Research Article

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Effects of load types and critical molar ratios on strength properties and geopolymerization mechanism

<https://doi.org/10.1515/rams-2021-0019>

Received Dec 13, 2020; accepted Dec 23, 2020

Abstract: In this study, the effect of $\text{SiO}_2/\text{Al}_2\text{O}_3$ (S/A), $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ (N/A) and $\text{H}_2\text{O}/\text{Na}_2\text{O}$ (H/N) molar ratios on bending and compressive strength of geopolymer were investigated. The geopolymerization mechanism was also analyzed from microstructure difference by FTIR. The experimental results showed that compressive strength and bending strength of geopolymer has an opposite reaction under different critical molar ratios. The increase of S/A molar ratio and the decrease of N/A and H/N molar ratios have resulted in an increase of the compressive strength. However, it caused a noticeable decrease in bending strength. The microstructure of geopolymer indicated that the degree of polymerization and cohesion of geopolymer have systematical depending on these critical molar ratios, making the mechanical properties of geopolymer susceptible to different types of loads. This paper reveals the relationship between the microstructure of geopolymer and different mechanical properties and helps to selectively prepare corresponding geopolymer for different loading patterns.

Keywords: geopolymer, critical molar ratio, load type, strength properties, mechanism

1 Introduction

Geopolymer is a kind of calcium-free inorganic silica-alumina cementitious materials, which involves the chemical reaction of aluminosilicate oxides with alkali polysil-

icates yielding polymeric Si-O-Al bonds at temperatures lower than 100°C or even at room temperature [1, 2]. It presents the properties of fast hardening [3], high early-strength [4], good mechanical properties [5, 6], high-temperature resistance [7] and a good immobilization system for heavy metals [8]. Recent research showed that the synthesis from industrial wastes and modification by nanotechnology have attracted close attention in the world [9–19].

Davidovits *et al.* [1] proposed that the amorphous to semi-crystalline three-dimensional silico-aluminate structures, the products of polymerization reaction, are of the Poly(sialate) type, the Poly(sialate-siloxo) type, the Poly(sialate-disiloxo) type, whose formation has important connections with S/Al, Na/Al and H/Na molar ratios [9]. A lot of researches show that mechanical properties of geopolymer are also related to these three molar ratios. Duxson *et al.* [20, 21] studied effects of Si/Al and Na/K on the microstructure and mechanical properties of geopolymer and found that as Si/Al increased, compressive strength and Young's modulus increased and then decreased, and reached the maximum when Si/Al reached 1.90 and Na/K was 0.5/0.5. He *et al.* [22] found that as the Si/Al increased from 2 to 4, the molecular structure also changed, resulting in a gradual increase in flexural strength and Young's modulus. Fletcher *et al.* [23] studied the microstructure and mechanical properties of geopolymer, when Si/Al changed from 0.5 to 300, and found that when Si/Al was greater than 24, no brittle failure occurred in the compressive specimens, and ductility gradually increased. Zhang *et al.* [24] studied the effect of $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ and $\text{H}_2\text{O}/\text{Na}_2\text{O}$ molar ratios on the preparation of PSDS geopolymer and found that the maximum compressive strength was 34.9 MPa, when $\text{SiO}_2/\text{Al}_2\text{O}_3 = 5.5$, $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3 = 1.0$, $\text{H}_2\text{O}/\text{Na}_2\text{O} = 7.0$. Khan *et al.* [25] studied the effect of Na/Al molar ratio and water-solid ratio on geopolymer adhesion, setting time, microstructure and temperature stability. Yusuf *et al.* [26] studied the effect of $\text{H}_2\text{O}/\text{Na}_2\text{O}$ molar ratio on microstructure and mechanical properties of geopolymer. It was found that High $\text{H}_2\text{O}/\text{Na}_2\text{O}$ had a negative effect on the degree of polymerization thereby leading to low compressive strength due

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Table 1: Chemical composition of raw materials

Raw materials	Al ₂ O ₃	SiO ₂	Na ₂ O	H ₂ O	others
Water glass	0.00%	27.35%	8.42%	64.23%	0.00%
Metakaolin	32.60%	59.72%	0.00%	0.00%	7.68%

to the presence of excessive pores and microcracks within the matrix. Zhang *et al.* [27] studied the reaction kinetics, phase formation and mechanical properties of metakaolin-based geopolymer by varying Si/Al molar ratios of 1.2-2.2 and Na/Al molar ratios of 0.6-1.2.

In view of the microstructure of geopolymer, researchers used XRD\SEM and other microscopic techniques to study it. Duan *et al.* [28]. investigated durability and microstructure of fly ash and metakaolin based geopolymer, they concluded that geopolymer presented better durability and denser microstructure compared to ordinary Portland cement when exposed to elevated temperatures and acid attack. Subaer *et al.* [29]. investigated the structure of the geopolymer using XRD. The XRD patterns revealed that Na-PS geopolymer consists of zeolite-X in conjunction with amorphous aluminosilicate, while Na-PSS geopolymer was amorphous with a broad hump in the region 20°–38° (2 θ). Duan *et al.* [30] investigated the effects of silica fume on properties of fly ash based geopolymer under thermal cycles. They concluded that the incorporation of silica fume optimizes the microstructure and improves the thermal resistance of geopolymer and the pores of geopolymer are also refined by the addition of silica fume. From the above results, as a new gel material, the microstructure analysis of geopolymers has yet to be further revealed.

Lots of studies have shown that Si₂O/Al₂O₃, Na₂O/Al₂O₃ and H₂O/Na₂O molar ratios are the key parameters that influence the microstructure and mechanical properties of geopolymer. Most of them mainly focus on the effects of these ratios on compressive strength of geopolymer. However, researches are rare especially on effects and relationships of different load types on the mechanical properties of geopolymer. So in this paper, we prepare Na-PSS geopolymer to study effects of different molar ratios on mechanical properties and microstructure of geopolymer and relationships between microstructure of geopolymer and different mechanical properties.

Table 2: Mix proportions

No.	SiO ₂ /Al ₂ O ₃	Na ₂ O/Al ₂ O ₃	H ₂ O/Na ₂ O
1	4.30	0.95	12.63
2	4.40	0.95	12.63
3	4.50	0.95	12.63
4	4.30	1.00	12.63
5	4.30	1.20	12.63
6	4.30	0.95	13.32
7	4.30	0.95	14.06

2 Materials and Methods

2.1 Raw materials

Relatively pure powdery metakaolin (MK) from Hangzhou, Junyi Chemical Co. was selected in this study. NaOH with a purity of 99.8 wt% and sodium silicate solution (water glass) with S/A molar ratio of 3.2 and solid content of 37% were used as alkaline reagents. Chemical compositions of raw materials above are summarized in Table 1.

2.2 Mix proportions

Based on previous studies, the primary S/A, N/A and H/N molar ratios were adjusted on a small scale to study the effect on mechanical properties and microstructure of geopolymer. Mix proportions are shown in detail in Table 2.

2.3 Mixing, curing, and testing methods

Alkaline activator was made by dissolving solid NaOH in a solution of water glass and water, the liquid been allowed to cool to room temperature before mixing with metakaolin. The dry metakaolin was stirred for 1 minute by an adjustable speed mixer, then the alkaline activator was added slowly and mixed for 3 minutes in low speed then stirred for 2 minutes in high shear mode. The expansion test of geopolymer paste was conducted according to cement mortar expansion test. The paste was poured in 40×40×160

mm³ molds. The molds sealed with plastic foil to prevent loss of water and left in room temperature for 24 hours then demolded and moved to the curing room with 90 humidity and temperature and kept there until appropriate testing age.

According to Mehta and Siddique results [31], geopolymer did well in early strength and in 3-days the mix has already reached more than 90% of 28-days strength.

2.4 Testing methods

2.4.1 Bending and compressive strength

The specimens were moved from the curing room and wiped by dried towel after curing for the specific ages. Three specimens for flexural strength and six specimens for compressive strength of each group were tested according to JGJ/T233-2011 [32].

2.4.2 FTIR test

Samples dried for 24 h, ground and sieved to pass sieve No. 0.3 mm. Then they were mixed with KBr (inert and does not show absorbance for radiation) to get better resolution for peaks. AVATAR370 machine was used to performed FTIR test. The resolving power is 4 cm⁻¹, and the scanning frequency is 32 times with a wavelength of 450–4000 cm⁻¹. Samples were tested using attenuated total reflectance.

2.4.3 NMR-MAS test

The specimens were kept at 20°C, and the hydration was stopped by pure alcohol. Before the test, the specimens were broken and ground on a 45 mm sieve to a residue ratio of less than 2%. The NMR-MAS were performed by using Avance III HD Solid state nuclear magnetic resonance spectrometer. Cross polarization was used for ²⁹Si and one pulse was used for ²⁷Al.

3 Results and discussion

3.1 Effects of critical molar ratios on mechanical properties of geopolymer

Figure 1 showed the effect of the S/A, N/A and H/Na molar ratios on bending strength, compressive strength and

expansion degree of geopolymer. In Figure 1(a) samples elaborated with varied ratios of S/A and fixed N/A and H/N ratios showed better results in expansion degree of geopolymer paste. Duxson *et al.* mentioned that developing of S/A molar ratio has a progressive effect on the tetrahedral aluminosilicate network strength, as the strength of Si-O-Si bond is stronger than Si-O-Al and Al-O-Al [21]. Furthermore, the rise of S/A ratio from 4.30 to 4.40 and 4.50 increased the fluidity resulting in a gradual reduction of bending strength for hardened geopolymer by 23% and 43%, while the com-

Figure 1: Effect of different molar ratios (a) S/A, (b) N/A and (c) H/Na on Bending strength, compressive strength and expansion degree of geopolymer

pressive strength increased by 3% and 9% for S/A 4.40 and 4.50 ratios respectively.

With fixed S/A and H/N molar ratios, as N/A molar ratio varies from 0.95 to 1.00 and 1.20, the fluidity increased, that lead to a gradual increase for the expansion degree of the geopolymer paste and bending strength of hardened geopolymer, while the compressive strength showed a dropping trend, Figure 1(b). For N/A molar ratio 1.00 the bending strength increased by 17% and compressive strength decreases by 6.5% compared to N/A 0.95. Other samples with N/A molar ratio 1.2 followed the same behavior where the bending strength increased by 27% and compressive strength decreases by 13%.

Figure 1(c) represents the effect of changing H/N molar ratio from 12.63 to 13.32 and 14.06 for the same $\text{Si}_2\text{O}/\text{Al}_2\text{O}_3$ and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ molar ratios. As the H/N increased each of the fluidity, expansion degree and bending strength increased except the compressive strength which decreased by 4% and 5% for H/N 13.32 and 14.06 respectively. The bending strength values rose by 2% and 7%.

3.2 FTIR analysis

FTIR absorption spectroscopy is well known for its sensitivity in characterizing materials with short-range structural order and has been useful for characterizing geopolymer. Figure 2 shows the main adsorption bands of geopolymer for different S/A, N/A and H/N molar ratios. The stretching vibration of O-H bond at 1645 and 3448 cm^{-1} beside the asymmetric stretching vibration (T-O-Si) at about 1020 cm^{-1} are related to non-solubilized particles where T = Si or Al, symmetric stretching vibration of CO_2 at about 873 cm^{-1} , symmetrically stretching vibration of Si-O at 698 cm^{-1} , bending vibration of Si-O-Al at 578 cm^{-1} and in-plane bending vibration of Si-O-Si and O-Si-O at 453 cm^{-1} respectively as in Figure 2(b) [33–35].

As per Figure 2(a), the stretching vibration is extremely sensitive to Si/Al composition ratio in structure for that by increasing the S/A ratio from 4.3 to 4.5 the corresponding peak may shift to lower frequency due to the more Si substitution by Al in IV fold coordination, which indicates that the partial replacement of SiO_4 species by AlO_4 will result in a change in the local chemical environment of Si-O bond [21]. Si-O bond has higher bond energy than Al-O bond, leading to higher cohesion in structure, which resulted in higher compressive strength for S/A molar ratio of 4.50 than 4.30 as mentioned before. The broad bands in the region of 1645–3448 cm^{-1} characterized the spectrum of stretching and deformation vibrations of OH and H-O-H groups from weakly bound water molecules which are adsorbed on the

(a)

(b)

(c)

Figure 2: Fourier transform infrared spectroscopy (FTIR) of geopolymer (a) FTIR of S/A molar ratio of 4.30 and 4.50, (b) FTIR of N/A molar ratio of 0.95 and 1.20 and (c) FTIR of H/N molar ratio of 12.63 and 14.06

surface or trapped in the large cavities between rings of geopolymeric products [35]. So the broader bands in the region for S/A molar ratio of 4.50 than 4.30 caused more weak regions and lead to lower bending strength.

With the increase of N/A molar ratio from 0.95 to 1.20, asymmetric stretching vibration (T–O–Si) shifted from 1020 cm^{-1} to 1014 cm^{-1} and bending vibration of Si–O–Al shifted from 578 cm^{-1} to 574 cm^{-1} leading to the lower compressive strength of geopolymer. The vibration in the region of $900\text{--}1300\text{ cm}^{-1}$ for N/A molar ratio of 0.95 is broader than 1.20 indicating that the increase of alkali concentration stimulated the dissolution and recombination of silicate components in raw materials so that more uniform geopolymerization products be generated, resulting in higher bending strength of geopolymer [24].

With a high H/N molar ratio, stretching vibration (T–O–Si) shifted from 1020 cm^{-1} to 1018 cm^{-1} and the region of $900\text{--}1300\text{ cm}^{-1}$ became broader leading to lower compressive strength due to the presence of excessive pores and microcracks within the matrix. At the same time, with high H/N molar ratio, formation of more gel phase $[\text{Na}_x(\text{AlO}_2)_y \cdot n\text{NaOH} \cdot m\text{H}_2\text{O}]$ made the polymerization between alkali silicate solution and aluminum-silicon complicated, contributing to the improvement of bending strength [25].

3.3 NMR-MAS analysis

Magic Angle Spectroscopy Nuclear Magnetic Resonance (MAS NMR) is an important microscopic technique for studying Si–Al structures, which can provide effective structural data for the study of geopolymers, especially MAS

Figure 3: ^{27}Al MAS NMR results of geopolymers

NMR of ^{29}Si and ^{27}Al . ^{27}Al MAS NMR can determine the coordination number of aluminum atoms in geopolymer and the corresponding basic unit, but cannot distinguish whether the geopolymer unit is PS type, PSS type or PSDS type, which requires ^{29}Si MAS NMR. Studies have shown that the chemical shift of ^{29}Si increases with the degree of polycondensation of the silicon tetrahedron, and with each alum tetrahedron connecting to silicon tetrahedron, the chemical shift of ^{29}Si increases by about 5 ppm. Therefore,

Figure 4: ^{29}Si MAS NMR spectrum of geopolymer with different molar ratios

^{29}Si MAS NMR can be used to determine the structure and dynamics of geopolymer systems.

The ^{27}Al MAS NMR results of geopolymers in Figure 3 showed that molar ratios had some influence on geopolymer structure. The extremely narrow peak at 58–61 ppm indicated a well-defined single Al environment which exists in the form of tetrahedral Al $\text{Q}_4(4\text{Si})$, and there were no low relative molecular mass like dimers and trimers in geopolymer structure, which mean geopolymer was a kind of silicon-aluminum compounds with spatial three-dimensional networks.

^{29}Si MAS NMR spectrum showed a broad resonance between –75 ppm and –120 ppm associated with very strong resonance at about –87 ppm and a small peak at about –96 ppm (Figure 4). Resonances in the ^{29}Si MAS NMR spectrum, namely –85 ppm, –87 ppm and –96 ppm, can be assigned to $\text{Q}_4(4\text{Al})$, $\text{Q}_4(4\text{Al})$ and $\text{Q}_4(3\text{Al})$ respectively. The spectrum showed that with Na/Al ratio and H/Na increasing, $\text{Q}_4(3\text{Al})$ disappeared, which indicated to some degree that there were more Al in the geopolymer structure.

4 Conclusions

This paper firstly studied the effect of load type on properties of geopolymers and use FTIR and NMR to explain the reason for different development of compressive strength and bending strength of geopolymer. The detailed experimental results were shown as follows.

- (1) $\text{Si}_2\text{O}/\text{Al}_2\text{O}_3$, $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ and $\text{H}_2\text{O}/\text{Na}_2\text{O}$ molar ratios all had significant influence on mechanical properties and microstructure of geopolymer.
- (2) The fluidity of the geopolymer can be enhanced by higher $\text{Si}_2\text{O}/\text{Al}_2\text{O}_3$, $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ and $\text{H}_2\text{O}/\text{Na}_2\text{O}$ molar ratios. But $\text{Si}_2\text{O}/\text{Al}_2\text{O}_3$, $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ and $\text{H}_2\text{O}/\text{Na}_2\text{O}$ molar ratios had different effects on mechanical strength with different load types. When increasing $\text{Si}_2\text{O}/\text{Al}_2\text{O}_3$ molar ratio or decreasing $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ and $\text{H}_2\text{O}/\text{Na}_2\text{O}$ molar ratio, bending strength tended to decrease and compressive strength tended to increase.
- (3) Results in the paper showed that bending strength increased while compressive strength decreased and bending strength decreased while compressive strength increased, which may be connected with the polymerization and cohesion of groups in the microstructure of geopolymer. The fluidity of fresh geopolymer was not related to the mechanical properties of geopolymer.

Acknowledgement: The authors want to thank the support by Zhejiang Key Research & Development Program (2020C04013) and National Natural Science Foundation of China (52078453).

Funding information: This work was supported by the Zhejiang Key Research & Development Program (2020C04013) and National Natural Science Foundation of China (52078453).

Conflict of Interests: Authors state no conflict of interest.

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