Selected Test Methods for Assessing Fresh and Plastic-State 3D Concrete Printing Materials

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Abstract. Materials' requirements for 3D concrete printing centre around printability and buildability. The concrete must be pumpable, extrudable, yet retain its shape after extrusion (fresh state) and stack over each other without yielding and buckling failure (plastic state). The nature of 3D printed concrete materials has driven research into adapting various test methods to assess these requirements. This study reports selected test methods evaluated for 3DCP in its fresh and plastic state – rheometry, unconfined compression tests, gravity-driven (slug) tests and slow penetration tests. The study further highlights their correlations, applications, and practicality for the two main types of printable materials – enhanced-thixotropic and accelerated-hydration concrete.

Keywords: Thixotropy · Accelerator · Slug test · Slow penetration test · Yielding and buckling failure · Rheometry

1 Introduction

The major requirements for 3D concrete printing (3DCP) can be categorised into pumpability, extrudability, printability, and buildability [1]. Two major approaches of materials formulation are widely used to achieve these – enhanced-thixotropic and accelerated-hydration concrete. The former allows for a time-limited reversible moderate increase in yield strength (structuration) of the fresh material by enhancing their thixotropy, using viscosity modifying admixtures, while the latter finds its origin in hydrates nucleation leading, in time, to an irreversible structuration of the material [2] using set accelerator near the printing head/nozzle.

These stringent requirements give rise to generally stiff fresh concrete with fast-changing materials properties that are not easily compatible with existing test methods. Therefore, new test methods are being developed by researchers to evaluate the material properties and guarantee that the target requirements are satisfied. Some of these selected test methods include rheology tests, unconfined compression, gravity-driven tests, and cone penetration tests [3].

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The rheometry tests are generally undertaken to evaluate if the printable material is pumpable. In addition, materials that are not too stiff can also be tested for rheology to ascertain that they retain their shape and have adequate structuration for buildability. The compression tests most often evaluate material properties for buildability, especially stiff materials. Accelerated materials are generally out of the scope of the former tests and easily lend themselves to penetration tests. Gravity-driven tests are targeted at the material's properties obtainable at the print nozzle for shape retention evaluation. The present study applied the selected test methods on two printable mixtures, enhanced-thixotropic and accelerated-hydration concrete for comparative purposes and application emphasis.

2 Materials and Methods

The mortar for printing consists of preblended dry (powder) materials of Portland cement, fly ash, silica fume (70, 20, 10%, respectively) and fine sand formulated for high-performance 3D concrete printing (Fig. 1a) [4, 5]. The enhanced-thixotropic mixture was formulated to allow satisfactory structuration and an open time of about 2 h. For printing, the mixture is pumped through a Ø33 mm and 5 m length pipe to a 20 mm nozzle attached to a robotic arm. For the accelerated-hydration mixture, a secondary dynamic mixer is attached to the printer head where an accelerator is dosed as a two-part system. These mixtures have been tested at Loughborough University laboratories to satisfy the requirements of 3DCP for targeted printing (e.g., Fig. 1b), and the results of their properties evaluation are reported in this paper.

3DCP mixture		Thixotropic	Accelerated
Pre-blended dry materials (kg/m^3)		2074	
Water	content (kg/m^3)	212	
	w/b ratio	0.26	
polycarboxylate ester-based superplasticiser (kg/m^3)		5.81	12.45
Cellulose-based consistency retainer (kg/m^3)		3.32	1.41
Aluminum-sulphate based accelerator		-	41.5

(a)

(b)

Fig. 1. (a) Materials' constituents for the high-performance 3D concrete printing (b) 2.5 m column build height within 3 h and cantilever build at 20, 30, 40 & 50° to the vertical

2.1 Rheometry and Plastic-State Compression Tests

HAAKE Viscotester iQ with a rotor vane in cup geometry was used to evaluate the rheological properties such as yield stress, modulus, plastic viscosity and structuration. A flow curve test was used to evaluate the dynamic yield stress and plastic viscosity [6].

This test is usually targeted at the pumpability of the mixtures. The static yield stress and its development over time (structuration), targeted at the extrudability and buildability (yielding failure) of thixotropic mixtures, was evaluated up to 60 min concrete age using a single sample [6–8]. The shear modulus of the thixotropic mixture, targeted at the buildability (buckling failure), was evaluated using the small amplitude oscillatory shear (SAOS) [8, 9] up to 2 h of concrete age.

The unconfined compression of the thixotropic mixture, targeted at the buildability (yielding and buckling failure), was evaluated to determine the compressive strength and modulus up to 2 h of concrete age (Fig. 2a) using a Ø70 × 140 mm mould. True strain measurements of the specimens under a 1 kN load cell were captured by a LaVision DIC measurement system.

(a) (b) (c)

Fig. 2. (a) Plastic-state compression setup, (b) Slug test setup, (c) Slow penetration test set up

2.2 Gravity-Driven (Slug) and Slow Cone Penetration Tests

For both the thixotropic and accelerated mixtures, an automated "slug" test system built at Loughborough University ABCE laboratory (Fig. 2b) [3, 10] was used to estimate the yield stress at the nozzle in real-time (Eq. (1a) for the evaluation of the extrudability/shape retention. Slow penetration of Ø100 × 200 mm samples under a needle attached to a 10 kN load cell (Fig. 2c) was also used to estimate the yield stress evolution (structuration) up to 2 h and is usually targeted at the buildability (yielding failure). Estimation of the yield stress is based on Eq. (1b) [11].

$$\tau = \frac{mg}{\sqrt{3}A}; \tag{1a}$$

$$\tau = \frac{F}{\pi r(\sqrt{r^2 + h^2})} \tag{1b}$$

where m – slug mass, g – acceleration due to gravity, A – nozzle cross section area, τ– residual yield stress, F – force, r – radius of cone needle, h – height of cone needle.

3 Results and Discussion

The steady-state properties (Fig. 3a) of the mixtures is such that segregation is avoided. The rheology tests seem to be sensitive to capture the two-stage material behaviour – the initial stage of restructuration and later stage of lower structuration rate. The plastic-state compression tests are not sensitive enough to capture the initial restructuration. These stages are critical during printing because this is when the deforming loads from overlaying filaments are resisted by the underlayers.

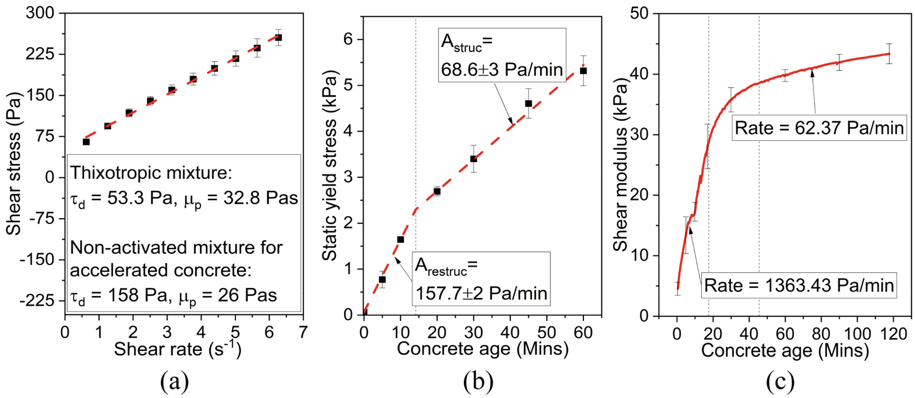
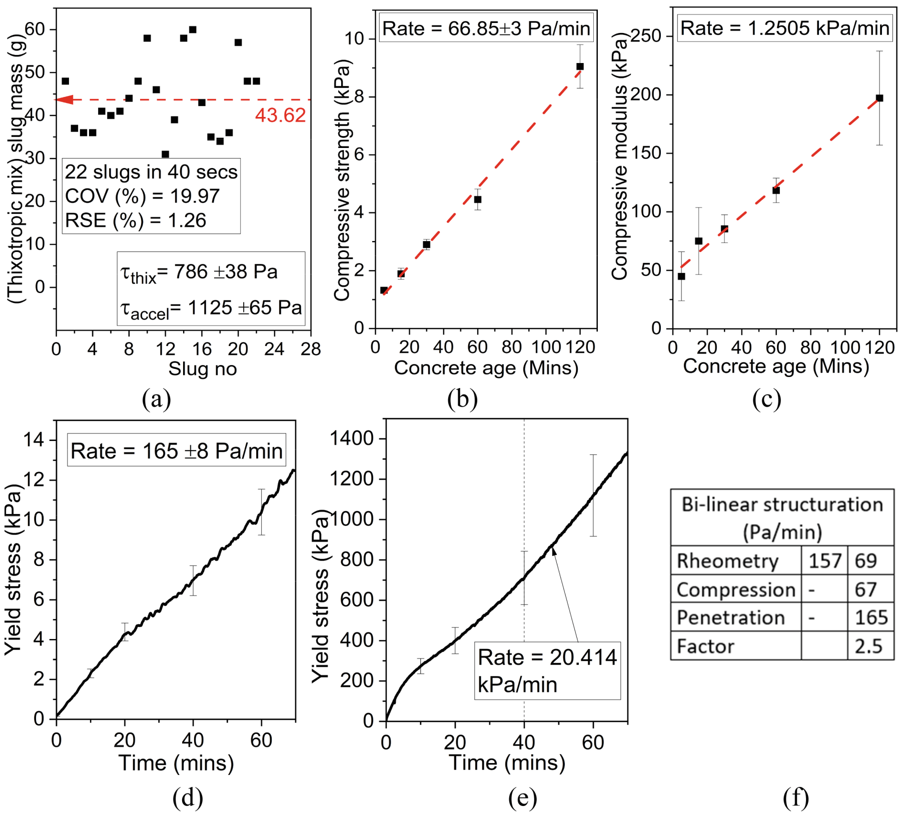


Fig. 3. (a) Flow curve test results (b) stress growth result (c) SAOS results (τ_d – dynamic yield stress, μ_p – plastic viscosity, $A_{restruc}$ – rate or restructuration, A_{struc} – rate of structuration).

As noted earlier, the application of rheometry to accelerated concrete is unrealistic but is adaptable to penetration test. Question arises on how penetration yield stress values correlate with rheometry values. Within the limits of this study, Fig. 4f shows that a factor of about 2.5 exists between the structuration rates of both methods. This can be traced to the origins of internal friction of the mixture, adhesion to cone and heave of displaced mortar. Assuming similar internal friction (particle interaction) within the accelerated concrete, this will yield a structuration rate of 8.2 kPa/min instead of 20.4 kPa/min. The compressive strength seems to have a similar structuration rate (67 Pa/min) with the shear static yield strength from the rheometry (69 Pa/min), but the relation of their stiffness (modulus) evolution seems unclear. The slug test results in Fig. 4a reveal that the yield stress at the printer's nozzle (786 Pa) is quite dissimilar to the dynamic yield stress envisaged at the rotor-stator of the pump (53 Pa in Fig. 3a). At the flow rate of 1.4 kg/min, there is a lag of about 7 min between the pump and nozzle, allowing for some sort of structuration within the pipe. This should be considered in the extrudability of 3DCP, which is obtainable from the 7 min yield stress evolution of Fig. 3a.



Bi-linear structuration (Pa/min)		
Rheometry	157	69
Compression	-	67
Penetration	-	165
Factor		2.5

Fig. 4. (a) Slug test results (b) Compressive strength of the thixotropic mix (c) Compressive modulus of the thixotropic mix (d and e) Penetration results of the thixotropic & accelerated mixtures (f) Relation between results.

4 Roadmap for Application and Conclusion

Table 1 highlights the application and suitability of the selected test method for evaluating the requirements of 3DCP. "Difficult" represents a situation where it is practically impossible or a struggle to yield reliable results. The table is subjective to the authors' experience, and the figure of the broken shear vane highlights the difficulty of trying SAOS with the accelerated concrete for a few minutes.

The rheometry is fairly easy to execute for a trained specialist but limited in the range of materials it can be tested with; very stiff, highly thixotropic and rapid setting materials usually exceed the torque limit of classical rheometers within tens of minutes. Plastic-state compression tests tend to be practicable for stiffer materials, and due to the plastic nature of the tested material can make the evaluation of compressive modulus quite tricky. The slug test is a new method for estimating yield stress and requires a specialist setup for its automation. It physically evaluates the material properties as it is obtained from the nozzle, unlike others that simulate the process. The slow penetration test result

Table 1. Test method application and suitability for HP-3DCP requirements

3DCP mixture	Thixotropic	Accelerated	Suitability
Rheometry	Rotational & SAOS - good	SAOS – difficult	Pumpability, extrudability, printability, buildability
Plastic-state compression	Fair	Very difficult	Buildability
Slug test	Good	Good/fair	Extrudability, shape retention
Slow penetration test	Difficult	Good	Buildability

can be subjective and higher in value due to influencing factors; within the limits of this study, its value deviated by a factor of 2.5 from the well-established rheometry method.

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