Optimization of Printing Parameters for Printing of Ordinary Portland Cement using a Custom-made 3D Printer

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Abstract. 3D printing techniques have recently received a lot of attention as a new technique for forming construction materials, due to benefits over the traditional cast. In this research, ordinary Portland cement (OPC) paste was formed using a 3D extruder. Optimized mixture and printing parameters were determined. Compressive strengths of the printed and the traditionally casted OPC paste were measured after the specimens were cured in saturated limewater for 7, 14 and 28 days, respectively. The result led to the conclusion that the most suitable mixture for printing was achieved at the water to cement ratio of 0.35 with water reducing and retarding admixture added. The optimum printing parameters were the printing speed of 60 mm/min and the feed rate of 120 ml/s. Compressive strength of the 5 x 5 x 5 cm³ hollowed and filled cubic printed specimens, were compared with casted cement paste. The strengths of both the printed and the casted specimens were higher with longer curing durations. The highest levels of strength were achieved at 12.36 MPa on the printed specimens and 80.88 MPa when testing the casted samples.

1. Introduction

Ordinary Portland cement (OPC) has been used for many years as a construction material. The traditional OPC forming method is by mold casting which limits freedom of design due to available formwork in the market. The method is time consuming and needs human labor to proceed. On the other hand, 3D printing or an additive manufacturing technique forms a product by adding cement layer by layer, resulting in borderless design. 3D printing has been used in various applications in several industries, such as in the architectural and medical industries for preparing prototypes and customizing complex geometry, because of its preciseness. The printing process is automated which requires less human labor and is less time consuming. The interest in using a 3D forming technique in building and construction applications has grown continuously. Extrusion [1-4] and powder bed [5, 6] techniques are two types of printer that are currently used in construction, as they offer higher safety, less material...
waste and less room for human error. Factors that affect the harden printed specimen are fresh properties of the material and printing parameters. The fresh properties are rheology and setting time, while the printing parameters are printing speed, pump flow rate, interval time of adjacent layers, nozzle height, nozzle shape and diameter [7]. The parameters vary according to printer and printed materials.

Filament for cement printing can be cement paste, mortar, and concrete [4, 8-10]. For example, a mixture of OPC, amorphous silica fume, polycarboxylic ether-based superplasticizer and viscosity modifier cement paste was printed by a customized 1 m³ steel printhead 3D extrusion-based printer [8]. The layer height was fixed at 7 mm, the interval duration between each layer was controlled at 100 s. The printing conditions were controlled by weight of the printed cement of 50±0.5 g. the 3D printer can also be used for fiber reinforced cement paste extrusion. Carbon, glass, and basalt were mixed with OPC, silica fume, water, water reducing agent and hydration inhibitor [11]. The pressure of 3 bar was applied to the mixture for the extrusion through a 2 mm nozzle with the printing speed of 30 mm/s. The height of each printed layer was 1.5 mm. Regarding OPC mortar, OPC was mixed with fine and coarse sand [9]. The ratio between sand and cement was 3.0 and the water to cement ratio was 0.5. The printing was done at a speed of 12 mm/s through a 25 mm x 15 mm nozzle.

It is significant to control the printing parameters since they affect the structure and properties of the printed cement. Therefore, in this research, the optimized mixture and printing conditions (printing speed, feed rate, and interval duration) were determined. The printed specimens were tested for the compressive strength at the curing age of 7, 14 and 28 days, and compared with the traditionally casted specimens.

2. Materials and experiment

2.1. Specimen preparation

Ordinary Portland cement (OPC) paste was prepared according to ASTM C305-20 [12]. Type I OPC clinker was mixed with water for 30 seconds with the water to cement (w/c) ratio of 0.35, 0.40 and 0.45. The mixture was stirred at low speed by a mechanical blender (C223 DTM, China) for 30 seconds. After that, the material around the bowl was scraped down within 15 seconds and mixed again at high speed for 1 minute. In addition, type B and D (ASTM C494) water reducing and retarding admixture (WRA, Sika Plastiment® R, Thailand) were introduced to the w/c 0.35 mixture. Adding WRA resulted in a reduction in the required amount of water for cement paste mixing to prevent bleeding behavior, and extension of the printability duration to prevent hardening of the paste inside the syringe. According to the product standard, WRA can be added to a mixture in the range of 200-500 ml/100 kg OPC, and the density is 5.7 g/cm³. Therefore, WRA concentration was experimented with, and the most appropriate concentration was 225 ml/100 kg OPC. WRA was firstly mixed with water and the ensuing process was the same as the ASTM standard. The freshly mixed paste was filled into a syringe for the extrusion. The nozzle of the syringe was an open circle with a diameter of 4 mm.

The printer was adapted from a fused deposition modeling (FDM) printer. After being filled, the syringe was fixed to a holder and pushed by a 3D printed plastic rod. The rod was connected to an electric motor with the maximum torque of 4.2 kg-cm.

To find optimized printing parameters, a 7 cm straight line was printed. The printing pattern was designed on Solidworks and converted to G-code using Slic3r. The printing speeds were 30, 40, 50 and 60 mm/min and the flow rates were 100, 105, 110 and 120 ml/min. The height of the nozzle head while printing was trialed and set at 7 mm from the substate, at this height the printed cement paste had the closest size to the Solidworks design.

The specimen for compressive strength measurement was prepared by printing cement paste into hollowed (Figure 1a) and filled (Figure 1b) cubic shapes with the dimension of 5 x 5 x 5 cm³. The printed specimen was left at an ambient temperature for 1 hour and kept in a box to prevent moisture loss. It was stored in a control room at 25°C and 50% relative humidity for 24 hours. Then, the specimen was
cured in saturated limewater for 7, 14 and 28 days. The same sized casted specimen (Figure 1c) was prepared by pouring the fresh cement paste into a cubic acrylic mold. For comparison purposes, the casted specimen was managed through the same process as the printed specimen.

![Figure 1](image1.png)

**Figure 1.** OPC paste specimens for compression test (a) hollowed printed (b) filled printed (c) casted

### 2.2. Characterization techniques

The chemical composition of OPC clinker was determined by X-ray fluorescence spectroscopy (XRF, Rigaku ZSX Primus, Rigasu, Japan), and particle size and particle size distribution were studied by Mastersizer 3000 (Malvern instruments Ltd., UK).

Vicat setting time and flowability were tested on freshly mixed OPC paste. Setting time was conducted according to ASTM C191-19 [13]. Initial and final setting times were evaluated using a 1 mm Vicat needle. Flowability of 0.35 with WRA added (0.35+WRA) was measured by a miniature slump cone test (Figure 2a), the measurement was adapted from a concrete slump cone test (ASTM C143/C143M-20 [14]). The height of the cone is 57 mm, and the original diameter is 38 mm. After mixing, the OPC paste was half-filled in an acrylic conical cone and tampered around on the top surface 20 times. The process was repeated on the other half. Excess paste was removed, and the cone was then lifted vertically. As can be seen from the Figure 2b, the specimen moderately collapsed, indicating that the mixture was not too wet or too dry. The diameter of the cement paste was measured 4 times, every 60 seconds up to 5 minutes and the average was applied. The flowability can be calculated using the equation:

$$%W = \left( \frac{d - d_0}{d_0} \right) \times 100$$

Where \%W is flowability, d is the average diameter of cement paste, and \(d_0\) is the original cone’s bottom diameter.

![Figure 2](image2.png)

**Figure 2.** (a) an acrylic conical cone for miniature slump test (b) mini slump tested OPC paste

A compression test of printed and casted specimen was carried out after the specimen were cured in saturated limewater for 7, 14 and 28 days by universal testing machines (UTM). The compressive strength of the printed specimen was tested using Instron 5567A with the loading speed of 0.1 mm/min and compared with the traditionally casted one at the same curing age. The compressive strength of the casted specimen was evaluated under a 50-ton Chun Yen testing machine (CY-6040A, China) according to ASTM C109/C109M-20a [15]. The strength in MPa was calculated by dividing the ultimate stress with the loaded area. The top and the bottom surfaces of each specimen were measured.
by a digital micrometer and an average was decided upon. At least three specimens from each mixture were tested and averaged for their compressive strength.

3. Result and discussion

3.1. Raw materials
The X-ray fluorescence (XRF) result shows that chemical composition of OPC clinker mainly composed of CaO and SiO$_2$ with the concentration of 69.95 and 14.72%, respectively. The other oxides that can be found at lower concentrations were SO$_3$, Fe$_2$O$_3$, Al$_2$O$_3$, B$_2$O$_3$, and MgO. The particle size of OPC clinker was distributed in a range of 0.128 - 127 μm. Most of the sizes of the particles were 17.2 μm with a specific surface area of 1381 m$^2$/kg.

3.2. Freshly mixed properties
Initial and final setting time of cement paste with w/c of 0.35, 0.40, 0.45 and 0.35+WRA were presented in Table 1. Initial and final setting time of freshly mixed OPC paste.” 0.35+WRA provided the longest initial and final setting time. It indicates that additional WRA in w/c 0.35 mixture can increase 56% of the initial setting time and 64% of the final setting time. The increment provides a benefit during the forming process, as extended printability of OPC paste. However, 3D printing is a layer-by-layer process, one requirement is that a printed layer must be strong enough to hold its shape and withstand the next layer’s weight. Consequently, the interval time between two printed layers is required to be extended. Otherwise, the structure collapses.

Table 1. Initial and final setting time of freshly mixed OPC paste

| w/c ratio | Initial setting time (min) | Final setting time (min) |
|-----------|---------------------------|-------------------------|
| 0.35      | 170                       | 255                     |
| 0.40      | 241                       | 358                     |
| 0.45      | 250                       | 345                     |
| 0.35+WRA  | 303                       | 400                     |

The flowability tests of w/c 0.35, 0.40, 0.45 and 0.35+WRA cement paste are presented in Figure 3. The flowability of all mixes were increased with time. Comparing the different w/c ratios, it can be discovered that for w/c 0.35 (Figure 3a), the diameter was only 3.7% larger when the paste was lifted for 5 minutes. Increasing the ratio to 0.40 (Figure 3b), the flowability of the cement paste rose to 20.92% at 5 minutes or 7.3% higher than that of w/c 0.35. A significant increase was observed when w/c ratio was increased to 0.45 (Figure 3c), the flowability increased by twice from w/c 0.40. From the numbers, it seems that higher w/c content provided better flowability, but the larger number was actually due to the bleeding. As a result, the WRA additive was introduced to w/c 0.35 (Figure 3d), and the flowability was approximately 5% higher than w/c 0.35 with a small bleeding detected. Therefore, 0.35+WRA cement paste was used as the filament.
0.35 (b) 0.40 (c) 0.45 and (d) 0.35+WRA cement paste

Figure 3. Flowability of (a) 0.35 (b) 0.40 (c) 0.45 and (d) 0.35+WRA cement paste

3.3. Printing parameter optimization

Printing speed of 30 and 40 mm/s gave a relatively symmetrical printed line with every feed rate. However, those speeds were too slow and caused the paste to harden inside the syringe. Increasing the printing speed to 50 ml/s with the feed rate from 100 to 120 ml/s, the line was asymmetrical due to too much material extruding at the beginning or at the end of the printing. When the feed rate was raised to 120 ml/s, the printed line was relatively symmetric with every speed rate. Therefore, a feed rate of 120 ml/s and a speed rate of 60 mm/s were chosen as the most appropriate conditions for maximizing the printability and productivity, because the material can be printed continuously without hardening inside the extruder during the process.

The most suitable mixture was also determined by varying w/c ratios. In general, higher flowability and longer setting time provide longer printability of the filament, consequently, the feed rate and speed rate can be lower. However, a significant disadvantage is that they lead to a longer duration for the cement paste to gain enough strength which means that the interval time of the printing must be longer and reduce the productivity. OPC paste hardened inside the syringe when the mixture of w/c 0.35 and 0.40 were used. Alternatively, bleeding of the printed specimen can be observed at w/c 0.45 due to high water content. Therefore, water reducing, and retarding admixture (WRA) was added to w/c 0.35 to get the most suitable mixture that did not harden inside the syringe and did not show any sign of bleeding. In addition, the mixture gained strength faster because of its lower water content. As a result, the subsequent layer can be printed quicker. The interval duration was shortened, and products can be produced more rapidly. Therefore, 0.35+WRA was used for the specimen’s preparation and the interval duration was 10 minutes.
3.4. Compressive strength

Load-displacement curve of 7 days-old hollowed cubic specimen is shown in Figure 4a. The load-extension graph was a nonlinear fluctuation, resulting from nonuniform stress distribution and breakage of each layer of the specimen. Moreover, the hollowed printed specimens were not symmetrical as can be seen in Figure 5a because the first layer did not have enough strength to hold its shape and withstood the following layers, subsequently the structure collapsed a little. Therefore, the 3D specimen design was changed from hollow cubic to filled cubic. In the case of filled printed cubic specimen, the specimen was more symmetrical in all directions and the lower layer can withstand the weight of the following layer as shown in Figure 5b. The stress was better distributed within the specimen, which was reflected in the linear load-extension curve (Figure 4b). The strength of 7, 14 and 28 days aged filled cubic specimens were 11.6±0.5, 11.7±0.3 and 12.4±0.2 MPa, respectively.

![Figure 4](image1.png)

**Figure 4.** Load-displacement curves of 7 days-old specimens (a) hollowed (b) filled printed cubic

![Figure 5](image2.png)

**Figure 5.** (a) hollowed cubic specimen (b) filled cubic specimen extruded by 3D printer

The OPC casted specimen showed higher compressive strength with a longer curing duration. The compressive strength of the specimen was 66.9±7.3 MPa at 7 days, 75.7±0.2 MPa at 14 days and 80.9±0.2 MPa at 28 days. Comparing the strength development of the casted with that of printed filled specimen, at the same curing duration from 7 to 28 days, the strength of the casted specimen was 21% higher while the printed showed only a 7% increase.

Lower compressive strength of the printed specimen than that of the casted one was also found in other research examples [4, 8]. The averaged compressive strength of 28 days aged 3D printed concrete was 8.3% and 28.5% decreased when compared with the casted concrete [16]. The decline in mechanical properties is mainly due to voids and weak bonding between adjacent layers. As in the traditional production, once the material is poured into a mould, it is tamped by a tamper according to the ASTM C305-20 process which can eliminate trapped voids and make the specimen denser, leading to better mechanical properties. Moreover, during the layer addition process, there is a possibility that air was trapped between the extruded layer which affected the stress distribution. It was reported that the nozzle shape also affected the strength development, as a rectangular nozzle created less void during the extrusion. The time gap between each layer possibly created a cold joint which lowered the bonding efficiency between each layer and resulted in losing moisture. Therefore, it is possible to improve the strength of the 3D printed specimens by altering the nozzle shape and size to optimize the design. Also,
the printed material can be changed to cement mortar which probably provides better early and later mechanical properties.

However, it should be noted that the strength development of 3D printed specimens was in an upward trend when being cured for 28 days, while that of casted specimens, showed a downward trend and displayed signs of beginning to be stable.

4. Conclusion

4.1. The mixture that provided the best extrudability without water bleeding was w/c 0.35 with WRA addition.

4.2. The best printing condition was the feed rate of 120 ml/s and printing speed of 60 mm/s in which the OPC can be continuously extruded without hardening in the extruder.

4.3. Although the 3D printed specimen provided lower compressive strength than the casted specimen, this method is still the future of construction building due to the ability of creating complex and customized designs.

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