Rheology Analysis of 3D Printed Geopolymer Based on High Calcium Fly Ash

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Abstract

The advancement of 3D concrete printing has focused on automation research in recent decades. 3D printing technology is adequate to reduce waste and improve efficiency for construction. Former research on 3D concrete printing used hydration cement based on ordinary Portland cement (OPC), which was not environmentally friendly. An alternative material to overcome problems with hydrated cement mortar is a geopolymer. Geopolymer mortar based on 3D printing is still in its infancy. Mechanical and rheological properties are the key parameters of this technology: yield stress, shear stress, and viscosity. The flow characteristics of 3D concrete printing represented the ability of material transfer along the system of the 3D printing machine. This research utilizes type C fly ash waste as the primary material for making geopolymer 3D-printed concrete. Variations of 8, 10, and 12 M of NaOH concentration were used to investigate the relationship between workability and quality of the geopolymer mortar. Workability testing of 3D concrete printing consists of several parameters: pumpability, extrudability, and buildability. Material identification, including rheology and flowability, is carried out to determine mortar specimens' pumpability, extrudability, and buildability. Several test approaches, such as slump flow, slump, shape retention, and rheometer tests using the vane shear approach method, were conducted to identify the rheological characteristics and flowability of the material. Based on testing of the material's workability, 10 M NaOH concentration variation is the most suitable material for future 3D printing material. The workability of 10 M NaOH is 177.5 mm and the the copressive strength is 25.84 Mpa. This variation meet ACI 318/318R – 14 criterion for building structure.

Keywords: 3D Printing; Fly ash; Geopolymer; Rheology

1. Introduction

The utilization of 3D printing in residential establishments is adequate to improve cost-effectiveness and relieve the construction process. OPC-based mortar is used to use as 3D printed material due to its [1], [2]. However, the use of OPC as a construction material is no longer environmentally friendly. [3] mentioned that cement production contributed about 5% of CO_2 gasses increment in the atmosphere per year. Based on data in 2020, the Portland cement industries release 40,3 million metric tons of CO_2 gases and trigger global warming with long-term effects [4]. As an alternative, geopolymer technology is inspected as substitutive material which is more economically and environmentally friendly [5].

Geopolymer technology has compound interactions between high-alkali materials (K-Ca) and poly(sialate-siloxo) that result in polycondensation reactions between the matrixes [5]. The most common materials contain high alkali compounds such as fly ash, metakaolin, mill scale, blast furnace slag, and rice husk ash [7]. Previous research has discussed the use of fly ash as an OPC substitution material to produce high-performance concrete [8]. The result showed that geopolymer concrete had better performance and durability compared to OPC-based concrete. Geopolymer concrete using fly ash has good retention in a destructive environment [6]–[8]. This technology is widely used as a replacement or supplementary for green material construction. One of the green construction concepts developed by researchers or industries worldwide is 3D printing technology.

3D-printed concrete technology was initiated by [9] in the 1990s to improve the precision model and efficiency for complicated geometric design called "contour crafting." Due to the formwork job, 3D printing technology for concrete may save 35-54% of total construction costs and 50-75% of total construction time.

Further research has developed the mortar mixing composition with measurements of parameters such as extrudability, buildability, and stiffness throughout the hardening process of 3D-printed specimens [10], [11]. Machine capacity has also played a significant role in the deposition process. The workability of fresh material is used as a measurement parameter to determine the printing machine's passing ability. Several methods were conducted to investigate the workability of fresh material for 3D concrete printing, such as rheology analysis [12]–[15].

Rheology is defined as the material's ability to deform and flow due to shear forces applied by the system [16]. Rheology is the most used method for determining the behavior of a material in its plastic state. Further investigation into the rheological is required to identify the specimen's flowability, workability, and buildability. The printing process distinguishes into a fresh state and a hardening state—the first state of the printing process focusses on the workability, pump ability, and extrudability of a specimen. Second, the specimen occupies its form to gain a hardening process and durability. In the fresh state, 3D printing material should have low dynamic stress to increase its flow and ease printing. However, the hardening state requires higher dynamic stress to preserve the shape from failure [17]. The addition of sucrose in 3D printng based-geopolymer could enhace the workability and reduce its hardening time to fullfil the workability requirement [18]. In other hand, the varition of NaOH variation could affect its mechanical properties of 3D printing based geopolymer [19], [20].

This research focuses on alkali-activator effect to assess the rheological and mechanical behaviour of 3D concrete printing geopolymer specimens. The variation of NaOH molar varies from 8M, 10M, and 12M. The use of sucorse based superplasticizer intends to enchace its flowability. Approaching methods use to conduct the result analysis of its rheological behavior. The yield and shear stress of plastic specimens determines rheological behavior. Vane shear testing mentioned in the [21] was adopted to find the yield and shear stress value as retrieved by [11], [14], [22]. In addition, compressive tests, slump tests, and visual judgment were carried out to support the results.

2. Method

2.1. Materials

The fly ash material used in this research is from PT YTL Paiton Jawa Power, Probolinggo, Indonesia. The composition of material compounds was characterized at the Laboratory of Energy, Directorate of Research and Community Service, Sepuluh Nopember Institute of Technology, Surabaya, Indonesia. The chemical composition was characterized to figure out the classification of fly ash. The chemical composition is shown in **Table 1**. The chemical characterization test result showed class C fly ash based on [23]. Fly ash (FA) powder is activated using sodium metasilicate (NaOH) flakes and sodium metasilicate pentahydrate ($Na_2SiO_3.5H_2O$) granular. The NaOH had 98% purity level. Local river sand with a maximum diameter of 2.38 mm was selected as aggregates. Aggregates were in oven-dry condition prior to use. The physical properties of fine aggregates are shown in **Table 2**.

Table 1. Chemical composition of fly ash.

Chemical Composition	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**K_2O**	**TiO_2**
FA (%)	24,9	9,5	30,6	27,2	1,27	1,3

Table 2**.** Physical properties of aggregates.

Max. Size [mm]	**Humidity [%]**	**Specific Gravity**	**Loose Density [kg/m^3]**	**Infiltration [%]**
2,38	5,83	2,78	1607,5	1,01

2.2. Mix Design and Preparation

Based on former, NaOH and N_2SiO_3 took an important role in high calcium fly ash based geopolymer concrete performance and workability [20], [24]–[26]. Since mixing proportion of high calcium fly ash based geopolimer concrete for 3D priting had not been reported yet. Three variation of mix proportion were prepared with NaOH molarity differences as is shown in **Table 3**. The precursors (FA, NaOH, and $Na_2SiO_3.5H_2O$) and aggregates were inserted into the grinding machine using 500 cycle with 13-14 rpm of speed to fine the particles and produce geopolymer cement refere to [6]. Dry mixing method was used to delay the hardening process of high calcium fly ash based geopolymer concrete effectively [27]. 5% silica fume of cementitious material were added to increase the stiffness of sepecimen [28]. 2% fine sucrose based supperplasticizer were added to control workability of concrete mixture [18]. All the dry components are mixed for 5 min to ensure the components are dispersed. The water is thoroughly poured into the mixer and continous stirring for 5 min until homogenous.

Table 3. Mix proportion of 3D geopolimer concrete printing specimens.

Material (kg/m^3)	**M8**	**M10**	**M12**
FA	568,10	568,10	568,10
NaOH	40,95	49,57	56,53
$Na_2SiO_3.5H_2O$	29,90	29,90	29,90
Sand	1380,00	1380,00	1380,00
Water	206,14	197,52	190,56
w/c (%)	29,00	27,00	26,00

2.3. Experimental procedures

2.3.1. Flowability Test

The flowability parameter indicates the ability of a 3D concrete printing specimen to mobilize along the printing system. [29]used alternative methods to investigate the flowability of 3D-printed concrete specimens using a slump flow test. The result of the slump flow test corresponded to the stress loss of the specimens. The slump flow test was conducted to investigate the flowability of 3D-printed concrete specimens based on [30]. Former researchers state threshold values that characterize printable concrete. [31] states that printable concrete ranges between 18 to 24 cm of slump flow. However, [32] gave a lower boundary of printable concrete ranging between 13 to 21 cm of slump flow. The slump flow test procedure begin by filling half of the mold ring with concrete specimens. Then, it was tamped 20 times uniformly distributed over the cross-section of the mold. Repeat until the specimens fill the mold. Discharge the mold over the specimens 1 minute early before it drops. After the mold is removed, Drop the flow table 25 times, and record the initial and final diameters of the specimens on the top of the flow table.

Figure 1. Flowability test.

2.3.2. Slump Test

The slump test conducted to investigate the correlation between the stress of material with its shape retention behaviour of specimens. A study by [29] showed the relationship between decreased slump value and material yield stress loss.This test method was did under concern of [33]. The slump test procedures begin by filling 1/3 part of the mold. Then, it was rodded 25 times and uniformly distributed. The steps were repeated until the mold was filled with specimens. Then, the mold was removed, and the deviation between the top side of the mold and the specimens was measured.

Figure 2. The slump test procedures.

2.3.3. Rheological Test

Since the rheological behavior of concrete can be investigated using rheometer apparatus, it has difficulties involving calibration and in situ testing. Rheology of materials is essential for understanding the behavior of materials in their plastic state. Deformation and changes in the shape of materials are heavily influenced by their flow characteristics. Mortar in its plastic state is categorized as a pseudoplastic material or a material with pseudo-plasticity, and it will harden over time due to the setting of the mortar.

The rheological testing conducted in this research involves using the vane shear test to determine the shear stress and evaluate the buildability of the 3D printed mortar mix. The test is performed every 15 minutes to monitor changes in shear stress in the 3D printed mortar filament. The vane shear test effectively represents the non-Newtonian properties of the Bingham model for various materials.

The relationship between the torque value from the vane shear test and the shear stress of the material is calculated using Equation 1.

$$\tau = \frac{T_M}{\frac{\pi D^3}{2}\left(\frac{H}{D} + \frac{1}{3}\right)} \tag{1}$$

Where:

t = Shear stress of the material, Pa

T_M = Torque reading from the vane shear test, N·m

D = Diameter of the vane blades, m

H = Height of the vane blades, m

3. Result and Discussion

3.1. Flowability Test

To determine the material's ability to flow through the mechanical system of a 3D printer, such as through pipes to the nozzle, the material must have good flowability characteristics. The effect of NaOH concentration on the material's flowability is shown in Figure 3. An increase in NaOH concentration by 2 molar reduces the flowability by 29.92%. The highest flowability is shown by the lowest concentration of 8M, which is 213.5 mm, followed by a decrease in flowability for NaOH concentrations of 10M and 12M, which are 177.5 mm and 153.67 mm, respectively.

Table 4. The flowability of 3D printed geopolymer mortar relative to changes over time.

No	Time	Molarity NaOH					
		8 M	10 M	12 M	8 M	10 M	12 M
	Minutes	(%)			mm		
1	0	114%	78%	54%	213.5	177.5	153.67
2	15	96%	68%	40%	196.25	167.5	139.75
3	30	82%	63%	44%	182.25	162.75	143.75
4	45	79%	42%	30%	178.5	141.75	129.67
5	60	82%	36%	30%	181.5	136	129.50
6	75	81%	33%	23%	180.75	132.5	123.33
7	90	77%	23%	25%	176.75	122.75	125.00
8	105	61%			161.25		
9	120	69%			168.5		
10	135	59%			158.75		
11	150	56%			156		

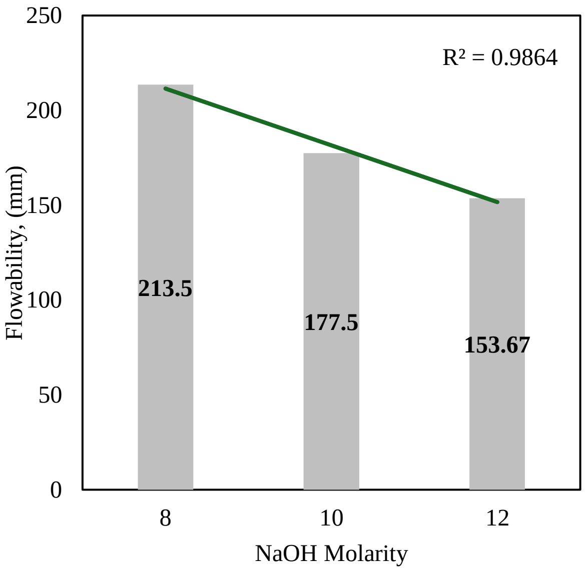

Figure 3. The relationship between the concentration of NaOH in the composition of 3D printed geopolymer mortar and the flowability (%) of the material.

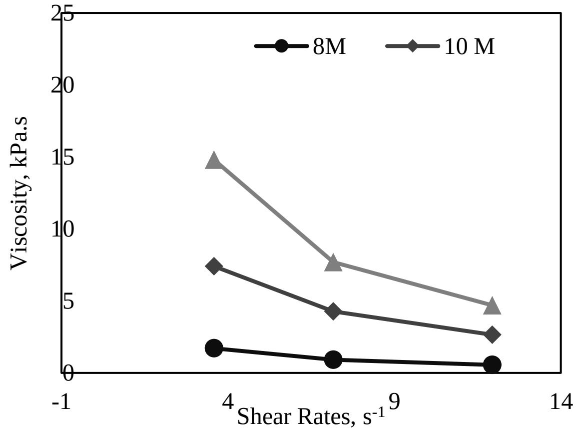

Figure 4. The relationship between NaOH concentration and the viscosity of geopolymer material.

An increase in NaOH concentration decreases the flowability of the material [34]. This is directly proportional to the relationship between NaOH concentration and the viscosity of the material. Figure 4 shows a decrease in material viscosity with an increase in NaOH concentration. The viscosities for NaOH concentrations of 8M, 10M, and 12M at the lowest constant speed of 30 rpm are 14.81, 7.41, and 1.74 Pa.s, respectively. Viscosity data were taken at 90 minutes to observe the trend of the effect of increasing NaOH concentration. Viscosity data for less than 90 minutes could not be compared because the viscosity for the 8M NaOH variation was too low to be read by the testing device.

The setting time of 3D printed mortar is identical to the machine's open time, during which the material can flow through the entire printing system. Figure 4.3 sequentially shows the workability of the material relative to time. The 8M mix composition has a relatively longer open time compared to 10M and 12M. This is due to the higher water content (water-to-solid ratio) in the mix. Lower NaOH concentrations result in a relatively longer setting time [19], [35]. Increasing the water content in the mortar mix also delays the setting time [36]. Laboratory testing yielded open times ranging from 1.5 hours to 2.5 hours. However, these results cannot be definitively applied to experiments using a 3D printer.

Table 4 shows the flowability values of 3D printed geopolymer mortar relative to changes over time.

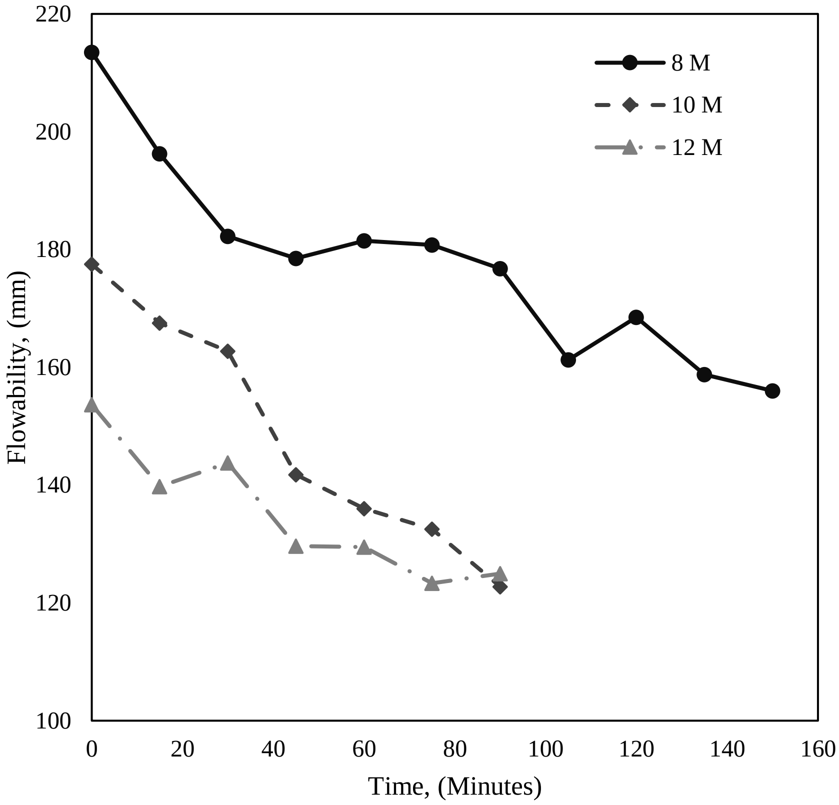

Figure 5. Graph of the Relationship Between Flowability Level and Time

Figure 5 shows a decrease in flowability over time. The reduction in flowability indicates the setting process in the 3D printed geopolymer mortar specimens, which start to lose their plastic phase and transform into solid mortar. Figure 5 illustrates the loss of workability in the 3D printed geopolymer mortar material as time progresses. This loss of workability is caused by the binding process and water absorption by the matrix in the mortar [37].

Slump Test

Table 5 illustrates the relationship between the slump values and the printable area for 3D printed geopolymer mortar with varying NaOH concentrations over time. The table provides data on slump measurements (in mm) for different NaOH concentrations (8M, 10M, and 12M) recorded at 15-minute intervals up to 150 minutes.

Table 5. The relationship between slump and slump flow values relative to the printable area based on [32].

No	Time	*Slump, mm*		
	Minutes	8M	10M	12M
1	0	11	4	2,5
2	15	8,5	4,5	1
3	30	5	2,5	0,5
4	45	4,5	1,5	1
5	60	5	1,5	0,5
6	75	4,5	0,5	0
7	90	2,5	0	0
8	105	2	0	0
9	120	1,5	0	0
10	135	0,5	0	0
11	150	0	0	0

The table highlights the impact of NaOH concentration on the slump and flowability of geopolymer mortar. Higher NaOH concentrations (10M and 12M) result in quicker loss of workability compared to the lower concentration

(8M), which retains its printable properties for a longer duration. This relationship is crucial for determining the appropriate mixture and timing for 3D printing applications in construction.

3.2. Vane shear test

The Table 6 presents the melting tensile strength, shear strength, and viscosity values of 3D printed geopolymer mortar at varying NaOH concentrations (8M, 10M, and 12M) over different time intervals, measured in minutes. The parameters assessed include yield stress (kPa), shear stress (kPa), and viscosity (kPa.s). At 0 minutes, yield stress data for 8M is unreadable (*), while 10M and 12M start at 0.04 kPa and 0.33 kPa respectively. Shear stress begins at 0.10 kPa for 10M and 0.42 kPa for 12M, with unreadable data for 8M. Viscosity at the start is unreadable for 8M but measures 0.03 kPa.s for 10M and 0.12 kPa.s for 12M. Over time, all parameters generally increase, with notable increments at 75 minutes where 12M reaches a peak yield stress of 5.40 kPa, shear stress of 5.72 kPa, and viscosity of 1.60 kPa.s. From 90 minutes onwards, data for 10M and 12M is marked as not done (**), and only 8M continues to provide measurable values, albeit with a reduction in viscosity and yield stress over time.

Table 6. Melting tensile strength, shear strength, and viscosity values of 3D printed geopolymer mortar for variations in NaOH concentration of 8M, 10M, and 12M.

Minutes	Yield stress, kPa			Shear stress, kPa			Viscosity, kPa.s		
	8M	10M	12M	8M	10M	12M	8M	10M	12M
0	*	0,04	0,33	*	0,10	0,42	*	0,03	0,12
15	*	0,23	0,77	*	0,31	1,04	*	0,09	0,29
30	*	0,73	1,43	*	0,99	1,77	*	0,28	0,49
45	*	1,18	3,41	*	1,44	3,43	*	0,40	0,96
60	*	1,72	3,25	*	1,87	3,43	*	0,52	0,96
75	*	2,09	5,40	*	2,29	5,72	*	0,64	1,60
90	0,61	2,56	5,31	0,62	2,71	5,41	0,17	0,76	1,51
105	0,59	**	**	0,62	**	**	0,17	**	**
120	0,54	**	**	0,62	**	**	0,17	**	**
135	0,50	**	**	0,52	**	**	0,15	**	**
150	0,50	**	**	0,42	**	**	0,12	**	**

* = Not readable by the tool; ** = Not done

4. Conclusions

Based on the research conducted, it can be concluded that increasing the concentration of NaOH decreases the flowability of 3D geopolymer mortar specimens, with an average reduction of 28%. The flow values for NaOH concentrations of 8M, 10M, and 12M are 213.5, 177.5, and 153.67 mm, respectively. The ability of the 3D geopolymer mortar filaments to maintain their shape is highly dependent on the material's stress value due to the slump flow. The 10M and 12M variations exhibit better resistance to slump compared to the 8M variation. Therefore, 3D geopolymer mortar with 10M and 12M NaOH concentrations can be developed as a material for 3D printing. To further enhance the flowability of 3D geopolymer mortar during the printing process, it is recommended to explore the use of various types of superplasticizers. These additives could help accommodate the flowability requirements necessary for effective printing while also addressing the issue of rapid setting times associated with high-calcium fly ash-based geopolymers.

Acknowledgment

The research was funded by the Building Materials and Structures Laboratory, Department of Civil Infrastructure Engineering, ITS. We would like to express our sincere appreciation to PT YTL Paiton Jawa Power for their generous support in providing the materials necessary for this study. Their contribution has been invaluable in facilitating our research efforts.

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