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Research Article

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Posted Date: September 15th, 2026

DOI: <https://doi.org/10.21203/rs.3.rs-10772036/v1>

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Additional Declarations: No competing interests reported.

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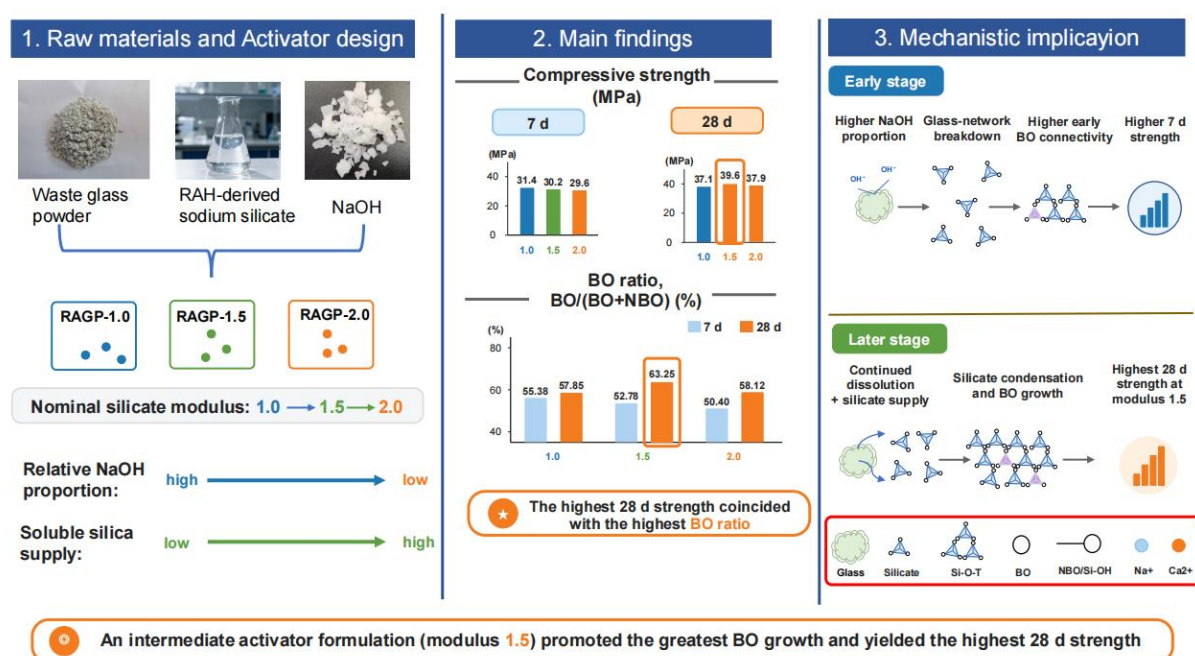
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ABSTRACT

Under the condition of an approximately constant Na_2O equivalent, with waste glass powder as the sole precursor, rice husk ash-based sodium silicate combined with NaOH was used to prepare rice husk ash-based sodium silicate-activated glass powder cementitious materials (RAGP) with moduli of 1.0, 1.5, and 2.0. Compressive strength, XRD, FTIR, and XPS analyses were performed to characterize the strength development and silicon-oxygen network evolution at 7 d and 28 d. The results show that the 7 d compressive strength decreased from 31.4 MPa to 29.6 MPa as the modulus increased; at 28 d, RAGP-1.5 achieved the highest compressive strength of 39.6 MPa, corresponding to an increase of 31.1%. Compared with the raw glass powder, the centroid of the amorphous diffuse peak of the hardened RAGP shifted toward higher angles by 2.13° – 2.33° , while the centroid difference among the different mix proportions did not exceed 0.19° , indicating that alkali activation altered the amorphous scattering environment of the glass powder without inducing a resolvable crystalline phase transformation. The main Si-O-T band of all specimens was located at 973 – 981 cm^{-1} and did not exhibit a consistent shift with curing age. In addition, the bridging oxygen ratio at 7 d decreased from 55.38% to 50.40% as the modulus increased; at 28 d,

the bridging oxygen ratio of RAGP-1.5 reached 63.25%, an increase of 10.47% relative to 7 d, consistent with the ranking of compressive strength. A higher NaOH proportion was favorable for early-age depolymerization of the glass network, whereas the later-age strength depended on the balance between continued glass dissolution and polycondensation of soluble silicon; within the present mix design range, the activator composition with a modulus of 1.5 yielded the highest 28 d compressive strength and the largest increment in bridging oxygen.

Graphical abstract



Keywords: rice husk ash-based sodium silicate; waste glass powder; alkali activation; activator; silicon-oxygen network

1 Introduction

With the rapid development of urbanization and manufacturing, waste glass has become a typical large-volume solid waste characterized by high output, stable chemical properties, and near-non-degradability under natural conditions. Its landfill disposal not only occupies land but also causes long-term persistence in the environment due to the difficulty of natural weathering of

glass, and its resource utilization rate remains relatively low [1]. Meanwhile, the production of conventional cementitious materials represented by cement is a high-energy-consumption and high-carbon-emission industry. Under the strategic goals of "carbon peaking and carbon neutrality" advocated by the United Nations, the development of low-carbon cementitious materials using industrial and agricultural solid wastes as raw materials has become a research hotspot in the field of building materials. Therefore, converting waste glass—a difficult-to-treat solid waste—into a high-value-added cementitious material, thereby simultaneously alleviating the pressures of both solid waste disposal and carbon emissions from building materials, is of significant environmental and engineering importance.

Existing studies have shown that glass powder does not merely act as an inert filler in an alkaline environment; OH^- can attack the Si-O-Si bonds in the glass network, promoting depolymerization of the silicon-oxygen network and forming silicate monomers or oligomers [2]. Subsequently, the dissolved silicate species reconstruct the silicon-oxygen network through polycondensation reactions, forming amorphous reaction products with cementitious capacity [3]. For systems using glass powder as the primary precursor, the reaction products typically exhibit pronounced silicate gel characteristics. In addition, when components such as aluminum and calcium are present in the precursor and participate in the reaction, the gel composition and local structure undergo further changes [4]. Therefore, the mechanical properties of alkali-activated glass powder systems depend not only on whether the glass powder dissolves, but also on whether an appropriate balance can be established between the dissolution and polycondensation processes.

In alkali activation reactions, sodium silicate generally serves dual roles as both an alkaline medium and a soluble silicon source, and the molar ratio of SiO_2 to Na_2O , the modulus is an

important parameter affecting the chemical state of the activator [5–7]. At lower moduli, the alkalinity of the system is relatively higher, which favors rapid depolymerization of the glass network; however, an insufficient supply of soluble silicon may limit subsequent gel polycondensation [6]. As the modulus increases, the content of exogenous soluble silicon increases, improving the conditions for gel network construction. It should be noted that an excessively high modulus may also reduce the effective alkalinity and inhibit the continued dissolution of glass powder [7]. It can thus be seen that the modulus of sodium silicate does not control the reaction in a unidirectional manner, but rather influences the structural evolution and macroscopic performance of the alkali-activated system by regulating the competitive relationship between the dissolution capacity of glass powder and the polycondensation capacity of the gel [8].

Although alkali-activated materials are regarded as low-carbon cementitious materials, their sustainability is largely constrained by the alkali activator. Commercial sodium silicate (water glass) is typically produced by calcining quartz sand and sodium carbonate at 1400–1500 °C, making it the component with the highest energy consumption, highest cost, and largest carbon emission contribution in the entire system, and thus the primary bottleneck limiting alkali-activated materials from truly achieving low-carbon status [9,10]. Therefore, developing low-energy, low-carbon alternative activators derived from solid waste is crucial for improving the sustainability of alkali-activated materials. Rice husk ash is a high-silicon agricultural waste generated during rice processing, with an SiO_2 content of up to approximately 90%, mostly present in an amorphous state, and possessing good alkali-dissolution reactivity [11]. Owoeye and Isinkaye [12] confirmed that self-prepared sodium silicate obtained through alkali dissolution (hydrothermal) treatment of rice husk ash with NaOH solution, when used as a substitute for

commercial sodium silicate as an activator, yielded alkali-activated materials with mechanical properties comparable to those of commercial sodium silicate systems.

Recently, the application of rice husk ash-based sodium silicate and glass powder in alkali-activated materials has attracted attention; however, existing studies have mostly focused on fly ash-slag, metakaolin, or composite precursor systems. In comparison, research using glass powder as the sole cementitious precursor and activated with self-prepared rice husk ash-based sodium silicate remains relatively limited. In particular, there is a lack of explanations, corroborated by macroscopic performance and multi-scale characterization, of how variations in activator modulus affect the reaction extent of waste glass powder, silicon-oxygen network reconstruction, and surface chemical state. Based on this, this study employs rice husk ash-based self-prepared sodium silicate and NaOH to constitute a composite alkali activator, uses glass powder as the cementitious precursor, and prepares paste specimens by adjusting the activator modulus. Using 7 d and 28 d compressive strength as macroscopic performance indicators, and combining X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and X-ray photoelectron spectroscopy (XPS), the phase characteristics, silicon-oxygen bond structure, and surface chemical state evolution of the glass powder reaction products under different modulus conditions are analyzed. On this basis, the mechanism of the effect of rice husk ash-based sodium silicate modulus on the alkali activation reaction of glass powder is discussed from the perspectives of three processes: glass powder depolymerization, silicate migration, and gel polycondensation. This study aims to provide a basis for the synergistic resource utilization of glass powder and rice husk ash, and to provide a reference for the composition design and mechanistic analysis of silicon-rich solid waste-based alkali-activated materials.

2 Experimental materials and methods

2.1 Raw materials

In this study, glass powder was used as the cementitious precursor, rice husk ash was used as the silicon source to prepare the sodium silicate solution, and NaOH was used to adjust the composition of the composite activator. The raw materials used included rice husk ash, glass powder, flake NaOH, and deionized water.

The rice husk ash was obtained from a biomass power plant in Hunan Province, China. The glass powder was produced from recycled waste glass through cleaning, crushing, and grinding, with a fineness of approximately 2000 mesh. The NaOH was a flake solid with a purity of 96%.

The chemical compositions of the rice husk ash and glass powder were determined using an X-ray fluorescence spectrometer, and the results are shown in Table 1. The SiO₂ content in the rice husk ash was 92.30%, indicating a relatively high silicon content, which enables its use as a silicon source for preparing sodium silicate. The SiO₂, CaO, and Na₂O contents in the glass powder were 68.52%, 11.53%, and 14.23%, respectively. The relatively high SiO₂ content and amorphous structure of the glass powder provide the basis for the dissolution of the glass network under alkaline conditions and the subsequent formation of silicate gel.

Table 1 Chemical composition of rice husk ash and glass powder (%)

Type	SiO ₂	Fe ₂ O ₃	CaO	Al ₂ O ₃	K ₂ O	MgO	Na ₂ O	SO ₃	TiO ₂
Rice husk ash	92.30	0.179	1.14	0.256	3.88	0.723	0.041	0.224	0.031
Glass powder	68.52	0.378	11.53	2.587	0.549	1.31	14.23	0.213	0.0579

XRD was used to analyze the phase characteristics of the rice husk ash and glass powder, and the results are shown in Figure 1. The XRD patterns of both raw materials exhibited broad

amorphous diffuse peaks, indicating that they contain a relatively high proportion of amorphous phase. In addition, minor diffraction peaks corresponding to crystalline SiO₂ could be observed in the rice husk ash.

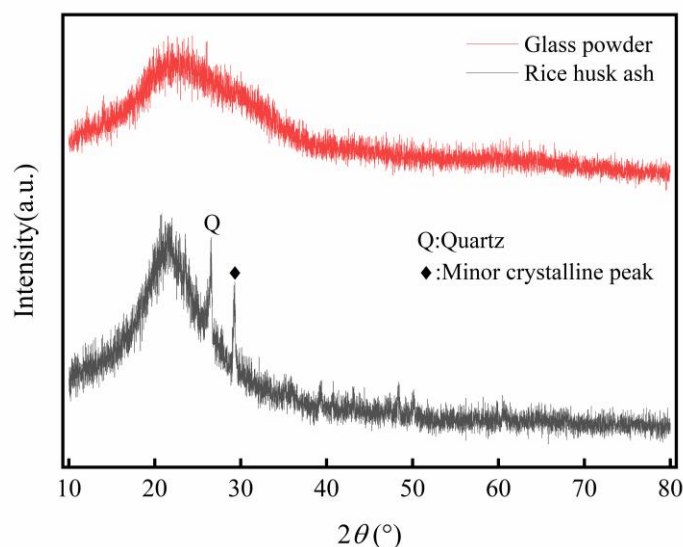


Figure 1 XRD of raw materials

2.2 Preparation of rice husk ash-based sodium silicate

Rice husk ash-based sodium silicate was prepared using a constant-temperature alkali dissolution method. For each batch, 171.4 g of flake NaOH was weighed and dissolved in 828.7 g of deionized water. After the NaOH was fully dissolved, 1000 g of rice husk ash was added to ensure sufficient contact between the rice husk ash and the alkaline solution. Subsequently, the mixed slurry was placed in a constant-temperature water bath at 60 °C and reacted continuously for 6 h under stirring at 500 rpm. After the reaction was completed and the mixture was cooled to room temperature, the undissolved residue was removed by filtration, and the resulting filtrate was the rice husk ash-based sodium silicate solution used in the subsequent tests.

The chemical composition of the filtrate was determined by inductively coupled plasma optical emission spectrometry (ICP-OES). The test results showed that the mass fractions of SiO₂ and Na

in the filtrate were 28.52% and 7.04%, respectively. After converting the Na content to Na_2O , its mass fraction was 9.49%. Based on the molar ratio of SiO_2 to Na_2O in the sodium silicate, the modulus of the prepared rice husk ash-based sodium silicate was calculated to be approximately 3.1.

2.3 Mix design and specimen preparation

To investigate the effect of activator modulus on the mechanical properties and microstructural evolution of RAGP, three groups of paste specimens were designed. In all three groups, glass powder was used as the precursor, with the dosage fixed uniformly at 520 kg/m^3 , and the activator modulus was adjusted by varying the relative dosages of self-prepared sodium silicate, NaOH, and added water. The mix design is shown in Table 2. The dosage of self-prepared sodium silicate was increased from 154 kg/m^3 to 308 kg/m^3 , while the dosages of NaOH and added water were correspondingly decreased, such that the overall modulus of the activator (accounting for the combined contribution of SiO_2 and Na_2O from both the sodium silicate and NaOH) was designed to be 1.0, 1.5, and 2.0, respectively. The specimens were designated as RAGP-1.0, RAGP-1.5, and RAGP-2.0, respectively.

Table 2 Mix design

Specimen No.	Modulus	Glass powder	Sodium silicate	NaOH	Water
RAGP-1.0	1.0	520	154	35.7	50
RAGP-1.5	1.5	520	231	26.8	20
RAGP-2.0	2.0	520	308	17.9	0

Specimen preparation procedure was as follows: each component was first weighed according to the mix proportions, and NaOH, self-prepared sodium silicate, and added water were mixed uniformly to form the composite alkali-activating solution. The glass powder was then slowly

added to the activating solution under low-speed stirring, and stirring was continued until the paste became homogeneous. Immediately after mixing, the paste was cast into 70.7 mm × 70.7 mm × 70.7 mm cubic molds. The specimens were kept in a drying oven at 60 °C for 12 h before demolding, and were then transferred to a constant-temperature and constant-humidity curing chamber, where they were cured at a temperature of 20±2 °C and a relative humidity of ≥95% until 7 d and 28 d.

2.4 Testing and characterization methods

Compressive strength was measured on 70.7 mm × 70.7 mm × 70.7 mm cubic specimens, which were loaded continuously to failure at a rate of 0.5 MPa/s at the ages of 7 d and 28 d. Three parallel specimens were tested for each group at each age, and the results were taken as the arithmetic mean.

XRD was performed using a Rigaku SmartLab SE X-ray diffractometer (Japan) to characterize the phase composition of the specimens, with a scanning range of 10°–80° (2 θ) and a scanning rate of 5°/min.

FTIR was performed using a Thermo Fisher Scientific Nicolet iS20 Fourier transform infrared spectrometer (USA) to characterize the chemical bond structure of the specimens, with a wavenumber range of 400–4000 cm⁻¹ and a resolution of 4 cm⁻¹.

XPS was performed using a Thermo Scientific K-Alpha X-ray photoelectron spectrometer (USA) to analyze the elemental composition and chemical state of the specimen surfaces. A full-spectrum scan was first conducted to identify the major elements on the sample surface, followed by high-resolution scans of the Si 2p, O 1s, and Al 2p characteristic peaks.

3 Results and analysis

3.1 Compressive strength

The compressive strengths of the RAGP systems at 7 d and 28 d are shown in Figure 2. Overall, the RAGP systems achieved considerable mechanical properties under ambient-temperature curing: the 28 d compressive strengths of all groups fell within the range of 37–40 MPa, and a continuous increasing trend was observed from 7 d to 28 d for all groups, indicating that the alkali activation reaction of RAGP proceeded continuously during curing, with gel continuously forming and densifying.

Regarding the 7 d early-age strength, the three groups exhibited a monotonic decrease with increasing activator modulus: RAGP-1.0 was the highest at 31.4 MPa, followed by RAGP-1.5 at 30.2 MPa, and RAGP-2.0 was the lowest at 29.6 MPa. Although the differences among the three groups were limited (approximately 6%), the direction of variation was consistent with the alkalinity of the activator. Given that the total Na_2O dosage was essentially constant across the three groups, the decrease in modulus mainly resulted from an increase in NaOH dosage, i.e., in the lower-modulus system, more alkali was present in the form of free OH^- , resulting in a higher initial pH. A higher alkalinity favors the rapid breakage of Si-O-Si bonds in the glass powder network and the dissolution of reactive silicon, thereby accelerating the early-age reaction process [13,14]. Consequently, RAGP-1.0, which was rich in free alkali, achieved a relatively higher strength at early age, while RAGP-2.0, with a relatively insufficient free alkali content and a weaker driving force for glass dissolution due to its high modulus, exhibited a slightly lower early-age strength.

By 28 d, the strength ranking changed to RAGP-1.5 (39.6 MPa) > RAGP-2.0 (37.9 MPa) > RAGP-1.0 (37.1 MPa), with the highest value occurring in RAGP-1.5 at the intermediate

modulus. In addition, the magnitude of strength development followed the same pattern: the increases from 7 d to 28 d for RAGP-1.5, RAGP-2.0, and RAGP-1.0 were 31.1%, 28.0%, and 18.2%, respectively, with RAGP-1.5 exhibiting the most pronounced later-age strength gain. This transition can be explained by the fact that the later stage of the alkali activation reaction is dominated by gel polymerization, and the improvement of later-age strength depends more on the polycondensation of dissolved silicate species to form a gel network with a high degree of polymerization and dense structure, for which a sufficient supply of soluble silicon is fundamental [15–17]. Although RAGP-1.0 exhibited sufficient early-age dissolution, its sodium silicate dosage was the lowest, resulting in a relative scarcity of silicon available for polycondensation; hence, gel development was limited and the later-age strength gain was the smallest. RAGP-2.0 had the most abundant silicon supply, but its relatively low free alkali content constrained the extent of glass dissolution and reaction, preventing it from fully exploiting the advantage of its high silicon content. RAGP-1.5, in contrast, achieved a more favorable balance between alkalinity and silicon supply, and thus attained the highest 28 d strength and the largest strength gain. It can thus be seen that an intermediate modulus exists in the RAGP system at which the later-age strength is optimized, which is consistent with the widely recognized existence of an optimal activator modulus in alkali-activated materials [18,19].

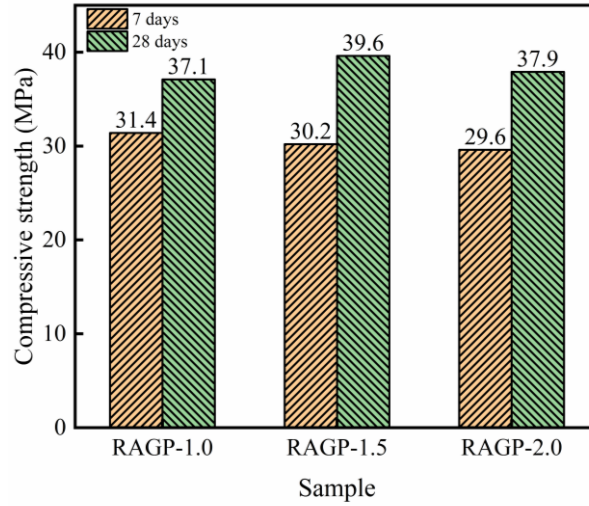


Figure 2 Compressive strength

3.2 XRD

Figure 3 presents the XRD patterns of the RAGP specimens at 7 d and 28 d.

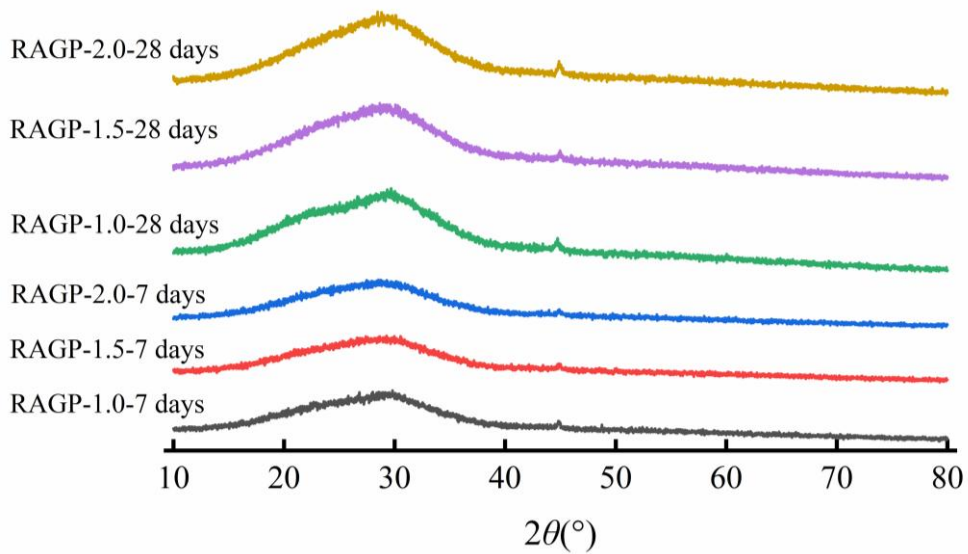


Figure 3 XRD patterns of RAGP pastes at 7 and 28 d

All six groups of hardened specimens exhibited a broadened amorphous diffuse peak in the range of approximately 20° – 40° , and no set of crystalline diffraction peaks attributable to newly formed reaction products was observed, indicating that the hardened bodies were mainly composed of amorphous phases or phases with low crystallinity [20]. A small number of low-intensity narrow peaks were present in the patterns; however, they did not form a

characteristic peak set suitable for reliable phase matching, and therefore their specific phase assignment is not discussed in this study.

As shown in Figure 1, the raw glass powder itself exhibited a distinct amorphous diffuse peak, with its peak top and centroid located at 22.47° and 25.36°, respectively. The diffuse peak in the hardened specimens includes scattering contributions from both the residual glass phase and the newly formed amorphous reaction products. XRD broad peaks alone cannot distinguish gel types such as N-A-S-H, C-A-S-H, or C-(N)-A-S-H, nor can the reaction degree of the glass powder be directly calculated [21]. Therefore, in this study, using the raw XRD data of the glass powder in Fig. 1 as the baseline, the same background subtraction and peak-position extraction method was applied to both the glass powder and the six groups of hardened specimens: the local background was subtracted within the range of 15°–42°, and the diffuse peak top position and scattering centroid were extracted. The centroid of the diffuse peak was calculated according to Eq. (1):

$$\overline{2\theta} = \frac{\int_{\theta_1}^{\theta_2} (2\theta) I_c(2\theta) d(2\theta)}{\int_{\theta_1}^{\theta_2} I_c(2\theta) d(2\theta)} \quad (1)$$

where $I_c(2\theta)$ is the diffraction intensity after background subtraction, and θ_1 and θ_2 are the lower and upper limits of the integration interval, respectively.

The extracted parameters are shown in Table 3. It can be seen that, after alkali activation, the peak tops of all six groups of hardened specimens shifted to 28.60°–29.58°, and the centroids shifted to 27.48°–27.69°. Compared with the raw glass powder, the centroid of the diffuse peak of the hardened specimens shifted toward higher angles by 2.13°–2.33°. This shift was much greater than the inter-group differences among the different hardened specimens, indicating that alkali activation altered the average scattering environment of the original glass network. In addition, the centroid difference among specimens with different moduli and curing ages did not exceed 0.19°,

indicating that the activator composition and curing process did not change the crystalline phase composition of the RAGP reaction products, and that their effect was mainly reflected in the local connectivity state of the amorphous network.

Table 3 Parameters of the amorphous diffraction features of raw glass powder and RAGP pastes

Specimen	Peak top $2\theta_{\max}/^{\circ}$	Centroid/ $^{\circ}$	Centroid shift relative to glass powder/ $^{\circ}$
Glass powder	22.47	25.36	—
RAGP-1.0-7d	29.52	27.56	2.21
RAGP-1.5-7d	28.68	27.48	2.13
RAGP-2.0-7d	28.6	27.5	2.15
RAGP-1.0-28d	29.58	27.5	2.14
RAGP-1.5-28d	29.22	27.62	2.27
RAGP-2.0-28d	29.3	27.69	2.33

3.3 FTIR

Figure 4 presents the FTIR spectra of the RAGP specimens at 7 d and 28 d. The absorption band compositions of the six groups of specimens were essentially consistent, indicating that the activator ratio and curing age did not change the types of the main chemical bonds in the hardened bodies. The broad absorption band at approximately 3450 cm^{-1} was attributed to the O-H stretching vibration of hydrogen-bonded hydroxyl groups, and the absorption band at approximately 1650 cm^{-1} corresponded to the H-O-H bending vibration of molecular water; both mainly originated from gel-bound water, adsorbed water, and surface hydroxyl groups [22].

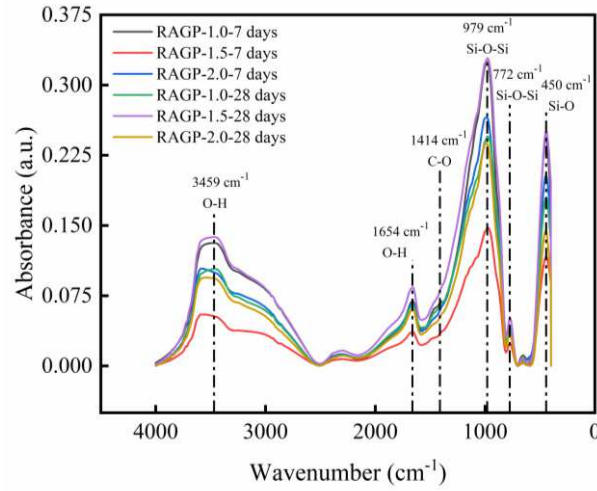


Figure 4 FTIR spectra of RAGP pastes at 7 and 28 d

The broad absorption band at 973–981 cm^{-1} was attributed to the asymmetric stretching vibration of Si-O-T (T is Si or Al) structural units, while the absorption bands at 772 and 450 cm^{-1} corresponded to the symmetric stretching vibration of Si-O-Si and the Si-O bending vibration, respectively [23,24]. These absorption bands indicate that an amorphous silicate or aluminosilicate network with silicon-oxygen tetrahedra as the basic structural unit was formed in the hardened RAGP bodies.

Since the Si-O-T main band simultaneously contains signals from both the reaction gel and the residual glass phase, the relevant parameters are used to characterize the average vibrational environment rather than serving as direct measures of gel content or reaction degree [25]. To compare the Si-O bonding environments of different specimens, the same smoothing and local baseline correction methods were applied to all six sets of spectra, and the peak position and apparent full width at half maximum (FWHM) of the Si-O-T main band, as well as the integrated area ratios of the O-H and H-O-H absorption bands relative to the Si-O-T main band, were extracted; the results are listed in Table 4.

Table 4 FTIR quantitative parameters of RAGP specimens

Specimen	Si-O-T/cm ⁻¹	FWHM/cm ⁻¹	H-O-H/cm ⁻¹	A_{O-H}	A_{H-O-H}
RAGP-1.0-7d	977	212.1	1655	0.641	0.037
RAGP-1.5-7d	973	212.8	1654	0.586	0.038
RAGP-2.0-7d	981	212	1656	0.648	0.041
RAGP-1.0-28d	972	221.7	1654	0.766	0.045
RAGP-1.5-28d	974	221.7	1655	0.731	0.044
RAGP-2.0-28d	981	205.6	1679	0.693	0.045

Note: FWHM is the apparent full width at half maximum of the composite Si-O-T main band after unified baseline processing; the integrated area ratios are used only for comparison among specimens processed with the same sample preparation and data processing methods.

At 7 d, the Si-O-T main band of the three groups of specimens was located at 973–981 cm⁻¹. The main peak of RAGP-2.0 was 7.7 cm⁻¹ higher than that of RAGP-1.5, while the peak position of RAGP-1.0 was intermediate between the two, indicating that the Si-O-T main band did not shift monotonically with increasing activator modulus. The apparent FWHM of the three groups of specimens was approximately 212 cm⁻¹, with an inter-group difference of no more than 0.8 cm⁻¹, indicating that the Si-O-T vibrational environments of the different mix proportions had similar distribution widths at 7 d. At 28 d, the Si-O-T main peak of RAGP-2.0 remained at approximately 981 cm⁻¹, which was 8.7 and 7.2 cm⁻¹ higher than those of RAGP-1.0 and RAGP-1.5, respectively. The higher main peak wavenumber indicates that the average vibrational environment of the Si-O-T bonds in RAGP-2.0 differed from that of the other two groups. However, RAGP-1.5 exhibited the highest compressive strength and bridging oxygen ratio at 28 d, while its FTIR main peak wavenumber was lower than that of RAGP-2.0. It can thus be seen that the Si-O-T peak position is controlled not only by the degree of gel network connectivity, but is also jointly influenced by the residual glass phase, the Si/Al composition, and elements such as Na and Ca

[26].

As the curing age increased from 7 d to 28 d, the Si-O-T main peaks of RAGP-1.5 and RAGP-2.0 shifted by only 1.0 and 0.5 cm^{-1} , respectively, which is below the 4 cm^{-1} test resolution; the main peak of RAGP-1.0 shifted toward lower wavenumbers by 4.9 cm^{-1} , which is also only close to one instrumental resolution unit. Therefore, none of the three groups of specimens exhibited a consistent shift toward higher wavenumbers with increasing curing age. This indicates that the continued dissolution of glass powder, gel formation, and changes in the proportion of residual glass exerted a superimposed effect on the composite Si-O-T main band, and that a single peak position is insufficient to describe the network structural evolution between 7 d and 28 d [27,28]. In addition, as the curing age increased from 7 d to 28 d, the apparent FWHM of RAGP-1.0 and RAGP-1.5 increased by 4.5% and 4.2%, respectively, while that of RAGP-2.0 decreased by 3.0%. Notably, the main band of RAGP-2.0 at 28 d was approximately 7% narrower than those of the other two groups, indicating a relatively smaller distribution range of its Si-O-T vibrational environment. This is because the Si-O-T main band is composed of multiple overlapping structural units, and the FWHM is relatively sensitive to the baseline position and peak-shape overlap [29]. It should be noted that, as the curing age increased, the $A_{\text{O-H}}$ and $A_{\text{H-O-H}}$ of the three groups of specimens increased synchronously, indicating that the contribution of water- and hydroxyl-related vibrations relative to the Si-O-T main band increased in the 28 d specimens. However, since the broad band at 3000–3800 cm^{-1} simultaneously contains contributions from bound water, adsorbed water, Si-OH, and hydrogen-bonded hydroxyl groups, and the moisture absorption state of the KBr pellet, pellet thickness, and sample dispersion degree all affect the absorption intensity [30–32], these area ratios cannot be directly converted into

bound water content, nor can they be used alone to determine the degree of polycondensation.

It should be emphasized that, within the range of 1300–1500 cm^{-1} , none of the specimens exhibited an independent absorption peak that could be reliably separated from the high-wavenumber side of the Si-O-T main band. Although this region may contain contributions from the asymmetric stretching vibration of carbonate ions [33,34], the available spectra are insufficient for quantifying carbonate content or phase type. Therefore, FTIR was not used in this study to evaluate the carbonation degree of the specimens.

3.4 XPS analysis

The XPS full-spectrum scanning results of the RAGP system at 7 d and 28 d are shown in Figure 5. Characteristic peaks of the main elements O, Si, Al, Na, and C could be identified in the spectra, among which the C 1s peak was attributed to adventitious carbon contamination adsorbed on the sample surface and was used as an internal standard for charge correction (284.8 eV). The presence of the characteristic peaks of O, Si, Al, and Na corresponds to the main components such as SiO_2 , Al_2O_3 , and Na_2O in the precursor glass powder and composite activator, indicating that the surface composition of the reaction products was dominated by silicate (aluminosilicate) gel and unreacted residues. In addition, the overall profiles of the six spectra were similar, indicating that no significant change occurred in the types of elements on the surfaces of specimens with different moduli and ages, and that the differences were mainly reflected in the chemical state distributions in the fine spectra of each element.

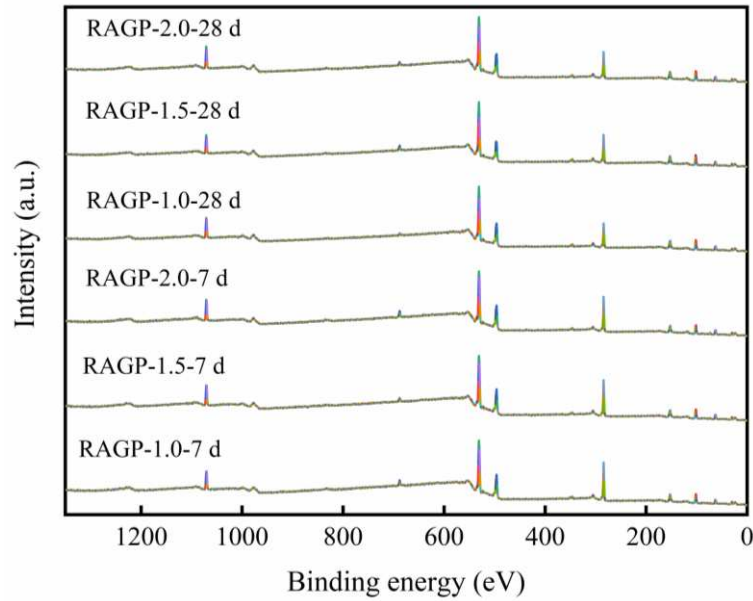


Figure 5 XPS full-spectrum patterns

To further reveal the connectivity state of the gel network, high-resolution O 1s spectra were deconvoluted, and the results are shown in Figure 6. Previous reports have indicated that the O 1s spectrum can be clearly deconvoluted by high-resolution fitting to separate the signals of bridging oxygen (BO, Si-O-Si) and non-bridging oxygen (NBO, Na-O-Si), as there is a distinct difference in their binding energies, and this method has been used to accurately quantify the relative proportions of bridging and non-bridging oxygen [35–37]. Accordingly, in this study, the O 1s spectrum was deconvoluted into three sub-peaks: a non-bridging oxygen peak (NBO) at a lower binding energy, corresponding to Si-O-Na and representing terminal oxygen not participating in network connectivity; a bridging oxygen peak (BO) at an intermediate binding energy, corresponding to Si-O-Si/Si-O-Al and representing oxygen involved in the three-dimensional network connectivity; and a hydroxyl/adsorbed water peak (OH/H₂O) at a higher binding energy, representing unreacted surface hydroxyl groups and adsorbed water. This peak deconvolution method is consistent with previous reports on chemical states such as Si-O-Na and Si-OH in alkali-activated systems [38,39]. The area percentage of bridging oxygen relative to the total

network oxygen (BO%) was used to characterize the degree of polymerization of the gel network, and the proportion of OH/H₂O relative to the total O 1s area was used to characterize the unreacted degree and water-containing state of the system; the results are listed in Table 5.

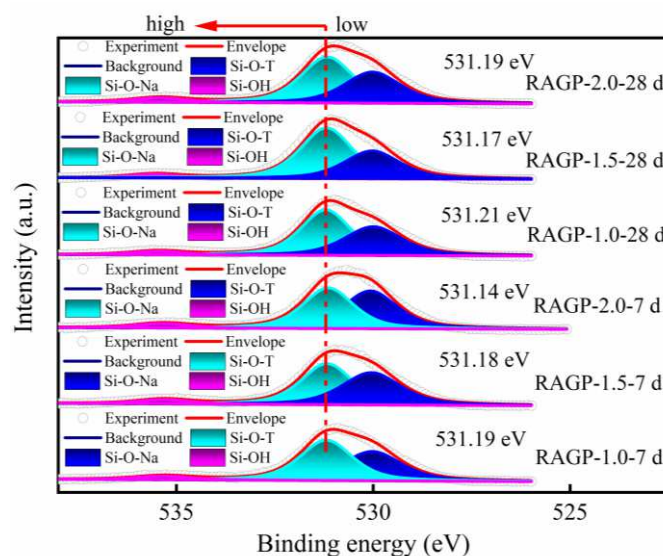


Figure 6 The fitting of the O 1 s line of RAGP

Table 5 O 1s deconvolution area results of the RAGP system

Specimen	Age	NBO area/proportion	BO area/proportion	OH area/proportion
		(%)	(%)	(%)
RAGP-1.0	7 d	44449/44.62	55167/55.38	5663/5.38
RAGP-1.5		53159/47.22	59315/52.78	6591/5.54
RAGP-2.0		56851/49.6	57764/50.4	6990/5.75
RAGP-1.0	28 d	57824/42.15	79373/57.85	6834/4.74
RAGP-1.5		57342/36.75	79564/63.25	7954/4.42
RAGP-2.0		46874/41.88	80662/58.12	5892/5.49

From Table 5, it can be seen that at 7 d, BO% decreased monotonically with increasing modulus, while OH% correspondingly increased monotonically, with a difference in BO% of 4.98% between RAGP-1.0 and RAGP-2.0. Since the dosage of sodium silicate increased with increasing modulus while the glass powder dosage was fixed, the total SiO₂ supply of the system should theoretically increase with increasing modulus; if the 7 d degree of polycondensation were

determined by the amount of silicon source supplied, BO% should have increased with increasing modulus and OH% should have correspondingly decreased, but the measured trend was opposite. This indicates that the reaction degree at 7 d was still governed by the dissolution rate of glass powder rather than by the amount of silicon source supplied. The lower-modulus system had a higher initial alkalinity, which promoted rapid breakage of Si-O-Si bonds in the glass network and rapid dissolution and in-situ polycondensation of reactive silicon-aluminum species; even though the total silicon source supply was relatively low, this allowed a higher bridging oxygen ratio and a lower unreacted hydroxyl/adsorbed water ratio to be simultaneously achieved at an early stage. In the higher-modulus system, insufficient free alkali meant that the dissolution reaction had not yet fully developed, and the excess soluble silicon was neither converted into a bridging oxygen network nor prevented from being accompanied by more unpolycondensed Si-OH and residual adsorbed water.

By 28 d, the BO% of RAGP-1.5 was 5.40% and 5.13% higher than those of RAGP-1.0 and RAGP-2.0, respectively, while the OH% of RAGP-1.5 decreased to the lowest among the three groups; both differences exceeded the maximum inter-group difference at 7 d. This indicates that the mechanism by which the modulus affects the reaction shifted with curing age: as the reaction time was extended, the dissolution of glass powder was no longer the limiting step, and the RAGP system accumulated a sufficient amount of soluble silicon-aluminum species, at which point the silicon-source advantage brought about by an increased modulus began to translate into actual network connectivity. This is similar to the conclusion in existing studies that the degree of gel polymerization during the polycondensation stage depends on a sufficient supply of soluble silicon [40–42].

Comparing the changes from 7 d to 28 d for the same mix proportion, the BO% of RAGP-1.0 increased by 2.47% and OH% decreased by 0.64%; the BO% of RAGP-1.5 increased by 10.47% and OH% decreased by 1.12%; and the BO% of RAGP-2.0 increased by 7.72% and OH% decreased by 0.26%. It can be seen that RAGP-1.0 exhibited both the smallest increase and the smallest decrease, possibly because most of the available silicon source had already been consumed by 7 d, leaving a limited material basis for subsequent polycondensation, and the unpolycondensed Si-OH consequently lacked the driving force for further conversion. Although RAGP-2.0 had an abundant silicon source, its persistently insufficient free alkali constrained continued dissolution, leaving the polycondensation reaction without replenishment, and its decrease in OH% was also relatively low among the three groups. RAGP-1.5 did not excessively consume its silicon source at 7 d and retained sufficient alkalinity to support subsequent dissolution, allowing dissolution and polycondensation to remain synergistic throughout the entire curing period, such that both bridging oxygen generation and hydroxyl consumption proceeded synchronously and fully. This phenomenon is similar to the pattern in alkali-activated systems whereby the reaction rate is jointly controlled by alkalinity and silicon supply [43,44].

In Figure 7, the Al 2p binding energies of the six samples were all within the range of 73.27–73.34 eV, consistent with the binding energy range of tetrahedrally coordinated aluminum (73.2–74.35 eV) and lower than that of octahedrally coordinated aluminum (74.1–75.0 eV) [45], indicating that aluminum in the RAGP system existed consistently in the tetrahedral coordination form. The inter-sample differences were only 0.01–0.07 eV, close to the instrumental resolution limit, indicating that the coordination state of aluminum did not change significantly with modulus or curing age, and that its charge was consistently balanced by Na⁺, stably participating in the

construction of the Si-O-Al network. Therefore, the differences in the degree of polymerization among the different mix proportions mainly originated from the Si network itself, rather than from changes in aluminum coordination.

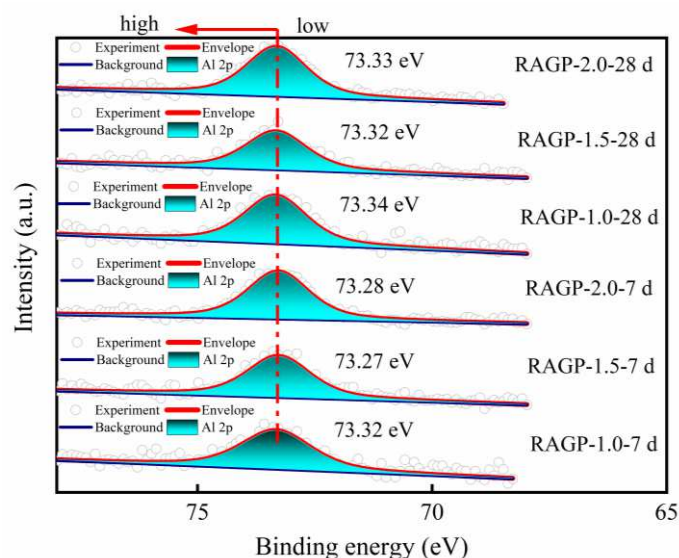


Figure 7 The fitting of the Al 2p line of RAGP

In Figure 8, the Si 2p binding energy ranged from 101.46 to 101.60 eV, with a difference of only 0.14 eV. RAGP-2.0 was higher than the other two groups at both ages, a result that is inconsistent with the O 1s results showing the highest bridging oxygen ratio for RAGP-1.5 at 28 d. This discrepancy may arise because Si 2p reflects the average binding energy of all silicon-containing species on the surface, including the unreacted residual glass powder skeleton. In addition, RAGP-2.0 had relatively little free alkali and incomplete dissolution of glass powder, and the high degree of polymerization of the residual skeleton raised the overall binding energy, but this portion does not represent the degree of polymerization of the gel network itself. Since the inter-group difference in Si 2p was much smaller than the difference in the O 1s bridging oxygen ratio, the assessment of the degree of polymerization was still primarily based on O 1s, with Al 2p illustrating the stability of aluminum coordination and Si 2p illustrating the difference in the

residual amount of glass powder.

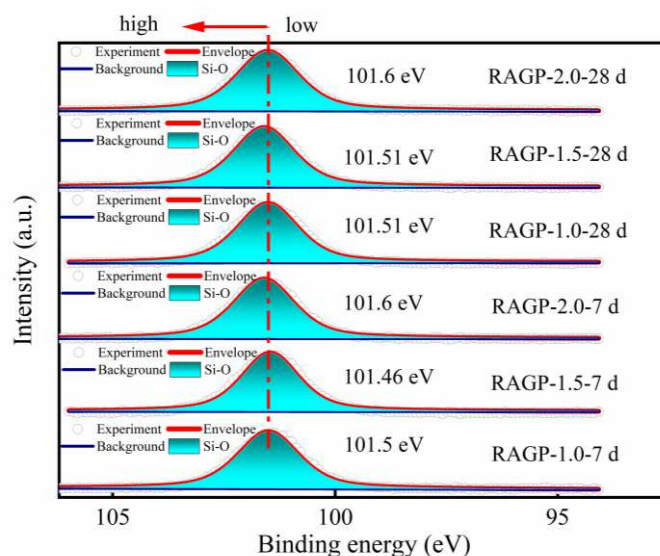


Figure 8 The fitting of the Si 2p line of RAGP

3.5 Structure-property relationship and reaction mechanism

Figure 9 summarizes the structural evolution process of the RAGP system from 7 d to 28 d under different activator compositions. It should be noted that, as the three groups of specimens varied in nominal modulus, the dosages of sodium silicate, NaOH, and added water also changed accordingly; therefore, Figure 10 reflects the effect of the overall activator composition under the mix design conditions of this study, rather than the independent effect of modulus as a single variable.

The XRD results showed that, after alkali activation, the centroid of the amorphous diffuse peak of the hardened specimens shifted toward higher angles by 2.13° – 2.33° relative to the raw glass powder, indicating that the average scattering environment of the original glass network was reconstructed. The centroid differences among specimens with different mix proportions and curing ages did not exceed 0.19° , and no resolvable newly formed crystalline phase was observed, indicating that the later-stage structural evolution mainly occurred within the amorphous network.

The FTIR results showed that all specimens exhibited a broad Si-O-T absorption band at 973–981 cm^{-1} , also indicating the presence of an amorphous network with silicon-oxygen tetrahedra as the basic unit in the hardened bodies. Since this absorption band simultaneously contains contributions from both the reaction products and the residual glass phase, the connectivity state of the gel network was mainly assessed based on the relative proportions of bridging and non-bridging oxygen from O 1s.

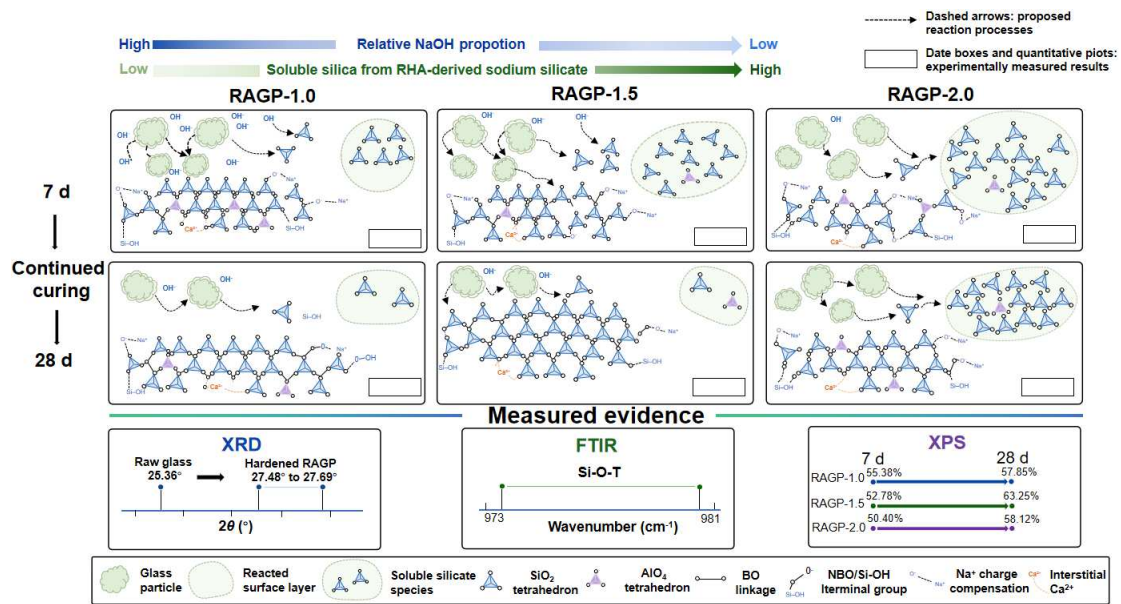


Figure 9 The structural evolution process of the RAGP

At 7 d, both the compressive strength and BO ratio decreased with increasing nominal modulus. The strength and BO ratio of RAGP-1.0 were 31.4 MPa and 55.38%, respectively, while those of RAGP-2.0 were 29.6 MPa and 50.40%. Under the condition that the total Na_2O equivalent was approximately constant, the mix with a lower nominal modulus had a higher proportion of NaOH, which may have provided stronger conditions for early-age depolymerization of the glass network, allowing the dissolved silicon-aluminum species to participate more rapidly in the construction of the Si-O-T network. As the proportion of sodium silicate increased, the exogenous soluble silicon in the system increased, but the proportion of NaOH decreased, which may have limited the

early-age dissolution of glass powder; consequently, RAGP-2.0 exhibited a lower bridging oxygen ratio and compressive strength at 7 d.

As the curing age increased from 7 d to 28 d, the strength and BO ratio of all three groups of specimens increased, but to different extents. The BO ratio of RAGP-1.0 increased by only 2.47 percentage points, while its strength increased by 18.2%, indicating that its early-age reaction proceeded rapidly, but the subsequent growth of network connectivity was limited. The BO ratio of RAGP-2.0 increased by 7.72 percentage points, with a strength increase of 28.0%, indicating that the high exogenous silicon supply promoted partial network reconstruction at a later stage, although the lower NaOH proportion may still have constrained the continued dissolution of glass powder. In comparison, the BO ratio of RAGP-1.5 increased by 10.47 percentage points, with a strength increase of 31.1%, reaching 63.25% and 39.6 MPa at 28 d, respectively. This result indicates that the intermediate activator composition simultaneously retained the alkaline conditions supporting continued glass dissolution and the soluble silicon supply for subsequent polycondensation, allowing the bridging oxygen network to continue growing between 7 d and 28 d.

Therefore, the strength development of the RAGP system can be understood as two interconnected processes: the early-age performance is mainly related to the depolymerization of glass powder and the formation of the initial network, for which a higher NaOH proportion is favorable; the later-age performance depends on whether continued dissolution and silicate polycondensation can proceed synchronously. An insufficient exogenous soluble silicon supply limits later-age network growth, while an excessively low NaOH proportion also reduces the capacity of glass powder to continuously supply reactive species. Within the mix design range of

this study, RAGP-1.5 achieved a relatively appropriate match between the two, attaining the highest bridging oxygen ratio and compressive strength at 28 d.

4 Conclusions

In this study, a composite activator composed of rice husk ash-based sodium silicate and NaOH was used, with glass powder as the sole precursor, to analyze the strength development and silicon-oxygen network changes of RAGP under three activator compositions. The following conclusions were obtained:

(1) The activator composition altered the strength development pathway of RAGP. The 7 d strength decreased with increasing nominal modulus, while at 28 d, RAGP-1.5 achieved the highest compressive strength of 39.6 MPa, with the highest-strength group shifting from RAGP-1.0 to RAGP-1.5.

(2) Alkali activation caused the centroid of the amorphous diffuse peak to shift toward higher angles by 2.13° – 2.33° relative to the raw glass powder, while the centroid difference among hardened specimens with different mix proportions and curing ages did not exceed 0.19° , and no resolvable newly formed crystalline phase appeared. The structural evolution of RAGP mainly occurred within the amorphous network.

(3) All specimens exhibited a broad Si-O-T absorption band at 973 – 981 cm^{-1} , but the peak position did not vary monotonically with nominal modulus or curing age. FTIR can be used to identify the presence of the silicon-oxygen network and its average vibrational environment, but cannot be used alone to determine the degree of connectivity of the gel network.

(4) The bridging oxygen ratio exhibited a consistent ranking relationship with the compressive strength at the same curing age. The bridging oxygen ratio of RAGP-1.5 increased from 52.78% at

7 d to 63.25% at 28 d, an increment of 10.47 percentage points, corresponding to the largest strength increase; within the mix design range of this study, the intermediate activator composition was more favorable for the coupling between continued glass dissolution and silicate polycondensation.

Acknowledgement

This research was supported by Zhejiang Provincial Natural Science Foundation of China under Grant No. LQN26E080040 and the Competitive Project of Quzhou Science and Technology Bureau Grant No. 2025K200. The authors gratefully acknowledge their financial support.

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