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# INFLUENCE OF ORGANIC ALKALI AND CELLULOSE ON THE PROPERTIES OF FLY ASH-BASED GEOPOLYMER

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## ABSTRACT

This study investigates the influence of organic alkali and cellulose on the properties of fly ash-based geopolymer materials. Geopolymers have shown promising applications in various fields, but their performance is significantly affected by the type and dosage of additives. The influence of organic alkali and cellulose on the properties of fly ash-based geopolymer materials has been extensively explored. To reveal the mechanism of these additives in the formation and performance regulation of geopolymers, this study used various characterization methods such as compressive strength testing, XRD analysis, FTIR analysis, and SEM observation. The experimental results show that the dosage of organic alkali has a certain effect on the strength of geopolymer, but the impact is relatively small. When only adding organic alkali, a dosage of twice only increases the strength from 5MPa to 6.3MPa. The addition of sodium silicate significantly improved the compressive strength of geopolymers. At a 5% organic alkali content, the addition of 5.5%, 11.0%, and 16.5% sodium silicate increased the compressive strength by 60%, 186%, and 396%, respectively. At a 10% organic alkali content, increasing by 47%, 256%, and 306% respectively, sodium silicate can promote the formation and expansion of the aluminosilicate network structure. The addition of cellulose exhibits a dual effect of first strengthening and then weakening, and its distribution and dosage have a significant impact on the properties of geopolymer. The cellulose dosages of 0.33%, 0.66%, and 0.1% increase by 0.9MPa, 2.4MPa, and 0.1MPa compared to those without addition. Through comprehensive analysis, this study reveals the correlation between organic alkali and cellulose in the microscopic characteristics and macroscopic performance of geopolymers, providing an experimental basis and theoretical guidance for the preparation of high-performance geopolymer materials.

**Keywords:**
*Organic alkali, Cellulose, Fly ash based geopolymer, Compressive strength, Microscopic characteristics*

## INTRODUCTION

Concrete, as the main material of most buildings today, has a huge usage. According to statistics, the world's cement production reached 4.4 billion tons in 2021(Garside, 2022). The production of cement requires a large amount of nonrenewable mineral resources such as limestone and clay, as well as a large amount of energy while generating a large amount of CO2, which has adverse effects on the environment. Fly ash geopolymer is a high-performance cementitious material generated from calcium-free or low-calcium aluminosilicate minerals under the action of activators. Its production process is energy-saving and environmentally friendly, and the sources of production materials are extensive (Miasin & Woon, 2020; Salleh & Roslan, 2015; Yang et al., 2023). Fly ash geopolymer has mechanical properties comparable to ordinary concrete, and even exhibits better performance in some aspects, such as high-temperature resistance, corrosion resistance, etc., which has attracted widespread attention from researchers. However, the brittleness of geopolymers greatly limits their development and application (Harmal et al., 2023). Therefore, reducing the brittleness of geopolymers and improving their toughness are important ways to develop high-performance geopolymers and expand their application scope. In addition, as the use of geopolymers becomes increasingly widespread, their consumption is also increasing, and their prices have significantly increased. Reducing the cost of geopolymers has become a focus of researchers' attention.

At present, researchers have conducted extensive research on improving the mechanical properties of geopolymers, consisting of adjusting the active components and content of raw materials

(Sitarz et al., 2022; Xu et al., 2021), selecting appropriate activators and activator ratios (Aiken et al., 2018; Xie et al., 2023), appropriate curing conditions (Sajan et al., 2021; Yılmaz et al., 2023), and adding fibers(Deng et al., 2023; Ganesh & Muthukannan, 2021; Punurai et al., 2018). Chindaprasirt et al. (2007) used Na2SiO3 and NaOH as activators to polymerize with fly ash. The resulting geopolymer sample was cured at 75 ℃ and had a compressive strength of up to 50 MPa after 7 days. Hervé et al. (2017; 2017) studied the effect of the concentration of the $H_3PO_4$ solution on the properties of geopolymers and found that as the concentration increased, the compressive strength of the polymer increased. Compared with alkali-based geopolymers, phosphate-based geopolymers had higher compressive strength. When the $H_3PO_4$ concentration was 10 mol/L, the highest compressive strength reached 93.8 MPa, while the compressive strength of alkali-based geopolymers was only 63.8 MPa. Siciliano et al. (2024) investigated the mechanical and microstructural evolution of kaolin-based geopolymers under temperature and pressure curing conditions. The research results showed that a curing temperature of 90 °C was conducive to the formation of regular nanopores, while higher temperatures helped to increase porosity. On the contrary, maintenance pressure can promote or inhibit material recombination, with a pressure of 20 MPa promoting this process and pressures above 40 MPa preventing it.

The above research focuses more on improving the compressive performance of geopolymers. Regarding how to reduce the brittleness of geopolymers, researchers mainly use the method of adding fibers to reduce their brittleness (Johari et al., 2023; Kong & Kamaruzaman, 2023). The disadvantage of the high brittleness of geopolymers greatly limits their application. Adding fibers as a common means to improve the brittleness of geopolymers has been widely studied and applied. Common fibers include nonplant fibers and plant fibers, among which nonplant fibers mainly include inorganic fibers and organic polymer fibers. Khalil et al. (2018) prepared lightweight geopolymer concrete containing steel fibers. The study showed that the addition of steel fibers increased the splitting and bending tensile strength of lightweight geopolymer concrete. When the steel fiber content reached 0.5% of the total volume, its bending strength at 28 days was 1.063 times that of specimens without steel fibers. Sukotasukkul et al. (2018) studied the bending performance of geopolymers under mixed steel fiber and polypropylene fiber conditions. The research results showed that the mixing of steel fibers can improve the bending strength, toughness, and residual strength of polypropylene fiber-reinforced geopolymers to varying degrees. Adding both inorganic fibers and organic polymer fibers can effectively improve the brittleness and mechanical properties of geopolymers, but inorganic fibers have problems such as high cost, difficult degradation, and easy environmental pollution. Organic polymer fibers have disadvantages such as high price and an unfriendly environment (Korniejenko & Łach, 2020; Shaikh, 2020).

Plant fibers in natural fibers have garnered considerable interest among researchers, stemming from their large reserves, renewability, low cost, and environmental friendliness. There have been many reports on adding plant fibers to enhance the mechanical performance of geopolymers during the preparation process. For example, researchers have added fibers such as cotton (Kozub et al., 2023), coir (Ayeni et al., 2022; Narasimha Swamy et al., 2017), pineapple fiber (Linhares et al., 2023; Zulfiati et al., 2019), sugarcane bagasse (Aran et al., 2021; Srinivas et al., 2021), etc. to geopolymers, and the reinforcement effect of geopolymers is excellent. Alomayri et al. (2013) systematically investigated the influence of cotton fiber orientation and content on the mechanical properties of fly ash-based geopolymers. The results indicate that when the orientation of cotton fabric is aligned horizontally with the load, it has greater load-bearing capacity and deformation resistance than when it is aligned vertically. Alshaaer et al. (2017) used 10% loofah fiber reinforced geopolymer to study the physical characteristics and mechanical properties of geopolymer composites. The compressive strength and flexural strength of geopolymer composite materials were found to have increased by 138% and 318%, respectively, with good durability. Huang et al. (2021) compared the effects of untreated and alkali-treated straw on the mechanical properties of slag-based geopolymer composites, and the results showed that the former had significantly lower flexural strength than the latter. Research has found that the compatibility between plant fibers and geopolymers, as well as the particle size and aspect ratio of plant fibers, can be regulated through the selection and adjustment of plant fiber materials to control geopolymer properties. However, this requires a lot of research work and the enhancement effect is limited. To fully utilize plant fibers, further research can be conducted on the mechanical properties of geopolymers reinforced by plant fibers, especially to improve their toughness. By improving the interaction between plant fibers and geopolymers, geopolymer properties can be improved. Sanfilippo et al.(2024) used alkali pretreatment to damage the wax layer on the surface of jute fibers, enhancing their hydrophilicity and increasing the interaction force between jute fibers and geopolymers. It found that the bending strength of jute fiber geopolymers increased by 53%. Ye et al.(2018) studied the effect of natural cellulose on metakaolin-based geopolymer. It found that the presence of an appropriate amount of cellulose had an internal curing effect on the geopolymer-based material, improving its microstructure and enhancing its flexural and compressive strength. However, excessive cellulose increased the porosity of the material and reduced its mechanical strength due to its increased water absorption rate.

The preparation of traditional geopolymers usually relies on inorganic bases (such as NaOH, and KOH) as activators. These inorganic bases not only pose safety hazards during production, storage, and transportation, such as strong corrosiveness, flammability, and explosiveness, but their high cost also limits the large-scale application of geopolymer materials. Exploring safer, more economical, and environmentally friendly activators has become the key to promoting the development of fly ash-based geopolymers. Organic bases, as a class of compounds with unique chemical properties and wide application potential, have gradually entered the field of research. The molecular structures of organic bases are diverse, and different organic bases have different functional groups and reaction activities, which provides the possibility for precise regulation of geopolymer properties. By selecting appropriate types and amounts of organic bases, precise control of geopolymer reaction kinetics, microstructure, and macroscopic properties can be achieved, thereby meeting the special requirements of different engineering fields for material properties. Lu et al. (Lu et al., 2022) used organic strong alkali sodium tert butoxide as an activator to prepare fly ash geopolymers and characterized the microstructure of fly ash geopolymers through various methods. The results indicate that C-OH in sodium tert butoxide can form C-O-Si bonds after hydrolysis with Si-OH in sodium tert butoxide and geopolymer, resulting in an intricately interlaced 3D network structure.

Based on this, this study intends to investigate the effects of organic alkali and cellulose on the properties of fly ash-based geopolymers. By systematically exploring the effects of different types and amounts of organic alkali and cellulose on the preparation process, microstructure, physical and mechanical properties, and durability of geopolymer, the aim is to reveal their mechanisms and laws of action and provide scientific basis and technical support for the efficient utilization of fly ash and the green development of geopolymer materials. For this purpose, we investigated how the concentrations of Si/Al, organic bases, and cellulose affect the geological polymerization pore structure, hydration products, and strength. The effects of organic alkali and cellulose on the chemical bonding, mineralogy, and mechanics of kaolin-based geopolymers were determined by FTIR, XRD, and SEM, respectively.

## EXPERIMENTAL

### *1. MATERIALS*

The activators are reagent-grade sodium tert butoxide (STB), sodium silicate (SS), and a mixture of STB and SS. The modulus of SS is 2.0, and the cellulose is purchased from Aladdin Reagent Co., Ltd. The fly ash is taken from the Laibin thermal power station in Guangxi, China. Si and Al are the main elemental components in the fly ash, as shown in Table 1. The total amount of oxides SiO2 and $Al_2O_3$ is close to 90% (wt), and the CaO content is 2.61%. It is a low calcium F-type fly ash with a particle size of about 14 μm.

Table 1: Main chemical composition (wt/%) of fly ash

| Material | $SiO_2$ | $Al_2O_3$ | CaO | MgO | $SO_3$ | $Fe_2O_3$ | $K_2O$ |
|---|---|---|---|---|---|---|---|
| Fly ash | 58.80 | 30.76 | 2.61 | 0.64 | 0.33 | 1.87 | 1.03 |

### *2. SAMPLE PREPARATION*

Firstly, through literature analysis and comparison, a composite design experiment was conducted on fly ash, STB, and SS. Set the dosage of STB to 5%~10% of the fly ash dosage, and the dosage of sodium silicate to 0~16.48% of the alkali activator dosage, while maintaining a constant water/binder ratio of 0.33, to prepare geopolymer by compounding with fly ash. The specific mix proportion is shown in Table 2.

According to the experimental requirements, water, STB, and SS are used to prepare the corresponding alkali activator. Then, a mortar mixer is used to mix it with a certain amount of fly ash at 1200r/min for 15 minutes to make the whole mixture uniform. The mixed slurry is placed on a vibration table for 5 minutes to remove the bubbles generated by high-speed mixing. Then, it is poured into a 40 × 40 × 40mm mold and covered with plastic wrap. After curing at 75 °C for 1 day, it is demolded and cured at room temperature and pressure for 28 days. The average of at least 3 parallel experimental results with the same physical and chemical properties is taken as the final data. Analyze the effect of adding different amounts of STB and SS on the compressive strength of geopolymers.

Then the optimal dosage of STB and SS is based on the results of compressive strength, and they are compounded into cellulose, STB, SS, and fly ash in different proportions to prepare fly ash-based geopolymers, with a cellulose dosage of 1-3g.

Table 2: Mix proportions for geopolymer.

| Samples | Si/Al | $H_2O$ | Fly ash | SS | STB | Cellulose |
|---|---|---|---|---|---|---|
| SA6-1 | 1.62 | 100 | 300 | 0.00 | 15 | - |
| SA7-1 | 1.72 | 105 | 300 | 16.48 | 15 | - |
| SA8-1 | 1.82 | 110 | 300 | 32.96 | 15 | - |
| SA9-1 | 1.92 | 115 | 300 | 49.45 | 15 | - |
| SA6-2 | 1.62 | 100 | 300 | 0.00 | 30 | - |
| SA7-2 | 1.72 | 105 | 300 | 16.48 | 30 | - |
| SA8-2 | 1.82 | 110 | 300 | 32.96 | 30 | - |
| SA9-2 | 1.92 | 115 | 300 | 49.45 | 30 | - |
| SA9-1C1 | 1.92 | 115 | 300 | 49.45 | 15 | 1 |
| SA9-1C2 | 1.92 | 115 | 300 | 49.45 | 15 | 2 |
| SA9-1C3 | 1.92 | 115 | 300 | 49.45 | 15 | 3 |

## *3. MEASUREMENT METHODS AND MICROSTRUCTURE CHARACTERIZATION*

The geopolymer slurry is mixed using a mortar mixer, and after curing in an oven, it is subjected to unconfined compressive strength testing on a cement automatic constant stress testing machine. After the strength test, the sample is crushed and ground to a powder that passes through a 200 mesh sieve. Conduct microscopic characteristic testing on the powder.

$2\theta$ is between 10 and 70°, an X-ray diffractometer (Rigaku, SmartLab SE, Japan) was used to examine the crystal structure of fly ash geopolymer in increments of $0.02°^{-1}$. The scanning speed was 5 seconds/step. The fly ash geopolymer was characterized using FTIR (Thermo Fisher Scientific Nicolet iS5, USA). The microstructure of fly ash geopolymer was observed by SEM (ZEISS, Sigma 300, Germany).

## RESULTS AND DISCUSSION

### *1. COMPRESSIVE STRENGTH*

The 28-day compressive strength results of geopolymer samples prepared by compounding organic alkali activators with fly ash in different proportions are shown in Figure 1. The compressive strength increases with the increase of the silicon-aluminum ratio under two different dosages of STB. When only adding STB, 5% and 10% alkali influence strength is somewhat increased, but not significant. This should be because the provided alkalinity can indirectly promote the polymerization reaction of geopolymer to form compressive strength to a certain extent, and fly ash itself does not have enough silicate to provide a geopolymer reaction. After increasing the dosage of SS, the strength gradually improved. When the mixed activator was 10% STB and 16.5% SS, the peak compressive strength of the geopolymer could reach 25.6MPa.

Considering that the addition of 16.5% sodium silicate has little effect on the strength, 5%, and 10% STB were ultimately chosen as activators for cellulose-based geopolymers to save costs in application. After adding cellulose, the strength is enhanced to a certain extent, but not significantly. After adding cellulose to the geopolymer, the compressive strength exhibits an initial upward trend, followed by a subsequent decline. From existing literature, on the one hand, a moderate amount of cellulose has an internal curing effect that can improve the internal structure of geopolymers, thereby increasing their strength; On the other hand, the strong polarity of cellulose and the high-density hydrogen bonds between molecules and their internal structures result in a diminished reactivity within the matrix (Rana et al., 2021), and the interfacial tension between cellulose and matrix reduces the density of composite materials(Azlina Ramlee et al., 2021). An excessive quantity may induce detrimental alterations to the internal micropores, such as increased porosity or fiber aggregation, thereby weakening compressive strength.

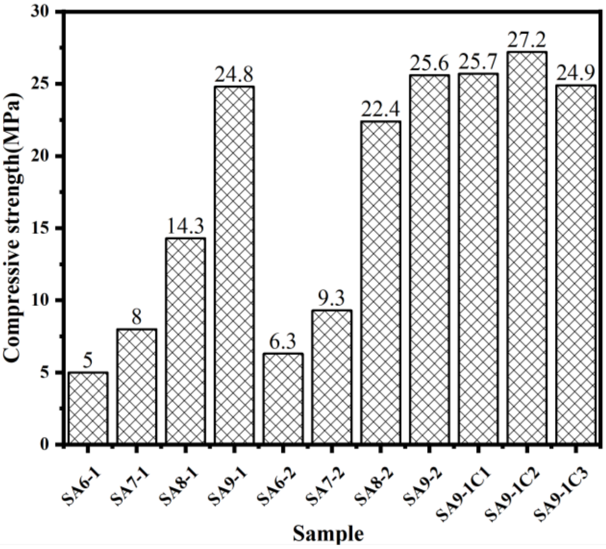

Figure 1: Compressive strength of geopolymers with different mix proportions at 28 days

## *2. XRD ANALYSIS*

To further investigate the effects of STB, SS, and cellulose on the microstructure of geopolymers, we conducted an XRD analysis. The XRD pattern reveals the changes in mineral phase composition and crystallinity in geopolymers, providing important evidence for understanding the changes in macroscopic properties. Figure 2 shows the XRD pattern, and the characteristic peak intensities of each sample phase are relatively close. Compared with the existing research on fly ash XRD, the characteristic peak intensity between 20° and 40° in the fly ash XRD spectrum is significantly weakened, indicating that the glass phase of fly ash is destroyed due to activation reactions (Kaze et al., 2018; Lemougna et al., 2014).

At different STB dosages, there were no significant signs of new phase formation or disappearance of the original phase in the XRD pattern of the geopolymer, indicating that STB mainly participates in the formation process of the geopolymer through amorphous or low crystallinity forms, and has little effect on the overall crystallinity. The observed phenomenon in the compressive strength test aligns with the notion that a rise in STB content results in a marginal enhancement of strength, without significant augmentation.

The primary reaction product of geopolymers, aluminum silicate (N-A-S-H), exhibits feeble diffraction peaks. The primary factor lies in the presence of silicon aluminate (N-A-S-H) primarily in an amorphous state within the reaction products of fly ash geopolymers.(Palomo et al., 2007; Phoo-ngernkham et al., 2014). With the increase of sodium silicate content, more obvious characteristic peaks of aluminosilicate appeared in the XRD pattern, and the peak intensity gradually increased, indicating that the increase of sodium silicate promoted the formation of aluminosilicate network structure and the improvement of crystallinity. The small peaks of SA8-1 and SA9-1 between 30° and 40° are more pronounced than others, indicating that there are more products of aluminosilicate (N-A-S-H).

After adding cellulose, the XRD pattern of the geopolymer sample doped with cellulose did not show significant changes at low cellulose content, and the XRD was almost consistent with SA9-1, indicating that it did not affect the types of geopolymer reaction products and no new substances were generated.

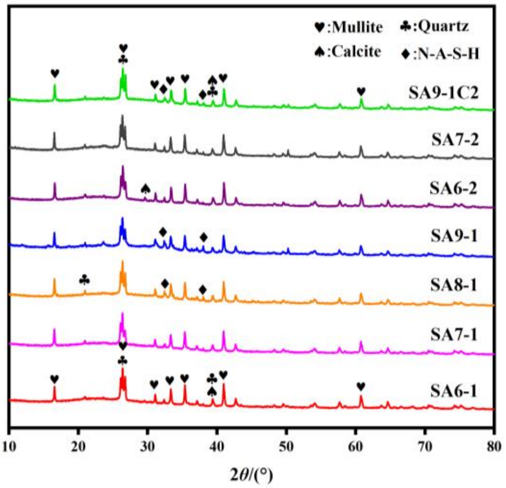

Figure 2: XRD patterns of geopolymers with different mix proportions

## *4. IR ANALYSIS*

The infrared spectra of fly ash-based geopolymers, ranging from 4000 to 400 cm$^{-1}$ are presented in Figure 3. The situation of different groups of polymers is similar. Separately, the vicinity of 3446 cm$^{-1}$ displays a broad absorption peak, indicative of the stretching vibration associated with $H_2O$ molecules., and 1645 cm$^{-1}$ is the -OH bending vibration peak of water in geopolymer. The signal appearing at 1488 cm$^{-1}$ is due to the presence of carbonate groups in the geopolymer. The broad absorption peak at 1050 cm$^{-1}$ is the asymmetric vibration peak of the Si-O-T bond (T can be Si or Al), which is widely accepted for studying the changes in the reaction products of alkaline aluminosilicate gel(Liu et al., 2020). The signal appearing at 460~480 cm$^{-1}$ corresponds to the stretching vibration of Si-O-Si, which is very sharp.

With the increase in STB content, no new characteristic peaks appeared in the FTIR spectrum, but the intensity and position of some peaks changed slightly. These changes may reflect the interaction between STB and geopolymer matrices, such as promoting the formation of geopolymer structures or regulating their surface properties through acid-base reactions. However, these changes did not directly lead to a significant increase in compressive strength, which may be related to the specific mechanism of action of organic bases in geopolymers.

After the addition of SS, the infrared absorption peak shifted around the wavenumber 1080 cm$^{-1}$, indicating that the active substance in the fly ash reacted with the STB. It can be observed that SA9-1 has the highest offset, indicating that with the increase of silicon content, the geopolymer reaction was complete, leading to an increase in Si-O-T bond aggregation. This also explains why the fly ash geopolymer synthesized with 5% STB and 16.5% SS as activators has the highest compressive strength.

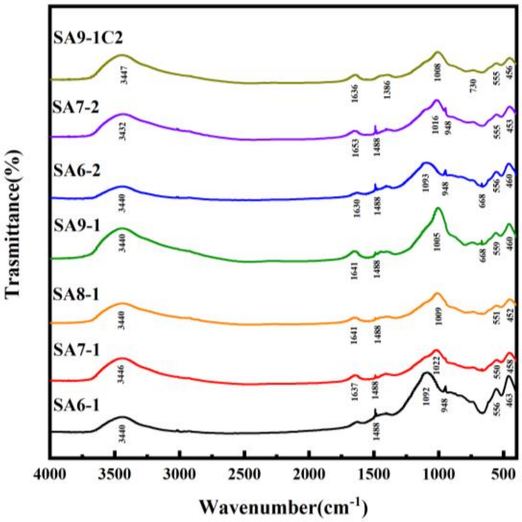

Figure 3: FTIR analysis of polymers with different mix proportions

## *5. MICROSTRUCTURE ANALYSIS*

To visually observe the microscopic appearance of the geopolymer and the effects of STB, SS, and cellulose on its structure, we conducted SEM analysis. SEM images display the microscopic appearance and pore structure of geopolymers, providing an important microscopic perspective for understanding their macroscopic properties. The sample was magnified 10000 times for observation. Figure 4 presents the outcomes of the SEM analysis conducted on the samples. We found many unreacted fly ash glass beads in SA6-1 and SA7-1. After doubling the alkali content, SA6-1 and SA7-1 glass beads were dissolved and reacted in large quantities. There are obvious cracks and pores in the SA8-1 sample. When the SS content is increased to 16.5%, the number of cracks decreases significantly and the slurry matrix becomes denser. When cellulose is added, the basic cracks and pores in the net slurry matrix are greatly reduced, and the density is higher than that of the SA9-1 sample. This fully indicates that when the amount of cellulose is reasonable, the internal curing effect is beneficial for its density and compressive strength. Cellulose can also serve as a nanofiller in the geopolymer matrix, increasing its density and enhancing its mechanical properties.

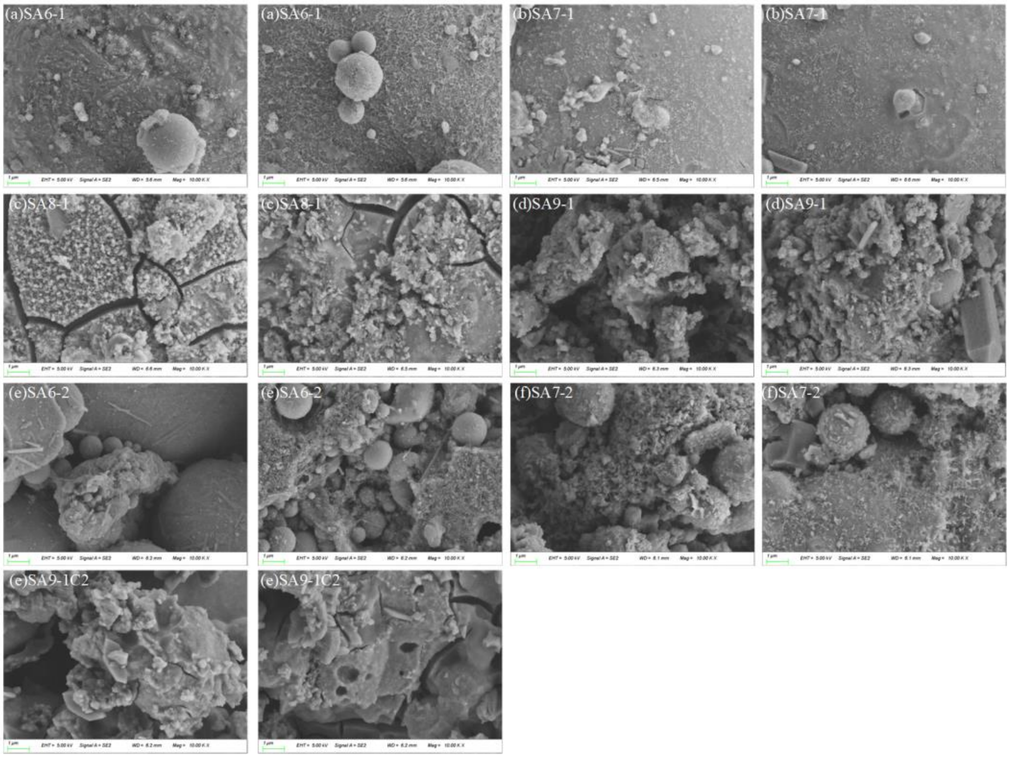

Figure 4: SEM images of geopolymers with different mix proportions

## CONCLUSION

This study systematically probed into the influence of STB, SS, and cellulose on the characteristics of geopolymers. Through various characterization methods such as compressive strength tests, XRD analysis, FTIR spectroscopy, and SEM observation, the different mechanisms of these additives in the formation and performance regulation of geopolymers were revealed.

Due to the low quality and low activity of the fly ash used, the effectiveness of STB as a single activator is limited. Therefore, the selection and dosage of organic alkali need to be further optimized in the preparation of high-performance geopolymers. The amount of SS has a notable influence on the compressive strength exhibited by geopolymers, and its increase promotes the formation and expansion of the aluminosilicate network structure, thereby significantly enhancing the strength of geopolymers. When the contents of STB and SS are 10% and 16.5%, respectively, the strength of the geopolymer reaches its maximum at 28 days. However, compared to the contents of 5% and 16.5% STB and SS, the increase is not significant. In practical applications, the content of STB can be adjusted appropriately to save costs. The addition of cellulose did not affect the types of geopolymer reaction products. At low dosages, cellulose enhances the compressive strength of geopolymers through physical reinforcement; However, at high dosages, negative effects such as fiber agglomeration lead to a decrease in strength. Therefore, it is crucial to reasonably control the dosage and dispersion state of cellulose to optimize the properties of geopolymers.

The contribution of this research is to reveal the effect mechanism of different additives on the properties of geopolymer and provide an experimental basis and theoretical guidance for the preparation of high-performance geopolymer. However, this research also has certain limitations, such as a limited selection range of additive types and dosages, and incomplete optimization of preparation process conditions. Future research can further explore more types of additives, optimize preparation process conditions, and conduct in-depth studies on the interaction mechanism between additives and geopolymer matrices to promote the application of geopolymer in a wider variety of fields.

## AUTHOR BIOGRAPHY

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