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# Simplified assessment for one-part 3D-printable geopolymer concrete based on slump and slump flow measurements

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# ABSTRACT

A combination of three-dimensional (3D) printing and one-part geopolymer concrete is an innovative and sustainable construction method to prevent environmental degradation. To perform well in a 3D printing application, one-part geopolymer concrete requires rheological specifications that will generate concrete that is printable. However, measuring rheological properties and interpreting the rheological results is time-consuming. It needs to have a standardized field-friendly method to assess the printability of one-part geopolymer concrete before printing. This study introduces a simplified way of predicting the printability of one-part geopolymer concrete for 3D printing with the use of a slump and slump flow diagram. Nineteen (19) mixtures of one-part geopolymer concrete with various water/binder ratios and different content of sodium tetraborate pentahydrate (borax) added were studied. The slump and the corresponding slump flow of these concrete mixtures were measured. Then, the actual printings were carried on to assess the printability of these mixtures. The open time of concrete was also determined. After 3 days of curing, the compressive strength of the concrete was evaluated. The investigation indicated that the mixtures with a slump in a range of between 15 mm and 30 mm accompanied by a slump flow of between 210 mm and 240 mm were found to be well-suited for 3D printing. Furthermore, the results figured out that to achieve a desirable open time for successful 3D printing, the borax content of 2–4% should be added while the appropriate water/ binder ratio of higher than 0.34 should be used.

## **1. Introduction**

Concrete is well known as the most widely used material in the construction field. With the tremendous growth in infrastructure and accommodation, it involves an increase in concrete and cement production annually [\[1\]](#page-10-0). Consequently, it leads to the overexploitation of natural resources and strongly affects various environmental issues [\[2\].](#page-10-0) Amongst various sectors in the construction field, the cement industry has been considered the major contributor to greenhouse emissions [\[3\].](#page-10-0) It releases approximately 8–9% of the global greenhouse gas emission [\[3\].](#page-10-0) Therefore, the cement industry imposes a great threat to environmental degradation, air

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pollution, and global climate change. Reduction of greenhouse gas emissions in construction during this period of development has been a huge challenge for academic researchers and policymakers [\[3,4\]](#page-10-0). Great efforts have been dedicated in the last decades to devise environmentally friendly binders that are potential alternatives to Portland cement in construction [\[5\]](#page-10-0) or methods to reduce the use of clinker in the cement industry [\[6,7\].](#page-10-0) Amongst various alternative binders, geopolymer has been widely accepted as the most feasible one  $[8,9]$ . Geopolymer is produced by the reaction between aluminosilicate precursors such as fly ash, ground granulated blast furnace slag, metakaolin, and alkaline activators which are usually sodium hydroxide, calcium hydroxide, potassium hydroxide, or sodium silicate. Many studies concluded that geopolymer concrete can provide comparative-or-better engineering properties and performances than concrete made from ordinary Portland cement  $[10-13]$  $[10-13]$  while the geopolymer emits little greenhouse gas and demands lower energy needed for manufacturing  $[4,5]$ . However, the inconsistent properties of precursors, difficulty in preserving alkaline activators, and low strength at the early age are primary causes that hinder the widespread application of geopolymer in the construction industry, where cast-in-situ concrete still plays an imperative role. Recently, great attention has been paid to one-part geopolymer, where the alkaline activator is in the solid form [\[14\].](#page-10-0) Therefore, the mixing of geopolymer concrete is similar to cement concrete, making it more promising to be used widely.

On the other hand, 3D concrete printing is an innovative and advanced generation of sustainable methods in the construction industry recently [\[15,16\].](#page-10-0) It possesses many advantages over traditional construction technology including labor intensity reduction, an increase in construction efficiency, and safety construction as well as improvement in architectural freedom creation [\[15,16\]](#page-10-0). Furthermore, this technology can help to reduce construction wastes and virtually eliminate the use of formwork materials, which account for 50% of construction cost and 70% of construction time [\[15,16\]](#page-10-0). Therefore, it helps reduce global solid wastes, and prevent environmental degradation as well as greenhouse gas emission due to the production of formworks [\[15,16\].](#page-10-0) 3D printing technology can be subdivided into different techniques according to their respective material concepts, equipment, and production steps [\[16\]](#page-10-0). Amongst these techniques, extrusion-based 3D concrete printing has been the most promising approach [\[16\]](#page-10-0). In this technique, concrete is extruded layer-by-layer via a gantry or a robotic arm in the absence of rigid molds. For satisfactory 3D application, the concrete mixture must satisfy certain fresh-state properties including pumpability, extrudability, and buildability [\[17,18\]](#page-10-0). From the rheological perspective, the printable concrete mixture must possess low viscosity during extrusion and high yield stress after deposition [18–[20\].](#page-10-0) Furthermore, the concrete mixture should have a sufficient open time, which is the interval time that fresh materials maintain good workability for printing [\[21\]](#page-10-0).

Recently, the combination of geopolymer concrete and 3D printing technology has been focused upon [22–[24\]](#page-10-0). The rheological properties of the geopolymer mixture were studied to evaluate the printability of geopolymer concrete. Based on that, the printable thixotropic zone was assessed while the open time was also determined [\[22\]](#page-10-0). It was pointed out that, the mixtures that were satis-factory for 3D printing required to have a minimum threshold limit level of 10000 [\[23,24\].](#page-10-0) Furthermore, the bonding strength between extruded layers strongly depended on the printing gap time between consecutive layers, the printing speed, and the nozzle standoff height [\[23,24\].](#page-10-0) The rheological properties of geopolymer mortar and Portland cement mortar were also compared. It was concluded that Portland cement mortar was preferable to thixotropic recovery behavior compared to geopolymer mortar, probably due to the absence of colloidal interaction in the geopolymer [\[23,24\].](#page-10-0) Furthermore, the type of alkaline activator solution,  $SiO_2/Na_2O$  ratio, and viscosity of the activator were imperative factors affecting the open time and buildability of the mixtures. The results reflect that the lower  $SiO_2/Na_2O$  ratio of the Na<sub>2</sub>SiO<sub>3</sub> solution resulted in the longer setting time and open time of the mixture, which is attributed to the lower rate of the geopolymerization process [\[25\]](#page-10-0).

3D printing technology using one-part geopolymer concretes has begun to draw attention lately [25–[27\].](#page-10-0) Noticeably, the precursors investigated in the one-part geopolymer mixture were usually a blend of fly ash and ground granulated blast furnace slag. Numerous studies indicate that the proper ground granulated blast furnace slag content in the fly ash-slag mix should be approximately 40–50% while about 8–15% of sodium metasilicate powder occurred to be appropriate content to activate the slag and fly ash [\[23,](#page-10-0) 25–[27\]](#page-10-0). Furthermore, it was observed that geopolymer activated with sodium metasilicate powder showed greater workability and lower yield stress than that of sodium hydroxide activated system. However, one-part geopolymer had a short setting time, which greatly hindered the printability of the concrete mixture [\[28\].](#page-10-0) Although some researchers have suggested that sodium tetraborate pentahydrate (borax) Na2B4O7.5H2O can help retard the setting time of geopolymer [\[29\]](#page-10-0), there are still few studies that can give an appropriate assessment of the effect of these components in retarding the setting time of geopolymer concrete.

Noticeably, most previous research assessed the printability of concrete via viscosity, yield stress, time-dependent performance, and the thixotropic behavior of the mixture. These parameters are measured and interpreted from a laboratory rheometer. However, the aforementioned tests and result interpreting are time-consuming. More to this point, interpreting rheological measurements is relatively complicated [\[30\]](#page-10-0) and rheometers may be inaccessible, particularly in the construction field, where the printability of concrete mixture should be assessed right before printing. Therefore, those parameters are not suitable to be taken place on each batch of concrete mix to simply give an appropriate judgment on whether the mixture can be printable to guarantee the quality of the structure. Finding a set of simple tests that are readily available in the construction field and can give an appropriate judgment about the printability of a concrete mixture is essential. In this research, standardized field-friendly tests including the slump and slump flow test were proposed to estimate the printability of concrete mixture for 3D printing applications. The rheological properties of materials can be evaluated by plastic viscosity and yield stress which can be assessed by rotational rheometers based on the Bingham model [\[31\]](#page-10-0). The simple linear Bingham model is expressed in Eq.  $(1)$ :

$$
\tau = \tau_o + \mu.\gamma \tag{1}
$$

Where: *τ* is shear stress.

*γ* is shear rate.

*τo*is dynamic yield stress.

In standardized field-friendly tests proposed, the slump value is related to the solid state of the microstructure, static yield stress and so buildability of the mixture, while the slump flow value is deemed as directly connected with dynamic yield stress and pumpability of the mixture [\[32\].](#page-10-0) With this simple and standardized method, the printability of the mixtures can be predicted in advance.

Due to the reasons above, the main aim of this study is to simplify the assessment process for the printability of 3D printing geopolymer concrete by using the slump and slump flow test. The geopolymer used was a one-part geopolymer where the precursors consisted of 50% fly ash and 50% ground granulated blast furnace slag. These precursors were then activated by 10% of sodium metasilicate. A printable region of one-part geopolymer concrete was also established, based on the slump-flow diagram.

It is noteworthy to mention that fresh geopolymer concrete is to lose its workability with time through early hydration. This loss can affect the rheological properties and printability of fresh concrete. The open time of fresh mixture, which is defined as the period that the fresh material still maintains good workability for printing, is one of the main measures to verify the printability of the mixture. Increasing the water/binder ratio can certainly improve the workability of concrete and reduce the loss of workability. However, it can lead to the reduction of the yield stress of concrete, making it to be unable to build. Besides, borax is able to extend the setting time of geopolymer, helping the geopolymer mixture to maintain its workability, as aforementioned. Nevertheless, there have been still few studies focused on this issue. Therefore, in this research, the effect of borax and water/binder ratio on the open time of one-part geopolymer concrete was also studied to evaluate the printability of one-part geopolymer concrete.

## **2. Experimental**

### *2.1. Materials and mix proportion*

Low calcium fly ash and ground granulated blast furnace slag were used as precursors to produce a one-part geopolymer binder. The chemical compositions of fly ash and slag are shown in Table 1. The particle distribution of fly ash and ground granulated blast furnace slag used were shown in [Fig. 1.](#page-3-0)

Sodium metasilicate pentahydrate granular Na<sub>2</sub>SiO<sub>3</sub>.5H<sub>2</sub>O with 29.5% Na<sub>2</sub>O and 28.5% SiO<sub>2</sub> served as activator was mixed with precursors while sodium tetraborate pentahydrate (borax) Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>.5H<sub>2</sub>O was added to adjust the setting time of one-part geopolymer binder. River sand had a maximum particle size of 2 mm and a fineness modulus of 2.1 was used to produce one-part geopolymer concrete with a sand/binder ratio of 1.5. After prescreening, a total of 19 concrete mixes with different water/binder ratios and borax contents, as presented in [Table 2](#page-3-0), were used in this research for pre-selecting printable region.

To prepare one-part geopolymer concrete, all powder components including fly ash, slag, sodium metasilicate pentahydrate, and sodium tetraborate pentahydrate (borax) were mixed with high shear mixer at 68 rpm for 3 min. Then three-fourths of water was added to the mixture and mixed for 3 min. Finally, the remaining water was added to the mixture following 5 min of mixing at 200 rpm.

#### *2.2. Slump and slump flow test*

The slump and flow slump tests were performed immediately upon the completion of mixing. The test procedure was carried out according to Tay et al. [\[32\]](#page-10-0). Two 25 mm layers of mortar were poured into a conical mold described in American Society for Testing and Materials (ASTM) C230 [\[33\]](#page-10-0) and each layer was tamped 20 times with the tamper. The top surface was flattened before the mold was then lifted. The difference in the height between the mold and the mortar is defined as the slump. Subsequently, the table was dropped 25 times in 15 s. The slump flow was recorded as the average diameter of the mortar along four lines as scribed on the tabletop.

The slump and slump flow of one-part geopolymer concrete were measured every 10 min until the slump flow was less than 160 mm. The prescreening test has pointed out that since the slump flow was below 160 mm the concrete was dry and unable to be used for printing.

### *2.3. Compressive strength*

**Table 1** 

After measuring the slump and slump flow, the cubic specimens of one-part geopolymer concrete were cast with a size of  $50\times50\times50$  mm to evaluate the strength development at 3 days. The specimens were then cured under ambient conditions. After 3 days, the compressive strength of the specimens was measured in accordance with ASTM C109/C109M-20 [\[34\]](#page-11-0).

Chemical composition of fly ash and ground granulated blast furnace slag (wt%).

|         |                  | $\sim$                 | $\sim$                         | $\sim$<br>____ |      |                 |        |            |      |
|---------|------------------|------------------------|--------------------------------|----------------|------|-----------------|--------|------------|------|
|         | SiO <sub>2</sub> | $\mathrm{Al_{2}O_{3}}$ | Fe <sub>2</sub> O <sub>3</sub> | CaO            | MgC  | SO <sub>3</sub> | $K_2O$ | Other<br>. | LOI  |
| Slag    | 36.70            | 12.61                  | 0.22                           | 47.32          | 2.05 |                 |        | 1.99       | 0.93 |
| Fly ash | 52.86            | 34.19                  | 3.85                           | 2.57           | 2.10 | 0.48            | 2.59   | 0.66       | 1.30 |

<span id="page-3-0"></span>

**Fig. 1.** Particle distribution of fly ash and ground granulated blast furnace slag.

**Table 2**  Mixing proportion of one-part geopolymer mixtures.

| Mixture ID     | Binder        |              |                           | Retarder-borax (wt% of binder) | Sand/binder | Water/binder |  |
|----------------|---------------|--------------|---------------------------|--------------------------------|-------------|--------------|--|
|                | Fly ash (wt%) | $Slag(wt\%)$ | Activator $Na2SiO3$ (wt%) |                                |             |              |  |
| G1             | 45            | 45           | 10                        | $\mathbf{0}$                   | 1.5         | 0.30         |  |
| G2             | 45            | 45           | 10                        | 0                              | 1.5         | 0.32         |  |
| G <sub>3</sub> | 45            | 45           | 10                        | 0                              | 1.5         | 0.34         |  |
| G <sub>4</sub> | 45            | 45           | 10                        | $\mathbf{0}$                   | 1.5         | 0.36         |  |
| G <sub>5</sub> | 45            | 45           | 10                        | $\overline{2}$                 | 1.5         | 0.30         |  |
| G6             | 45            | 45           | 10                        | 2                              | 1.5         | 0.32         |  |
| G7             | 45            | 45           | 10                        | 2                              | 1.5         | 0.34         |  |
| G8             | 45            | 45           | 10                        | 2                              | 1.5         | 0.36         |  |
| G <sub>9</sub> | 45            | 45           | 10                        |                                | 1.5         | 0.28         |  |
| G10            | 45            | 45           | 10                        | 4                              | 1.5         | 0.32         |  |
| G11            | 45            | 45           | 10                        | 4                              | 1.5         | 0.34         |  |
| G12            | 45            | 45           | 10                        | 4                              | 1.5         | 0.28         |  |
| G13            | 45            | 45           | 10                        | 5                              | 1.5         | 0.28         |  |
| G14            | 45            | 45           | 10                        | 5                              | 1.5         | 0.30         |  |
| G15            | 45            | 45           | 10                        | 5                              | 1.5         | 0.32         |  |
| G16            | 45            | 45           | 10                        | 5                              | 1.5         | 0.34         |  |
| G17            | 45            | 45           | 10                        | 6                              | 1.5         | 0.28         |  |
| G18            | 45            | 45           | 10                        | 6                              | 1.5         | 0.30         |  |
| G19            | 45            | 45           | 10                        | 6                              | 1.5         | 0.32         |  |

## *2.4. Visual inspection for printable assessment and open time test*

After carrying out the slump and slump flow tests, the printability of the mixture was assessed by printing a simple specimen composed of one deposited filament with a length and width of  $1100 \times 120$  mm, as presented in Fig. 2. The deposited filament had a width and height of 40 mm and 10 mm. The discontinuity of the filaments was carefully inspected to evaluate the printability of the one-part geopolymer concrete. Following that, the printable region of the mixture was proposed based on the slump and slump measurement.

Furthermore, the open time of the one-part geopolymer mixture was also measured by inspecting the continuity and disruption of the filament carefully. The open time of the mixture was considered as the time that the discontinuity occurred [\[35\]](#page-11-0).

## *2.5. Verification for the printable region*

The printable region proposed based on the slump and the slump flow diagram were verified by assessing the buildability of the



**Fig. 2.** Schematic illustration of the printed specimen for printability test (dimensions in millimeters).

<span id="page-4-0"></span>one-part geopolymer mixture on the actual printing test. Three mixes that were both inside and outside of the proposed printable region were chosen to be printed in an actual printing test using a gantry concrete printer, as presented in Fig. 3. The procedure of the printing test was carefully described in a study by Tran et al. [\[36\].](#page-11-0) In the extrusion-based printing test, all printing parameters were fixed. The flow rate, which influences directly the dimensions of printed layers, was 25 ml/s, while the printing speed was 60 mm/s under the extrusion of a cylinder nozzle having a 50 mm diameter. A square structure with a side length of 500 mm was printed. This structure was composed of 20 layers and the height of each layer was 10 mm, whereas the width of the concrete filament was 40 mm.

## **3. Results and discussions**

#### *3.1. Slump-slump flow measurements and printable region*

Immediately after the mixing, the slump and slump flow were measured. The slump-slump flow diagram is presented in [Fig. 4](#page-5-0). Based on the slump-slump flow diagram and the careful visual inspection taken on the finishing surface and the continuity of deposited filaments printed by one-part geopolymer concrete as presented in [Fig. 5,](#page-5-0) 19 mixtures prepared can be classified into 6 groups.

Group 1 included G16 and G19, as shown in [Fig. 4,](#page-5-0) which had water/binder ratios of 0.34 and 0.32 while the borax contents were 5% and 6%, respectively. The fresh concrete mixtures of this group were extremely flowable with a slump higher than 30 mm and a slump flow of approximately 240 mm. The concrete of this group can flow freely through the nozzle but was unable to hold its shape after deposition, as expressed in Fig.  $5(a)$ . The bleeding water can also be seen clearly.

Group 2 covered G15 and G18, as presented in [Fig. 4](#page-5-0). The slump of the mixtures was lower compared to group 1, about 25–30 mm. The fresh concrete mixtures of this group were still easy to be extruded through the nozzle, nevertheless, the filament was able to hold its shape after deposition although the slump flow was still very high, approximately 240 mm. Noticeably, the surface of the deposited filament was very fine and smooth as presented in [Fig. 5\(](#page-5-0)b). It is noteworthy to mention that the concrete mixtures in groups 1 and 2 contained a high content of borax, from 5% to 6%, but those in group 2 were mixed with lower water/binder ratios, 0.32 and 0.30 in G15 and G18, respectively, compared to those mixtures in group 1. Furthermore, the slump and slump flow of one-part geopolymer mixtures adding borax were not always associated. While group 1 and group 2 expressed approximate slump flow measurements, the slump was significantly different.

Similar to group 2, the fresh concrete of group 3, which encompassed G4 and G8, was continuously extruded through the nozzle and was able to hold its shape after deposition. The slump of one-part geopolymer mixtures in this group varied from 15 mm to 20 mm while the slump flow measurements were approximately 230 mm. The surface of the filament was fine and smooth, as presented in [Fig. 5\(](#page-5-0)c). However, the carefully visual inspection of the filament surface of those mixtures in group 3 showed that the surfaces were slightly drier than those in group 2, a water/binder ratio of 0.36 was used, which was higher than the mixtures in group 2. It is possibly due to the low borax content used in the mixtures of group 3 which range from 0% to 2%, compared to those in group 2 which utilized 5–6%.

Group 4 comprised G3, G7, G11, G12, and G14, which had a slump of 10–14 mm and a slump flow of 205–215 mm, as presented in [Fig. 4.](#page-5-0) These mixtures happened to be able to extrude through the nozzle and hold its shape after deposition. However, the surface of the filament was very dry and rough, as shown in Fig.  $5(d)$  although the approximate water/binder ratio to those mixtures in group 1,



**Fig. 3.** The gantry concrete printer used in actual printing test.

<span id="page-5-0"></span>

**Fig. 4.** Slump and slump flow of one-part geopolymer concrete mixtures.



**Fig. 5.** Visual inspection of deposited filaments of group 1 (a), group 2 (b), group 3 (c), group 4 (d), group 5 (e), and group 6 (f).

except G7 and G14. Obviously, adding of borax content about 4–6% can significantly improve the workability of the one-part geopolymer mixture.

The last two groups including group 5 and group 6 were unable to be printed, as presented in Fig. 5(e) and (f). While group 5, which covered G2, G5, G6, G10, and G17 with the slump and slump flow of 5–10 mm and 190–200 mm, respectively, seemed disruptively <span id="page-6-0"></span>extrudable, the mixtures of G1, G9, and G13 in group 6, which presented the slump and slump flow of 0–5 mm and 150–160 mm, were unable to be extruded, as presented in Fig.  $5(e)$  and (f).

Based on these observations, it seemed only groups 2 and 3 were able to be printed well. The one-part geopolymer mixtures of these groups with a slump between 15 mm and 30 mm and a slump flow of between 210 mm and 240 mm were extrudable through the nozzle while their deposited filament was able to hold their shape well with a fine and smooth surface. Visually, the fresh mixtures in group 4 including G3, G7, G11, G12, and G14 may have sufficient workability to be extruded through the nozzle of the printer at the beginning. However, the experimentation showed that these mixtures happened to lose their slump and flow slump rapidly. The slump and slump flow of concrete mixture are strongly dependent upon the water/binder ratio and the addition of borax. Increasing the water/binder ratio or borax content can help improve the slump and slump flow, as shown in [Fig. 4](#page-5-0). Additionally, it helps to maintain the workability of the geopolymer. The one-part geopolymer concrete mixtures of group 4 were mixed with either low water/binder ratios or low borax content, leading to the fast setting of one-part geopolymer. Therefore, these were not suitable for 3D printing.

Based on the results, the appropriate slump is between 15 mm and 30 mm and a slump flow of between 210 mm and 240 mm will be appropriate for printing, as presented in [Fig. 4.](#page-5-0) However, this proposed region still needs to be verified by actual printing, which was carried out and discussed below.

## *3.2. Effects of water/binder ratios and borax added on open time and early compressive strength*

It is well-known that one-part geopolymer concrete has a low setting time and loses its workability rapidly. This low setting time hinders it to be used in 3D printing, the innovative technology that can help not only save the time and cost of construction but also decline environmental degradation. Therefore, the open time of one-part geopolymer concrete needs to be evaluated to verify its capacity to be used in 3D printing technology. In this research, the effects of borax content and water/binder ratios on the open time of the one-part geopolymer concrete were evaluated. The results are presented in Fig. 6.

It can be seen clearly that both water/binder ratios and borax content can affect the open time of one-part geopolymer concrete. With the increase of either water/binder ratio or borax content, it can successfully improve the open time of the concrete mixture. Furthermore, at the same content of borax, the open time increased when the water/binder ratio went up. Notably, when no borax was added, only the mixture with a water/binder ratio of 0.36 showed a noticeable open time, which was about 10 min after mixing.

During the early stage of fresh concrete mixture, the dissolution of the precursor plays an imperative role in the workability and setting time of geopolymer concrete. High water content in the concrete mixture can help to impede the flocculation of colloidal particles, sustain workability, and improve the open time of the concrete mixture. This is the result of the lower viscosity of water compared to that of the alkaline activating solution. The viscous property of the alkaline activating solution significantly affected the colloidal interactions between particles and the formation of the well-percolated network [\[37\]](#page-11-0). It is well known that the viscosity of water is much lower than that of the alkaline activating solution. Thus, adding more water can help to reduce the viscosity of the activating solution, leading to the decline of the viscosity of the suspending solution of the geopolymer system [\[38\].](#page-11-0) Therefore, it helps to reduce the colloidal interactions between particles and gels in the system. Consequently, the cohesion of gel interactions formed at the early age in the geopolymer matrix can be weakened, then prolonging the open time of the one-part geopolymer mixture.

The increase of the borax content can also improve the open time due to its capacity for retarding the setting of geopolymer. Since the water/binder was less than 0.32, the impacting rate of borax on improving the open time was extremely limited. It can prolong the open time only since the borax contents were higher than 4%, as expressed in Fig. 6. However, with the water/binder ratios higher than 0.32, the efficiency of borax on one-part geopolymer concrete improved significantly. As the result, the open time of one-part geopolymer concrete increased greatly with the borax content, particularly on those mixtures that had borax content higher than 4%. In one-part geopolymer concrete with a water/binder ratio of 0.36, the use of 2% borax can extend the open time from 10 min to 24 min. It happened to be more productive since the borax content increased to 4% and 5%, where the open time reached 70 min and 110 min, respectively. Although the open time was not improved by adding 2% borax in the one-part geopolymer mixture with the water/binder



**Fig. 6.** Effect of borax content on open time at different water/binder ratios.

<span id="page-7-0"></span>ratio of 0.34, the same effect occurred in the mixtures blended with 4% and 5% borax, where the open time extend to 20 min and 90 min, respectively.

The retarding mechanism of borax is mainly due to the formation of  $[BO<sub>4</sub><sup>5</sup>]$  tetrahedron in the geopolymer matrix where it plays an imperative role in retarding the polymerization process of  $[SiO_4^4]$  and  $[AlO_4^5]$   $[29]$ . In the activation process,  $[SiO_4^4]$  and  $[AlO_4^5]$ tetrahedrons are dissolved in the alkaline medium. At the same time, the dissolution of sodium tetraborate pentahydrate produces [BO $_4^5$ ] tetrahedrons in the mixture. The polycondensation between [BO $_4^5$ ] and [SiO $_4^4$ ] tetrahedrons happens easily. However, the polycondensation between [BO $_4^5$ ] and [AlO $_4^5$ ] tetrahedrons or between [BO $_4^5$ ] tetrahedrons themselves are very hard, maintaining the [AlO<sub>4</sub><sup>5</sup>] concentration in the suspending solution of the mixture, hindering the flocculation of the fresh mixture of one-part geopolymer concrete. Therefore, it can retard the setting process of the one-part geopolymer. However, the retarding effect of borax can be weakened by increasing the concentration of alkali. Since the concentration of alkali in the medium is high, it can rapidly activate and dissolve the precursors, increasing the concentration of  $[SiO<sup>4</sup><sub>4</sub>]$  tetrahedrons in the suspending solution, and improving the viscosity [\[29\].](#page-10-0) Therefore, it can increase the colloidal interactions between particles and gels in the one-part geopolymer concrete.

Although the increase of borax content can help to retard the setting time and improve the open time of one-part geopolymer concrete, it can affect the strength evolution of one-part geopolymer concrete at the early age, particularly since the borax content blended was higher than 4%. Fig. 7 expresses the compressive strength of one-part geopolymer concrete at 3 days.

At the same water/binder ratio, the raising of borax content from 0% to 2% helped to slightly increase the compressive strength of one-part geopolymer concrete at 3-day age. However, the decline of compressive strength was observed when the borax increased higher than that. It is noteworthy to mention here that when the borax added was 6%, the compressive strength of the one-part geopolymer was negligible after 3 days, regardless of the water/binder ratios in the mixes. This finding is in good agreement with the report of Oderji et al. [\[39\]](#page-11-0), in which a significant increase in compressive strength was seen when the borax content was raised from 0% to 2% of the precursor mass, yet the compressive strengths declined when the borax content was increased further.

Derived from the results observed in [Figs. 5 and 7](#page-5-0), to acquire an open time longer than 20 min, which is sufficient for 3D printing [\[23,24,35\]](#page-10-0), it is rational to recommend the amount of borax added to one-part geopolymer concrete should be from 2% to 4% while the water/binder ratio should not be less than 0.34.

#### *3.3. Verification for proposed printable region*

To verify the acceptability of the printable region of the one-part geopolymer concrete proposed, three random mixes of G11, G12, and G8 were chosen for the actual printing experiment. A square structure with a side length of 500 mm was printed, as expressed in [Fig. 3.](#page-4-0) This structure was composed of 20 layers and the height of each layer was 10 mm, whereas the width of the concrete filament was 40 mm. In these mixtures, G11, and G12 are located outside the printable region. However, while G11 possessed lower measurements of both slump and slump flow compared to the proposed printable region, G12 showed the measurement of slump flow of 215 mm, which is in the proposed range, as presented in [Fig. 4](#page-5-0). Nevertheless, G8 located inside the proposed printable region**.** 

The actual printing showed that G11 with a borax content of 4% and water/binder ratio of 0.32 was able to be continuously extruded through the nozzle at the beginning, as presented in Fig.  $8(a)$ . However, it performed poorly in the real printing test, where the extrusion of one-part geopolymer concrete was disrupted, as shown in [Fig. 8](#page-8-0)(b). Furthermore, the surface of the printed filament was very dry and rough. It is probably due to the short open time of G11, which was nearly 0 min, as shown in [Fig. 6.](#page-6-0)

The poor performance of G11 can be attributed to the short open time of this mixture. And obviously, the short open time of G11 sourced by the rapid loss of workability of this mixture, which is evaluated via slump flow of the mixture, as expressed in [Fig. 9,](#page-8-0) which was caused by the lack of water in G11 due to the low water/binder ratio, which was 0.32. With that low water/binder ratio, the concentration of alkali in the medium was high. The precursors can rapidly be activated, increasing the dissolved  $[SiO<sub>4</sub><sup>4</sup>]$  tetrahedral concentration in the suspending solution. And then, the viscosity of the suspending solution improved. Consequently, it increased the colloidal interactions between particles and gels in the one-part geopolymer concrete, which made the loss of slump flow happen



**Fig. 7.** Compressive strength of one-part geopolymer concrete at 3 days.

<span id="page-8-0"></span>

**Fig. 8.** Extrusion (a) and printed structure (b) using G11.



**Fig. 9.** Loss of slump flow on G11, G12, and G8.

rapidly. As a result, the interface between layers of G11 was poor. Furthermore, the cold joint at the surface probably happened due to excessive drying and quick setting  $[40, 41]$ . These causes made G11 unable to be printed.

G12 with a higher water/binder ratio, which was 0.34, presents a greater slump flow compared to that of G11, as presented in Fig. 9. Furthermore, the higher water content in G12 helped to decline the viscosity of the suspending solution in the mixture, causing the reduction of the colloidal interactions between particles and gels in the system. That high water/binder ratio and the retarding effect by adding 4% borax in G12 helped it to sustain the slump flow longer than that of G11. Consequently, the one-part geopolymer concrete of G12 was continuously extruded through the nozzle. The printed structure from this mixture can reach the designed height, which included 20 layers, as presented in Fig.  $10(a)$ . However, the surface of this structure was dry and rough with numerous cavities, as shown in [Fig. 10](#page-9-0)(b). The rough finishing surfaces of printed layers were able to create voids between extruded layers, which makes the interfacial bond between the layers weak. Therefore, this mixture should not be used for printing.

On the other hand, G8 with a borax content of 2% and a water/binder ratio of 0.36 was in the center of the proposed printable region, as expressed in [Fig. 4](#page-5-0). Although G8 was mixed with relatively high water/binder, the compressive strength of this mixture at 3 days was 16.7 MPa, as shown in [Fig. 7,](#page-7-0) which met the requirements of 3D printing structures [\[23,24\].](#page-10-0) Regardless of the low borax content added, the high slump flow at the beginning was still able to help the mixture of G8 sustain its workability, maintaining the open time of approximately 24 min, as presented in [Fig. 6,](#page-6-0) which was sufficient for the printing process  $[23,24]$ . Therefore, in the actual 3D printing process, the concrete was easily and continuously extruded through the nozzle. And the printed structure reached the designed height of 20 layers with good surface finishing, where it was very fine and smooth, as presented in [Fig. 11](#page-9-0). That fine and smooth surface can limit voids and cavities occurring between concrete layers, helping it to improve the bonding between layers.

Based on the actual printing tests and the observation of slump loss on G8, G11, and G12, the accuracy of the printable region proposed in [Section 3.1](#page-4-0) can be confirmed.

<span id="page-9-0"></span>

**Fig. 10.** Printed structure (a) and finishing surface of printed structure (b) using G12.



**Fig. 11.** Printed structure (a) and finishing surface of printed structure using mixture G8.

# **4. Conclusions**

This study investigated the feasibility of utilizing the slump and slump flow tests to assess the suitability of one-part geopolymer concrete for 3D printing applications. Based on the experimental findings, the following conclusions can be drawn:

- 1) The mixture with a slump between 15 mm and 30 mm accompanied by a slump flow in a range of between 210 mm and 240 mm was found to be suited to 3D printing technology using one-part geopolymer concrete. Based on this finding, a printable region of one-part geopolymer concrete using slump-slump flow diagram was proposed. The actual printing tests performed have confirmed the accuracy of the proposed printable region.
- 2) Deriving from the printable region, the open time can be measured by using the slump flow test, which was determined as the period from when the concrete mixing was completed to when the slump flow of fresh concrete was maintained from 210 to 240 mm.
- 3) The open time of one-part geopolymer concrete for 3D printing was significantly dependent on borax content and water/binder ratio. Increasing the borax content can successfully improve the open time, however, the compressive strength of one-part geopolymer concrete was significantly reduced while the borax content was over 4%.
- 4) With the one-part geopolymer comprised of fly ash/slag ratio of 1, to obtain a suitable open time of longer than 20 min, which is sufficient for 3D printing, the borax content of 2–4% and the water to binder ratio of higher than 0.34 were strongly recommended.

### **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## **Data availability**

Data will be made available on request.

#### <span id="page-10-0"></span>**Acknowledgment**

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