Using Calcium Oxide and Accelerator to Control the Initial Setting Time of Mortar in 3D Concrete Printing

by Antoni Antoni

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Abstract. In recent years, 3D printing has attracted a lot of attention in the construction industry. Compared with general concrete construction, 3D concrete printing has higher flexibility in creating concrete's shape and design. 3D concrete printing requires the precise control of fresh concrete properties such as flowability, extrudability, and resistance to segregation during printing process. The initial setting time of the concrete also needs to be controlled as it needs to adhere to the next layer and then harden rapidly in order to support the upper layer. This study proposes a method to control the initial setting time of the concrete for the 3D printing process by using a mixture of calcium oxide powder and accelerators. The study showed that using 5–10% calcium oxide and 2–4% accelerator by mass of cement, the initial setting of the concrete can be varied. It is also shown that adding only accelerator prolongs the setting time of the mixture due to the plasticizer contained therein. By using calcium oxide powder, the initial setting time of the concrete can be hastened and the combination of calcium oxide powder and accelerator can reduce the initial setting time while maintaining good workability of the mixture. The addition of accelerator also increases the early compressive strength of the concrete mixture.

Keywords: 3D concrete print, calcium oxide, accelerator, initial setting time, compressive strength.

1 Introduction

Three-dimensional (3D) printing, also known as "additive manufacturing," is an advanced manufacturing process that can automatically generate complex geometric shapes from a computer-assisted model [1,2]. Currently, limited forms of building structures are available due to restricted shapes of formwork but with 3D concrete printing method various complex shapes can be constructed [3]. The 3D concrete printing construction method still faces many challenges that need to be address simultaneously [4], from the construction process[5–8], printing machines and its precision and tolerance [9,10], design and structural modelling [11,12], and the hardened properties of the fabricated member [13–15]. The material used in 3D printing has several factors that need to be considered, including extrudability, shape retention, thixotropy and buildability [16–18].

In 3D concrete printing, extrudability can be defined as the ability of a material to be pumped out through the extruder or nozzle without any interference/blockage in the pipe [19]. Like extrudability, shape retention is also an important factor for 3D concrete printing. After extrusion, the material must retain its shape according to the dimensions of the extruder. Thixotropy can be defined as the time interval during which the material loses its extrudability; for 3D printing concrete, this is always earlier than the setting time [20].

In 3D printing, buildability is a challenging problem. To overcome this, the material that has been extruded must produce sufficient viscosity and yield stress before the second layer begins to fall on it [21,22]. The main challenge is to determine the best mix of materials to be able to flow out from the printing nozzle smoothly as well as to make a mortar that does not experience slump or sagging and self-compaction after extrusion [23,24]. Although these two requirements contradict each other, they can be achieved by successively separate processes. First, the material must be extrudable and maintain its shape when applied to a printing bed. Second, the layer that has been applied must not sag with the application of the layer above it. Finally, the material must have good bonding between layers in order to achieve better rigidity and strength. Therefore, materials that have high yield stress and low viscosity are the best materials for this method [25,26].

The exact composition of the binder and aggregate, and the particle size distribution should be designed carefully for better printability of different construction designs [27–29]. Various additives, such as superplasticizer, retarder, and accelerators, can be used to increase the strength of the printed material. An accelerator is not sufficient to produce such material if the ratio is not adjusted correctly while an inadequate retarder does not allow the material to be pumped through hoses, which can damage the pump and the distribution system [26].

One behavior of the fresh concrete properties that also influence the buildability is the windows of time for the best printing result [30,31]. After the addition of water into the mixture, certain time need to be elapsed for the chemical reaction to proceed before the fresh concrete exhibit a suitable yield stress and viscosity for the printing process. This behavior further complicated the printing process and the material need to be precisely controlled for the optimum results. The optimum period is closely related to the initial setting time of the mortar, as it is varied with the mixture composition of the fresh mortar.

This study explores the use of local sand and cement as the material for the 3D concrete printing process and calcium oxide powder and accelerator to control the initial setting time of the mortar mixture. The high content of calcium oxide in cementitious mixture is known to cause flash setting in concrete [32], and adding small amount of it can hasten the setting time of the concrete and give the concrete structural strength to sustain the following layer. To modify the yield stress and viscosity of the mortar matrix, accelerator with plasticizer effect is also added in to the mixture. By adding or reducing the amount of the admixture, the setting time hence the printing time can be controlled. Such a parametric material study can show the behavior of the additive and can be used as a guide to modify the mix design to comply with machine and design requirements. The evaluated fresh and hardened mortar properties include workability, initial setting time, and compressive strength.

2 Experimental Study

2.1 Materials and Mix Design

The fresh and hardened behavior of 3D-printed concrete is studied by investigating the mortar mixture with various admixture. The fine aggregate was sourced from Lumajang quarry in East Java, the cement used was Ordinary Portland cement from Semen Indonesia, the calcium oxide (CaO) powder was obtained from a local producer, and the accelerator used was Sikacim from SIKA. The fine aggregate was graded before mixing to ensure uniform consistency. The Fineness Modulus of the sand used was kept at 2.19. CaO was selected to increase the strength and to speed up the initial setting time and accelerator was added to improve the workability and early age strength of the mortar. Before deciding on the mix design for this study, several preliminary tests were conducted to investigate the dosage required for each parameter, additional admixture such as superplasticizer and calcium hydroxide was also considered as the potential candidate to accelerate the initial setting time, however the effect was found to be reversed, i.e. the initial setting time of the mixture was prolonged.

The mix design for 3D concrete printing was compiled from other studies where the mass ratio of sand to cement can be varied from 0 to 2.5. Adding more sand can increase the yield stress but susceptible to bleeding occurrence, while the addition of fine powder such as fly ash, nano silica, or nano clay can improve the cohesion of the mixture [16,28,33]. Hence for the current study, the ratio of sand to cement was selected at 0.5 to reduce the need of other fine powder material in the mixture. The high amount of cement in the mixture also can provide a high strength buffer due to the imperfect condition of the printing process, the bond between layers, and uncertainty in the mortar compaction. Water to cement ratio was kept at 0.3 from the preliminary trial that give the optimum workability. The resulting mix designs investigated in this study are shown in Table 1.

Table 1. Mixture codes and mix design of the mortar

Mix Code	w/c	Water (gr)	Cement (gr)	Sand (gr)	CaO (gr)	Accelerator (gr)
C					-	-
CA2	0.3	60	200	100	-	4
CA4					-	8
CCo5					10	-
CCo5A2	0.3	60	200	100	10	4
CCo5A4					10	8
CCo10					20	-
CCo10A2	0.3	60	200	100	20	4
CCo10A4					20	8

2.2 Specimen Preparation and Testing

Materials and equipment were prepared according to the mix design. Mixing was done by dry mixing cement and sand evenly before pouring water into the mixture. The accelerator was then slowly added while stirring followed by the CaO powder. After the mixture was uniform, it was poured into the flow table cone to measure its flow diameter in the flow table test according to ASTM C230 [34]. The test was carried out at room temperature ranging from 28 to 30°C and at a relative humidity of 78–80%. The target flow diameter can be correlated with the yield stress of the mortar, and the flow diameter of 13-23 cm is suitable to be used as the printing material for the 3D concrete printing process, with the optimum value at 15-19 cm, according to Tay et al [30].

The mixture was then cast into 5 cm cube formworks for compressive strength test and filled halfway into 15 cm cube molds for the setting time test. The setting time test was done using a mortar penetrometer and the penetration stress recorded in correlation with time since the addition of water. The mortar penetrometer test was conducted in according to ASTM C403 [35].

The compressive strength specimens were cured in water at room temperature until one day before the compressive strength test. The compressive strength test is conducted at 3, 7, 14, and 28 days from casting, with three replications using universal testing machine.

3 Results and Discussion

3.1 Preliminary Study

A preliminary study is needed to determine the material variables that would be used as the boundary condition in this study. The change of initial setting time is an essential factor in 3D concrete printing and the dosage of admixtures that influence the change is still unknown.

The materials considered as the admixture were calcium oxide, calcium hydroxide, accelerator, and superplasticizer. It was observed that each admixture has a different effect on the initial setting time. The addition of superplasticizer always causes longer initial setting time because the water to cement ratio was kept constant for all mix designs. Combination of superplasticizer and calcium oxide or calcium hydroxide always resulted in longer setting time than the control (cement only) mixture. Adding calcium hydroxide only also has a slower initial setting time than the control mixture, similar to the superplasticizer, even though it is combined with an accelerator.

Meanwhile, the accelerator has a similar initial setting time with the control mixture and the addition of calcium oxide powder always produced a mixture with faster initial setting time.

After conducting a preliminary test, superplasticizer and calcium hydroxide were not used because they did not accelerate the initial setting time. When a longer initial setting time is needed, for example, with long printing cycle time, these admixtures can be considered. Accelerator, when combined with calcium oxide, produced faster initial setting time than the mix design control. The CaO powder dosage used was 5% and 10% of the cement mass and the accelerator was used at 2% and 4% of the cement mass; thus, their combined effect on the initial setting time could be observed. The maximum limit for CaO was set at 10% due to possible flash setting and low workability at higher CaO dosage.

3.2 Workability Control

The flow diameter measures mortar workability in the flow table test. The accelerator dosage is limited to 4% because the initial setting time can be extended further when adding dosage beyond that, as the accelerator chosen in this research also contain some plasticizer. The result of the flow diameter of the mixture is shown in Fig. 1.

Adding CaO to the mortar mixture reduced the workability slightly while the accelerator produced a larger flow diameter. Addition of CaO at 5% and 10% reduced the flow diameter from 14.6 cm to 14 cm and 11.8 cm, respectively. The addition of 2% and 4% accelerator, by mass of cement, increased the mixture flow to a pumpable condition. This result showed that CaO and accelerator could be added together to control the initial setting time and workability of the mortar mixture.

The mixture composition of the mortar still need to be adjusted with the parameters of the printing machine, thus the optimization process of the mix design still excluded the addition of supplementary cementitious materials into the concrete mixture. Furthermore, to control the consistency of the mixture, viscosity modifying agent (VMA) can also be added into the mixture when the flow diameter is too high [36].

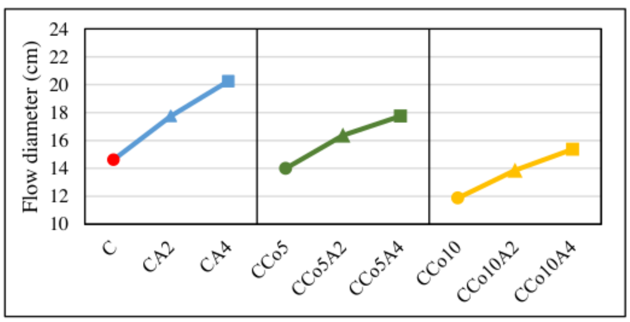

Fig. 1. Flow diameter of the fresh mortar with variations of additive.

3.3 Initial Setting Time

The initial setting time test was carried out using the mortar penetrometer at 15-minute intervals for all of the mixtures. The initial setting time is defined as the time needed until the penetration pressure reached 500 psi, however the changes of the penetration resistance can give the indication of the hardening process in the mortar mixture. Higher penetration resistance showed increase of stiffness and strength to sustain the upper layer in the 3D printing process. This hardening process is essential in reducing the elastic buckling and plastic collapse at high upper layer count [37].

The change in the mortar's hardening process can be observed by its initial setting time as seen in Fig. 2. The solid red line was the control mixture without any admixture. It can be seen that the time needed to reach the initial setting time decreases with increasing CaO dosage. The reduction of setting time was shown to progress at exponential rate with the increase of CaO dosage. At 5% dosage, the setting time was reduced 21 minutes, while from 5 to 10% dosage, the initial setting time reduction was 40 minutes. A faster setting time occurred when adding CaO mostly because of the increase of temperature due to the exothermic reaction of the CaO. At higher CaO dosage the mixture could cause very rapid setting even flash setting and could reduce the workability of the mixture.

On the contrary to the CaO, the initial setting time increased with increasing accelerator dosage. The mortar mixture with addition of accelerator only have longer initial setting time compared to the control mixture, however the benefit of adding accelerator is found on the increase of workability and the early strength of the mortar. The combination of CaO and accelerator changes the initial setting time at a different rate and can be utilized to control the behavior of the fresh concrete in the 3D printing process.

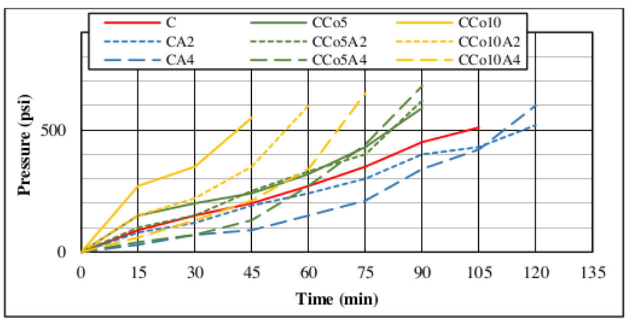

Fig. 2. Penetration pressure into the fresh mortar to measure the hardening process with time.

3.4 Compressive Strength

The compressive strength test was carried out to examine the effect of adding CaO and accelerator on the mortar strength. The concrete compressive strength test was carried for each variable at 3, 7, 14, and 28 days. The results of the mortar compressive strength test are shown in Fig. 3.

The compressive strength of the mortar was very high due to the low sand to cement ratio and low water to cement ratio. The high cement content also aimed to cause a faster initial setting time and increase the cohesion of the mixture. The use of accelerator was also found to increase the early strength and later strength of the mortar mixture. Adding 5% CaO did not have any detrimental effect on the compressive strength. At 10% CaO, there was a reduction in the compressive strength of the mortar. However, with the addition of accelerator, the final strength of the mixture for each CaO series can be increased and is higher than the control mortar.

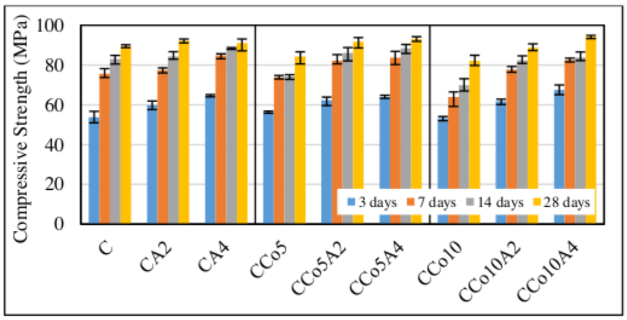

Fig. 3. Increase of compressive strength of the mortar specimen with age.

The effect of using an accelerator on the initial setting time tends to be less beneficial but not detrimental because it does not slow down the initial setting time. However, when considering its benefit to increase the compressive strength, it is more beneficial because the accelerator increases the initial and final compressive strength. This is shown by the CA4 mixture strength at 3 days of 64.6 MPa while the 28-day compressive strength was 91.1 MPa.

Using only CaO reduced the initial and final strength of the mortar with a higher strength loss at higher CaO dosage. The CCo10 achieved a 3-day strength of 53.3 MPa and 28-day strength of 82.0 MPa, and lower than that of the control mortar. The best combination of faster initial setting time and compressive strength was found with the CCa10A4 mixture with an initial setting time of 60–90 minutes and the highest 28-day compressive strength of 94.3 MPa.

4 Conclusions

The results of this research lead to the following conclusions:

- The combination of calcium oxide and accelerator as an additive can control the initial setting time of the concrete material needed in the 3D concrete printing process. Calcium oxide can cause a higher reaction rate in the concrete while the accelerator can control the concrete flowability and increase the compressive strength.
- Calcium oxide can hasten the initial setting time of the concrete; however, the dosage should be limited to 10% to avoid possible flash setting or rapid hardening at higher dosage.

- Mortar workability needs to be increased by the use of accelerator as the calcium oxide tends to reduce the workability. The target flow diameter should be greater than 13 cm in the flow table test to ensure a good extrusion process.
- By changing the CaO and accelerators dosage, the optimum printing window can be adjusted to achieve the best rheological properties of the fresh concrete. However the optimum dosage need to be determined to comply with the printing machine parameter and concrete mixture.

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