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# Fresh and Hardened Properties of High Calcium Fly Ash-Based Geopolymer Matrix with High Dosage of Borax

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## Abstract

Geopolymer is synthesized by mixing material rich in alumina and silica, such as fly ash, with a highly alkaline solution to form a hardened matrix. However, when using high calcium fly ash as a precursor, a flash set frequently occurs, i.e., the mixture hardens very rapidly before casting can be completed. The use of borax as an additive has been reported due to its potential to prolong the setting time. In this study, the use of a significantly higher dosage of borax is explored. The results show that the addition of borax up to 20% of fly ash, by mass, into the alkaline solution prolongs the setting time by up to 90 min. Conversely, the addition of higher amounts of borax tends to decrease the compressive strength of the geopolymer, whereas adding a small amount of calcium oxide into the mixture increases the strength marginally, especially when the borax content is small. However, the amount of calcium oxide should be limited because at higher content, the effectiveness of borax to prolong the setting time is reduced.

**Keywords** Geopolymer · High calcium fly ash · Workability · Flash set · Borax · Calcium oxide · Compressive strength

## 1 Introduction

The use of fly ash as cement substitute material has become common in the construction world, especially for partial substitution (Huang et al. 2013; Rashad 2015). The application of fly ash can be increased even further when fly ash is used as the main binder by geopolymeric reaction. Fly ash, either with low calcium (class F) or high calcium (class C) content, is a material rich in silica and alumina, and can be used as a precursor in making geopolymer. The activation of the source material occurs in the presence of a highly alkaline solution, commonly a combination of sodium hydroxide and sodium silicate (Wattimena et al. 2017; Yildirim et al. 2011). The ratio and content of the two alkalis determine the characteristics of the geopolymer concrete (Arioz et al. 2012; Hardjito et al. 2004; Mustafa et al. 2011; Rangan 2010; Rattanasak and Chindaprasirt 2009; Thunuguntla and Gunneswara Rao 2018).

The use of high calcium fly ash as a source material results in a high-strength geopolymer (Antoni et al. 2017;

Chindaprasirt et al. 2007; Guo et al. 2010). The geopolymerization and hydration reactions may occur concurrently in the matrix when high calcium fly ash is used (Antoni et al. 2016a; Li et al. 2013). The high calcium fly ash used had similar calcium content with ground-granulated blast-furnace slag (GGBFS), which can be beneficial in increasing the compressive strength of the geopolymer (Sethi et al. 2018). Both reactions can improve the quality of the resulting matrix. However, the use of high calcium fly ash has a shortcoming because high calcium content can cause a flash set with insufficient time to work with the mixture. The fresh mixture hardens only 5–7 min after the addition of alkaline solution (US Patent no US8202362B2 2012). Mixing fly ash classes F and C can slow down the setting time. However, it may cause a decrease in the concrete's compressive strength (Boonserm et al. 2012). The application of sucrose as a retarder in the geopolymer is one of the possibilities; however, there is a strength reduction tendency with added sucrose (Assi et al. 2018; Kusbiantoro et al. 2013). Other efforts to prolong the setting time of high calcium fly ash geopolymer have been done previously with less success (Antoni et al. 2017; Topark-Ngarm et al. 2015).

Adding a small amount of borax with dosage within the range of 1–7% by mass of fly ash was found to deter the occurrence of flash setting marginally; however, the

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initiative confirms the potential of borax to be used as a retarder in the geopolymeric system. Initial setting time is increased with the increase in borax dosage (Antoni et al. 2016b). However, the extension of setting time was found to be insufficient. The initial setting time was extended up to 25 min with 7% borax. This period allows the production of research specimens but is not enough time for casting fresh geopolymer for a large-scale application. The use of borax in a geopolymer mixture, aside from extending the setting time, was also shown to increase the compressive strength marginally (Nazari et al. 2014; Nicholson et al. 2005). The addition of borax containing boron element was shown to produce a good quality geopolymer (Taveri et al. 2018).

Meanwhile, the addition of CaO (calcium oxide) to geopolymer concrete may increase the compressive strength of concrete (Diaz et al. 2010; Temuujin et al. 2009). Temuujin et al. (2009) showed that by adding calcium oxide to the fly ash, the compressive strength of geopolymer concrete was increased. The calcium oxide replacement of about 1–3% of fly ash by mass was shown to increase the strength of the geopolymer. Nevertheless, there was no discussion about its influence on the setting time of the geopolymer mixtures.

In this study, the effect of the addition of borax and calcium oxide on the fresh and hardened properties of the high calcium-based geopolymer mortar was investigated. Higher dosages of borax, from more than 7% and up to 20% of fly ash, were added into the geopolymer mixture to prolong the setting time. Furthermore, CaO was added to simulate the use of fly ash with higher pH and to explore the possibility to increase the compressive strength. If the rapid setting time problem can be solved, then a huge amount of high calcium fly ash currently available can be utilized as precursor for producing geopolymer, thereby creating another alternative to recycle this waste to become a high-quality and green construction material.

## 2 Materials and Methods

High calcium fly ash was obtained from a pulverized coal combustion power plant in Paiton, East Java, Indonesia. This study utilized two samples of fly ash (denoted as FA1 and FA2) that were taken at different times. Two rapid indicator tests, i.e., pH and percentage of mass retained on #325 sieve tests (Antoni et al. 2015), and an X-ray fluorescence (XRF) test were performed on both fly ash samples. The pH was measured using two methods according to ASTM D5239 (2012) and Davidovits (2008, p. 394). Particle fineness was determined using sieve #325 (45  $\mu\text{m}$ ) with sieve shaker for 10 min. The sand used was from a local quarry in Lumajang, East Java, Indonesia. Borax used was in the form of sodium tetraborate pentahydrate. Calcium oxide (CaO) used was sieved, passing sieve #30 (0.6 mm), and retained at sieved

#50 (0.3 mm). The alkaline solution used in this study was sodium hydroxide (NaOH) flake and sodium silicate type 52. The ratio of sodium silicate solution to sodium hydroxide solid was set as 2.5, by mass. The NaOH solution was 8 M.

The composition of the mixtures and their corresponding coding are shown in Table 1. The sand-to-fly ash ratio for all mixtures was fixed at 2, and the water-to-fly ash ratio was set at 0.25. Hence, a 1 m<sup>3</sup> mixture would require 680 kg fly ash, 1380 kg sand in saturated surface dry condition, 54.4 kg NaOH solid in 170 kg water, and 135 kg of sodium silicate solution for F1B0 and F2B0 mixtures. The use of borax was calculated as an addition into the alkaline solution to the percentage of fly ash, by mass, while the calcium oxide was calculated as a fly ash partial replacement, by mass.

Sodium hydroxide solution was prepared the day before use and mixed with sodium silicate before mixing with the dry mix of fly ash and sand. When borax was used, the borax flakes were added to the sodium hydroxide solution, while it was still warm. For all specimens, curing was performed in the oven at temperature of 60 °C for 24 h. The compressive strength test was conducted at 7, 14, and 28 days, with three replications. The initial and final setting times were measured on the geopolymer paste, i.e., mortar mixture without any sand, using a Vicat needle (ASTM C191 2004), in room environment.

## 3 Results and Analysis

### 3.1 Fly Ash Composition

The fly ash used in this study was analyzed in advance to determine its composition and chemical properties. The results of rapid analysis of fly ash using pH and fineness measures are correlated with the XRF measurement. The outcomes of the rapid analysis are reported in Table 2. Determination of pH was carried out using two methods. The difference between the two measurements is the ratio of fly ash to the aqueous solution. It is shown that the pH level of fly ash used is around 11. This indicates that the added fly ash has a high probability of causing rapid

**Table 1** Coding for mixture composition of the geopolymer mortar

|           | Fly ash 1 (FA1) | Fly ash 2 (FA2) | Fly ash 2 (FA2)<br>partly replaced with<br>3% CaO (FA2C3) |
|-----------|-----------------|-----------------|---|
| Borax 0%  | F1B0            | F2B0            | F2C3B0  |
| Borax 5%  | F1B5            | F2B5            | F2C3B5  |
| Borax 10% | F1B10           | F2B10           | F2C3B10   |
| Borax 15% | F1B15           | F2B15           | F2C3B15   |
| Borax 20% | F1B20           | F2B20           | F2C3B20   |

**Table 2** Rapid evaluation of the fly ash quality

| Material | pH in accordance to ASTM D5239 (20 FA/80 H <sub>2</sub> O) | pH in accordance to Davidovits (10 FA/100 H <sub>2</sub> O) | Fly ash particles retained on #325 (%) |
|----------|--|---|--|
| FA1      | 11.3   | 11.2  | 12                                     |
| FA2      | 11.6   | 11.5  | 8                                      |
| FA2C3    | 12.3   | 12.1  | –                                      |

or flash setting (Davidovits 2008). Replacing 3% of FA2 with calcium oxide increased the pH from 11.6 to 12.3. The particles of the fly ash were found to be fine, with about 88–92% passing sieve #325, which well exceeds the requirement of the ASTM C618 Standard of 65% passing sieve #325 (ASTM C618 2010).

The results of the XRF test are reported in Table 3. Both fly ash samples were found to be high calcium fly ash (class C), with total content of SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub> of 67.8% and 66.29% for FA1 and FA2, respectively, and the CaO level of more than 10% and with a very low loss on ignition (LOI) value. FA2 has higher CaO content than FA1. This difference in CaO content is indicated from the pH of the two fly ash samples, i.e., FA2 shows higher pH than FA1. Although the MgO content was quite high, the result from Li (Li et al. 2019) suggested it could have beneficial effect in reducing the shrinkage.

**Table 3** Chemical composition of the fly ash used

| Component | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | TiO <sub>2</sub> | CaO   | MgO  | K <sub>2</sub> O | Na <sub>2</sub> O | SO <sub>3</sub> | MnO <sub>2</sub> | P <sub>2</sub> O <sub>5</sub> | LOI  |
|-----------|------------------|--------------------------------|--------------------------------|------------------|-------|------|------------------|-------------------|-----------------|------------------|-------------------------------|------|
| FA1       | 35.46            | 16.91                          | 15.43                          | 0.75             | 16.98 | 7.23 | 1.32             | 2.83              | 1.72            | 0.18             | 0.26                          | 0.40 |
| FA2       | 34.29            | 16.62                          | 15.38                          | 0.73             | 18.18 | 7.52 | 1.35             | 2.97              | 1.63            | 0.17             | 0.25                          | 0.36 |

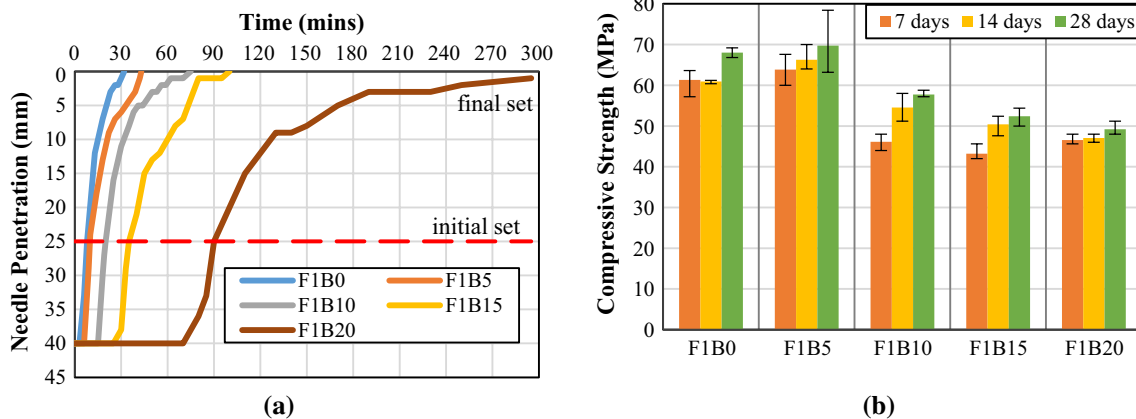
### 3.2 Setting Time and Compressive Strength

Setting time for each mixture composition of geopolymer paste was measured using the Vicat needle apparatus. Setting time testing was carried out at room temperature (33–35 °C). The compressive strength test was performed on 5 cm mortar cube specimens with three replications for each testing age of 7, 14, and 28 days.

Figures 1, 2, and 3 show the results of the Vicat needle penetration with time and the compressive strength test results for FA1, FA2, and FA2C3, respectively. Calculations of the initial and final setting times of the geopolymer paste are shown in Table 4.

It is shown that the setting time of geopolymer paste was extended significantly with the increase in borax added to the alkaline solution. Without borax addition, the initial setting time was measured at 8, 6, and 5 min for FA1, FA2, and FA2C3, respectively. These are very fast setting times that hardly leave any time for the geopolymer to be cast. The faster setting time was found to be correlated to the higher calcium oxide content in the mixture (Antoni et al. 2016c), although there might be an effect from the room temperature. The setting time of geopolymer tends to be shorter in warmer climates (Antoni et al. 2016b). The use of 5% borax only marginally extended the initial setting time. A previous study reported the use of up to 7% borax in the mixture and showed a similar result (Antoni et al. 2016c).

The addition of 10% borax extended the initial setting time by up to 17–20 min, making the geopolymer more



**Fig. 1** a Vicat needle penetration at the geopolymer paste using FA1 and b the compressive strength test for FA1 mortar with various amounts of added borax

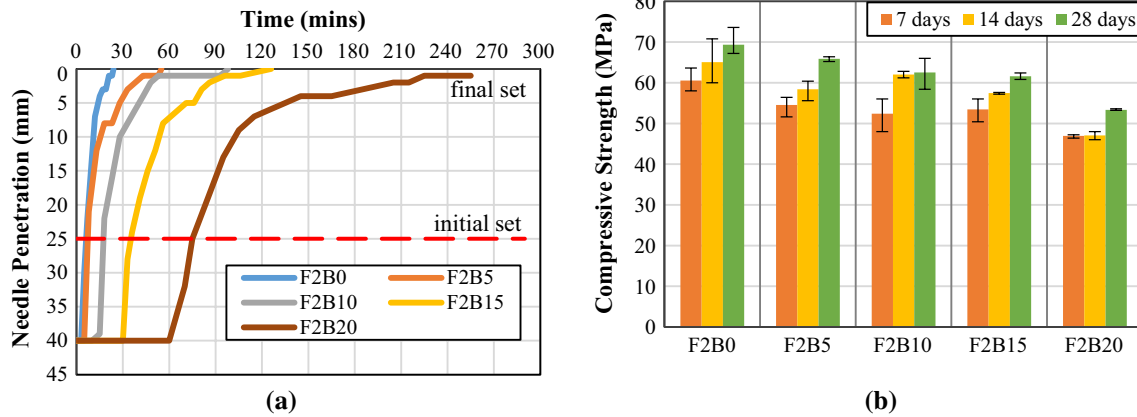


Fig. 2 a Vicat needle penetration at the geopolymer paste using FA2 and b the compressive strength test for FA2 mortar with various amounts of added borax

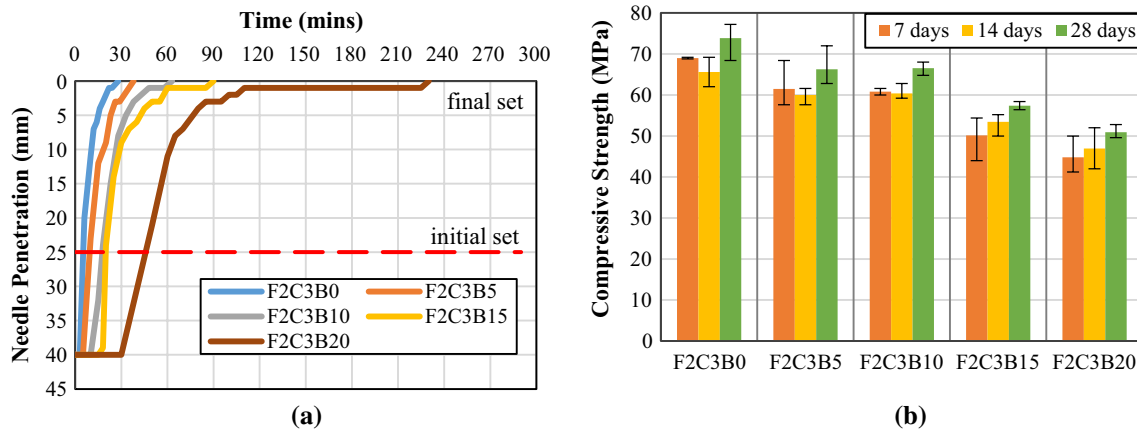


Fig. 3 a Vicat needle penetration at the geopolymer paste using FA2C3 and b the compressive strength test for FA2C3 mortar with various amounts of added borax

Table 4 Initial and final setting time of the geopolymer paste

| Borax (%) | Initial setting time (min) |     |       | Final setting time (min) |     |       |
|-----------|----------------------------|-----|-------|--------------------------|-----|-------|
|           | FA1                        | FA2 | FA2C3 | FA1                      | FA2 | FA2C3 |
| 0         | 8                          | 6   | 5     | 32                       | 24  | 28    |
| 5         | 10                         | 7   | 9     | 43                       | 55  | 38    |
| 10        | 20                         | 17  | 18    | 75                       | 98  | 63    |
| 15        | 35                         | 35  | 20    | 100                      | 126 | 90    |
| 20        | 90                         | 75  | 46    | NA                       | NA  | 230   |

manageable to cast a small volume of mixture without fear of flash setting. At 15% borax, the initial setting time was extended further and at 20%, the initial setting time was extended up to 90, 75, and 46 min for the paste with FA1, FA2, and FA2C3, respectively.

At 20% borax in FA1 and FA2, it was observed that the paste did not experience the final set for up to 24 h. This

could be due to the higher dosage of borax addition which causes retardation of the mixture reaction and, therefore, takes longer to harden.

Adding 3% CaO into the mixture enabled the fresh geopolymer paste with FA2C3 to undergo a final set in about 4 h, whereas the other two mixtures were not fully set after 24 h. The high pH of the fly ash increased the reaction rate

of the geopolymer paste. Geopolymer paste with FA2C3, with highest pH, showed the shortest initial setting and final setting time even at higher borax dosage.

The addition of 5% borax in FA1 (F1B5) is shown to marginally increase the compressive strength of geopolymer mortar compared with F1B0. However, further dosage addition showed a decrease tendency in the compressive strength. The compressive strength of geopolymer mortar was reduced from 69 MPa to 57 MPa for F1B5 to F1B10. Further addition of borax more than 10% reduced the compressive strength further.

On FA2, the addition of borax reduced the compressive strength of geopolymer mortar compared with those without any borax. Geopolymer mortar samples of F2B5, F2B10, and F2B15 showed lower rates of decrease in compressive strength, whereas there was a significant drop in compressive strength from 61 MPa to 53 MPa for F2B15 to F2B20 samples. This shows the limit of borax usage in the geopolymer mixture. A similar trend was observed on FA2C3. The use of borax reduced the compressive strength; however, a significant drop of compressive strength is observed at 15% borax (F2C3B15). This drop in strength was not observed in the previous study due to lower borax dosage (Antoni et al. 2016c), and its strength reduction was not as pronounced as found by Nicholson et al. (Nicholson et al. 2005). The effect could be due to the difference in mixture composition and fly ash material.

The correlation between the amount of borax addition to the compressive strength and the initial setting time of geopolymer mortar mixtures is shown in Fig. 4. Increasing the borax dosage significantly prolonged the setting time of fresh geopolymer mixture. However, concurrently, this caused a reduction in the compressive strength. The initial setting time of FA2C3 was shown to diverge at 15% borax content. This shows that with the higher calcium content in fly ash, the addition of borax is no longer effective in increasing the setting time.

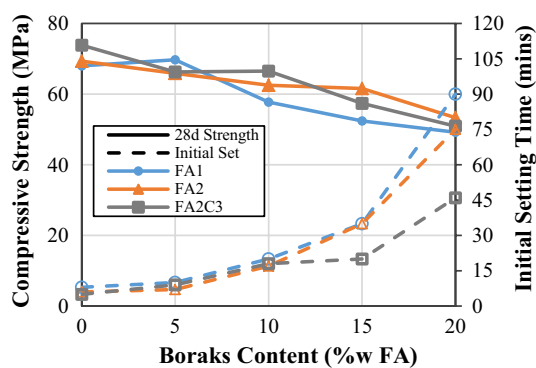


Fig. 4 Effect of borax content on compressive strength and initial setting time

Calcium oxide replacement in FA2C3 was shown to cause higher compressive strength compared with FA2 when no borax is used. The higher strength could be due to higher geopolymeric reaction rate due to the heat released during mixing and the availability of calcium compound that could react with the fly ash. According to Temuujin et al. (2009), the addition of CaO to a geopolymer mixture reduces the compressive strength of mortar when cured at 70 °C. However, the use of CaO in this study increased the compressive strength of geopolymer mortar when no borax was used. Temuujin used 14 M NaOH solution, whereas this study used 8 M NaOH. A high level of NaOH molarity may interfere with the reactivity of CaO.

FA2C3 shows a noticeably higher compressive strength compared with FA2 for lower borax dosage of up to 10%. However, at higher dosage of 15% and 20%, the compressive strength of FA2C3 is lower compared with the ones of FA2. This could be due to the faster setting time of mixtures with lower borax content. High dosage of borax compound in the mixtures delays the geopolymeric reaction.

The effectiveness of borax addition in the geopolymer mixture to prolong the initial setting time seemingly depends on the calcium content and, therefore, correlates with the pH of the fly ash. Figure 5 shows the initial setting time plotted against the pH of the fly ash. The higher the pH of the fly ash, the faster the setting times of the geopolymer mixture. The effectiveness of borax addition in increasing the setting time of geopolymer mixtures is reduced with the higher pH of the fly ash used as a precursor for the geopolymer. Partly replacing fly ash with a small amount of CaO increases the pH of the mixture and, thus, will accelerate the setting time. Consequently, this will cause the addition of borax to deter the flash setting of geopolymer mixture to become less effective.

The addition of borax at high dosage reduces the compressive strength of high calcium fly ash-based geopolymer mortar. This creates a trade-off between the fresh properties against the hardened properties of geopolymer. Sufficient

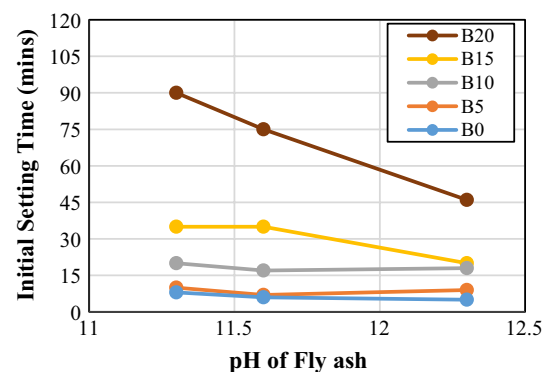


Fig. 5 Influence of pH on setting times with various borax percentage

initial setting time needs to be ensured in making geopolymer from high calcium fly ash. This should be achieved at the lowest possible borax dosage addition to ensure the attainment of good compressive strength. The upper limit of borax addition can be considered as the highest amount that does not reduce the compressive strength of the geopolymer considerably compared with those without borax.

### 3.3 Microstructures

The geopolymer microstructures were analyzed by observing micrographs of the samples obtained from the compressive strength specimens. Scanning electron microscope–EDX Carl Zeiss Evo MA10 was used at various magnification factors. Figure 6 shows micrographs of F2B0 at 1000 $\times$  and 3000 $\times$  magnification. It is shown that the microstructure of geopolymer matrix consisted of a dense structure with

unreacted fly ash distributed in the matrix. The degree of the reactivity of the fly ash can be correlated with the area of the fly ash particles still visible after geopolymeric reaction. The inert fly ash remains as unreacted sphere particles, while the reactive fly ash particles are dissolved in the alkaline solution and condensed as geopolymer matrix. F2B0 specimen has a high compressive strength, which is attributed to the high volume of condensation of the geopolymer matrix.

Figure 7 shows the influence of borax addition on the matrix of the geopolymer of FA2. At 10% and 20% borax addition, the majority of the geopolymer matrix was similar to F2B0 but with higher amount of artifacts of other materials. It was also shown that F2B20 has a higher number of small pores or air voids trapped within the geopolymer matrix.

The microstructure of FA2C3 geopolymer matrix is shown in Fig. 8. The microstructure of F2C3B0 (Fig. 8a)

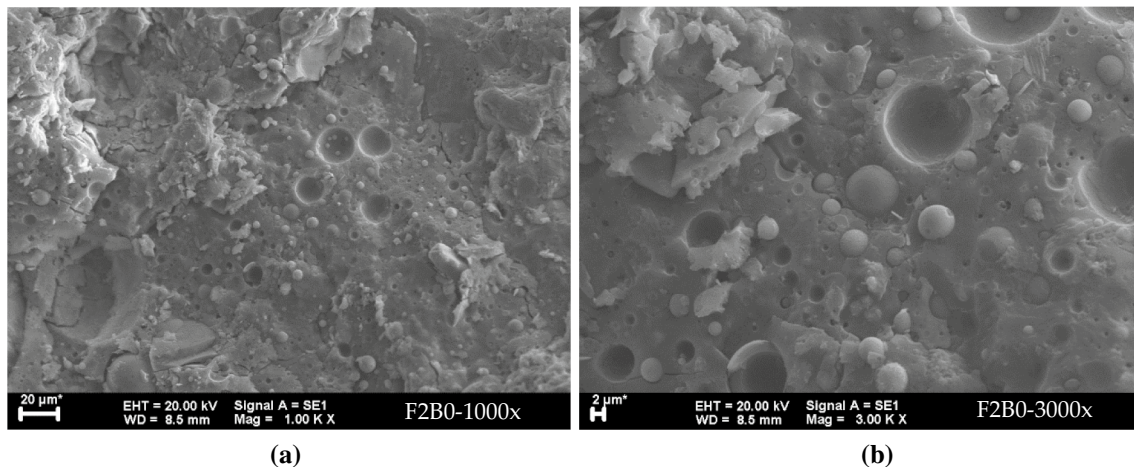


Fig. 6 SEM micrographs for F2B0 geopolymer at **a**  $\times 1000$  and **b**  $\times 3000$  magnification

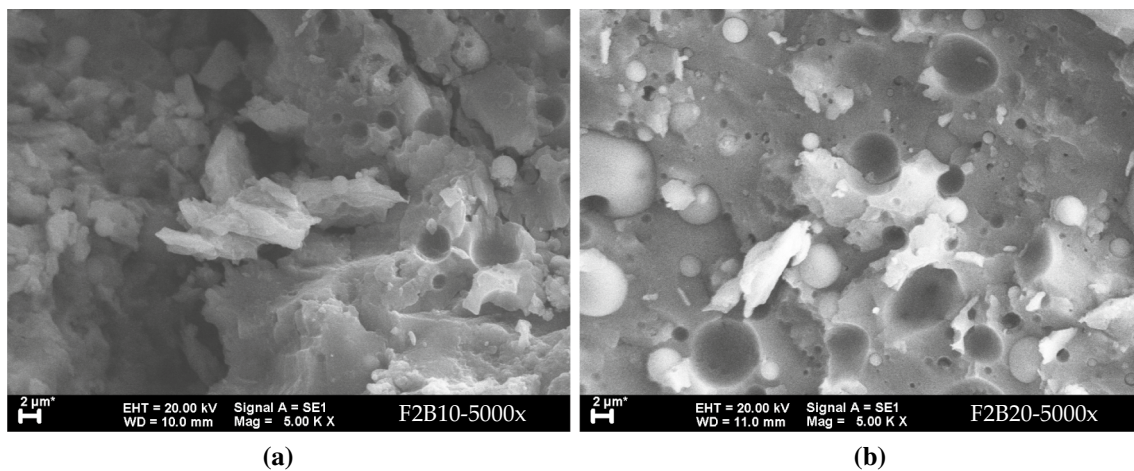
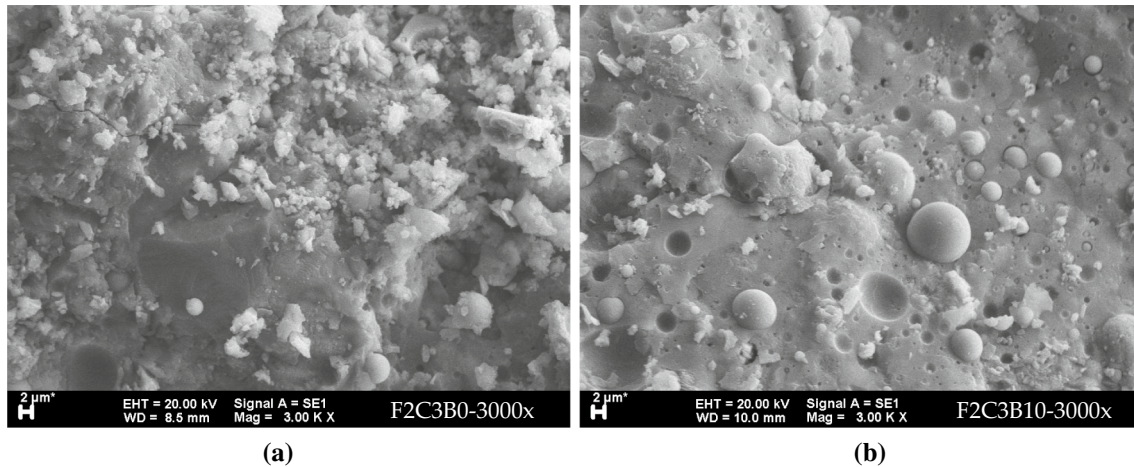


Fig. 7 SEM micrographs for **a** F2B10 and **b** F2B20 geopolymer at  $\times 5000$  magnification





**Fig. 8** SEM micrograph for **a** F2C3B0 and **b** F2C3B10 geopolymer at 3000× magnification

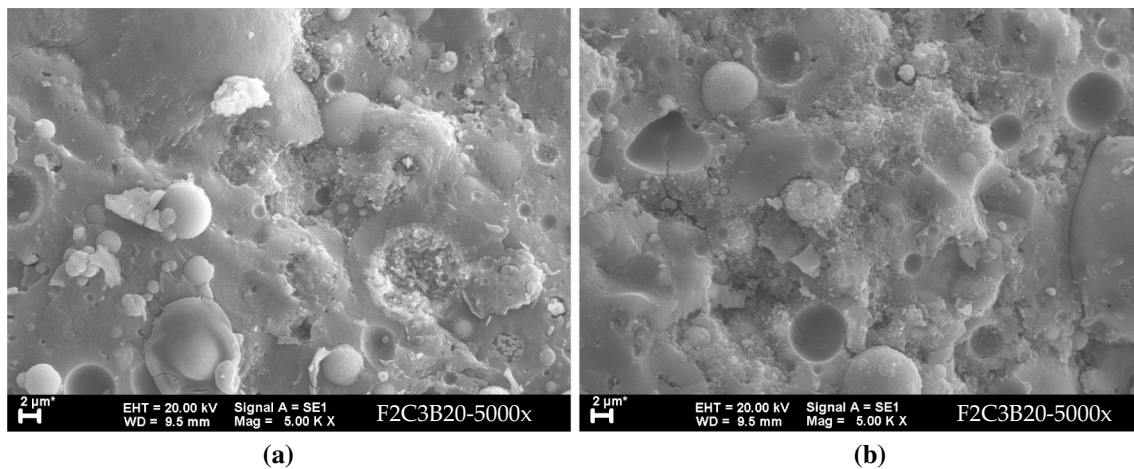
reveals some crumbly powders that are not similar to the other geopolymer matrices. These particles could be the result of a reaction between fly ash and calcium oxide that increases the compressive strength of the geopolymer matrix compared with F2B0. The F2C3B10 matrix also shows a high volume of air voids in the geopolymer matrix. This condition is similar to the one of F2B20 as shown in Fig. 7. The presence of these air voids could be the reason for the lower compressive strength of geopolymer mortar at higher borax concentration.

Figure 9 shows the microstructure of F2C3B20 mortar at two locations. The failure plane of F2C3B20 matrix looks less smooth compared with those of previous specimens. Lower compressive strength of the mortar could be due to the weak interaction between hydration products and the geopolymer matrix.

## 4 Conclusions

Based on the results of this study, the following conclusions can be drawn:

1. Increasing the amount of borax in high calcium fly ash-based geopolymer prolongs the setting time of fresh mixture significantly and deters the occurrence of flash setting. The use of 20% borax in high calcium fly ash-based geopolymer matrix resulted in initial setting time of about 75–90 min, whereas the control mixture without borax resulted in flash setting.
2. Adding up to 20% borax to the fly ash-based geopolymer decreases the compressive strength of the mortar by up to 30% compared with the control geopolymer without



**Fig. 9** SEM micrograph for F2C3B20 geopolymer at ×5000 magnification at locations (a) and (b)

any borax. Thus, the trade-off of fresh and hardened properties needs to be considered.

- The addition of small amounts of CaO increases the compressive strength of high calcium fly ash-based geopolymer mortar marginally when borax is not used. However, it also speeds up the setting time and reduces the effectiveness of borax to prolong the setting time.
- The microstructures of geopolymer matrix using high calcium fly ash reveal a combination of geopolymer and hydrates products as a result of pozzolanic reaction between fly ash and calcium oxide.

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### Compliance with Ethical Standards

**Conflict of interest** On behalf of all authors, the corresponding author states that there is no conflict of interest.

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