Synthesis and Properties of Geopolymer Mortar with Sodium Metasilicate Nonahydrate

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1. INTRODUCTION

The production of cement is one of the largest industries in the world. By calcinating the limestone, the production process consumes plenty of energy and releases large amount of CO_2. Recently, geopolymer materials [1] have attracted large attention to become alternative for ordinary Portland cement (OPC) because of its eco-friendly production process using industrial by-product and low CO_2 emission. Traditional "two-part" geopolymers are synthesized by mixing alkaline solutions with the aluminosilicate precursors. However, the alkaline solutions such as sodium hydroxide and water glass are usually highly corrosive and viscous. Different from "two-part" geopolymers, "one-part" geopolymers using solid activators could be prepared similarly with ordinary cementitious materials, making the precast process user-friendly and easily-handled.

In this study, the fly ash (FA) and blast furnace slag (BFS) based geopolymer activated by solid sodium metasilicate nonahydrate ($Na_2SiO_3 \cdot 9H_2O$) is synthesized. The optimum mixing ratio is obtained by orthogonal design of experiment. The effect of different superplasticizers is studied to increase the workability.

2. EXPERIMENT

Orthogonal design of experiment, applied in this research, is a study on the effect of multiple factors and levels to find out the optimum level combination. In this experiment, BFS content ratio (BS%), alkali-binder ratio (A/B), binder-sand ratio (B/S), water-binder ratio (W/B), are selected as factors. For each factor, 3 levels are decided based on the previous research [2]. The levels for each factor are described in **Table 2-1**. A $L_9(3^4)$ orthogonal array is applied and is described in **Table 2-2**.

Table 2-1: Levels for each factor

Level	BS%	A/B	B/S	W/B
1	30%	4%	40%	45%
2	45%	5%	45%	50%
3	60%	6%	50%	55%

A/B is the mass ratio of Na_2O from activator to binder

The experiment data was analyzed with two approaches: analysis of range and analysis of variance (ANOVA). Analysis of range gives the order of different influential level. ANOVA checked the statistical association between factors and objective functions by f-test. f-value is calculated by Eq. (1).

$$f = \frac{SS_S/df_S}{SS_E/df_E} \quad (1)$$

Here, SS_S stands for the sample variance and SS_E stands for the error variance. df_S and df_E are the degree of freedom of sample and error which equals to 2 in this experiment. The f-value is put into f-distribution to find probability of significance. The significance level α equals to 0.05 in this experiment. For p<0.05, there exists a significant statistically association between the factor and objective function.

Two superplasticizers, polycarboxylate- based (PC) and naphthalene-based (NF) are used to improve the workability of geopolymer pastes with the dosage of 0.5% (PC1, NF1) and 1.5% (PC2, NF2). One admixture borax with the dosage of 2% (Borax1) and 6% (Borax2) are used as well. A control group (CG) with no addition of superplasticizers was also introduced for comparison. The mixing ratio is settled as BS%=60%, A/B=0.06 and W/B=47.5%.

For compressive strength test, the specimens were casted into cylinder mold with Φ5×10cm. All the specimens are cured at a room with constant temperature (20±0.5℃) for 7 day. A plastic film was wrapped after final setting to prevent the specimen from water evaporation. The specimens were unmolded after 7 days for testing.

Table 2-2: $L_9(3^4)$ orthogonal array

No.	Factors			
	BS% (A)	A/B (B)	B/S (C)	W/B (D)
1	1	1	1	1
2		2	2	2
3		3	3	3
4	2	1	2	3
5		2	3	1
6		3	1	2
7	3	1	3	2
8		2	1	3
9		3	2	1

3. RESULTS AND DISCUSSION

3.1 Workability

Table 3-1 described the analysis of range for flow. It can be judged that W/B and BS% is the most and least influential factor for flow. **Table 3-2** described the results of ANOVA for flow. Since BS% is the least influential factor for flow, it is chosen as the error source in ANOVA. Only W/B has a statistically significant association with the flow. The mixing ratio combination for the largest flow is A1B1C3D3.

3.2 Compressive strength

Table 3-3 described the analysis of range for compressive strength. It can be judged that BS% and A/B are the most and least influential factor for compressive strength (7d). **Table 3-4** described the results of ANOVA for compressive

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strength. Since A/B is the least influential factor for compressive strength, it is chosen as the error source in ANOVA. Both BS% and W/B have a statistically significant association with the compressive strength while BS% is more dominant. The mixing ratio combination for the largest compressive strength is A3B3C1D1.

Table 3-1: Range analysis for flow

Avg (mm)	BS%	A/B	B/S	W/B
Level 1	**239.00**	**249.33**	223.00	168.00
Level 2	230.00	239.33	213.33	239.67
Level 3	214.33	194.67	**247.00**	**275.67**
Range	24.67	54.67	33.67	107.67
Order	4	2	3	1

Table 3-2: ANOVA for flow

Source	SS	df	MS	f	p
D	18024.2	2	9012.1	19.28	0.049
B	5083.6	2	2541.8	5.44	0.155
C	1802.9	2	901.4	1.93	0.341
A (Error)	934.9	2	467.4	-	-

Table 3-3: Range analysis for compressive strength

Avg (MPa)	BS%	A/B	B/S	W/B
Level 1	8.07	19.91	**24.21**	**29.02**
Level 2	26.50	21.87	21.75	20.05
Level 3	**30.12**	**22.91**	18.73	15.62
Range	22.05	54.67	5.47	13.41
Order	4	2	3	1

Table 3-4: ANOVA for compressive strength

Source	SS	df	MS	f	p
A	839.24	2	419.62	60.36	0.016
D	280.01	2	140.00	20.14	0.047
C	45.05	2	22.53	3.24	0.236
B(Error)	13.90	2	6.95	-	-

3.3 Superplasticizers

The fresh and hardened properties of geopolymer pastes with different superplasticizers are shown in **Figure 3-1** and **Figure 3-2**.

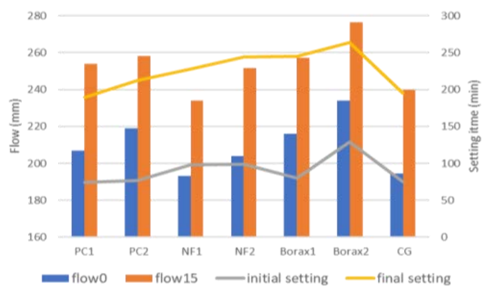

Figure 3-1: Fresh properties of geopolymer pastes

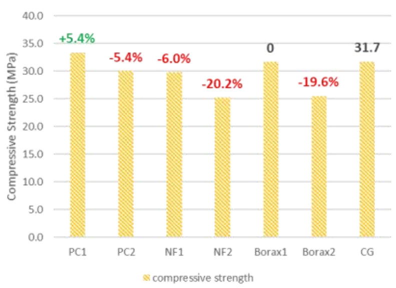

Figure 3-2: Compressive strength (7d) of geopolymer pastes

To Summarize, PC can improve the flow value and has a positive effect on compressive strength under small dosage. Borax could prolong the setting time and improve the flow value significantly while has negative influence on compressive strength under large dosage. NF does not work effectively compared with PC and Borax.

3.4 Stress-strain relationship

The samples used for the stress-strain curves are prepared with the mixing ratio of No. 5 with combination of A2B2C3D1. It is considered as the optimum mixing ratio in this experiment. The result is described in **Figure 3-3**. Here, lateral tensile and vertical compressive strain are indicated by plus and minus.

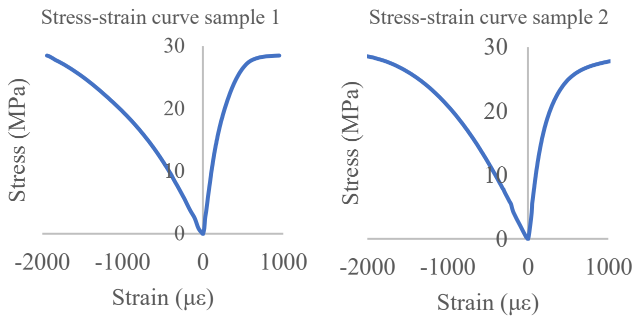

Figure 3-3: Stress-strain curves of geopolymer mortar

The compressive strength, elastic modulus, and Poisson ratio is 28.4MPa and 28.6MPa, 25.2GPa and 25.8GPa, 0.24 and 0.22 respectively for sample 1 and sample 2.

4. CONCLUSIONS

This study confirmed the feasibility of using solid $Na_2SiO_3 \cdot 9H_2O$ as sole activator to synthesize FA/BFS based geopolymer mortar. By taking care of both compressive strength and workability, an approximate area of mix ratio is given in **Table 4-1**.

Table 4-1: Recommended area of mix ratio for $Na_2SiO_3 \cdot 9H_2O$ activated FA/BFS based geopolymer mortar

<table>
<tr><td rowspan="2">Factors</td><td>BS%</td><td>A/B%</td><td>B/S%</td><td>W/B%</td></tr>
<tr><td>50±10</td><td>5±1</td><td>45±5</td><td>45±5</td></tr>
<tr><td rowspan="2">Objectives</td><td colspan="2">Flow (mm)</td><td colspan="2">7d compressive strength (MPa)</td></tr>
<tr><td colspan="2">200</td><td colspan="2">30</td></tr>
</table>

PC is suitable superplasticizers under dosage of 0.5% for $Na_2SiO_3 \cdot 9H_2O$ activated geopolymers which can increase the flow without sacrificing the compressive strength.

REFERENCES

[1] Joseph Davidovits, Geopolymer Chemistry and Applications, 5th edition, Geopolymer Institute, 2020
[2] Shaoqi HUANG et al., Workability and compressive strength of Na2SiO3-activated geopolymer mortar cured at ambient temperature, JCI Annual Convention, Vol. 41, No. 1, pp2003-2008, 2019