

# Measurement of tensile bond strength of 3D printed geopolymer mortar

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## Abstract

The structural capacity of construction joints in concrete bridges, deck and pavements mainly depends on the bond strength between the old substrate and new overlaid concrete. Sometimes, a mismatch in the properties of old and new concrete may lead to early age failure and shortened service life. Since in 3D concrete printing (3DCP), the whole object is made by layer by layer, bond strength is considered as one of the key parameters to ensure stability in the structure. For understanding bond mechanism, it is essential to measure bond strength at the interface between new and old layer and investigate significant parameters affecting this property. In this direction, our current work targets to analyse tensile bond strength of 3D printed geopolymer mortar with respect to *printing time gap between layers*, *nozzle speed* and *nozzle standoff distance*. A novel formulation of fly ash based geopolymer was made and printed using four-axis automated gantry system. Experimental findings reveal that the bond strength is a function of state of interface material between two nearby layers which can be influenced by material strength development rate and 3D printing parameters.

**Keyword:** 3D concrete printing, Geopolymer, Tensile bond strength and Interface properties

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## 1. Introduction

In recent years, 3D concrete printing (3DCP) is one of the emerging technologies that can minimize the supply chain in the construction process by automatically producing building components directly from digital model without human intervention and complex formwork. All around the globe, universities as well as industries are exploring this technology to accredit for various construction applications [1-3]. Though technology is advancing, use of conventional raw materials, especially, ordinary Portland Cement (OPC) as a printable material is creating negative environmental impact by directly releasing CO<sub>2</sub> into the atmosphere [4]. In this regard, use of local materials can be one of the alternative to reduce carbon foot print while targeting the global demand for sustainability [5]. Geopolymer, invented by French scientist Davidovits, is one among such materials that uses locally available by products such as fly ash, slag as key ingredients and exhibits superior properties compare to ordinary Portland cement (OPC). Geopolymerization is a geosynthetics process that chemically binds silicon and aluminum rich products in an alkaline medium and forms a 3-dimensional network (by poly-condensation) which makes the concrete strong, durable, and chemical resistant [6,7]. Owing to these significant properties, in this research, we conceptualized to utilize geopolymer in 3DCP process where custom made geopolymer mortar was laid down by a 4-Axis gantry system while following the deigned 3D computer-aided design (CAD) model. During printing, *nozzle standoff distance, printing speed and time gap between layers* were adjusted to investigate its impact on bond strength of printed specimen.

As a new technic, the results of bond strength in 3D printed structures are limited. For the first time, Le at al. [8] investigated the bond strength of 3D printed specimens at different time gaps. The specimens with varying time gap (0 minutes to maximum 7 days) were tested in tension and their bond strengths were found to be reduce exponentially with time i.e. lowest strength is found

in the specimens with higher printing time gap. This strength reduction was explained by the non-uniform shrinkage of the newer and older layers of the specimens. Typically, bond strength between the old and new substrates depends on the surface and moisture conditions of the existing concrete surface. In conventional concrete research, Gillette [9] observed that free water on the existing surface weakened the bond strength. However, Pigeon and Saucier [10] found no influence of moisture condition on the bond strength. Furthermore, Austin et al. [11] confirmed saturated surface dry (SSD) state of the existing surface as most favourable condition to obtain higher bond strength. These different results provided by authors might be attributed to the differences in materials for overlay and substrate, environmental conditions, and testing methods.

Creation of the bond can be explained in terms of specific and mechanical adhesion [12-14]. Specific adhesion is evaluated by studying the interfacial and surface forces acting at the interface whereas mechanical adhesion comes from interlocking effect induced by roughening concrete surface. Since in concrete printing, layers are deposited automatically by the printer, there is a need to study effects of printer parameters on bond formation mechanism between two subsequent layers. **To the best of our knowledge, no study has been reported on interfacial bond strength measurement of 3D printed geopolymers mortars and therefore,** in this research, we attempt to investigate the bond strength with respect to different printing parameters such as printing speed, printing time gap between layers and nozzle standoff distance etc. In the following sections, these parameters were briefly introduced and subsequently, the bond strength was characterised by altering one of these parameters while keeping the rest at their constant values.

## 2. Overview of 3D concrete printing

### 2.1 3D concrete printing processes

3D concrete printing (3DCP) is one of the advanced manufacturing technologies which is capable of automating our conventional construction process by minimizing the need of complex form work and 24×7 human resources [15]. Like fused deposition modelling (FDM), fresh concrete (mortar) was extruded in this process in a layer by layer manner with help of gantry or robotic system as shown in figure 1. A concrete pump was separately used to drive the concrete from hopper to the extruder, i.e attached to the 4-Axis gantry printer. With the help of a programmable logic controller (PLC), the pump flow rate and robot speed were controlled simultaneously to deposit the mortar as per the CAD model.

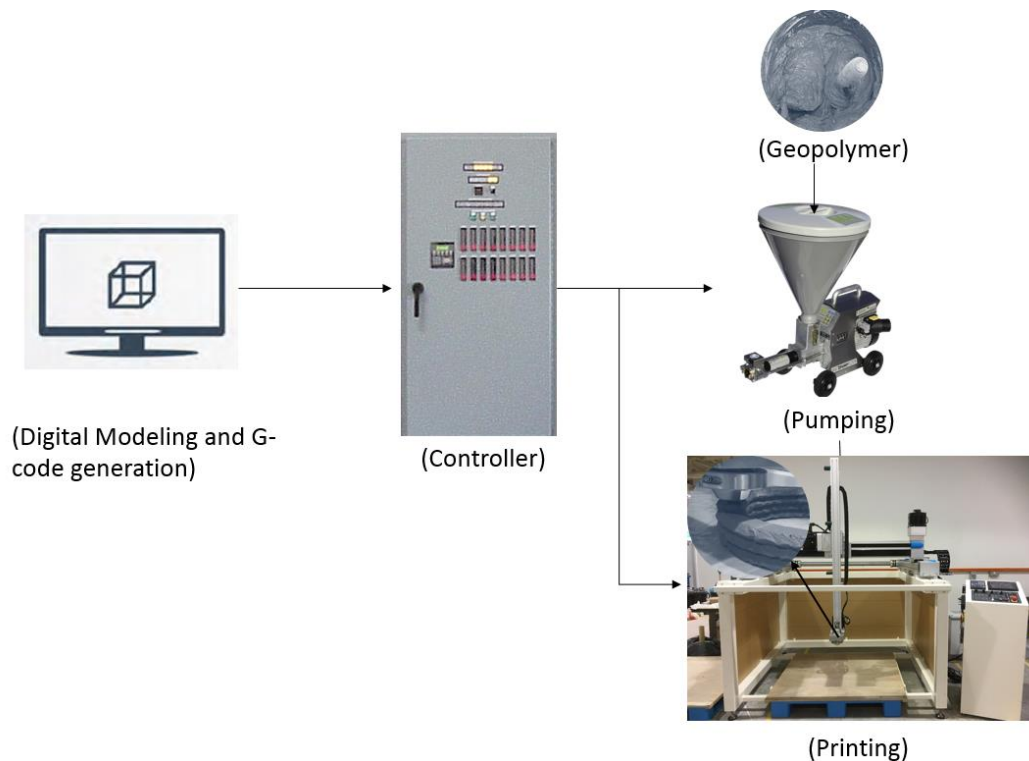


Figure 1: Process flow of geopolymer 3D printing

Developments for concrete printing started in the mid-1990s in California, USA, when Khoshnevis introduced a technique termed **Contour Crafting** (CC) (Figure 2(a)) [16]. As an automated construction process, CC seeks to increase safety standards (both for occupants and laborers) and construction efficiency at a time when Labor efficiency is alarmingly low, and skilled workforces are vanishing. Besides CC, in recent years, pioneering work also have been done by the *University of Loughborough, U.K.*, *Chinese company Winsun, Xtree at France, Apis cor at Russia* and *Minibuilders team at Institute of Advanced Architecture of Catalonia, Spain* with an aim to provide affordable and dignified housing to people across the world (Figure 2) [17].

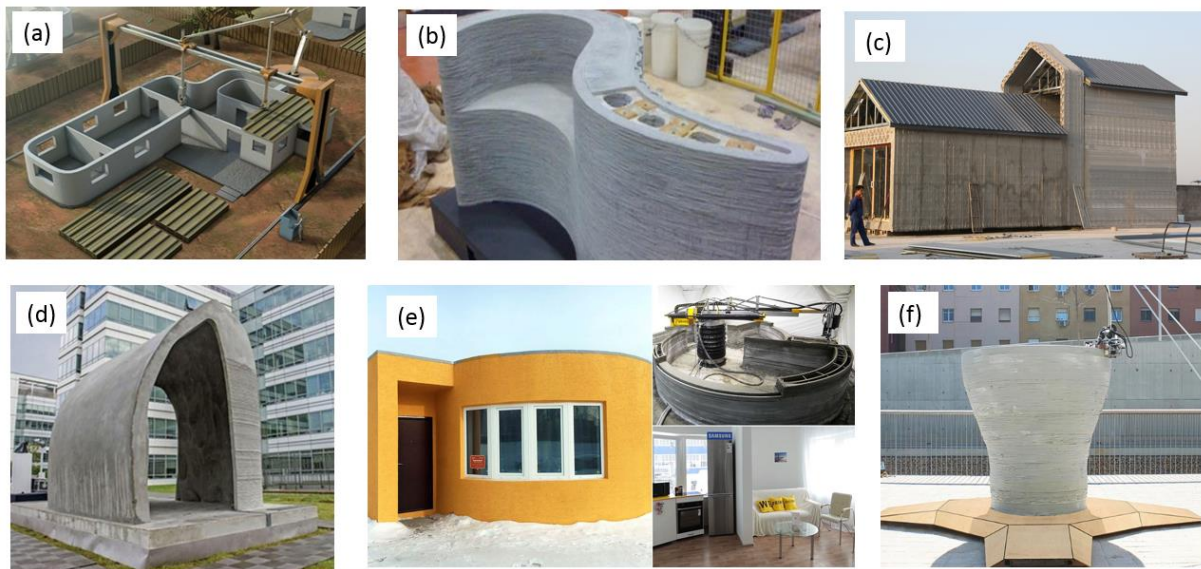


Figure 2: Noteworthy examples of concrete printing by (a) Counter crafting, USA (b) University of Loughborough, U.K (c) Winsun, China (d) Xtree, France (e) Apis-cor, France and (e) Mini-builder, Spain [17]

Compared to traditional casting, it can be realized that, 3DCP produces less material wastages, minimize human resource utilization and reduce formwork cost which incurs 40% of the total budget [18]. In terms of application, it allows design freedom to architect and designers for rendering complex structures that can be printed with different materials combination as per the requirement. Figure 3 shows some of the complex structures designed and printed at Nanyang Technological University (NTU), Singapore with different material combinations using automated gantry and 6-Axis robotic system. Though in recent days, 3DCP is capable for producing complex elements that are difficult to achieve in traditional practices, challenges such as structural strength, reinforcement placement, proper standard development are impeding this technology for its wide spread application and in this regard, in-depth investigations are needed to improve its structural, mechanical and durability properties.



Figure 3: 3D concrete printing research at Nanyang Technological University (NTU), Singapore

## 2.2 Material and machine parameters

Right formulation of material is one of the major challenges for 3DCP. Since no formwork is used in this process, rheology of the material should be carefully controlled in the sense that it must be able to be pumped through extruder and at the same time able to maintain its shape after being extruded out it [19,20]. A further challenge is that the printed layers should support the weight of subsequent layers that are being deposited by the printer on top of each other. Proper amount of binders, aggregates and their particles size distribution must be scrutinized for better printability of different construction materials. A range of different additives such as plasticizers, retarder and accelerator can be used to improve the printability of materials. Insufficient accelerator could make the material as such that doesn't set, or takes a long time to set, whereas insufficient retarder may allow the material to set in the hose pipes, thereby damaging the pump and material delivery system.

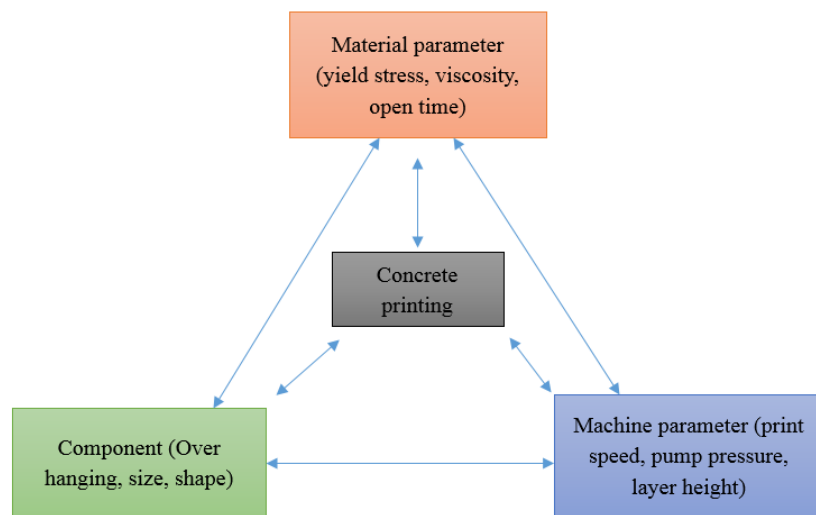


Figure 4: 3D concrete printing components

Literature reveals that, in 3DCP, both material and printer plays an important role which need to be balanced with complexity of the digital model (Figure 4) [28]. As discussed earlier, the material

present in the hopper (shown in figure 1) must have sufficient open time (time between adding water to the mix and the time until the material is pumpable) till end the printing process to avoid clogging in the hose pipe. Based on the open time and model complexity, printing parameters can be optimized to ensure continuous extrusion. Rapid hardening mortar is suggested to be mixed and printed simultaneously, so the wet material need not to wait for a long time in the hopper, rather it will be deposited immediately after mixing, thus allowing more layers to be stacked up on each other. Regardless of slow or fast hardening material, state of interface material (figure 5) is highly related to the tensile bond strength between layers and this property changes with change in printing parameters such as print speed, printing time gap between layers etc. Therefore, the aim of this paper is to investigate the bond strength property with different *time gap between layers, nozzle standoff distance and printing speed* while printing a novel geopolymer mortar developed for concrete printing application. The outcome of this investigation is believed to helpful to the 3DCP users in understanding the bond strength behavior of printed geopolymer mortar.

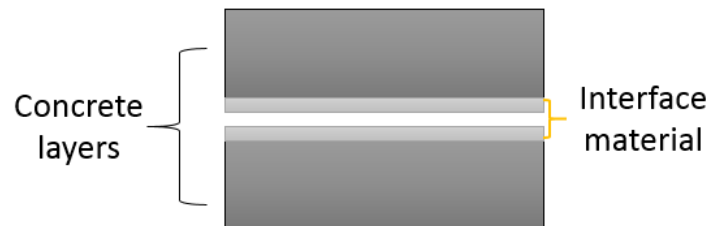


Figure 5: A typical bonding mechanism in layered concrete

### 3. Material and Methods

#### 3.1 Collection of raw material and characterization

The main precursor of geopolymer i.e. fly ash was collected from China and classified as class F as per ASTM C618-12a standard [21]. The detailed chemical compositions of as-received class F fly ash are 49.10% SiO<sub>2</sub>, 39.35% Al<sub>2</sub>O<sub>3</sub>, 3.48% Fe<sub>2</sub>O<sub>3</sub>, 2.94% CaO, 0.328% Na<sub>2</sub>O, and 1.4%

loss on ignition. Ground granulated blast-furnace slag (GGBS) and undensified micro silica grade 970 (silica fume) were provided by Engro and Elkem Pvt. Ltd., Singapore respectively. The Scanning electron microscope (SEM) images of as-received GGBS (irregular) and fly ash (mostly spherical) particles are shown in Figure 6.

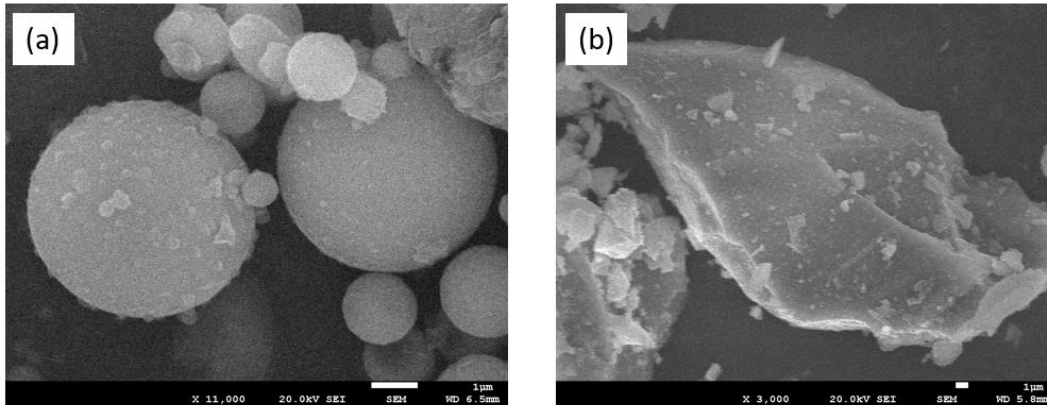


Figure 6: Fe-SEM images of (a) fly ash (b) GGBS

To characterize the reactivity of raw materials, X-ray diffraction (XRD) analysis were carried out by Bruker D8 advance setup for 2theta values between 10 to 120 degree using CuK $\alpha$  source at room temperature. It is clear from the obtained pattern (figure 7) that, GGBS is mostly amorphous and fly ash only behaves as amorphous between 10 to 30 degree. This amorphous nature of the raw materials accelerates the geopolymerization process as it is easy to dissolve in the alkaline solution like sodium or potassium silicate.

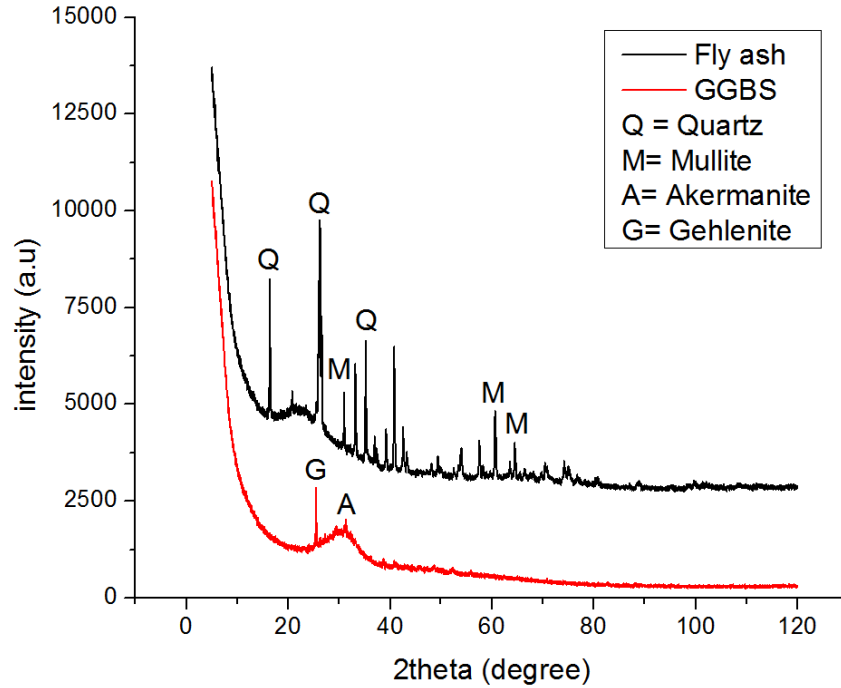


Figure 7: X-ray diffraction pattern of fly ash and GGBS

Liquid potassium silicate (k-Silicate) supplied by Noble Alchem Pvt. Ltd, India was used as alkaline reagent. For user-friendly purpose, k-Silicate of molar ratio=2 was considered with 40% solid content. As-received k-silicate was directly used in the geopolymer formulation without any addition of sodium or potassium hydroxide. Viscosity of the silicate was recorded around 18-20 cps in room temperature ( $25 \pm 3$  degree) condition. As aggregates, we used fine river sands of particle size less than 1.15 mm as-obtained directly from the supplier.

### 3.2 Geopolymer formulation and printing

In this research, printable geopolymer was prepared by adding thixotropic additives to the conventional geopolymer mortar. First, all the raw materials were weighed as per mix design given in Table 1 which was derived based on our preliminary test runs. Fly ash, slag and silica fume were added together in a Hobart mixer and mixed uniformly for two minutes at lowest speed. Then

k-Silicate was added into the mix to make the geopolymer binder and mixing was continued for another two minutes till it becomes like a slurry. With a thirty second pause, river sand was added slowly to the binder and mixed properly at highest speed around one minute. At the end, slight addition of water was allowed to make the mortar workable for the printing application.

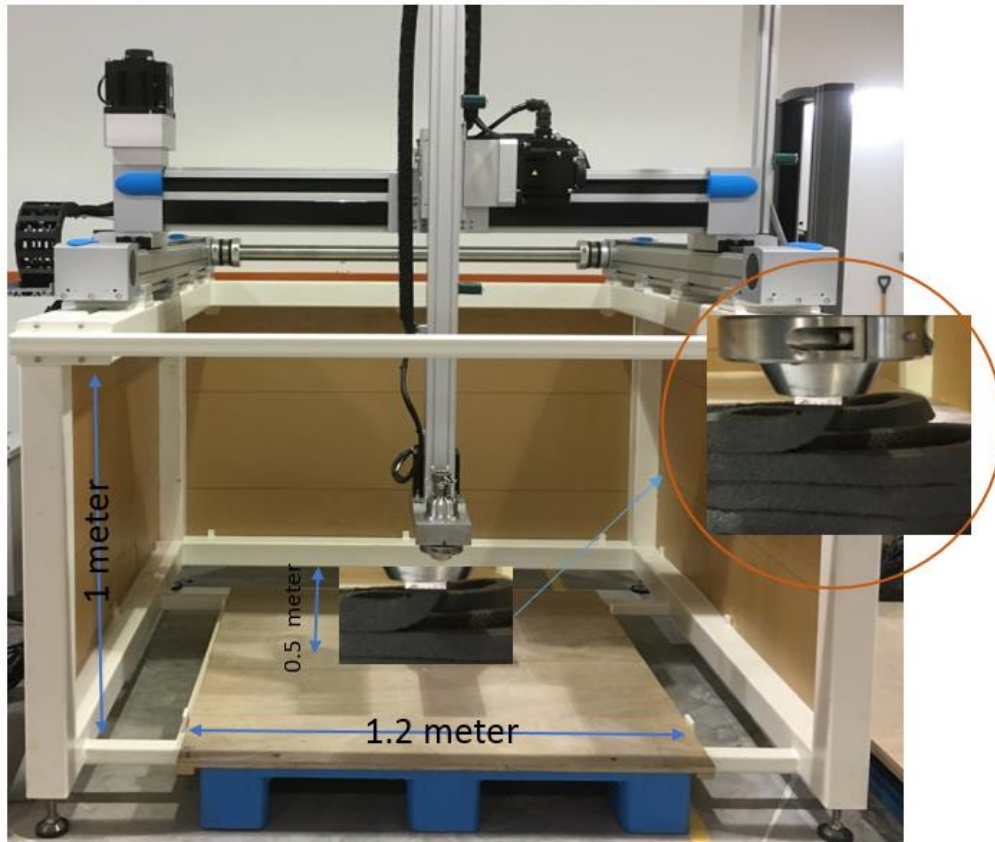


Figure 8: Geopolymer printing using 4-Axis gantry system

The fresh geopolymer mortar was placed directly in to the hopper of the pump and some material was pumped through the hose pipe (2.5-meter-long and 25 mm diameter) for one minute to avoid discontinuity in the extrusion process. For printing, the gantry speed was initially set at  $70\text{mm/sec}$  with respect to lowest flow rate of the pump i.e 1.5 lit/min. Figure 8 shows the complete

geopolymer printing process carried out in 1×1× 1.2 meter (length×width×height) workspace using rectangular and square nozzles.

**Table 1: Mix design of 3D printable geopolymer**

<b>Materials</b>	Fly ash	Slag (GGBS)	Silica Fume	Sand	K-Silicate	Water	Additives
<b>Content (Kg/m<sup>3</sup>)</b>	572.34	35.52	101.86	1219.74	140.739	144.09	10.05

### 3.3 Rheology of geopolymer

As mentioned earlier, fresh property of the material plays an important role in 3DCP and hence must be carefully studied prior to use for printing. The motivation behind rheology investigation is to get true information about thixotropic behavior and open time of the material i.e the time limit for the material to be extrudable through the nozzle. Here, we used viskomat XL rheometer from Schleibinger Testing Systems to measure Bingham parameters (yield stress and viscosity) of geopolymer. After loading the material in to the viskomat container, rotational velocity was increased from zero to sixty rpm in one minute followed by one minute constant shear at sixty rpm and then again getting down to zero in one time interval (see figure 9). **From the obtained torque and velocity (in rpm) information, shear stress, static yield stress and viscosity were calculated using our vane probe dimensions and container geometries [22].**

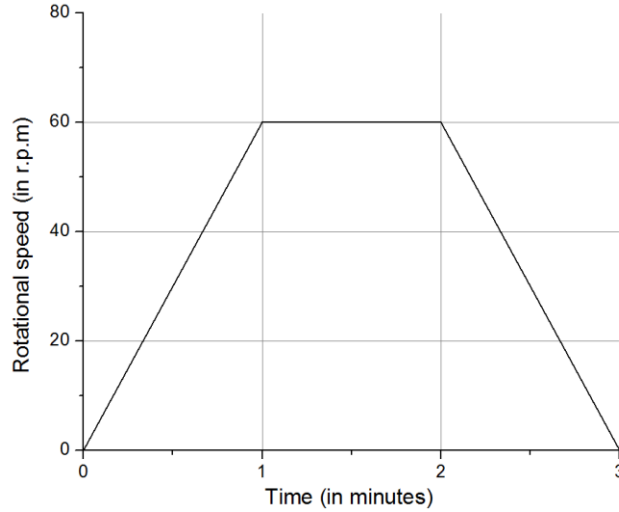


Figure 9: Rheology input to the rotational rheometer

Twenty liters of geopolymer mortar was used to track the change of shear stress with time and this was accomplished by conducting the test in five-minutes time interval starting from the mixing of geopolymer mortar. The obtained results and trend analysis was discussed in section 4.

### 3.4 Tensile bond test

To investigate the effects of printing parameters such as (a) printing time gap between layers (b) printing speed and (c) nozzle standoff distance (figure 10), geopolymer specimens were printed and then tested using INSTRON 5960 dual column tensile test machine for the bond strength (tensile). Selection of these parameters are purely based on our preliminary investigations and printer limitations [23]. 30 x 15 mm and 20 x 20 mm nozzles were used respectively for the experimental set up shown in figure 10 (a) and (b), (c). It is important to be note that, all the specimens in Figure 10(b) & (c) were prepared from the same batch of mix with a maximum three minutes printing time gap between two layers whereas, in case of figure 10(a), samples having less than 20 minutes printing time gap were from same batch while rest (35 min, 3h and 6h) were

printed with different batch of material for their top and bottom layers. Due to short open time of the material, it was difficult to use same batch of materials after 20 minutes.

For bond test, minimum 3 to 4 specimens of 50 mm length were extracted from a 350-mm long geopolymer block by sawing in a diamond cutter. Then two custom made mild steel plates were glued to both sides of specimen using rapid hardening adhesive (X60) supplied by HBM and testing was carried out at a rate of 0.05 mm/sec. Bond strength for each specimen was calculated by considering effective area of bonding and failure load. The average bond strengths were reported and discussed in the following section.

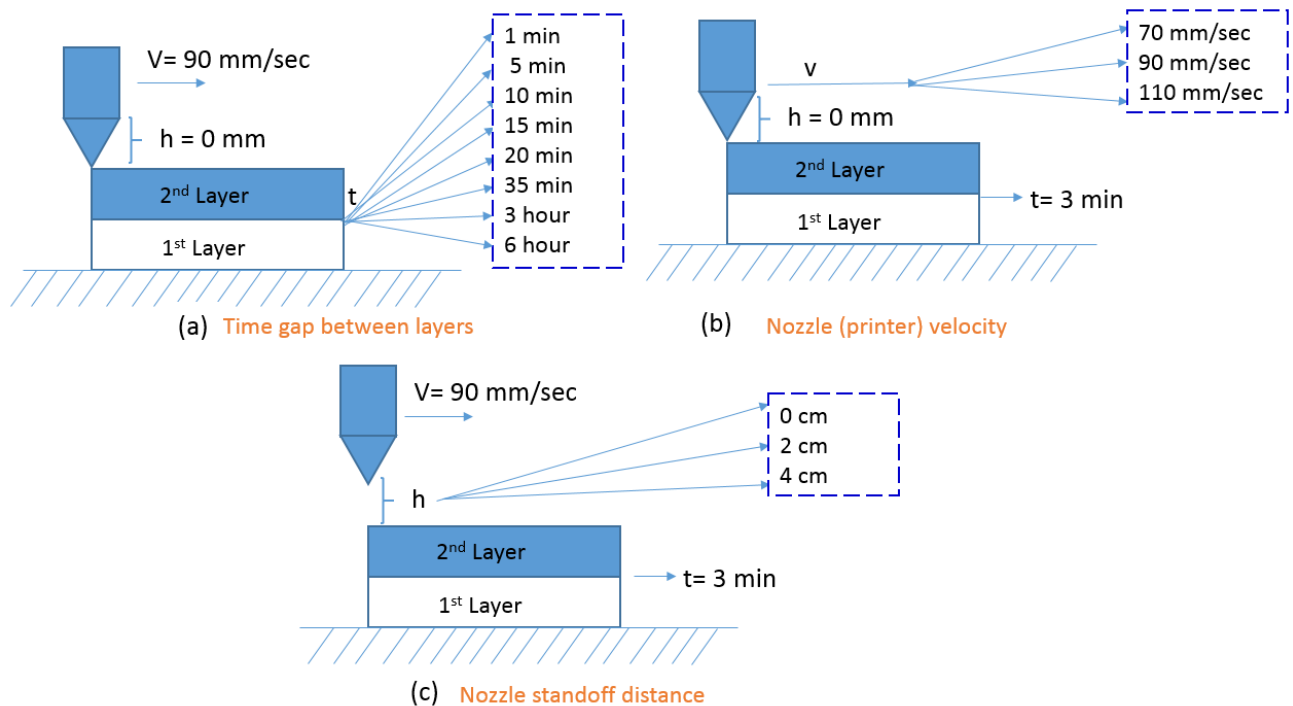


Figure 10: Effect of (a) printing time gap between layer (b) nozzle speed (c) nozzle standoff distance

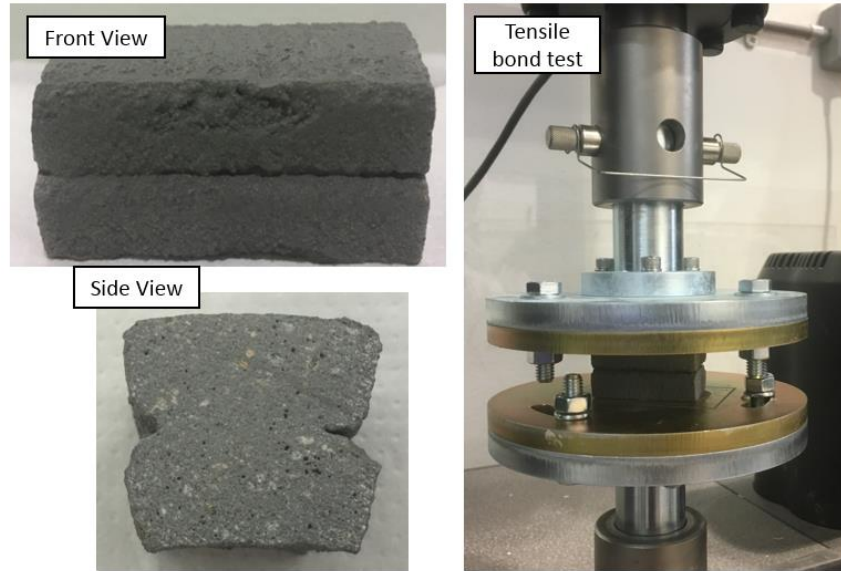


Figure 11: Tensile bond strength setup

## 4. Experimental results

### 4.1 Fresh and hardened properties of the geopolymer

Fresh property of the geopolymer, especially rheology, in terms of Bingham parameters (yield stress and viscosity) were obtained for every five-minute interval from the Viskomat T-N (torque-rpm) relationship. Like normal concrete, it was observed that yield stress and viscosity of geopolymer mortar increases with time due to poly-condensation reaction that usually occurs in this material [24,25]. Figure 12 (a) and (b) reveals the increase of yield stress and viscosity of the material after 20 min was quite significant and this stage of material indicates that the material has reached to its open time limit, thus further not extrudable. Therefore, the *time gap intervals* in section 3.4 were studied up to twenty minutes for same batch material.

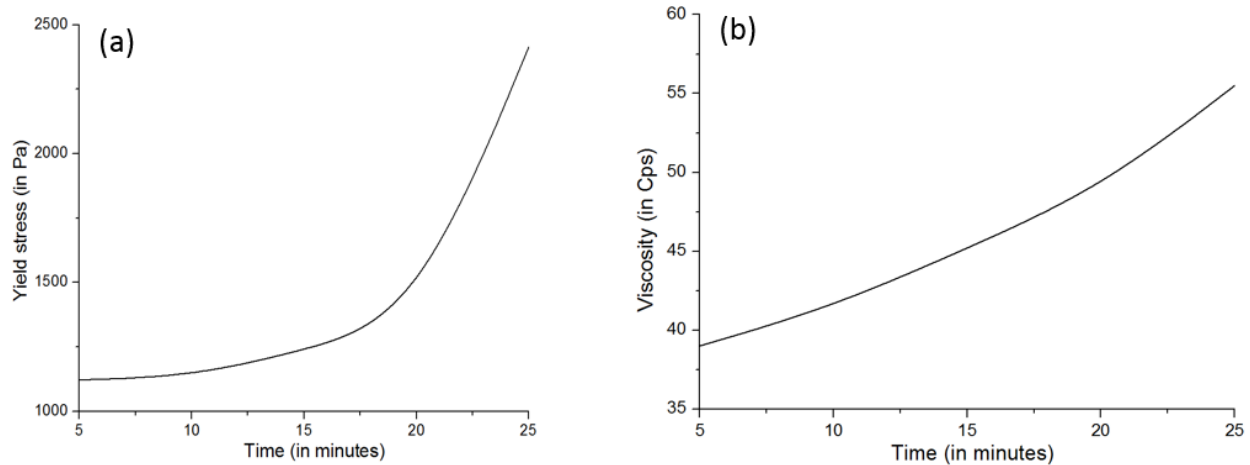


Figure 12. (a) Yield stress (b) viscosity trend of geopolymer mortar

Like fresh property, 28 days hardened properties of casted geopolymer such as compressive strength (50 mm cubes), tensile strength (dumbbell shape specimens with cross section 30 mm x 45 mm and gauge length 160 mm) and 3-point bending strength (40 x 40 x 160 mm<sup>3</sup> prism specimens) were measured as per BS EN 196-1:2016 standard. All test specimens were cured and tested in the ambient conditions. Table 2 reports the average strength results obtained by testing three samples for each test. Density of the printed geopolymer was found to be 2050 kg/m<sup>3</sup> i.e. higher than casted specimen (1900 kg/m<sup>3</sup>) probably because of high pressure exerted during the extrusion process.

Table 2. Hardened properties of geopolymer at 28 days

Compressive strength	Tensile strength	Flexural strength
36 MPa	1.63 MPa	5.05 MPa

#### 4.2 Effect of printing time gap between layers

Printing time gap between the layers is one of the most important property that governs the bond strength of printed structures. It depends on the perimeter of the 3D-Component and the printing speed. Here, as mentioned earlier, initially 350 mm long two layers (W30×H15) were deposited

one over another with time gaps of 5,10, 15 and 20 minutes. After twenty minutes, material was difficult to pump, therefore a fresh batch of material was deposited over the 1<sup>st</sup> layer with 35, 180 and 360 minute time gap intervals.

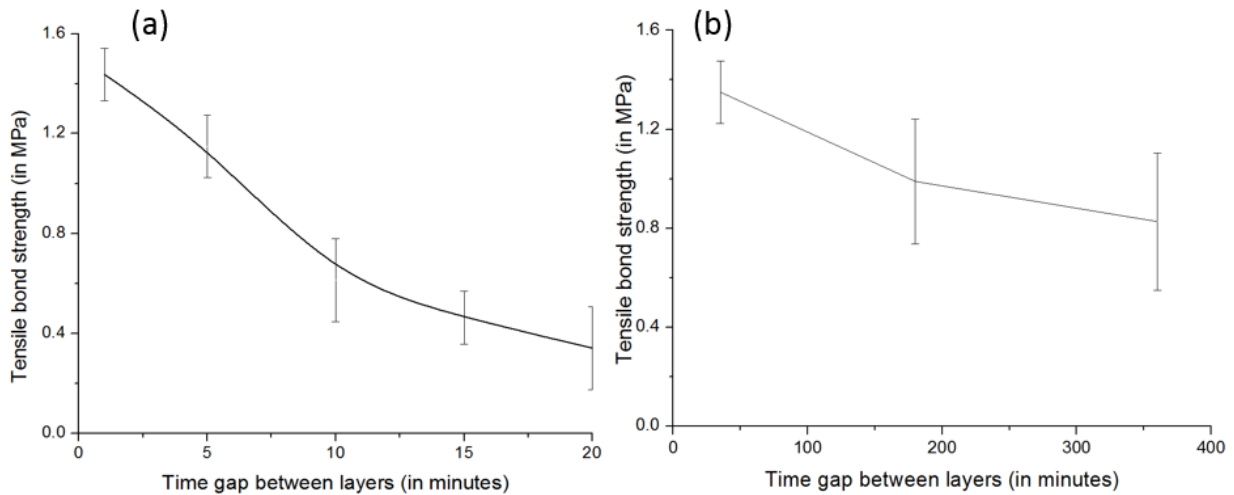


Figure 13: Effect of time gap for (a) same (b) different batch material

Figure 13 shows the result of tensile bond test with error bars, obtained by dividing maximum tensile force with effective bond area in between the two layers. The bond strength of 3D printed geopolymer was found to be decreasing with increasing time gap. Such decrement is understood as the result of interface layer property changes with time. Interface layer can be considered a thin film of layer, composed of very fine particles, that is usually present in the outer surface of fresh material. It also acts as a lubrication layer during pumping of concrete. However, when a fresh material starts to become dry after extrusion (with time), this interface layer diminishes and does not hold up the layers tightly. It can be seen from figure 14 that, for 1 min time gap between layers, the interface surface (after failure) is more rough compare to the 15-min time gap printing. This confirms the importance of the interfacial layers in bond strength measurement.

In geopolymer printing, due to sticky nature of potassium silicate, bonding between layers seems to be quite significant in first few minutes, however due poly-condensation process, material slowly becomes stiff and the bond strength decreases accordingly. A similar trend is also reported by group of researchers from Loughborough University, U.K by investigating possibility of using high strength cementitious material for large scale construction [8]. From the finding of figure 13 (b), it can be confirmed that bond strength for deposition of fresh geopolymer over the old geopolymer does not change much with time gap more than one hour, however for 35 minutes, the strength was found to be higher because of freshness of material. **The reduction in the bond strength for long printing time gap can be explained by considering a moisture exchange phenomena that states, when the bottom layer becomes drier with past of time, it absorbs more water from the freshly deposited top layer and simultaneously, some air present inside the bottom layer escapes out of it. This air stays entrapped at the interface and causes poor bond strength performances in the printed specimens [26,27].**

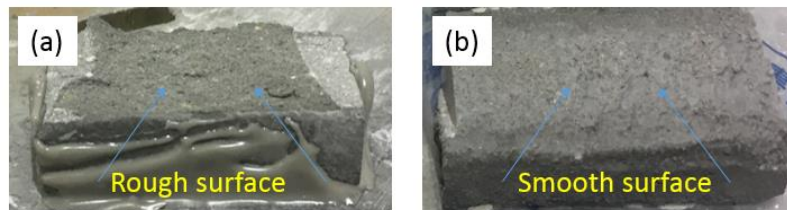


Figure 14. Failure patterns of bond test for (a) one (b) fifteen-minute time gap

#### **4.3 Effect of nozzle (printing) speed**

The role of nozzle speed in 3D printing process has been investigated by many researchers, however in concrete printing this effect is significantly visible due to thicker bead width compare to other extrusion based printing process [28,29]. In general, optimum printing speed was

determined by considering material property and pump flow rate. A balance between these parameters ensures a constant bead width throughout the printing process.

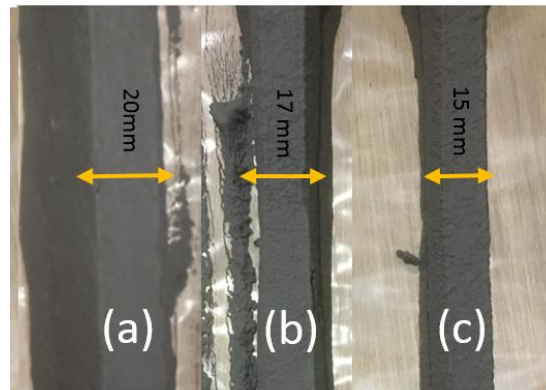


Figure 15. Effect of nozzle speed on bead width of printed geopolymer

Figure 15 shows the effect of three different speeds i.e (a) 70 mm/sec (b) 90 mm/sec and (c) 110 mm/sec on the printed geopolymer bead width. It's obvious to get smaller bead width for higher printing speed at a constant pump flow rate. In terms of, tensile bond strength, the effect was found to be not much different for the three different speeds, rather a wide variation was noticed in the bond strength values. Though the average bond strengths of specimens are close to each other, there was slight reduction in strength for 110 mm/sec speed which can be attributed to a combination of size effect and strain gradients in the cross section of the specimens, caused by the different speeds of the concrete printer [30].

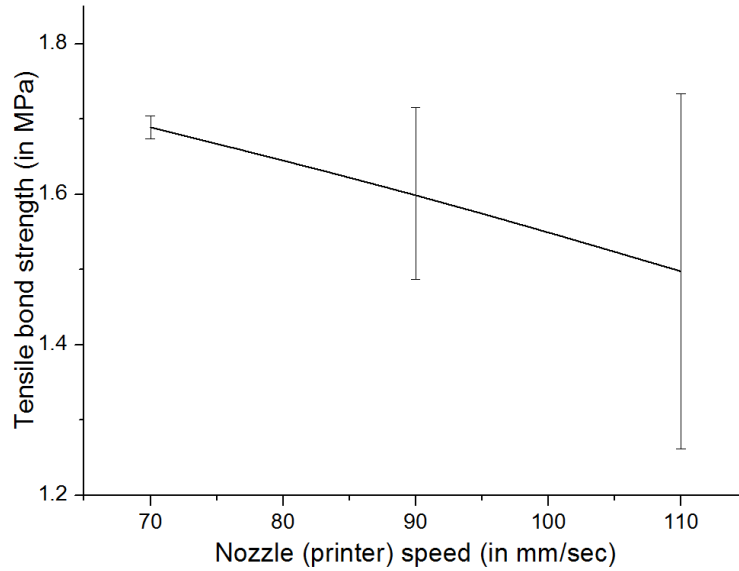


Figure 15. Effect of nozzle speed on tensile bond strength

#### 4.4 Effect of nozzle standoff distance

Position of nozzle above the printing surface (standoff distance) is one of the printer parameters that not only influences the bond strength but also specimen surface quality. The default standoff distance is considered equal to the nozzle section width, as this allows smooth deposition of filament and avoids interaction between the print head and filament [31]. In this research, the bond test specimens are printed and tested for different standoff distances considering default standoff distance as zero as shown in figure 16.



Figure 16. (a) Zero (b) Two (c) Four-millimeter nozzle standoff distances during geopolymer printing

Experimental findings (see figure 17) reveal that with decrease in standoff distances, bond strength increased and a wide variation is observed for two and four millimeter distances. A close look of the printing process helped us to understand that this variation was caused by slump/deformation of the bottom layer when a layer was getting deposited over it. Due to this deformation, the original standoff distance becomes large and at times, this phenomenon resulted in inaccurate bead deposition pattern, when the standoff distance goes beyond certain limit for the said material.

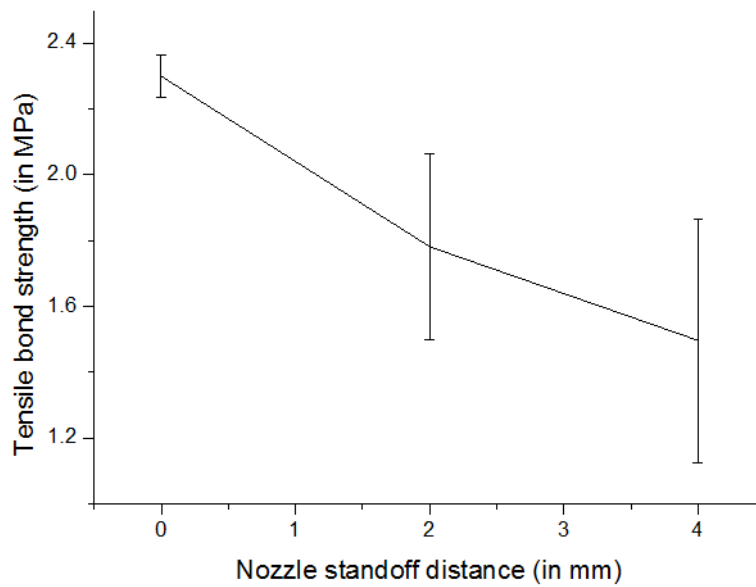


Figure 17. Effect of nozzle standoff distance on tensile bond strength

A recent research by Bos et al. [31] suggests to print layers by pressing the previously deposited layer (instead of depositing the layers from certain height) which can improve the interface adhesion and hence improve structural properties of the printed product. However, pressing of layers may affect the dimensional integrity and accuracy of the print geometry as it randomly changes the bead dimensions based on the intensity of the pressing between depositing layers.

## 5. Discussion and concluding remarks

Good bond is a key factor for providing monolithic action in 3D printing of concrete. Due to automatic layer deposition, there is a need to investigate interfacial bond strength, and its influencing factors for improving the structural performance. Traditional methods of surface preparation such as sand blasting, scrubbing have already been well studied in the literature in terms of their advantages, limitation for improving bond strength, however, in extrusion based printing, there are very few studies that reveals the complete picture of bond formation with respect to printing parameters. Therefore, this study takes the opportunity to explore the effects of different possible printing parameters on bond strength of geopolymer mortar. A novel formulation is made to print the geopolymer and tested in tension by varying printing time gap, printing speed and nozzle standoff distance considering capabilities of the gantry printer. **It can be reiterated from the experimental finding that, for the same batch of material, larger time gap between layers reduces the strength while effect of printing speed and nozzle standoff distance are better at their lower values. All these parameters are complement each other and must be limited to an optimum range to ensure smooth and continues printing. Depending upon the material and size, shape of the specimen, printer parameters can be optimized to improve the process performance. In this work, we set the open time around twenty minutes while targeting printing of the 350-mm tool path, however this can be adjusted as require by changing the mix composition of geopolymer.**

Following single layer printing, future work can be done on bond strength measurement in case of inter and intra layer adhesion by depositing multiple layers next to each other. Most importantly, interface mechanisms are needed to revealed by some advance scientific equipment like X-ray absorption, neutron radiography, NMR while comparing the findings of our experimental results. Additionally, researchers may take their interest on a different mode of bond test, i.e. shear bond

test for different combinations of material and nozzle sizes to examine their influences on layer adhesion phenomenon.

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