

# Optimisation of mixture properties for 3D printing of geopolymer concrete

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## ABSTRACT:

Freedom of design, customisation, automation, waste minimisation, reduced labour and building complex structures with cheaper materials are the main initiatives for developing 3D printed structures. The fresh properties of concrete are the most important aspects of a successful 3D printing. Concrete requires high workability for extrusion, optimum open time and high early strength in order to support the subsequent layers for 3D printing. Therefore, a mixture design that can satisfy these requirements is needed. Geopolymer concrete is a sustainable solution to traditional Portland cement-based concrete that uses waste materials. In addition, the controlled alkali-activation of geopolymer precursors in order to achieve optimum setting and workability compared to Portland cement provides freedom of mixture design for 3D printing. This paper will investigate the fresh properties of geopolymer mixtures in order to find an effective mixture that is compatible with 3D printing technology and can be also strong enough to stand as a structure. Rheology (workability), open time and compressive strength, as well as, printing parameters such as extrusion pressure and printhead speed was studied in order to achieve a successful geopolymer mixture for 3D printing.

**Keywords -**

**3D Printing; Mixture Design; Geopolymer Concrete; Additive Manufacturing;**

## 1 Introduction

Additive manufacturing technology (in particular 3D printing of concrete structures) has been introduced to the construction industry for more than a decade. However, 3D printed structures are still under-developed and there exist only a few successful trials which resulted in full-scale production.

Khoshnevis B. [1] developed the contour crafting (CC) technology for the automated construction of buildings and infrastructure on earth and other planets.

CC is a similar technology to inkjet printing. This method uses high pressure in order to extrude the concrete through a large nozzle of the printhead attached to automatic cranes which move in XYZ directions. On the other hand, the D-shape method was developed by Enrico Dini. The D-shape printer consists of a stationary horizontal frame (the base) with four perpendicular beams at each corner. The printhead is attached to the beams and moves upward (vertical direction). The printhead has approximately 300 nozzles (20 mm apart) spanning the entire base and utilises a magnesium-based binder to fuse sand or stone dust particles in a process of additive manufacturing. A limited number of successful full-scale 3D printed structures got publicity worldwide namely, the 3D printed residential house in Amsterdam (2014) by Dus Architects and also the mass production of 3D printed houses in Shanghai by WinSun (2014).

The benefits of additive manufacturing are freedom of design, reduced labour, customisation, automation, waste minimisation, and building complex structures with cheaper materials. Despite these benefits, there exist few drawbacks that would require further research and development in order to adopt this technology to the next generation of the construction industry. Printing of subsequent layers of materials results in void formation between them thereby causing additional porosity during the manufacturing process. The void formation can reduce mechanical performance because of the reduction in adhesion between the printed layers. Anisotropic behaviour is also common in 3D printed structures. As the result, properties of material inside each printed layer is different compared to that of at the boundaries between layers or the mechanical properties in each direction is different (affected by the orientation of the printed layers). The layer-by-layer appearance of the sides of 3D printed concrete (opposite to the flat sides of casted concrete) is another challenge owned by the nature of additive manufacturing. These challenges need to be addressed by new materials and machine design.

The fresh properties of concrete are the most important aspects of successful 3D printing of concrete. The possibility of aforementioned challenges can be reduced by controlling these properties. Not only the

printability of the mixture is determined by fresh properties of concrete but it also substantially affects mechanical properties of concrete after setting [2]. Rheology of the mixture determines the workability and extrude-ability of the concrete for smooth printing of layers with good shape-stability, less void formation and reduced chance of blockage of the nozzle. The rheological behaviour of concentrated suspensions (e.g. concrete) changes versus time and is of great importance for a hassle-free printing [3]. Even minor changes in rheological behaviour during the time-span of printing can potentially affect the properties of the 3D printed structure. Another important fresh properties of concrete for 3D printing is open-time. There exist an optimum time for the printed layer to get initial strength in order to hold subsequent layers but still wet so the layers can fuse together before final setting. The open-time is related to the initial setting time of the concrete. A sufficient open time is required to support subsequent layers [4]. A well-designed concrete mixture needs to satisfy the requirement of controlled rheology, optimum open-time and suitable strength for a successful 3D-printed structure.

## 2 Background

A limited number of studies on the effect of the mixture design on fresh properties of 3D printed concrete has been carried out, which are briefly discussed.

Gosselin C. et al [5] developed a printing process method which controls the rheology of the mortar for a longer time-span without a reduction in early strength. This method pumps the accelerator and the premix mortar from two separate reservoirs via separate tubes and then combines it at the printhead before the extrusion. As the result, rheological behaviour before extrusion slowly changes (because of no accelerating admixtures in the mortar reservoir) but after the addition of the admixture at the tip of the nozzle, the mixture can set quickly and get a higher early strength to hold subsequent layers. The reduced open-time results in faster printing and higher buildability. Paul S.C. et al [2] investigated different concrete mixtures and found that the rheological properties of concrete mixtures, especially thixotropic behaviour (changes in the rheological behaviour versus time), is an influencing factor for pumping and printing.

Zareiyan B. and Khoshnevis B. [6] suggested the smaller maximum aggregate size and higher cement to aggregate content which results in higher strength and better interlayer adhesion. The increased thickness of the layers with more time lapse between subsequent layers increased the adhesion between layers but decreased the compressive strength of the printed structure. A shorter setting time is also shown to increase the possibility of cold joints between layers [6]. Kazemian A. et al [7]

showed that the addition of silica fume and nano-clay can remarkably increase the shape-stability (the stability of the printed layers against settlement and deformation caused by printing of the subsequent layers) of 3D printed cement paste.

### 2.1 3D printing of geopolymer concrete

Geopolymer (or alkali-activated materials) is a concrete-like binder with approximately 80-90% less associated CO<sub>2</sub> emissions and mechanical, durability and thermal properties comparable with or exceeding those of normal concrete made of Portland cement [8, 9]. Geopolymer is made in a process of alkali activation of aluminosilicate-based materials (precursors) such as blast furnace slag, fly ash, silica fume or metakaolin [10]. The fresh properties (as well as mechanical properties) of geopolymer concrete greatly change based on the mixture design selected from a variety of precursors, activators, different ratios between them and water to solids (w/s) ratio [11-13]. The limitless mixture design of geopolymer with vastly different fresh properties compared to that of Portland cement (the common binder of normal concrete) introduces more possibilities to design a suitable mixture for 3D printing.

Xia M. and Sanjayan J. [14] investigated geopolymer 3D printing based on a powder-bed method which consists of ground blast furnace slag, sand and ground anhydrous sodium silicate (alkali activator). The liquid binder in order to fuse the powders based on a 3D pattern was water with a small amount of 2-Pyrrolidone. The printed cubes had a considerably low strength of 0.9 MPa with the dimensional expansion of less than 4%. Post-treatment of the samples at a higher temperature (60°C) in an alkali solution substantially increased the strength up to 16.5 MPa showing a great extent of unreacted powders after printing and before the heat-treatment.

This study looks into finding a suitable mixture design for 3D printing of geopolymer paste by an inkjet method similar to contour crafting (smaller scale). Fresh properties of the paste i.e. rheology and open-time, as well as strength, are the key experimental factors in order to find an optimum mixture design of the precursors, activators and w/s ratio. Finally, required pumping pressure and printing speed necessary for successful 3D printing was studied.

## 3 Materials and methods

### 3.1 Materials and mixture design

A mixture of ground granulated blast furnace slag (hereinafter slag) from Australian Builders, fly ash from Cement Australia and silica fume from Redox was used as geopolymer precursors. The chemical composition of

the precursors are displayed in Table 1 (determined by X-ray fluorescence (XRF) analysis and excluding loss on ignition). The ratios of slag: fly ash: silica fume were 3:1:0.5 based on the preliminary results of fresh properties and strength of different mixtures of the geopolymer precursors tested in the laboratory.

Table 1. Chemical composition of the precursors determined by X-ray fluorescence (XRF)

	Fly ash	Slag	Silica fume
Na <sub>2</sub> O	0.81	1.51	0.00
MgO	1.27	5.56	0.00
Al <sub>2</sub> O <sub>3</sub>	25.13	14.01	0.00
SiO <sub>2</sub>	42.09	32.83	96.85
P <sub>2</sub> O <sub>5</sub>	1.10	0.07	0.00
SO <sub>3</sub>	0.41	2.28	3.15
K <sub>2</sub> O	0.41	0.35	0.00
CaO	13.56	41.90	0.00
TiO <sub>2</sub>	1.44	0.53	0.00
MnO	0.18	0.22	0.00
Fe <sub>2</sub> O <sub>3</sub>	13.16	0.50	0.00
Total	100.00	100.00	100.00

Sodium meta-silicate powder (SiO<sub>2</sub>: 50 wt.%, Na<sub>2</sub>O: 46 wt.% and water: 4 wt.%) was used as the activator. Sodium silicate is a corrosive material and needs to be used carefully. However, there is no negative impact on the environment by using this activator. Two ratios of 8 and 10 weight % of the activator per total mass of precursors and three water to solids (w/s) ratios i.e. 0.31, 0.33 and 0.35 were used as variables. In total, 6 samples were tested for rheological behaviour, open-time (initial setting time) and compressive strength. The codes of these samples are shown in Table 2. The optimum mixture based on the fresh properties and strength results was used for 3D printing where optimised printing speed and pumping pressure was trialled.

Table 1. Samples codes of the six geopolymer mixtures

Activator per solid wt. %	w/s ratio		
	0.31	0.33	0.35
8%	G8-31	G8-33	G8-35
10%	G10-31	G10-33	G10-35

## 3.2 Test methods

### 3.2.1 Rheology

A Haake Rheometer (Viscotester 550) was used to measure the yield stress of the different mixtures. The paste was mixed for 5 minutes using a plenary Hobart mixer and poured into a cylindrical plastic container (d: 50 mm and h: 150 mm). The vane H with four perpendicular blades (width: 5mm and height: 25 mm) was inserted into the paste until the blades were entirely submerged. After 60 seconds resting in order to dissipate any accumulated stress after the insertion of the vane into the paste. The rotation rate of 0.2 rpm was applied until the maximum torque was recorded. The measurement was repeated three times and the paste was hand-mixed (to avoid settlement of particles) and rested before each measurement. The maximum torque was converted to yield stress based on the geometry of the vane H corresponding to the methodology used in previous works [15, 16]. The average of three measurements was reported. Yield stress versus time at an interval of 15 minutes up to 45 minutes was measured for each mixture in order to understand the rheological behaviour of the mixtures as time passes.

### 3.2.2 Initial setting time

The Vicat needle was used to conduct the initial setting time test of the samples and was related to the open-time. The test was conducted according to ASTM C191-13. The fresh sample was penetrated by the Vicat needle and the penetration depth was recorded versus time. The penetration readings were done at 15 minutes intervals. The approximated initial setting time was recorded when a penetration of less than 25 mm was achieved. The penetration was repeated for each mixture three times (freshly made each time). The average of three measurements was reported.

### 3.2.3 Compressive Strength

Compressive strength was measured using a Technotest compression testing instrument with a loading rate of 1 N/(mm<sup>2</sup>.s). The paste was mixed for 5 minutes using a plenary Hobart mixer and poured into a 50 mm cubic moulds, sealed with a plastic bag, cured at around 25°C for 21 days and then were tested. Two parallel surfaces of the samples were smoothed using a sandpaper before applying the load. The maximum load before failure was divided by the surface area under load in order to calculate the compressive strength. Five samples were tested; the compressive strength results were averaged and reported.

### 3.2.4 3D printer

A 3D-Bioplotter inkjet printer from envisionTEC was used for printing of the geopolymer paste on a small scale. A nozzle with an internal diameter of 1.65 mm was used. The paste was mixed for 5 minutes using a plenary Hobart mixer and poured into special plastic syringes. A controlled pneumatic pressure at the back of the syringe is used by this printer to extrude the materials from the nozzle. Horizontal speed of the head of the printer is also controllable. A combination of horizontal speed, pressure and nozzle size (in addition to the rheology of the mixture) determine the thickness of the printed layer. A 50×50 mm rectangular hollow column was designed by Solidworks and used as the input CAD file for 3D printing. The thickness of wall was 3.5 mm or approximately twice the diameter of the nozzle (in order to accommodate to layers side-by-side).

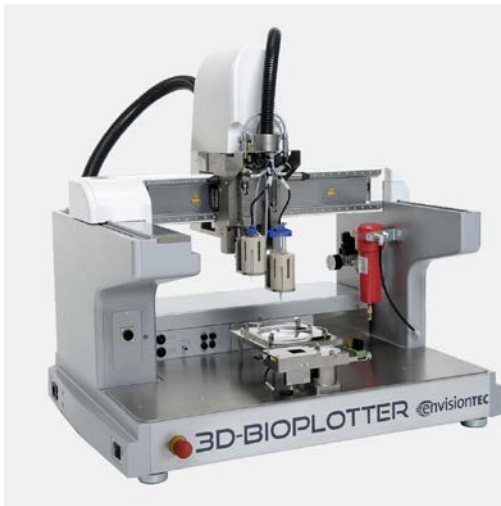


Figure 1. The inkjet 3D printer used in this study to print geopolymer

## 4 Results and discussion

### 4.1 Rheology of the geopolymer mixtures

Unlike Portland cement, the rheological behaviour of the alkali-activated materials (geopolymer) is less well known. This is due to the complexity of the chemical environment and reactions taking place, in addition to different physical properties of the main precursors. For example, an alkali silicate-activated slag has an extremely complicated solution environment with high ionic strength and alkalinity, which is a very challenging environment in understanding rheological behaviour [11]. On the other hand, rheology modifiers such as common superplasticizers (that improves workability without increasing w/s) in Portland-cement based materials

underperform in geopolymers [17]. Despite the recent aims for designing a suitable superplasticizer for geopolymer concrete [18], those superplasticizers having a minor effect on the rheology of geopolymers compared to that of Portland cement-based systems.

Internal forces between particles in a concentrated suspension of geopolymer are, namely, short-range repulsion forces, normal and shear stresses, fluid pressure, electric double layer forces, inertial and hydrodynamic effects and sedimentation [19]. The balance of these forces determines the yield stress (as one of the main rheological parameters of the fluid). The relationship between concrete slump (a common workability test in the construction industry) is complicated but normally higher slump height (higher fluidity) corresponds to lower yield stress [20]. Better pump-ability and easier flow which is essential for extrusion require lower yield stress and viscosity. However, a very low yield stress material does not hold its shape after extrusion which results in collapsing and self-levelling (not desired in 3D printing). Therefore, an optimum yield stress which allows easier flow of geopolymer for extrusion during 3D printing but at the same time avoid self-levelling and collapsing of the printed layer of concrete (which also needs to support successive layers of concrete) is required.

Figure 2 shows yield stress of the six samples of geopolymers for 3D printing. The effects of the w/s ratio and the wt.% of the activator are shown for the mixture of solid precursors used in this study.

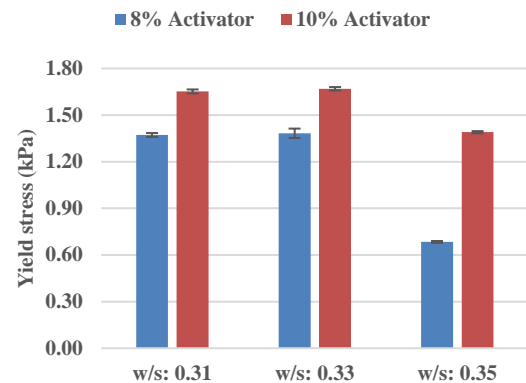


Figure 2. Yield stress of the geopolymer mixtures at different wt.% of activator and w/s ratios

The higher w/s ratio results in an excess water which is no longer used to fill the voids and therefore covers the surface of particles and separates them. The separation of particles by the excess water results in a reduction of bonding forces between particles hence it reduces the yield stress. The substantial reduction as the result of increasing w/s ratio from 0.31 to 0.35 for the sample with 8 wt. % activator is clear in Figure 2. The increase of

water from w/s of 0.31 to 0.33 did not change the yield stress. The reason is possibly because of the fact that the amount of excess water for both w/s ratios are negligible in order to facilitate the particle movement and reduce the yield stress. In another word, the sample with w/s of 0.31 is very dry (unsaturated with water) and needs a substantial amount of water before any changes in rheological behaviour takes place.

The addition of alkali activator provides a higher pH and different electrolyte environment in the fresh binder paste (compared to Portland cement-based binders), causing differences in the surface chemistry of particles and their interactions. For instance, the dissolution of slag particles at higher pH (provided by the higher amount of alkali activator) can affect the size of the slag particles and their packing thereby affecting the amount of excess water [11]. Besides the fact that high concentration of ions in the fluid between particles may also cause flocculation of particles thereby increasing the force between particles and yield stress.

In general, an increase in activator dosage results in faster reaction and a higher rate of yield stress increase as previously shown in [11] and also in this study (Table 3). Yield stress can depend on time and shear history which is known as thixotropy. The alkali reaction as time passes affect inter-particle forces via the formation of new chemical or physical bonds between the particles. It also can increase the volume fraction of solids in the fluid as some of the water molecules becomes chemically bound to the solids during the reaction. Therefore, water consumption during the reaction process of geopolymer reduces the amount of excess water which is necessary to disperse slag aggregates. As the results, yield stress increases as time passes.

Table 3. Yield stress (kPa) vs. time of the geopolymer samples (values above 2 kPa shows the sample is semi-hard and yield stress is not measurable)

Sample codes	t=0 min	t=15 min	t=30 min	t=45 min
G8-31	1.37	>2 (hard)	-	-
G8-33	1.38	1.68	1.95	>2 (hard)
G8-35	0.68	1.37	1.76	1.98
G10-31	1.65	>2 (hard)	-	-
G10-33	1.67	2.00	>2 (hard)	-
G10-35	1.39	1.48	1.76	>2 (hard)

The higher amount of water and lower amount activator slows down the yield stress increase by reducing the pH and diluting the system. The only system that can be workable for an extended period of time of 1 hour is the sample with the lowest amount of activator

and highest w/s among the samples (G8-35). This sample also has the lowest initial yield stress which was good for extrusion but not suitable for shape-stability after the extrusion. The next options which have prolonged workability (up to 45 minutes) but higher initial yield stress for better shape-stability are G8-33 and G10-35. The required pumping pressure is directly affected by the yield stress of the sample, thereby if the yield stress changes quickly, the pressure also needs to be adapted for homogenous printing. Therefore, minimum changes in yield stress vs. time-span of printing is desired,

## 4.2 Open-time of the geopolymer mixtures

The suitable time-span to print the subsequent layer of concrete is considered as open-time which is directly affected by the initial setting time of concrete. An optimum open-time is required for the printed layer in order to quickly get a minimum strength to support subsequent layers but long enough so the printed layer is still wet and the subsequent layer can fuse into the previous layer. The printed geopolymer transitions from a fluid material to a semi-solid and then a solid material because of the reaction of the precursors and the activator.

The initial setting time of the geopolymer mixtures with different ratios of water and activator to solid precursors are shown in Figure 3. It can be seen that higher wt.% of the activator (10%) results in considerably lower setting time because of a faster reaction, higher pH and a higher rate of dissolution of particles. Also, a higher w/s ratio causes prolonged setting time by lowering the pH, the rate of dissolution and reaction. The initial setting time can be as low as about 35 minutes for G10-31 up to 230 minutes for G8-35. As mentioned, very short setting time (open-time) although is a sign of getting strength quickly to support the subsequent layers, it can result in less adhesion (fusion) between layers and most importantly the mixture can lose workability for printing very quickly.

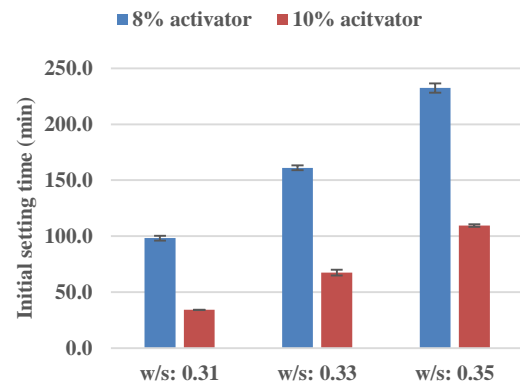


Figure 3. Initial setting time of the mixtures at different wt.% of activator and w/s ratios

The lower setting time can also increase the chance of the blocking the pipes or hardening the sample inside the reservoir. Table 3 shows that G10-31 with an initial setting time of about 35 minutes, is only print-able for less than 15 minutes and after that, yield stress increased and the paste is almost not pump-able (extrude-able). On the other hand, very long setting time (e.g. G8-35) can result in less strength which may not hold the subsequent layers resulting in deformation of bottom layers because of the mass of top layers (inferior shape-stability). It must be noted that sometimes initial yield stress is enough to support a limited number of subsequent layers so then prolonged setting time (slower reaction) would not be problematic (even beneficial for homogeneity of printing) in this case.

It must be noted the reaction also affect the fluidity and extrude-ability of the mixture because the rheological properties are also changing vs. time (Table 3). Reduction in changes of yield stress vs. time is required for homogeneity of printing. However, the samples with lower yield stress changes vs. time showed higher initial setting time (comparing Figure 3 and Table 3). Therefore, a combination of open-time, initial yield stress and changes of rheology vs. time is important to find a suitable geopolymer mixture for 3D printing. And sometimes one property needs to be sacrificed for the other based on prioritising the requirement for printing that can be a better homogeneity, higher number of layers (improved build-ability), easier extortion (better workability) and etc.

#### 4.3 Compressive strength of the geopolymer mixtures

Apart from the fresh properties of the geopolymer mixtures for successful printing, the mechanical properties such as compressive strength must be sufficient enough for a structure. Figure 4 shows the 21-day compressive strength of the geopolymer mixtures with different amount of activator and water to solid ratios.

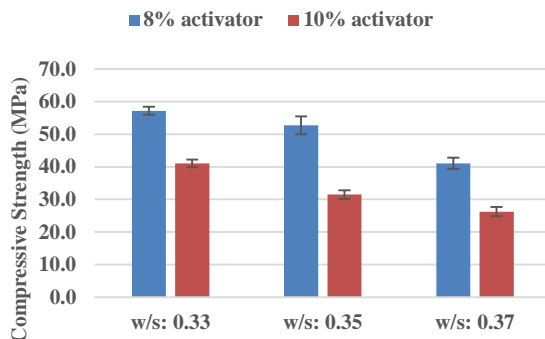


Figure 4. Compressive strength of the geopolymer mixtures at different wt.% of activator and w/s ratios at 21 days

The samples with a lower percentage of the activator (8 wt.%) showed higher strength compared to the samples with 10 wt.% of the activator. Lower alkalinity drive formation of a particular geopolymer gel which increases the mechanical performance [8]. Also, higher w/s normally results in a lower strength of geopolymers as shown in Figure 4.

It must be noted that the samples with 10 wt.% of the activator, despite having faster reaction evidenced by lower initial setting time and faster changes in workability (higher rate of increased yield stress vs. time), showed lower compressive strength. Therefore, 8 wt.% of activator seems a better option for 3D printing considering all these factors.

#### 4.4 3D printing of the geopolymer mixture

Considering, three factors of rheological behaviour, open-time and compressive strength, the samples with 8 wt.% of the activator showed higher strength, increased open-time and reduced change of yield stress vs. time compared to the sample with 10 wt.% of activator. Among the samples with 8wt.% of the activator, G8-3, the yield stress changes increased rapidly (Table 3) and after 15 minutes the workability massively dropped. Therefore, the other two mixtures with higher w/s which are G8-33 and G8-35 are considered for 3D printing.

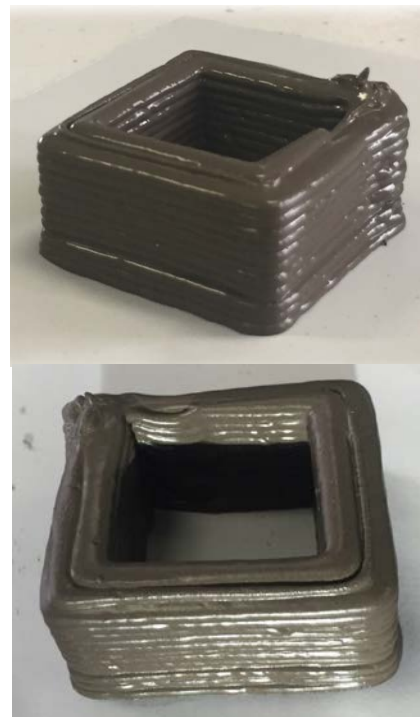


Figure 5. The 3D printed sample of the G8-33 geopolymer mixture (dimensions: 50×50×30 mm)

For G8-35, the printed geopolymer, after extrusion from the nozzle, showed insufficient shape-stability (the printed filaments collapsed and started to spread). This is because of the considerably lower yield stress of this sample as shown in Figure 2. The deformation was significant after printing of the third layer. Therefore, it is concluded that yield stress of around 0.7 kPa for this sample was insufficient for self-supporting of the printed layers. On the other hand, printing of G8-33 was successful as shown in Figure 5 (12 layers and each wall has two filaments printed side-by-side).

Apart from an optimum mixture design, a proper extrusion by the 3D printer is also essential to get the desired quality. Two critical parameters (pumping pressure and printhead speed) considerably affect the printing quality. Pressure is important to ensure an extrusion of a continuous filament from the nozzle. Higher pressure can also push the previously printed layers and affect the shape-stability of the object. The speed of the printhead also determines the time gap between printing of successive layers. Higher speed also may result in discontinuation of the filament (skipping) while printing (which results in air gaps) and it adversely affects shape-stability of concrete (quickly printed layer on top of each other provides a short time span for concrete to set and self-support the structure). Therefore, a combination of speed and pressure in addition of the size of the nozzle determines the thickness of each printed filament; lower speed and higher pressure cause thicker filaments (because the extruded volume of the paste exceeds the volume of material required for a specific length) and vice versa.

The optimum pressure for printing of G8-33 is around 70 to 90 kPa (about 60 times higher than the yield stress of the material) and the optimum speed for this pressure is around 25 mm/s (printing of each layer takes approximately 20 s). The printing of the sample in Figure 5 takes about 4 minutes which is considerably smaller time period compared to the setting time of this mixture. The reservoir capacity of the printer was limited so printing of a larger sample was not possible in one load (reloading takes a considerable amount of time which can affect the quality and homogeneity of the printed object).

The shape-stability of the sample was very good as shown in Figure 6 and except the first layer (because of the uneven substrate), the thickness of other layers are similar with negligible deformation after printing of the successive layers. As the result of the negligible changes in yield stress of the material in the short time period of printing, it can be concluded that the initial yield stress of G8-33 (i.e.  $\sim 1.4$  kPa) was enough for self-supporting of the number of printed layers. The average thickness of each layer is  $2.5 \pm 0.2$  mm (i.e. approximately 1.5 times larger than the internal nozzle diameter). The thickness of the layer can be controlled by the extrusion pressure

and speed of the printhead as well as nozzle diameter. Also, the fusion of the layers seems excellent and no air-gaps between layers is recognizable (Figure 6).

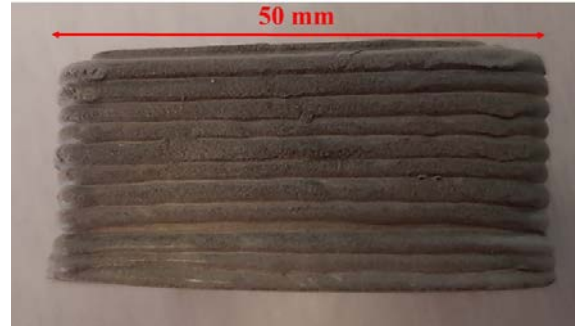


Figure 6. A side view of the 3D printed sample of the G8-33 geopolymer mixture

## 5 Conclusions

The material properties of a few geopolymer mixtures for 3D printing was studied. Rheology (workability), open-time and compressive strength as well printing parameters such as pumping (extrusion) pressure and printhead speed were studied in order to achieve a successful geopolymer mixture for 3D printing.

In particular, the effect of percentage of the activator and water to solid ratio was studied. The optimised mixture has 8 wt.% of the activator and water to solids ratio of 0.33. Consistent filaments of this mixture were successfully printed through a 1.65 mm diameter nozzle with negligible deformation after printing because of higher initial yield stress and minor changes in yield stress versus time for this mixture. The open-time (setting time) was also high enough to get an excellent fusion (adhesion) between the layers. The compressive strength of the sample was around 50 MPa at 21 days. The optimum extrusion pressure and the printhead speed for the optimised mixture were found to be around 70-90 kPa and 25 mm/s, respectively. However, long-term durability and load-bearing capacity such as the development of internal stresses (which can result in cracking) needs to be investigated in the future studies.

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