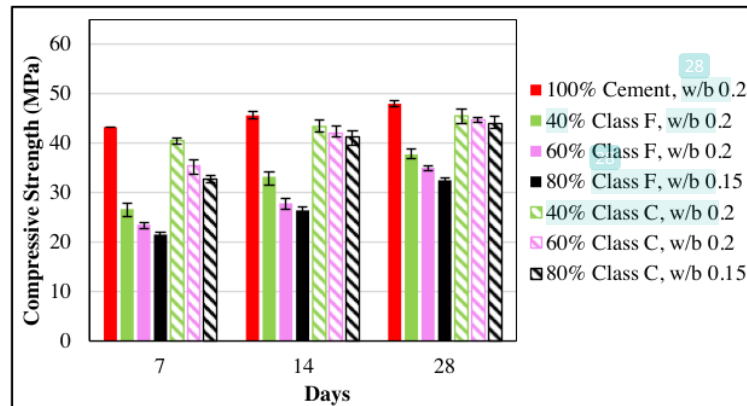


Figure 5. Compressive Strength of Mortar at Various Fly Ash Percentages

5.3. The Compressive Strength of VHVFA Mortar Activated with Low-Molarity Sodium Hydroxide Solution

In Figure 6, the compressive strength of VHVFA mortar (80% fly ash, 20% cement) using Class F and Class C fly ash is shown for 7, 14, and 28 days. The series of mortar mixtures was activated with NaOH solutions of 0.01, 0.1, 0.3, 0.5, and 1 M as chemical activators, and with water only. The use of NaOH solution as an activator with different low molarities on test specimens using either Class F or Class C fly ash results in a consistent increase in mortar strength with age. The increase of compressive strength in the VHVFA mortar using Class F fly ash activated with low-molarity NaOH is significantly noticeable at an early age (7 days). At 7 days, there is a 25% increase in compressive strength in the mortar activated using 0.5 M NaOH (26.78 MPa) compared to 21.49 MPa in the mortar with water instead of NaOH solution. At 28 days, the change in compressive strength becomes insignificant. The use of low-molarity NaOH as a chemical activator provides additional hydroxyl ions,

compensating for the low percentage of cement (20%) in the mixture. The hydroxyl ions obtained from NaOH react with silicates from the fly ash and form additional CSH (calcium silicate hydrate) in the mortar, thereby increasing its compressive strength. The results of this study are consistent with previous research where Class F fly ash would enhance its early compressive strength when activated with a low-molarity NaOH activator (Pratiwi et al., 2020)

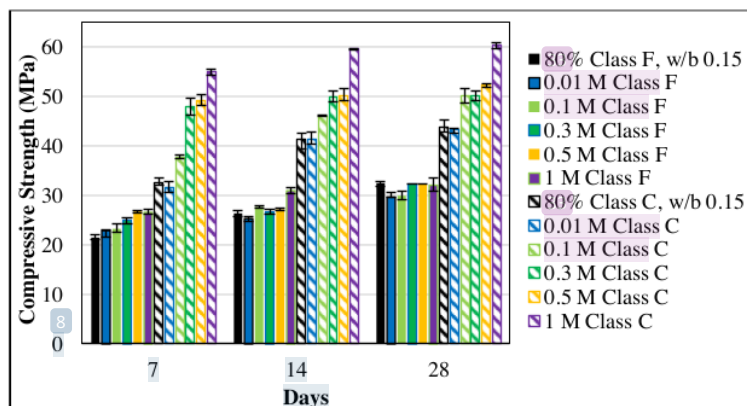


Figure 6. Compressive Strength of VHVFA Mortar Activated with Various NaOH Molarities

In the mortar using Class C fly ash, the use of low-molarity NaOH activator increases both early-age (7 days) and later-age (28 days) compressive strength, as observed in Figure 6. Chemical activation using NaOH with increasing molarities is seen to enhance the compressive strength of the tested mortar both at an early (7 days) and later (28 days) age. There is a 68% increase in compressive strength (to 54.92 MPa) compared to the control mortar (from 32.64 MPa) at the early age (7 days), exceeding the compressive strength of the mortar using 100% cement (43.23 MPa).

At a later age (28 days), the compressive strength of the mortar using 1 M NaOH activator (60.36 MPa) was 26% higher compared to the mortar using 100% cement (47.94 MPa) or 38% compared to the control mortar (43.83 MPa). In the more reactive Class C fly ash, the release of hydroxyl ions from NaOH to form additional CSH in the mortar continued even at a later age (28 days) although the rate of compressive strength increase is not as rapid as during the early age (7 days).

5.4. The Effect of Activation Time on Compressive Strength

Activation time refers to the time during which fly ash is mixed with water or an NaOH (alkaline) solution before being combined with other materials such as cement, sand, and superplasticizer. Sufficient activation time ensures that the fly ash in the mixture has reacted completely with water or the NaOH solution. If the activation time is too long, then the fly ash may set prematurely, interfering with the subsequent hydration process and reducing the compressive strength. The given activation time breaks down the silicate ions in the fly ash first. The effects of activation time (0 minutes and 30 minutes) conducted at room temperature (28°C) on the compressive strength of mortars activated using NaOH with low molarity (0.01, 0.1, 0.3, 0.5, and 1 M) can be observed in Figure 7 and Figure 8, which show the compressive strength achieved at 7, 14, and 28 days, the “-30” denotes 30 minutes of activation time.

As shown in Figure 7, the compressive strength of VHVFA mortar using Class F fly ash increased 7 days. The mortar activated for 30 minutes using a 0.5 M NaOH activator showed a 29% increase in compressive strength, from 21.49 MPa in VHVFA mortar without activation to 27.81 MPa; however, no significant change is observed at 28 days. The time of activation (30 minutes) compared to direct activation (0 minutes) did not show a significant influence on the compressive strength. The increase in compressive strength through pre-activation is consistent with the effects observed when the mortars were activated with NaOH solution only. The use of low-reactivity fly ash (Class F) compared to Class C fly ash in VHVFA mortar tested in this study over a short period

(30 minutes at room temperature) did not result in significant differences in its compressive strength enhancement because it requires a longer activation time and higher temperatures to comply with the Arrhenius law (Arjunan et al., 2001).

In Figure 8, it is illustrated that conducting compressive strength tests on VHVFA mortar, utilizing Class C fly ash alongside a low molarity NaOH activator, significantly amplifies the compressive strength of both the early (7 days) and later (28 days) age mortar specimens. The mortar activated for 30 minutes using a 1 M NaOH activator showed a 79% increase in compressive strength to 58.34 MPa at the early age (7 days) and 43% to 62.77 MPa (28 days) compared to the mortar without any activation from 32.64 MPa (7 days) and 43.83 MPa (28 days).

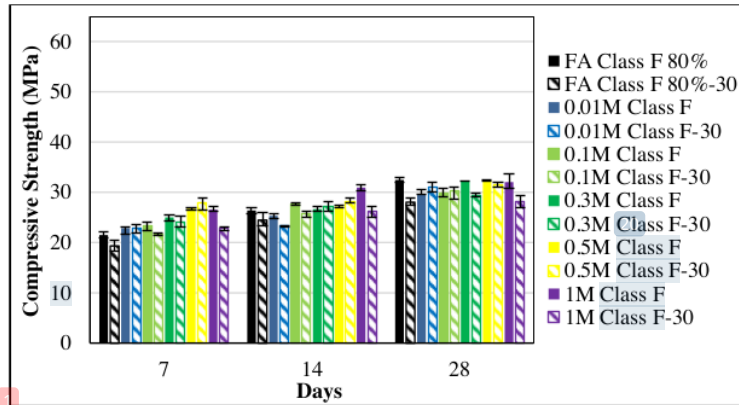


Figure 7. The Effect of Activation Time on Compressive Strength of VHVFA Mortar with NaOH Activator Using Class F Fly Ash

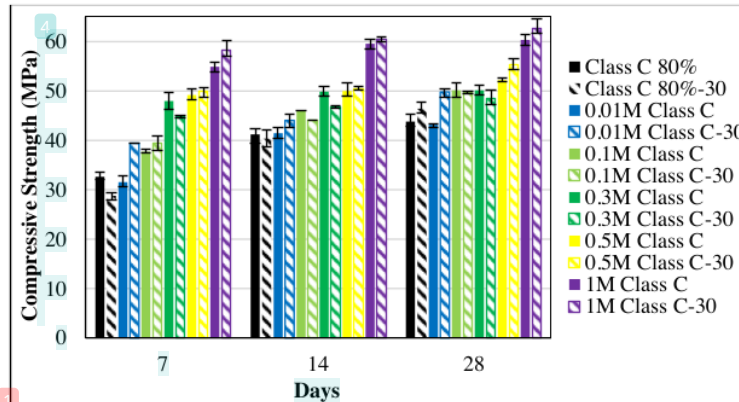


Figure 8. The Effect of Activation Time on Compressive Strength of VHVFA Mortar with NaOH Activator Using Class C Fly Ash

5.5. The Effect of Activation Time and Activation Temperatures on Compressive Strength in VHVFA Mortar with NaOH 0.3M

Testing the effect of activation time and activation temperature as well as activation time and curing at high temperature in VHVFA mortar using 0.3 M NaOH, refers to significant results from previous research (Pratiwi et al., 2020). The selection of 0.3 M NaOH molarity also considered its ease of implementation in the field and preliminary trials conducted earlier. This study utilizes a low-molarity alkali solution (0.3 M NaOH) in order to ensure that the reaction occurring is a hydration and not polymerization reaction as in geopolymers. The “-A60” denotes 60°C of activation temperature, “-A80” denotes 80°C of activation temperature.

In Figure 9 and Figure 10, the effect of activation time (0 minutes, 30 minutes, and 2 hours) and activation temperature (room temperature (RT), 60°C (A60), and 80°C (A80)) on VHVFA mortar activated with 0.3 M NaOH is shown for both Class F and Class C fly ash. Testing using Class F fly ash in VHVFA mortar activated for 2 hours at 60°C with 0.3 M NaOH as an activator (compressive strength of 29.07 MPa compared to 21.49 MPa without activation) resulted in a 35% increase in compressive strength at an early age (7 days) and a 12% increase (compressive strength of 36.28 MPa) at a later age (28 days) compared to VHVFA mortar without activation (compressive strength of 32.47 MPa).

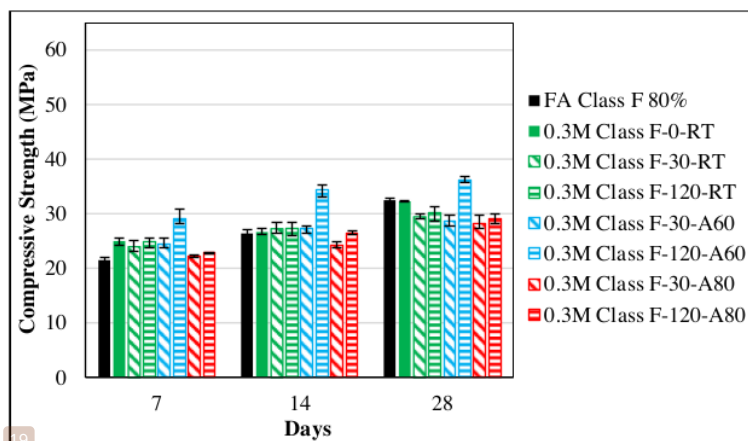


Figure 9. The Effect of Activation Time and Activation Temperature on Compressive Strength in VHVFA Mortar with NaOH 0.3 M Using Class F Fly Ash

Similarly, VHVFA mortar using Class C fly ash activated for 2 hours at 60°C consistently increased the compressive strength at all ages (7, 14, and 28 days). There was an 63% increase in compressive strength at an early age (compressive strength of 53.25 MPa from 32.64 MPa) and a 38% increase (from 43.83 MPa to 60.65 MPa) at a later age (28 days) compared to mortar without activation. Consistent with previous test parameters, the percentage increase in fly ash with alkali activation and thermal activation decreased at a later age (28 days) compared to an early age (7 days). Sodium hydroxide requires sufficient time to fully dissolve as an alkali activator before reacting with other materials. There is an optimum value for activation time and activation temperature in the mixture, which is influenced by the characteristics of fly ash and the mix design used. Excessive activation time and too high a temperature can have negative effects, leading to the premature setting of the mixture. Other studies have shown that activation for 2 hours at a temperature of 90°C resulted in a significant increase in compressive strength (Arjunan et al., 2001) while another study indicated a lower activation temperature of 60°C (Wang & Ishida, 2019). Insufficient activation time may result in an incomplete reaction of fly ash. Higher temperatures can increase the rate of chemical reactions in general, following the Arrhenius Law.

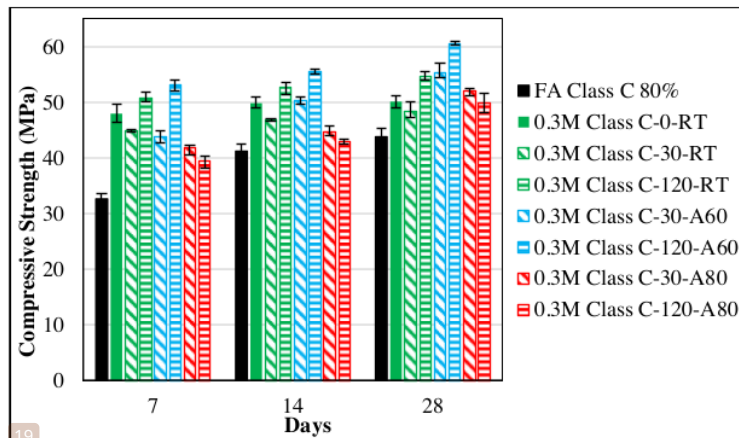


Figure 10. The Effect of Activation Time and Activation Temperature on Compressive Strength in VHVFA Mortar with NaOH 0.3 M Using Class C Fly Ash

5.6. The Compressive Strength of Activation Time and Curing at Higher Temperature in VHVFA Mortar with 0.3M NaOH

One of the testing parameters in this study is the curing process both at room temperature (RT) and higher temperatures. The curing process is the post-treatment of test specimens after the mold opens for a certain period. Sufficient water needs to be available in the mixture for complete hydration reactions to occur. The purpose of curing in the post-mixing and casting treatment of mortar is to prevent excessive loss of water from the mixture due to evaporation. According to the Arrhenius law, temperature is one of the factors affecting the rate of chemical reactions. Higher temperatures are required to stimulate the rate of hydration reactions in the mixture.

Testing of the samples was carried out to investigate the effect of post-casting sample curing for 24 hours and for samples activated immediately (0 minutes), after 30 minutes, and after 2 hours, at room temperature (28°C) and at higher temperature (60°C, and 80°C). Immediately after, all the test specimens were stored at room temperature until the compressive strength test. The results of the curing treatments and temperature on compressive strength are shown in Figure 11 and Figure 12. The results of the curing treatment on VHVFA mortar using Class F fly ash with 0.3 M NaOH as the chemical activator can be observed. There is no apparent increase in compressive strength in the test specimens at an early age (7 days) although there is a negative effect on compressive strength at a later age (28 days). The curing treatment that was conducted without adding water vapor but only elevating the temperature of the test specimens did not accelerate the reaction; instead, it caused the release of water vapor from the test specimens, which hinders the hydration process that requires water. The “-C60” denotes 60°C of curing temperature, “-C80” denotes 80°C of curing temperature.

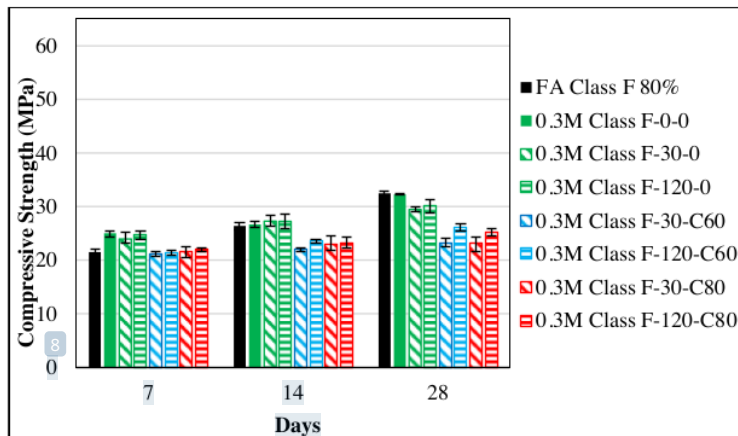


Figure 11. The Compressive Strength of Activation Time and Curing on Higher Temperature in VHVFA Mortar with 0.3 M NaOH Using Class F Fly Ash

The results of the curing treatment for 2 hours at 80°C on VHVFA mortar using Class C fly ash with 0.3 M NaOH as the activator can be observed. There is an increase in compressive strength at the early age (7 days) by 16% (from 47.97 MPa to 57.32 MPa) compared to the same mixture without curing at a higher temperature, and by 43% (from 32.64 MPa) compared to the mortar without any activation. In the testing for the later age compressive strength (28 days), a positive effect of the curing treatment is observed by 15% on a similar mortar without curing (compressive strength of 59.25 MPa from 50.15 MPa) and by 26% compared to the mortar without any activation (compressive strength of 43.83 MPa). The percentage of compressive strength development in the curing treatment decreases in the later age (28 days) from 43% to 26%. The curing treatment on Class C fly ash does not have the same effect as using Class F fly ash because Class C fly ash, due to its more reactive nature, undergoes further reactions during activation, reducing the evaporation effect during curing.

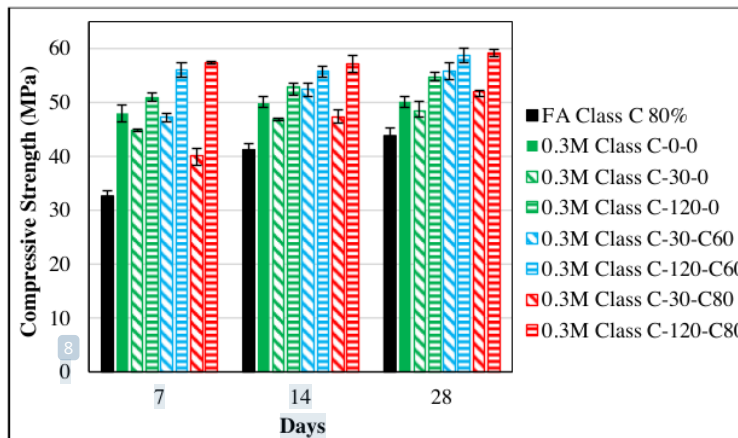


Figure 12. The Compressive Strength of Activation Time and Curing on Higher Temperature in VHVFA Mortar with 0.3 M NaOH Using Class C Fly Ash

5.7. The Effect of Low-Molarity Alkali Solution and Thermal Activation on Compressive Strength in VHVFA Mortar

In Figure 13, the effects of thermal activation and alkali activation treatments can be seen to yield the highest increase in compressive strength for both Class F and Class C fly ash in VHVFA mortars in this study. It is

observed that chemical activation using 0.3 M NaOH and an activation time of 2 hours on VHVFA mortar (20% cement and 80% Class F fly ash) can increase the compressive strength by 35% (29.07 MPa) compared to the mortar without any activation (20% Class F fly ash and 80% cement without additional activation) (21.49 MPa) at 7 days. The compressive strength is 67% for the mortar using 100% cement (43.23 MPa) at an early age (7 days). At a later age (28 days), chemical activation using 0.3 M NaOH and an activation time of 2 hours at 60°C on VHVFA mortar (20% Portland Pozzolana Cement and 80% Class F fly ash) can increase the compressive strength by 12% (36.28 MPa) compared to the mortar without any activation (20% Class F fly ash and 80% cement without additional activation) (32.47 MPa). The compressive strength is 76% for the mortar using 100% Portland Pozzolana Cement (47.94 MPa). The activation treatment with higher-temperature (60°C) activation for 2 hours and chemical activation using 0.3 M NaOH on these specimens can increase their compressive strength for both early age (7 days) and later age (28 days) mortar.

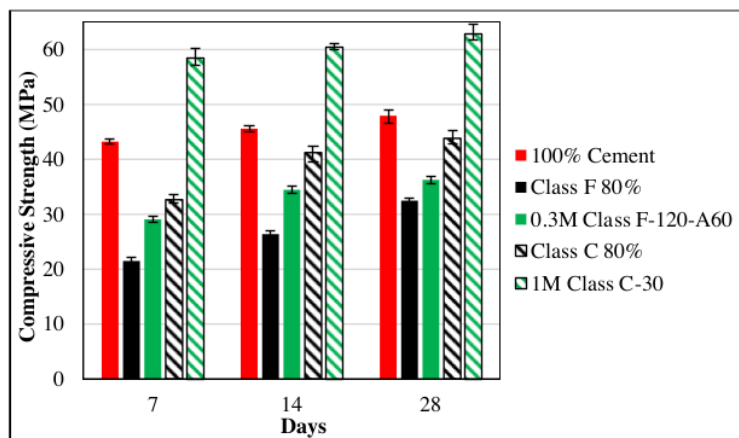


Figure 13. The Effect of Low Molarity Alkali Solution and Thermal Activation of VHVFA Mortar on Compressive Strength

In Figure 13, it is also observed that chemical activation using 1 M NaOH pre-activation 30 minutes at room temperature (28°C) on VHVFA mortar (20% cement and 80% Class C fly ash) can increase the compressive strength by 79% at 7 days (58.34 MPa) compared to mortar without any activation (20% Class C fly ash and 80% cement without additional activation) (32.64 MPa). The compressive strength is 35% higher than the mortar using 100% cement (43.23 MPa). This means that with this activation treatment, the compressive strength of the mortar surpasses that of the specimens made with 100% cement. At a later age (28 days), chemical activation using 1 M NaOH and activation for 30 minutes at room temperature (28°C) on VHVFA mortar (20% cement and 80% Class C fly ash) can increase the compressive strength by 43% (62.77 MPa) compared to mortar without any activation (20% Class C fly ash and 80% cement without additional activation) (43.83 MPa). This compressive strength is 31% higher than the mortar using 100% cement (47.94 MPa). The chemical activation treatment with 1 M NaOH and activation for 30 minutes at room temperature (28°C) on the specimens can increase the compressive strength for both early (7 days) and later (28 days) age mortar.

5.8. SEM Analysis of VHVFA Mortar Using Low Molarity Alkali Solution and Thermal Activation

The SEM images of VHVFA mortar using fly ash Class F and Class C (at 28 days) can be seen in Figure 14 and Figure 15. Figure 14(a) shows the fly ash Class F in VHVFA (very-high-volume fly ash) mortar without any activation while Figure 15(a) uses fly ash Class C. Significant results for both Class F and Class C fly ash on the mortar that has been further activated, shown in Figure 14(b) for fly ash Class F and in Figure 15(b) for Class C. As shown in Figure 14(b), it can be observed that the molecular structure of the fly ash Class F undergoes minimal damage caused by the hydration reaction of the cement in the mortar, activated using 0.3 NaOH for 2 hours at 60°C, which resulted in the highest compressive strength. This finding indicates a low reactivity level of this Class F fly ash may increase compressive strength when activated.

9 Unlike the Class F fly ash, the condition of the Class C fly ash in Figure 15(b) appears to have undergone further reaction as evidenced by significant damage to the fly ash particles in the VHVFA mortar activated for 30 minutes using 1 M NaOH at room temperature, which resulted in the highest compressive strength for the Class C fly ash samples (Salami et al., 2021; Xu et al., 2023; Shi et al., 2024).

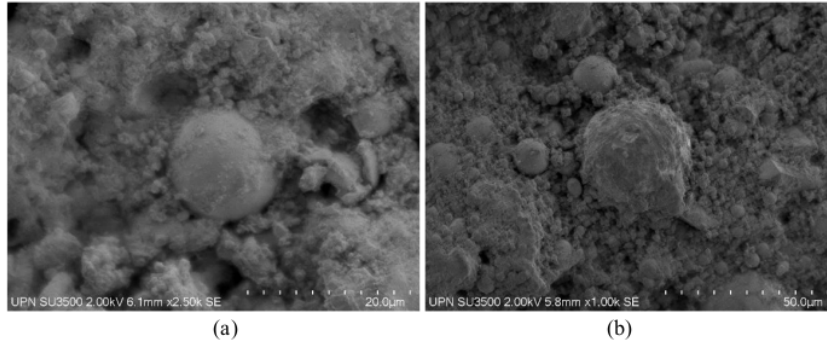


Figure 14. The SEM Images of VHVFA Mortar (80% fly ash and 20% cement) Using Fly Ash Class F, (a) without activation and (b) activated for 2 Hours, with 0.3M NaOH at 60°C

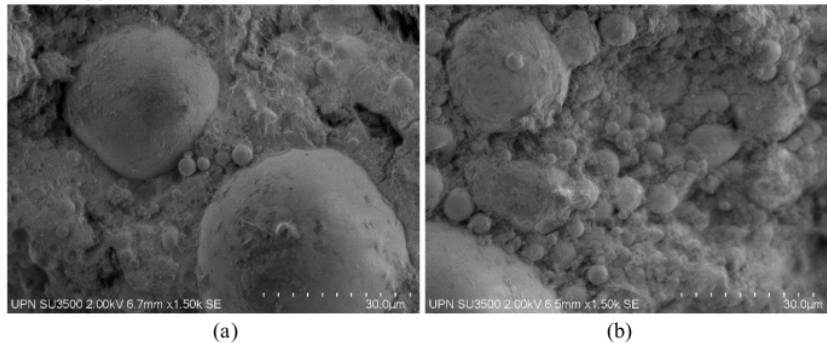


Figure 15. The SEM Images of VHVFA Mortar (80% fly ash and 20% cement) Using Fly Ash Class C, (a) without activation and (b) activated for 30 Minutes, with 1M NaOH at room temperature

6. Conclusion

6 This study reveals the potential use of VHVFA mortar in construction industry. From the experiment of very-high-volume fly ash (VHVFA) mortar (80% fly ash and 20% cement) with thermal activation treatment and chemical activation (low-molarity NaOH solution), some details conclusions are explained below:

1. Study shows that VHVFA mortar with thermal and chemical activation improves compressive strength significantly at 7 and 28 days for both Class F and Class C fly ash, more significant results with Class C fly ash.
2. Low-concentration alkali solutions (0.01 – 1 M NaOH) and thermal activation are more effective with Class C fly ash, enhancing its compressive strength and workability.
3. Chemical activation (0.3 M NaOH) for Class F fly ash at 60°C for 2 hours pre-activation time and 1 M NaOH for Class C fly ash for 30 minutes pre-activation time at room temperature significantly improve the compressive strength of VHVFA mortar.
4. Pre-activation times and temperatures affect the compressive strength of VHVFA mortar differently for Class F and Class C fly ash.

2 Declarations

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2

Declaration of Competing Interest

The authors declare no conflict of interest.

Data Availability Statement

The statements in the paper are properly cited in the manuscript and no additional data is available.

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