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# The strength properties of high calcium fly ash geopolymer specimens incorporating mud-volcanic and limestone variations

A Wardhono <sup>1</sup>, Y Risdianto <sup>1</sup>, M F Sofianto <sup>1</sup>, M T Nur <sup>1</sup> and A H Pradipta <sup>1</sup>

<sup>1</sup> Department of Civil Engineering, Universitas Negeri Surabaya, Indonesia

ariewardhono@unesa.ac.id

**Abstract.** The use of mud-volcanic and fly ash as cement replacement materials is one alternative solution to overcome environmental issues due to the mudflow disaster and pollution from cement production process. This paper objective is to identify the use of mud-volcanic and class C fly ash with high calcium content as waste materials in the development of fly ash geopolymer incorporating limestone. The variations of limestone to fly ash are 0.10:0.60, 0.2:0.5, 0.30:0.40, 0.40:0.30, and 0.50:0.20, respectively. The mud-volcanic ratio was kept at 0.30 to maintain the total weight of specimen at 1.00. The compressive strength was performed at 7, 14 and 28 days age, and the specimens were cured at ambient temperature. The depth penetration test was performed by vicat test. The results show that the highest strength value of 16.38 MPa was achieved by fly ash/mud-volcanic geopolymer with ratio of limestone to fly ash of 0.30:0.40. The initial setting time of 90 minutes indicates that the early reaction of fly ash/mud-volcanic geopolymer shows a comparable setting time to the normal concrete with initial setting time of 45 minutes. These results indicate that fly ash and mud-volcanic can be used as cement replacement materials.

## 1. Introduction

The volcanic mudflow disaster since 2006 in Sidoarjo Indonesia has caused material losses for the affected environment. The volcanic mudflow continues to occur to this day and it has reached the area of impact affected around 10 square kilometers and a depth of 30 meter [1-3]. The use of volcanic mudflow as a basic material for construction materials has been carried out to overcome the large volume of mud-volcanic [4-6]. The investigation results of previous studies indicate that the mud-volcanic has a high silicate, aluminate and ferrite content [7].

The use of cement, also known as Portland Cement (PC), as concrete-based material has been widely used as construction materials. However, the cement production process has a significant issue to the environment [8, 9]. The production of 1 ton of cement also delivers 0.7 to 1 ton of carbon dioxide (CO<sub>2</sub>) gas, which is one of the main cause of global warming problems [10]. However, the global cement production has grown rapidly in recent years. It is one of the largest emissions source of anthropogenic CO<sub>2</sub> gas [11]. The use of cement replacement materials is required to be encouraged to cope with this issue. One alternative solution is to utilize fly ash, a waste material from coal combustion process, as cement replacement materials [12, 13].

The utilization of fly ash as a fully cement replacement material has been extensively investigated. Fly ash as a precursor material is combined with an alkali activator solution, a mix of sodium hydroxide



(NaOH) and sodium silicate, and it is known as geopolymer [14, 15]. The basic reaction of geopolymer is a polymer reaction between silicate and aluminate formed a Si-O-Al- matrix through a geopolymerization process. However, the main issue of using geopolymer is the requirement of high curing temperature to accelerate the geopolymer reactions due to the low calcium content in fly ash material [16, 17].

This paper reports the utilization of mud-volcanic and fly ash as waste materials in the development of fly ash-based geopolymer mortar. Fly ash with high calcium compound was used as the primary material of geopolymer specimens. The mud-volcanic from the volcanic mudflow disaster in Sidoarjo, Indonesia, was used for this research. The variations of limestone was implemented to investigate the fly ash/mud-volcanic geopolymer behavior. The addition of limestone was applied to overcome the high temperature issue during the geopolymer curing process. The strength properties of geopolymer specimen was identified by a compressive strength at the age of 28 days. The depth penetration test (Vicat test) was performed to investigate the setting times of geopolymer specimens.

## 2. Research methodology

### 2.1. Materials

Fly ash from a coal power plant Indonesia was used as a geopolymer specimen primary material. It has a high ferrite (54.67%), low silicate (13.28%) and aluminate (4.82%) contents. It is categorized class C fly ash with high CaO content of 16.24% (> 10%) and the total content of  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  is 72.77% (> 50%) in accordance with ASTM C618 [18]. Mud-volcanic materials were collected from the disaster of mud eruption in Sidoarjo, Indonesia, while limestone material was available from the market material distributor. All materials chemical compositions were determined by X-Ray Fluorescence (XRF) test using PANalytical test equipment. The chemical compositions of fly ash, mud-volcanic, and limestone are listed in Table 1.

**Table 1.** Chemical composition of fly ash, mud-volcanic, and limestone (mass %).

Materials	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	K <sub>2</sub> O	TiO <sub>2</sub>	Mn <sub>2</sub> O <sub>3</sub>
Fly ash	13.28	4.82	54.67	16.24	1.38	0.86	0.51
Mud-volcanic	26.64	8.02	46.17	8.57	3.35	2.25	0.61
Limestone	-	-	0.34	96.12	-	-	-

The solution of alkaline activator was prepared by mixing sodium silicate and 10 Molar sodium hydroxide (NaOH). The 10 Molar NaOH solution was produced by dissolving 400 grams of NaOH pellet to 1 liter of water. The sodium silicate to NaOH ratio of 1.5 was applied for this research.

### 2.2. Mix design

The details of fly ash/mud-volcanic geopolymer (FAMVG) mortar mix design are listed in Table 2.

**Table 2.** Mix proportions of fly ash/mud-volcanic geopolymer (FAMVG) mortars

Mixture	Portland Cement	Lime stone	Fly ash	Mud-Volcanic	Fine aggregate	Sodium silicate	NaOH 10 M	Water
PC	1	-	-	-	3.0	-	-	0.45
FAMVG1	-	0.10	0.60	0.30	3.0	0.212	0.141	-
FAMVG2	-	0.20	0.50	0.30	3.0	0.212	0.141	-
FAMVG3	-	0.30	0.40	0.30	3.0	0.212	0.141	-
FAMVG4	-	0.40	0.30	0.30	3.0	0.212	0.141	-
FAMVG5	-	0.50	0.20	0.30	3.0	0.212	0.141	-

The total fine aggregate in geopolymer mortars was maintain to 75% by volume for all mixtures, while the total weight of binder (fly ash, limestone, mud-volcanic, and activator solution) was kept to 25.3%. The density of fine aggregate, fly ash, and mud-volcano was 2.604, 2.895, and 2.533, respectively, while the water solid ratio (w/s) of 0.21 was applied.

### 2.3. Curing models

Control mortar specimen was cured at ambient temperature (24°C - 30°C) before tested. In general, a geopolymer specimen is required to be cured at high temperature, however due to the inclusion of limestone, a room temperature curing process was applied for FAMVG mortars.

### 2.4. Testing mortar specimens

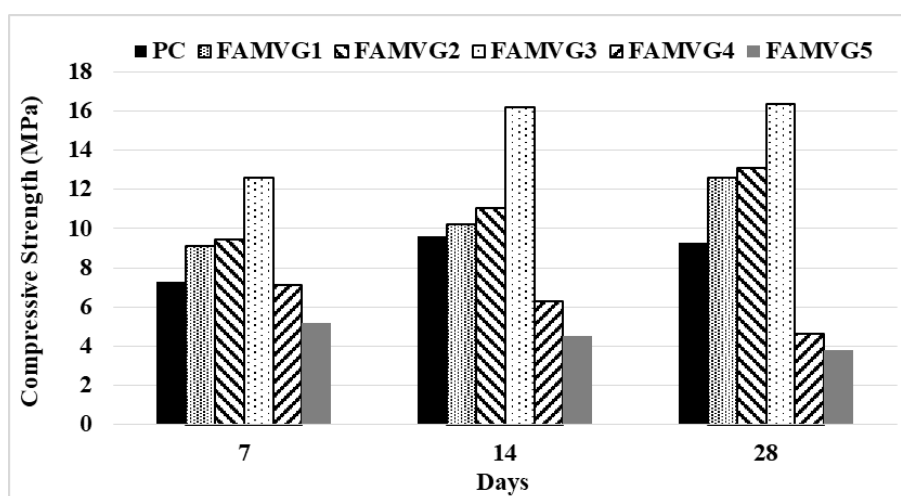
A cube steel mold of 5 x 5 x 5 cm<sup>3</sup> is used to make the FAMVG mortar specimens. The strength performance of FAMVG specimens was performed by compressive strength and vicat tests. The test of compressive strength was carried out at 7, 14, and 28 days in accordance with ASTM C109 [19]. The depth penetration test (Vicat test) was undertaken in accordance with ASTM C191 [20] to identify the setting time of FAMVG paste as the results of mud-volcanic and limestone variations.

## 3. Results and discussion

The strength performance of FAMVG specimens for all mixtures are shown in Table 3 and Figure 1. All FAMVG mortar specimens demonstrate a better strength performance compared to PC control except for FAMVG4 and FAMVG5 throughout 28 days.

**Table 3.** Compressive strength of FAMVG mortars

Mixture	Compressive strength (MPa)		
	7 days	14 days	28 days
PC	7.30	9.60	9.30
FAMVG1	9.10	10.24	12.58
FAMVG2	9.45	11.04	13.10
FAMVG3	12.64	16.20	16.38
FAMVG4	7.10	6.30	4.60
FAMVG5	5.20	4.50	3.80



**Figure 1.** The strength performance of fly ash/mud-volcanic geopolymer mortars

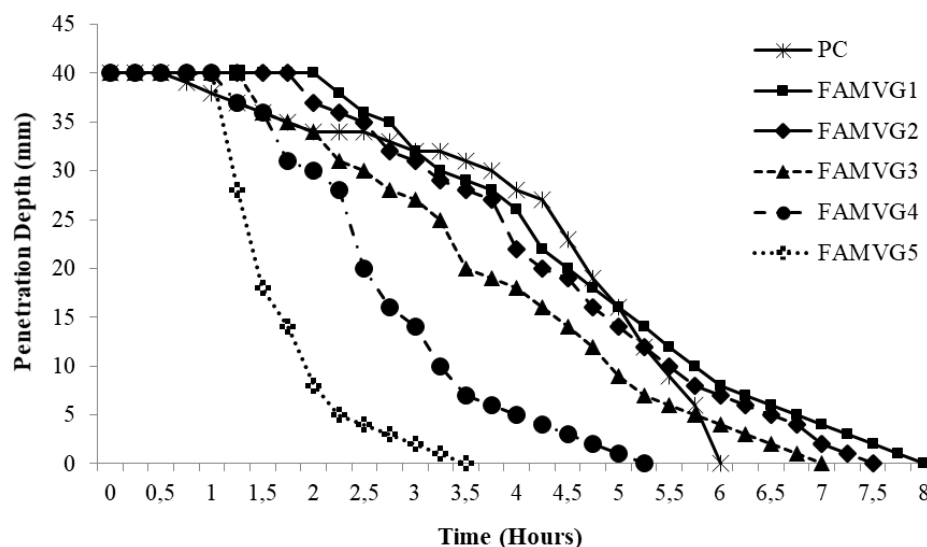
The highest early strength (7 days) of 12.64 MPa was shown by FAMVG3 (30% limestone) compared to the PC control with the strength merely of 7.30 MPa. The FAMVG3 also exhibits the

highest strength throughout 28 days with the strength of 16.20 MPa and 16.38 MPa at 14 and 28 days, respectively. A comparable compressive strength was also performed by FAMVG2 with the initial and final strength of 9.45 MPa and 13.10 MPa, respectively. Furthermore, FAMVG1, FAMVG2, and FAMVG3 geopolymer mixtures demonstrates a significant improvement in strength along with time.

In addition, inclusion of limestone more than 30% tends to lessen the strength of FAMVG specimens. FAMVG4 (limestone 40%) and FAMVG5 (limestone 50%) mortar specimens exhibit a lower strength performance compared to all mixtures. FAMVG5 specimen with the strength of 5.20 MPa and 3.80 MPa achieves the lowest compressive strength at 7 days and 28 days, respectively. Moreover, the higher addition of limestone tends to hinder and decrease the strength development of FAMVG specimens. The addition of 40% limestone decreases the strength of FAMVG4 specimen by 35.2% from 7.10 MPa at 7 days to 4.60 MPa at final strength. Further addition of limestone by 50% in FAMVG5 decreases the strength by 26.9% from 5.20 MPa to 3.80 MPa. Similar finding was found by [6], the author found that the addition of limestone more than 30% on fly ash-based geopolymer specimen using mud-volcanic as sand replacement material tends to reduce the strength performance.

Indeed, the addition of limestone improves the strength performance of fly ash/mud-volcanic geopolymer mortars as shown in FAMVG1, FAMVG2, and FAMVG3. The geopolymer reaction involves the primary reaction between silicate and aluminate to form geopolymer matrix [21]. However, due to the low aluminate compound in fly ash raw material, it causes un-reacted silicate during the geopolymerization reaction [22]. The addition of limestone increases the calcium content and drive a reaction between calcium and silicate. This reaction produces a calcium-silicate-hydrate (C-S-H) compound similar to the traditional concrete, which improves the strength performance of FAMVG specimens.

However, a high limestone inclusion tends to reduce the strength performance as shown in FAMVG4 and FAMVG5 specimens. It might attributes to the excess calcium compound, which does not react with silicate. This excess calcium forms a calcium hydroxide compound which leads to a concrete crack due to the material expansion [23]. This leads to the strength reduction throughout the time. Furthermore, the inclusion of limestone significantly governs the setting time rate of fly ash/mud-volcanic geopolymer specimens as shown in Figure 2.



**Figure 2.** The depth penetration test fly ash/mud-volcanic geopolymer mortars

Based on the results of vicat test, the standard setting time (initial and final) rate of normal concrete (PC control) was 0.75 hours and 6 hours, respectively. The fly ash/mud-volcanic geopolymer tends to

have a longer initial and final setting rate. The FAMVG1 with 0% limestone addition has initial and final setting time rate of 2.25 hours and 8 hours, respectively. Despite fly ash/mud-volcanic geopolymer can be made at ambient temperature, it requires a longer time to achieve its structural integrity. The inclusion of limestone significantly affects the setting time rate. The inclusion of 10% limestone affected the initial setting time rate from 2.25 hours to 1.25 hours and 8 hours to 7.5 hours for the final setting time. FAMVG3 specimen demonstrates a similar setting time rate with the initial and final setting time of 1.5 hours and 7 hours. The fastest setting time rate shows by FAMVG5 with limestone addition of 50%. It has initial setting time of 1.25 hours and final setting time of 3.5 hours. The accelerate setting time rate might be caused by two reaction, a reaction between silicate and aluminate (Si-O-Al-) and reaction between calcium and silicate (C-S-H).

#### 4. Conclusion

This research deals with the strength properties of high calcium class C fly ash/mud-volcanic geopolymer with variations of limestone. Based on the findings, it can be concluded that:

- A mix of high calcium, mud-volcanic, and limestone can be used as cement replacement material in geopolymer specimen due to the similarity compositions of silicate and aluminate.
- The addition of limestone in fly ash-mud volcanic geopolymer significantly affects the strength performance. The 30% inclusion of limestone gives the highest strength of 16.38 MPa at the final 28 days.
- High addition of limestone significantly decreases the strength performance of fly ash-mud volcanic geopolymer throughout the time due to the un-reacted excess calcium.
- The setting time rate of fly ash-mud volcanic geopolymer governs by the inclusion of limestone. It accelerates the setting time (initial and final) rates, which affects the hardening in room curing temperature.
- High inclusion of limestone at 50% significantly increases the setting time rate of fly ash-mud volcanic geopolymer in 60 minutes at final setting time. However, it tends to decrease the strength due to the difficulty during the casting process.
- The use of fly ash and mud-volcanic is one alternative material for cement replacement in construction material. It also helps to overcome the high volume of mud volcanic problem and global warming issue cause by cement production.

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