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Experimental analysis on rheological properties for control of concrete extrudability

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Abstract. In this study, we examined the relationship among the rheological properties, workability, and extrudability in the construction of concrete structures using additive manufacturing. We altered the component materials (binder type, water-binder (W/B) ratio, sand ratio) to assess their effect on the rheological properties experimentally. The results indicated that the W/B and sand ratios had the largest effect on the rheological properties. In particular, when the sand ratio increased, it indicated that adjusting the sand ratio would facilitate control over the rheological properties. Additionally, we compared the rheological properties with the results of a traditional workability evaluation, namely the table flow test. This indicated the possibility of inferring the rheological properties by using traditional methods. Finally, we evaluated extrusion quantity according to table flow. The extrusion rate was 350 g/s for a flow of 210 mm and 170 g/s for a flow of 130 mm, indicating that extrusion rate increased as flow increased; however, we concluded that a flow standard of approximately 140-160 mm is suitable for controlling the actual extrusion quantity and rate.

Keywords: 3D printing; additive manufacturing; concrete; flowability; extrudability; rheological properties

1. Introduction

3D printing construction technology started with US contour crafting in the late 1990s (Khoshnevis 2004) and has increased substantially worldwide since 2010. Loughborough University (Le *et al*. 2012a; Le *et al*. 2012b), Surrey University (Airey *et al*. 2013; Alwi *et al*. 2013), and TU Eindhoven (Bos 2015) have all researched 3D printing for construction, as well as the concrete materials suitable for different printing devices, and they successfully constructed creative architectural structures. Loughborough University (Le *et al*. 2012a) used a high-performance concrete mix for 3D printing and evaluated the inherent properties such as flow characteristics and setting time. Extrudability and buildability were examined through an extruder, and an optimal mix proportion for the device was selected. Surrey University (Alwi *et al*. 2013) conducted a study on equipment development and control methods by examining the compatibility of the extruder with various mixes.

Table 1 Concrete properties and their definition

Required property	Definition
Workability	Fluidity required to transport mixture to the extrusion devices
Extrudability	Fluidity required to extract mixture from the printer
Buildability	Support capability of each layer for additive manufacturing
Open time	Time in which extrudability is retained after mixing

The produced concrete must have a certain workability from pumping to the moment of extrusion in the printing phase; after extrusion, workability must be lost quickly to develop strength for layering. The properties required for concrete used in 3D printing are workability, extrudability, buildability, and open time (Le *et al*. 2012). In this study, the terms are defined based on previous studies and our experiences, as shown in Table 1.

An important performance in construction 3D printing is the fluidity of the material. For optimal performance of the printing, the material must be extruded continuously, and after extrusion, the material must be printed with the appropriate layer width and layer height according to the design. From the perspective of extrudability, when the fluidity is high, more materials are produced than the planned output, and when the fluidity is low, breakage occurs. Fig. 1 shows a situation where sufficient extrudability is not developed due to low fluidity. Fig. 1 all three specimens were printed with the same screw rotation

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(a) (b) (c)

Fig. 1 Effect of extrudability for construction 3D printing

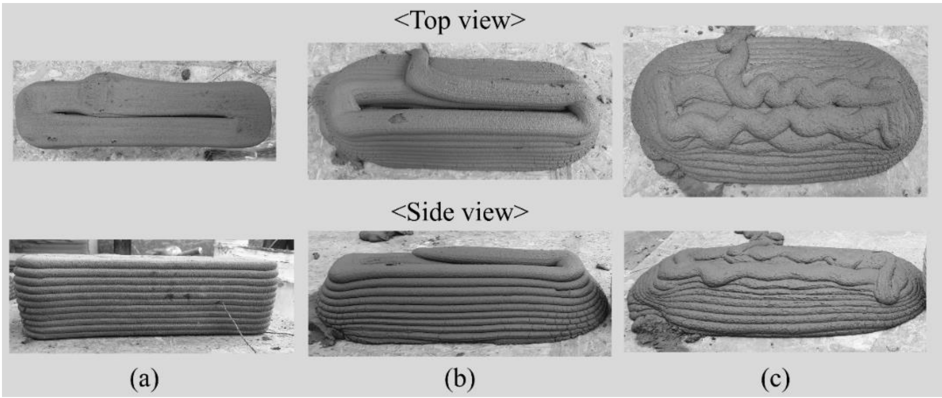


Fig. 2 Effect of buildability for construction 3D printing

speed, nozzle traveling speed and layer height. Fig. 1(a) shows an example of stacking with sufficient extrudability, while Fig. 1 (b) and (c) show a phenomenon in which buildability is sufficient but continuous output is not possible due to insufficient extrudability.

Conversely, there are cases where you have enough extrudability but not enough output due to a lack of buildability. Also, Fig. 2 all three specimens were printed with the same screw rotation speed, nozzle traveling speed and layer height. Fig. 2(a) shows an example of a test specimen printed with sufficient extrudability and buildability. However, in the case of Fig. 2(b) and (c), spreading and displacement occurred after printing due to insufficient buildability.

As shown in Fig. 1 and Fig. 2, the extrudability and buildability of the material affect the printing quality. This study focuses on the methods for controlling the rheological properties of concrete for 3D printing based on previous research concerning the relationship between the component materials and the rheology of concrete.

Normally, the rheological characteristics of fresh concrete can be quantified using concrete rheology measurement methods. There are multiple models for the rheological behavior of unhardened concrete, including the Bingham and Herschel-Bulkley models (Banfill 2003, Banfill 2006). The Bingham model has been used in recent studies on concrete 3D printing to explain the relationship between 3D printers and concrete (Le *et al*. 2012, Zhang *et al*. 2018). The rheological properties of concrete can mainly be described by yield stress and plastic viscosity. As shear stress increases past the yield stress, concrete that behaves as an elastic solid starts to behave as a fluid. This is because plastic viscosity dominates fluid behavior. These component materials are known to greatly affect the rheological properties of concrete depending on the type and amount of concrete used (Jang *et al.* 2018, Hu and Wang 2011, Paiva *et al*. 2012, Jiao *et al*. 2017, Tahar *et al*. 2017, Ma *et al*. 2018, Sui *et al*. 2018, Bauchkar and Chore 2017).

2. Materials and test methods

2.1 Materials

The main materials used for the experiment were three types of binders, ordinary Portland cement (OPC), fly ash (FA), and silica fume (SF). The raw material analysis was conducted for silica sand used as fine aggregates. As the admixture, a polycarboxylic acid high water reduction agent (HWRA) was used, and a cellulose thickener (viscosity agent) and 3 mm length nylon fiber were used to secure the buildability. The materials used for the experiments were analyzed for raw materials according to KS standards.

2.2 Mix proportion for paste

The mixing experiment was conducted after separately conducting the paste (mixture of binder and water) and mortar (mixture of binder, water and fine aggregate) tests.

Table 2 Mix proportions in test (In case of paste)

Mix	W/B[1]	Weight (g)				
		Water	Binder			Additive
			OPC[2]	FA[3]	SF[4]	HWRA[5]
W/B 25%	0.25	2000	8000	-	-	-
W/B 28%	0.28		7143	-	-	-
W/B 30%	0.3		6667	-	-	-
W/B 35%	0.35		5714	-	-	-
W/B28%-FA10%	0.28		6429	714	-	-
W/B28%-FA20%			5714	1429	-	-
W/B28%-FA30%			5000	2143	-	-
W/B28%-SF5%			6786	-	357	-
W/B28%-SF10%			6429	-	714	-
W/B 28%-HWRA 0.1%			7143	-	-	7
W/B 28%-HWRA 0.3%			7143	-	-	21
W/B 28%-HWRA 0.5%			7143	-	-	36

[1]W/B-water-binder ratio; [2]OPC-ordinary Portland cement; [3]FA-fly ash; [4]SF-silica fume; [5]HWRA-high water reduction agent

Table 3 Mix proportions in test (In case of mortar)

Mix	Mix ratio			Weight (g)				
	W/B	Weight ratio (paste/total)	Weight ratio (sand/total)	Water	OPC	Sand	Viscosity Agent	Nylon Fiber
W/B 28% -Sand 50%	0.28	0.5	0.5	1000	3571	4571	-	-
W/B 30% -Sand 50%	0.30	0.5	0.5		3333	4333	-	-
W/B 40% -Sand 40%	0.40	0.6	0.4		2500	2333	-	-
W/B 40% -Sand 50%	0.40	0.5	0.5		2500	3500	-	-
W/B 40% -Sand 60%	0.40	0.4	0.6		2500	5250	-	-
W/B 50% -Sand 50%	0.50	0.5	0.5		2000	3000	-	-
W/B 28% -VA 0.2%	0.28	0.5	0.5		3571	4571	7	-
W/B 28% -VA 0.4%	0.28	0.5	0.5		3571	4571	14	-
W/B 28% -Fiber 0.5%	0.28	0.5	0.5		3571	4571	-	18
W/B 28% -Fiber 1.0%	0.28	0.5	0.5		3571	4571	-	36

The paste was tested to evaluate the effects of W/B, replacement ratio of cementitious material (FA and SF), and HWRA dosage on the rheological properties. The rheological properties of paste were evaluated by altering W/B (25%, 28%, 30%, and 35%), with the change normalized to a mix containing fixed W/B and OPC contents. The rheological properties were evaluated further by altering the binder type and ratio: FA (10%, 20%, and 30%) or SF (5% and 10%). The mixing ratios used in the experiment are given in Table 2.

2.3 Mix proportion for mortar

The mortar mix was set up to measure the rheological properties according to changes in W/B and paste-sand ratio. As shown in Table 3, mix proportions are consist with varying W/B (30%, 40%, and 50%) and paste-sand ratio (60-40, 50-50, and 40-60% weight). To ensure the homogeneity of the material, the composite material was subjected to dry mixing for 3 min before the addition of water. To uniformly hydrate the mix after drying, water was added over a period of 1 min, after which mixing was performed for 3 min. Also, viscosity agent and nylon fiber were used in the mix proportions.

2.4 Compressive strength

Cubic specimens (50×50×50 mm) were molded according to ASTM C 109 for compressive strength measurements. The conditions in which the additive manufactured mortar was exposed to the outside air immediately after printing were simulated, and the test specimens for compressive strength measurement was also exposed to the outside air under the same conditions as the printed specimen to cure. Compressive strength was measured five times per formulation (age 1, 3, 7, 14 and 28 days), and the average value was measured by measuring the three specimens per age. 0.3 MPa/sec was applied to the loading rate for compressive strength measurement.

2.5 Measurement of rheological properties

Changes in the component materials are known to affect yield stress and plastic viscosity in rheology (Jiao *et al.* 2017). This study used both paste and mortar to evaluate the rheological properties of the component materials. Normally, concrete composite materials are in a fluid state immediately after mixing, where it behaves similar to a Bingham fluid (Jang 2009). Although Bingham fluids exhibit an elastic behavior below the yield stress, fluid behavior above the yield stress is dominated by plastic viscosity.

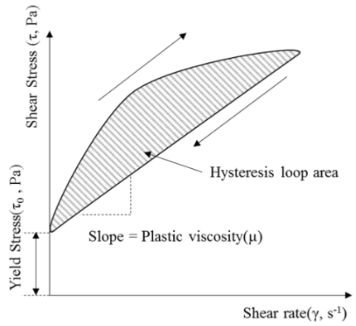
In this study, rheological measurements were taken using Brookfield's DV-III programmable rheometer and thermostatic chamber, which is capable of holding both paste and mortar at a constant temperature. To measure the plastic viscosity and yield stress of the paste, rheology measurements were performed by applying a paste to the rheometer immediately after mixing.

Preset profiles for flow curves as time-dependent step-like ramps, as shown in Fig. 3, here with 15 steps corresponding to 15 measuring points for ascending. The step speed is 10 sec per step. Each step is controlled by rotational speed, 10 rpm. A maximum speed is 150 rpm after 150 sec from the start as shown in Fig. 3.

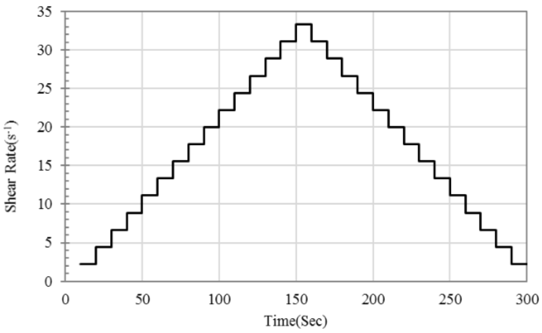
$$\tau = \tau_0 + \mu \cdot \gamma, \quad (1)$$

where τ is the shear stress [Pa], τ_0 is the yield stress [Pa], γ is the shear rate [s^{-1}], and μ is the plastic viscosity [Pa·s].

After the test, a linear regression analysis of the measured shear stress was used to conduct an analysis through the Bingham model. The rheological evaluation



(a) Bingham model



(b) Preset of rotational speed

Fig. 3 Setting for test of rheological properties using a rheometer

was conducted by measuring variations in shear stress according to variations in shear rate. To avoid the hysteresis of the measured values due to the hysteresis loop area, the shear rate was measured by setting the stepwise descent.

2.6 Measurement of extrudability

The effect of controlling rheological properties on the extrudability of 3D printers is examined using a screw pump type extruder. 3D printing for construction comprises three stages: production (mixer), pumping (peristaltic pump), and printing (screw type extruder). A single screw extruder was constructed with an extrusion capacity of 5 HP, a maximum capacity of 50 L, and an extrusion nozzle diameter of 25 mm.

Also, rheological properties and a traditional workability evaluation were performed and compared to analyze the relationship between rheological properties and workability. We evaluated the correlation between rheological properties and results of mortar flow test as well as the effect of rheology on the extrusion performance by using an extruder. For successful 3D printing, the extrusion rate of the material must be perfectly controlled according to the extrusion rate required by the extruder. We conducted an experiment to analyze the impact of rheological properties and workability on extrudability. The experimental process is shown in Fig. 4 and the test equipment are shown in Fig. 5

The table flow test, ASTM C 230 (ASTM 2014) was performed simultaneously with the rheological evaluation and used as basic data to evaluate the relationship between rheology and workability using viscosity agent mix proportion in Table 3.

To evaluate the performance between rheology and the table flow test, the yield stress and plastic viscosity were controlled with a viscosity agent. For the test, only the viscosity agent was increased by 0.2 and 0.4% relative to

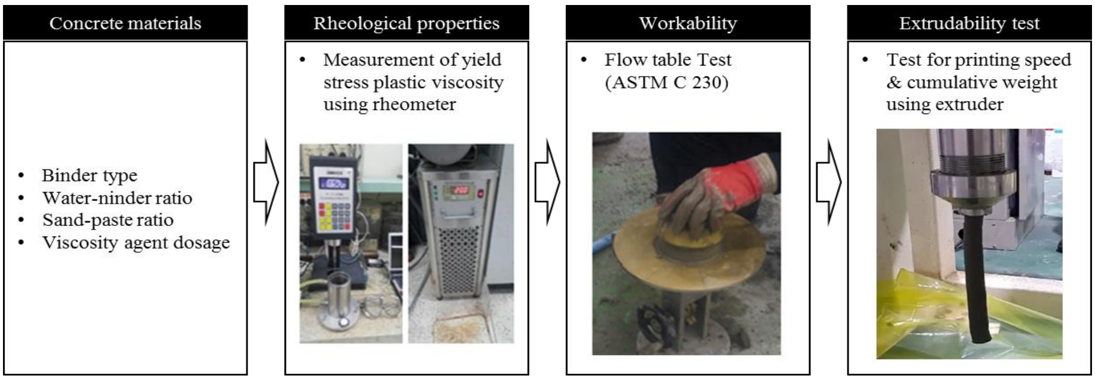
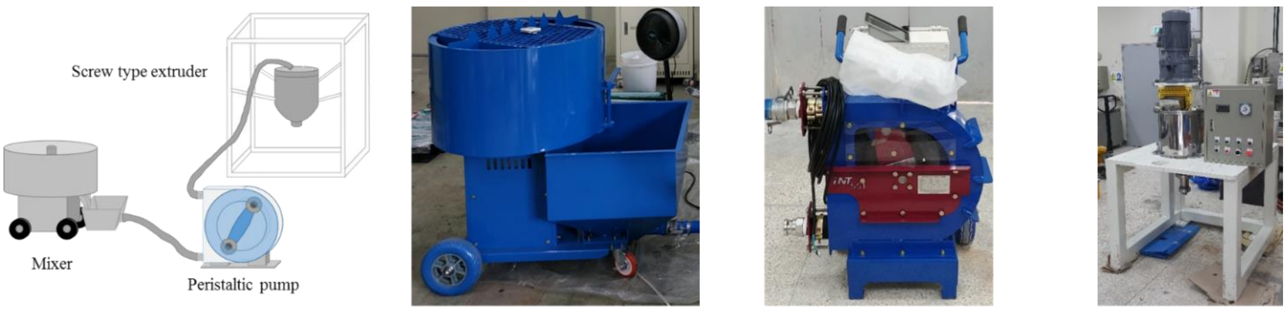
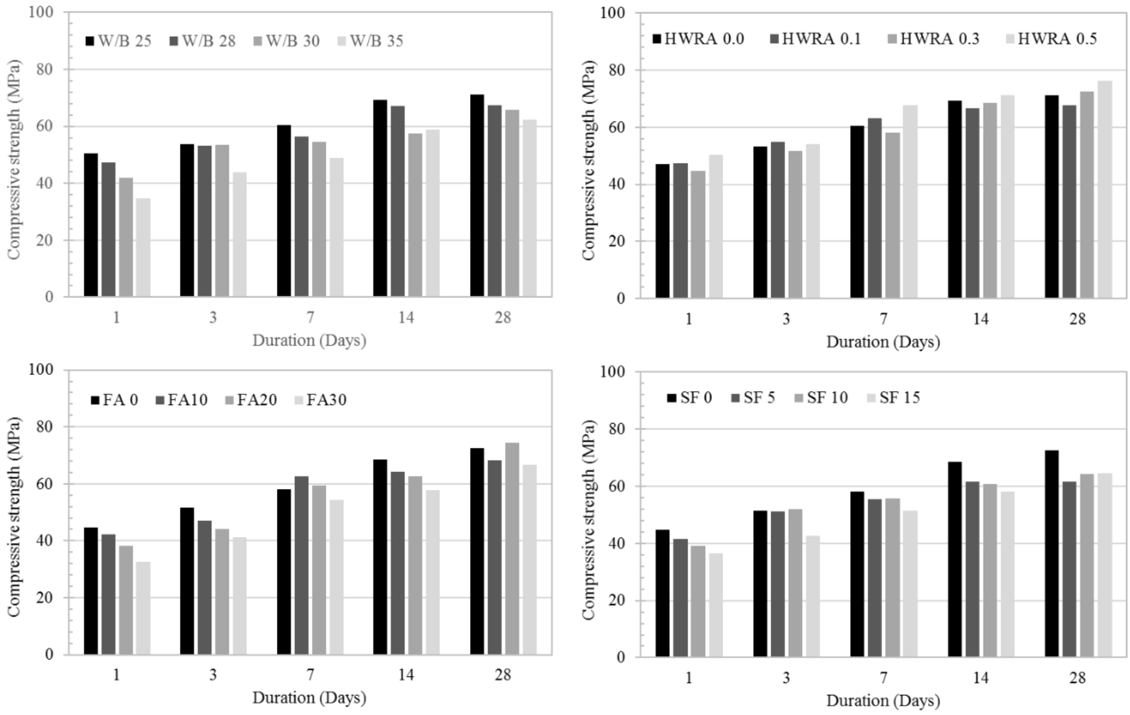


Fig. 4 Experimental process

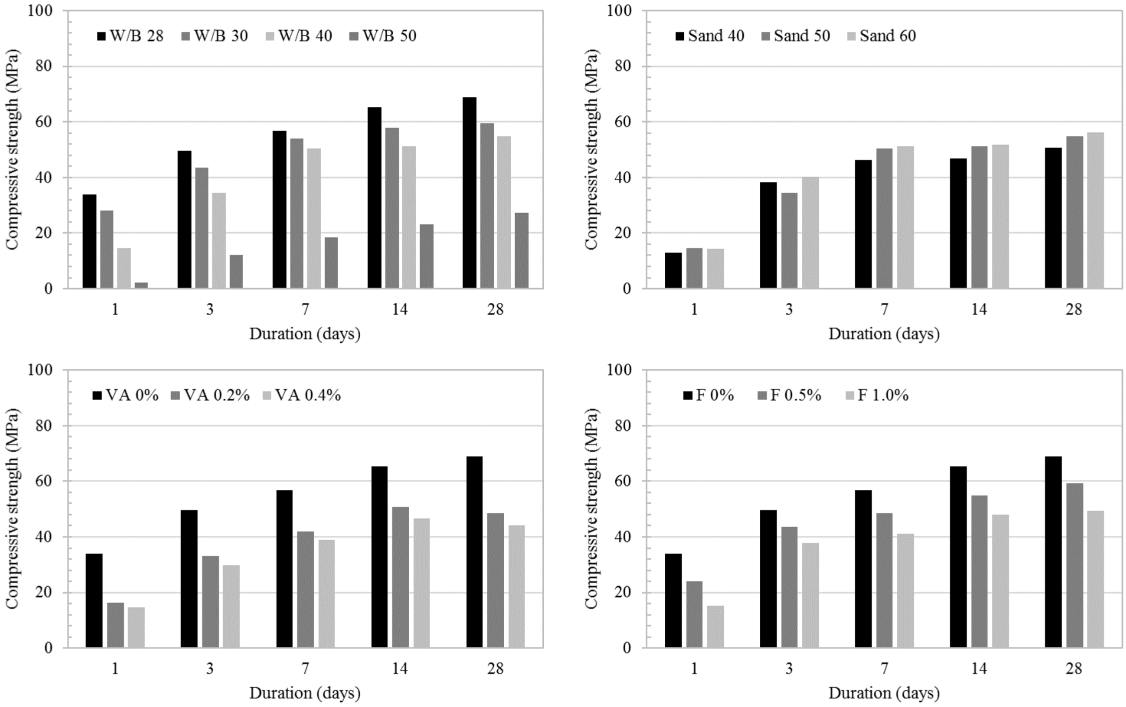


(a) Extrudability test system (b) Mixer (c) Peristaltic pump (d) Screw type extruder

Fig. 5 Extrudability test system (mixer - peristaltic pump - screw type extruder)



(a) Compressive strength of paste (W/B, HWRA, FA, SF)



(b) Compressive strength of mortar (W/B, Sand, Viscosity agent dosage, Fiber usage)

Fig. 6 Compressive strength: (a) paste; (b) mortar

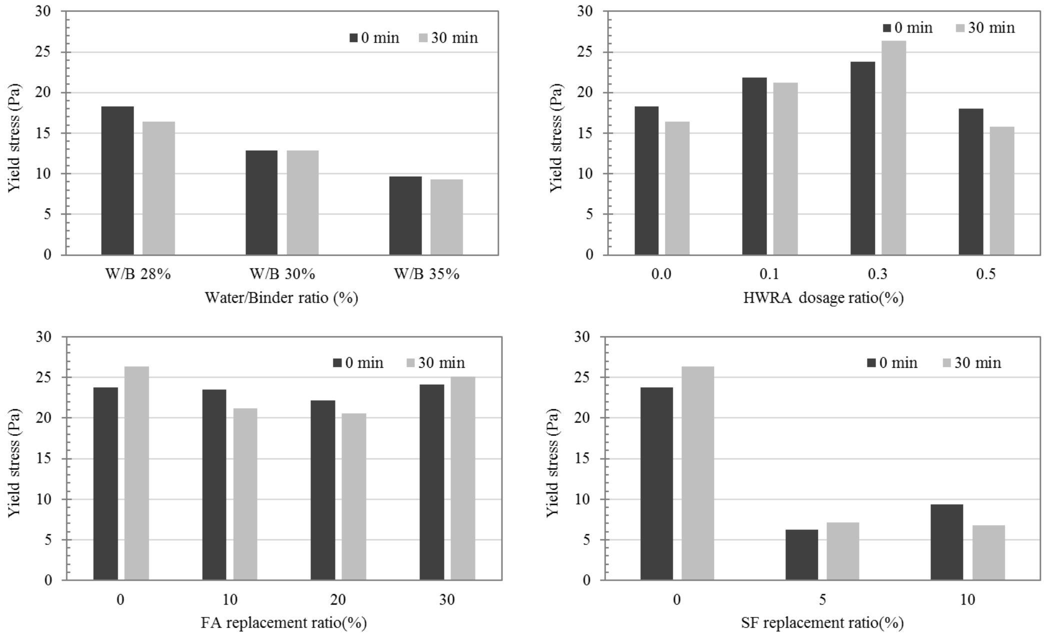
the weight of the binder based on a W/B 28% mix proportion of mortar. Both the rheological properties and table flow test results were conducted immediately after mixing to prevent changes in properties due to time. The screw pump type extruder was utilized to check the variation of the rheological properties by using a viscosity agent, and when extruding at a constant RPM, the change of extrusion rate with time was observed.

3. Results

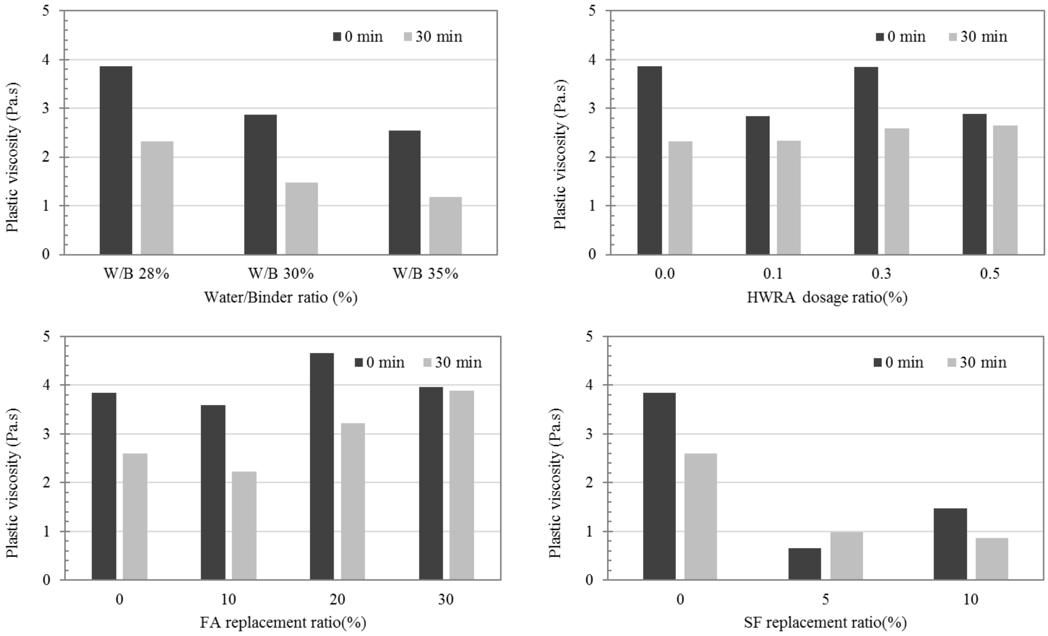
3.1 Materials

3.1.1 OPC

As a result of analyzing OPC according to KS L 5201, specific gravity 3.13 g/cm^3, specific surface area is 3,542 cm^2/g, and hydraulic cement has compressive strength of



(a) Yield stress of paste (after 0 min, 30 min)



(b) Plastic viscosity of paste (after 0 min, 30 min)

Fig. 7 Rheological properties of paste (after 0 min, 30 min): (a) yield stress; (b) plastic viscosity

3rd day 23.3 MPa, 7th day 36.6 MPa, 28th day strength 48.5 MPa appeared. In the case of the setting time, the initial setting time was evaluated at 263 minutes and the final setting time was 360 minutes.

3.1.2 Fly ash

Fly ash was analyzed according to KS L 5405. Specific gravity was 2.25 g/cm^3 and specific surface area was 3,232 cm^2/g. Loss on ignition was 2.9%, activity index was 28% 81%, 91 days 99%.

3.1.3 Silica fume

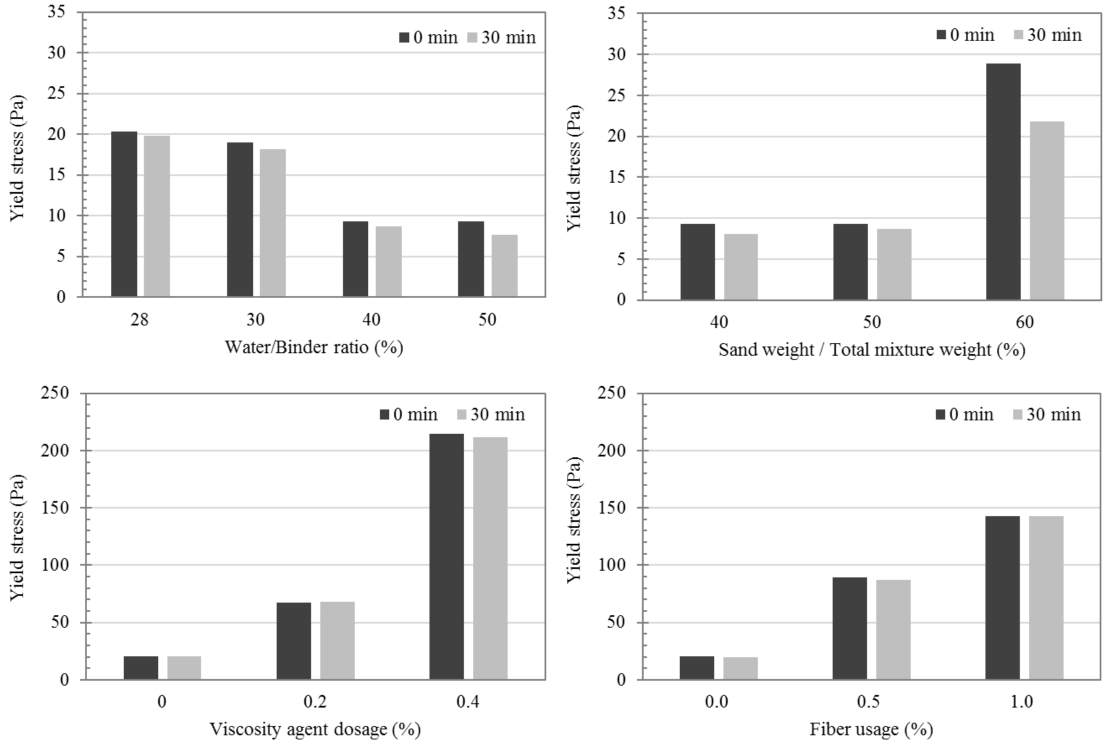
According to KS F 2567, silica fume showed 91.3% SiO_2 content, 19.3 cm^2/g specific surface area, and 4.4% remaining 45-μm (No. 325).

3.1.4 Fine aggregate

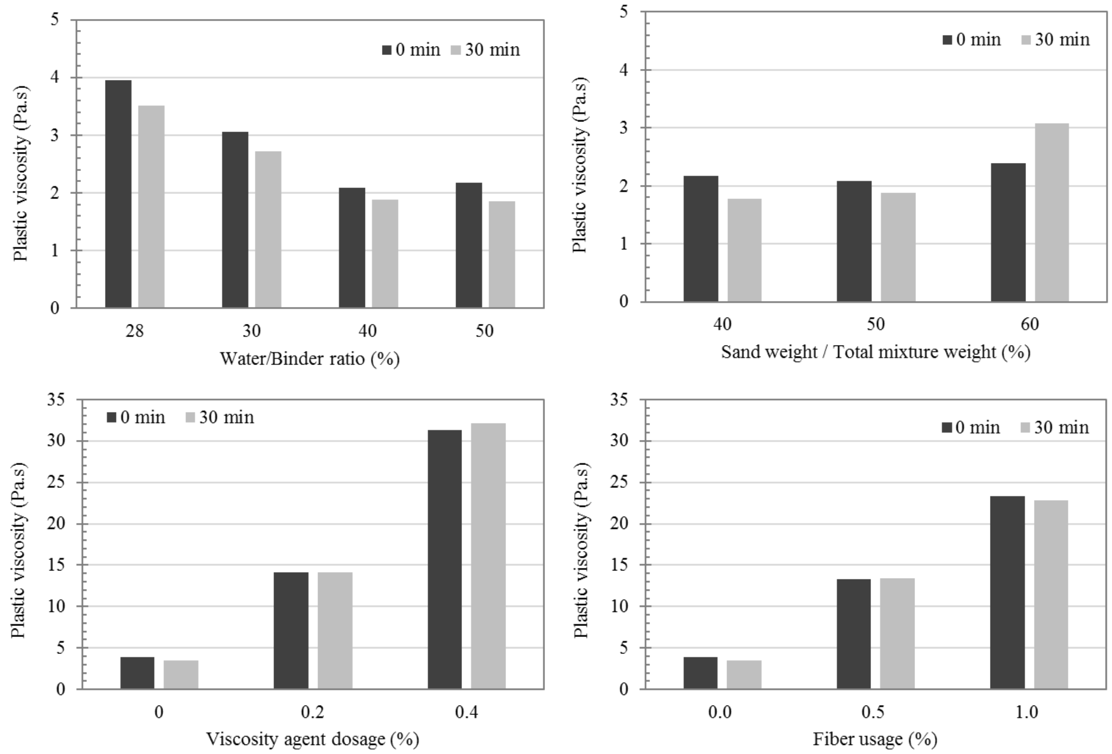
Fine aggregates (silica sand) were 1.20% level as a result of evaluating the water absorption rate according to KS F 2504. As a result of evaluating the 0.08 mm sieve passage according to KS F 2511, the passage was 0.7%, which is lower than 1.0%, and satisfies the criteria.

3.2 Compressive strength

3.2.1 Paste



(a) Yield stress of mortar (after 0 min, 30 min)



(b) Plastic viscosity of mortar (after 0 min, 30 min)

Fig. 8 Rheological properties of mortar (after 0 min, 30 min): (a) yield stress; (b) plastic viscosity

The strength was measured using the material used in the rheological properties experiment.

As *W/B* increases, it was confirmed that the strength decreased. An increase in W/B 1% at age 28 days was assessed to cause a decrease in intensity of about 1 MPa.

There is little change in strength depending on the amount of HWRA used. The HWRA used in this study was evaluated to affect only the fluidity in the fresh state. Paste strength according to the FA and SF substitution rates does not seem to affect the change of substitution rate.

However, if FA is replaced, early (age 1 and 3 days) strength expression is late, at 7 days of age, the intensity gradually developed, almost equal to that of OPC.

3.2.2 Mortar

In the case of Mortar, the change in strength due to W/B was clear. With the increase of W/B, the tendency of strength decrease was clearly seen. In particular, in the case of W/B 50%, a sharp decrease in strength was observed compared to *W/B* 40%.

Depending on the sand usage, no significant change occurred within the maximum of 10 MPa. In the case of Sand 60%, however, a certain level of strength was always expressed compared to other formulations.

In the case of using a viscosity agent, the amount used is small but it has a significant negative impact on strength. The amount of VA was 0.2% of the amount of binder, which was considerably less, but the strength decreased by about 30%. When the amount of VA was increased to 0.4%, the strength was further lowered, which is thought to be because VA interferes with the hydration of the binder and water.

When fiber was applied, the strength decreased depending on the amount of use as in VA. However, although the amount used was more than twice that of VA, the decrease in intensity occurred relatively little.

3.3 Rheological properties

3.3.1 Rheological properties of paste

W/B is the most influential factor for determining flowability and mechanical properties such as strength. Accordingly, when using concrete as a 3D printing material, W/B is one of the most important factors for determining extrusion characteristics and range of strength.

In this study, we evaluated the effect of W/B in OPC using paste at W/B ratios of 25%, 28%, 30%, and 35%. However, results of 25% W/B could not be obtained due to out of range of rheometer. Except for 25% of W/B, the results are shown in Fig. 7. As shown in Fig. 6, as W/B increased, the yield stress and plastic viscosity decreased. Notably, the decrease in yield stress and plastic viscosity is smaller when W/B increases from 28% to 30% than from 30% to 35%. Hence, the rheological properties are not linearly inversely proportional to W/B, but they are sensitive to specific W/B ranges.

Two types of cementitious material used as a binder, FA and SF, were evaluated according to the replacement ratio of OPC. FA additions gave a high overall yield stress regardless of the substitution rate compared to the control mixture (W/B 28% OPC100), while for plastic viscosity, the initial value showed a range similar to that of the control mixture. Although it was difficult to determine a tendency based on substitution rate immediately after mixing, the value gradually increased as the substitution rate increased in the re-measurement 30 min after mixing, most likely owing to the activation of the electrochemical distribution characteristics of the binder over time. We infer that it is difficult to reflect all of the material's rheological properties directly after mixing.

3.3.2 Rheological properties of mortar

Mortar was used to adjust the aggregate ratios and evaluate rheological properties. As shown in Fig. 8, an increase in the sand ratio led to increases in the yield stress and plastic viscosity. In particular, when the sand ratio was increased from 50% to 60%, the yield stress improved by approximately 20 Pa immediately after blending. This indicates that the higher the incorporation of aggregate, the greater the initial pressure required for rheological behavior. However, when the sand ratio was increased from 40% to 50%, the same increase in incorporation rate had negligible effect on the yield stress. This could be because as the used paste ratio decreased from 60% to 50%, the main factor affecting the yield stress was paste rather than sand. Thus, to control rheological properties using sand, we postulate that a proportion exceeding 50 % wt. is preferable.

3.4 Effect of fluidity on extrudability

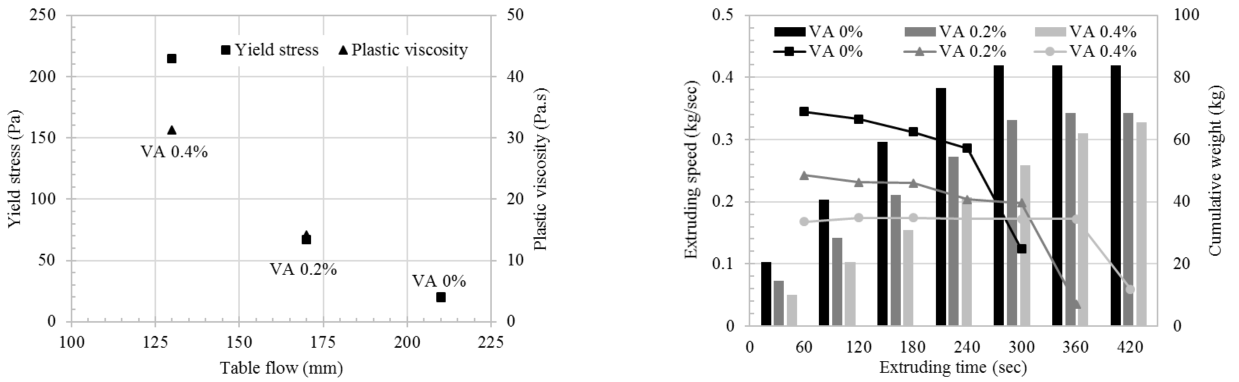
Mortar was used to assess the effect of thickener on rheological properties. The results confirmed that the yield stress and plastic viscosity increased sharply as the amount of thickener increased. The initial flow was 210 mm, and the amount of viscosity agent was used 0, 0.2 and 0.4% by weight of cement. We showed the tendency of the yield stress and the plastic viscosity to increase sharply when using viscosity agent in Fig. 8. The use of viscosity agent greatly influences the workability and rheological changes in concrete. Comparing this to the effect of the binder and aggregate type and content or W/B on workability, when controlling the rheological properties through the use of a thickener, it is preferable to control it by using a very small amount owing to the sharp changes caused by viscosity agent use.

Based on the results of the flow test using 3 mix proportions(VA 0%, VA 0.2% and VA 0.4%), the flows of 210, 170, and 130 mm were derived. The extrudability of these flows with time is shown in Fig. 9.

The amount of concrete extruded over time is represented in the bar graph, with the accumulated amount indicated on the right vertical axis. The extruded amount measured every 60 s is indicated by a line graph

Approximately 35 L of mortar was pumped into the extruder, and the extruded amount was measured by weight (kg) every 60 s after the start of extrusion. The extruding speed of each mix was greatly affected by the workability measured by the flow test result. In a mix with a flow of 210 mm (VA 0%), the initial extrusion amount was approximately 350 g/s, indicating that the extrusion speed was significantly greater than that of the other mixes. However, the extrusion speed decreased with time, exhibiting a sharp drop after only 4 min. Extrusion was completed before 3 min. In contrast, at a flow of 130 mm (VA 0.4%), the extrusion rate was approximately 170 g/s—nearly half that for the flow of 210 mm. However, this rate was constant irrespective of time. This indicates that flow is a critical factor for controlling extrusion speed in a concrete extruder; as it is lowered, the extrusion rate is improved.

On the other hand, the graph shows that the final extrusion output at a flow of 130 mm is approximately 17 kg less than that at a flow of 210 mm-this material remains in the nozzle and screw of the extruder, owing to a lack of extrusion load due to self-weight. Accordingly, when the



(a) Relationship between table flow and rheological properties (b) Changes in extrudability over time

Fig. 9 Relationship between rheological properties and extrudability

flow is low, the extrusion amount must be planned while considering the amount of material remaining in the transfer and extrusion system. From Fig. 9, the minimum rheological properties for quantitative extrusion were found to be over 200 Pa for yield stress and 30 Pa.s for plastic viscosity, which is equivalent to about 130 mm for table flow.

4. Conclusions

We conducted an experimental study on the relationships among material properties, rheological properties, flowability, and extrusion rate for extrudability in the construction of concrete structures through 3D printing method. The conclusions of this study as follows:

- The purpose of this study was to suggest the minimum rheological properties that can be controlled to exert quantitative extrusion over time in terms of material extrusion. When the 3D printing method is applied, it must be extruded according to the designed extrusion amount for the lamination method of equipment so that precise construction can be performed. Therefore, this study aims to analyze the minimum fluidity of materials that can be quantitatively extruded with the same amount of material per unit time at constant speed in terms of rheology. In addition, the effects of the constituent materials on the fluidity and rheological properties were examined, and the method to control the rheological properties by changing the composition of the material were examined.
- As shown in Fig. 9(b), VA 0% and VA 0.2% can be seen that the relatively high fluidity compared with the VA 0.4% is expressed and the extrusion rate decreases with time. This suggests that when the 3D printing method is applied, the material is extruded at a higher speed than the design speed of the equipment, making precise control of the extrusion amount difficult. According to the results of Fig. 9, the minimum limit for quantitative extrusion of 3D printing materials was about 200 Pa or more for yield stress and about 30 Pa.s for plastic viscosity. In case of yield stress, Le T.T. It is shown that it is similar to the minimum value proposed by *et al*. 300 Pa, and it can be seen that the definition of extrudability indicates the range of quantitative extrusion.
- As a result of examining the range of rheological properties by using paste, it was confirmed that HWRA had no significant effect and showed an effect of up to about 5 Pa depending on the replacement ratio of FA. The use of SF has been shown to reduce yield stress up to about 20 Pa and plastic viscosity to about 3 Pa.s. The most significant effect is W/B in the paste results. When W/B decreases from 30% to 28%, yield yield increases by 6 Pa and plastic viscosity increases by about 1 Pa.s. It is expected to increase significantly.
- Mortar test showed that the highest effect was the viscosity agent and fiber. In particular, the viscosity agent increased yield stress about 50 Pa and plastic viscosity about 10 Pa.s by applying 0.2% of the binder weight. In the case of fiber, yield stress increased about 69 Pa and plastic viscosity increased about 10 Pa.s when 0.5% of the binder weight was applied.
- Through these results, yield stress and plastic viscosity can be increased as the paste matrix becomes dense even at the same W/B and material composition, and as a result, it is judged that proper extrudability can be obtained. On the other hand, as the sand ratio was increased from 50% to 60%, yield stress was increased about 20 Pa and plastic viscosity was increased by about 0.3 Pa.s. The yield stress was increased by improving the internal density between cement matrices, but the plastic viscosity was considered to be increased slightly because the effect of increasing the tensile force between paste matrices was small.
- As a result, it was confirmed that the minimum rheological properties satisfying extrudability were yield stress of 200 Pa and plastic viscosity of 30 Pa.s. The rheological properties related to buildability require further study along with the printing test, and the buildability control using viscosity agent, fiber, sand ratio and W/B is necessary for effective printing.

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