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## Effect of Na<sup>+</sup> on hydration degree of alkali activated metakaolin polymer

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**Abstract.** In recent years, geopolymeric gel materials have become a hot research issue due to their good mechanical properties, durability and excellent chemical stability. In this paper, Na<sup>+</sup>-metakaolin soil polymer was prepared by activating metakaolin with a combination of NaOH and sodium silicate. Taking Na<sup>+</sup> influence on the hydration degree of alkali-activated metakaolin soil polymer as the goal, we selected Na<sub>2</sub>O equivalent and the modulus of the alkali activator, two most important factors for Na<sup>+</sup> content in the system, as the object of our studies. The effect of Na<sub>2</sub>O equivalent and modulus on the degree of hydration of geopolymers was analyzed by testing the compressive strength of metakaolin land polymers and microscopic SEM, FT-IR and XRD. The results show that the optimal activation environment for metakaolin is 18 % Na<sub>2</sub>O equivalent and 1.5 modulus. When the Na<sub>2</sub>O equivalent is less than 18 %, as the Na<sub>2</sub>O equivalent increases, the degree of hydration of the metakaolin land polymer deepens. When it is greater than 18 %, with the Na<sub>2</sub>O equivalent, the first stage of the geopolymer hydration reaction is suppressed and the degree of kaolin hydration is partially weakened. When the modulus is less than 1.5, as the modulus increases, the rate of the first stage of geopolymerization is accelerated, and the degree of hydration is deepened. When the modulus is greater than 2.0, as the modulus increases, the geopolymerization hydration reaction is suppressed during the second and third stages, the formation rate of geopolymeric gel is slowed down and the degree of hydration is weakened.

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### 1. Introduction

In the past few decades, geopolymer cementitious materials prepared by alkali activated aluminosilicate materials have become a hot research area. Compared with ordinary Portland cement, geopolymer cementitious materials have the same excellent compressive strength and excellent durability, high temperature resistance, shrinkage and chemical stability. At the same time, due to their low emission and low energy consumption preparation technology, geopolymers are considered the most promising building material to replace OPC in the 21<sup>st</sup> century at the world market [1].

Kaolin in the aluminosilicate precursors as the most pure natural ingredient has attracted researchers' attention. Alkaline activated solution plays an important role in the geopolymer reaction or hydration degree of metakaolin geopolymer, which greatly affects its mechanical properties. In recent years, some researchers carried out experimental observation by isothermal calorimetry (ICC) and

thermogravimetric analysis (TGA). The effect of alkaline activator (type, modulus and ionic concentration) on the formation process of geopolymer was revealed gradually. The results showed that alkaline sodium silicate solution had good activation effect on metakaolin. The content of  $\text{Na}^+$  and  $\text{Si}^{4+}$  in the activation system played an important role in the degree of hydration and mechanical strength of geopolymers [2–5].

Researchers have basically reached a consensus on the formation process of geopolymers in metakaolin, which can be divided into three stages: 1) dissolution and reconstruction of aluminosilicate in metakaolin, 2) the regenerated silicon tetrahedron and aluminum oxide tetrahedron are condensed to form small geopolymer gels, 3) the small aggregates of small gels are continuously aggregated to form highly polymerized geopolymers [6–9].

J.P. Gevaudan's studies have shown that alkali content affects the amount of silica used in geopolymer water and the process, determines the type and structure of final polymer formation, and affects the overall properties of geopolymers, such as permeability and porosity [10]. They believed that the increase of pH value promoted the dissolution of aluminosilicate in the first stage, and  $\text{Na}^+$  played an important role in the second stage. At the same time, the higher the concentration of the activator, the higher the rate of condensation of tetrahedron and the higher the order of the structure. Based on this study, clear relationships were found between the reaction processes and chemical deformations. Furthermore, a conceptual model of the reaction processes and corresponding chemical deformation is proposed based on experimental results and theoretical calculations [11]. The effect of alkaline cation on the compressive strength of metakaolin polymer was studied. The results showed that the compressive strength of metakaolin polymer was highly dependent on the metal cation of the activator.  $\text{Na}^+$  polymer had higher compressive strength than the  $\text{K}^+$  polymer at the same Si/Al ratio and alkali metal ion concentration with higher concentration of  $\text{Na}^+$  observed; higher compressive strength of the geopolymer is formed [10, 11].

In this paper, we consider the influence of  $\text{Na}^+$  on the hydration degree of alkali activated metakaolin geopolymer, selecting two factors controlling the content of  $\text{Na}^+$  in the geopolymer system as the object. The factors were the modulus of basic sodium silicate and the  $\text{Na}_2\text{O}$  equivalent. In addition, we analyzed the role of  $\text{Na}^+$  in the three stages of the geopolymerization and the effect on the overall hydration degree.

## 2. Methods

### 2.1. Raw materials

This experiment uses metakaolin produced by ASUS mineral products company of Lingshou, China as raw material for preparing metakaolin polymer. Its chemical composition is shown in Table 1. The activator is composed of NaOH and sodium silicate composite activator, of which sodium silicate is industrial grade sodium silicate produced by Harbin Qiang Li sodium silicate factory (50 Bo Mei degree). NaOH provides analytical pure sodium hydroxide (NaOH, purity 96 %) for China National Pharmaceutical Group Chemical Reagent Co., Ltd.

**Table 1 Chemical composition of metakaolin (weight%).**

$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{TiO}_2$	$\text{Fe}_2\text{O}_3$	MgO	GaO	$\text{Na}_2\text{O}$	$\text{K}_2\text{O}$
52	45	1.5	0.5	0.5	0.2	0.2	0.1

### 2.2. Preparation of metakaolin polymer

In this paper, the preparation method of the metakaolin polymer is made of paste, and its preparation method is strictly referenced to the national standard cement mortar strength testing method (IOS method). The local test procedure has been adjusted because of the particularity of the metakaolin polymer paste.

1. Solid analytical pure sodium hydroxide was added the sodium silicate to produce a solution complying with the modulus requirements of of the test. In this process, the problem of exothermic heat will occur, so after adding sodium hydroxide to stir and dissolve it, we need to seal it with plastic film and keep it for 24 hours.

2. The water consumption (deionized water) was added to the quantitative sodium silicate in the experiment, so that it can be mixed evenly and placed at normal temperature (not placed in the control group).

3. The metakaolin solution and the solution in second step was added into the mortar mixer (planetary cement mortar mixer). The mix process took 240 seconds, and then the mixture was stirred slowly for 60 s, and quickly for 180 s.

4. The colloid obtained in the third step was supplied into the mortar test mold of 40 cm×40 cm×160 cm, because the geopolymer paste is thicker than the cement paste, so it is necessary to manually compact the polymer paste and then place it on the sand shaking table for 60 s.

5. Polyethylene plastic film was used to seal the surface of the mould to prevent the water from evaporating rapidly, and then the trial mould was put into the curing box for maintenance.

The specimen composition is shown in Table 2.

**Table 2 Specimen composition table.**

Paste number	modulus	Na <sub>2</sub> O equivalent	Water solid ratio	Paste number	modulus	Na <sub>2</sub> O equivalent	Water solid ratio
M <sub>1-8</sub>	1	8	0.5	M <sub>2.0-8</sub>	2.0	8	0.5
M <sub>1-12</sub>	1	12	0.5	M <sub>2.0-12</sub>	2.0	12	0.5
M <sub>1-18</sub>	1	18	0.5	M <sub>2.0-18</sub>	2.0	18	0.5
M <sub>1-24</sub>	1	24	0.5	M <sub>2.0-24</sub>	2.0	24	0.5
M <sub>1-30</sub>	1	30	0.5	M <sub>2.0-30</sub>	2.0	30	0.5
M <sub>1.5-8</sub>	1.5	8	0.5	M <sub>2.5-8</sub>	2.5	8	0.5
M <sub>1.5-12</sub>	1.5	12	0.5	M <sub>2.5-12</sub>	2.5	12	0.5
M <sub>1.5-18</sub>	1.5	18	0.5	M <sub>2.5-18</sub>	2.5	18	0.5
M <sub>1.5-24</sub>	1.5	24	0.5	M <sub>2.5-24</sub>	2.5	24	0.5
M <sub>1.5-30</sub>	1.5	30	0.5	M <sub>2.5-30</sub>	2.5	30	0.5

### 2.3. Mechanical properties of metakaolin polymer

The mechanical properties test of the metakaolin polymer is strictly referenced to the national standard GB/T17671-1999 cement mortar strength detection method (IOS method). Compressive strength and flexural strength of metakaolin polymer slurry at the ages of 3 days, 7 days and 28 days are tested by compressive strength test machine and bending strength tester respectively. The average value of the same three specimens is taken as the corresponding compressive and flexural strength.

### 2.4. Microscopic characterization of metakaolin polymer

At the same time, mechanical tests were carried out to collect the microscopic test samples of the polymer mortar of the mountain ridge. The size of the specimen was about 50–5000 cm<sup>2</sup>. The specimen was chosen from the naturally shedding polymer parts after the strength test, and was selected for scanning electron microscope (SEM) for the Korean company EM-30 plus.

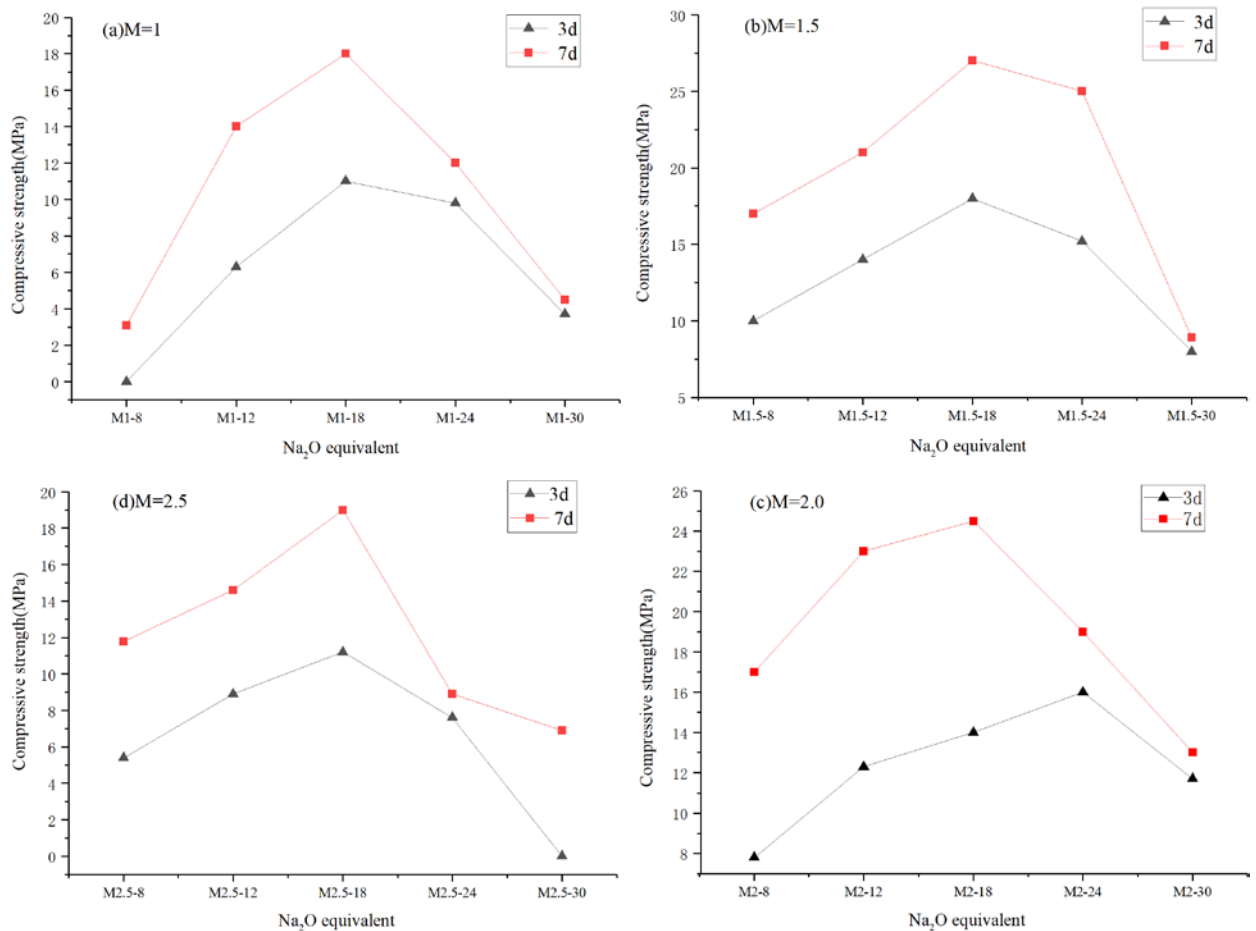
After the strength test, the samples of 1 cm<sup>3</sup> were selected for infrared spectrum and X-ray diffraction test. The samples were processed before the test, and the collected samples were ground in the mortar to make the particle diameter of the sample as small as possible.

## 3. Result and Discussion

### 3.1. Effect of Na<sub>2</sub>O equivalent on compressive strength of metakaolin polymer

Na<sub>2</sub>O equivalent and strength effect of MK geopolymer is shown in Fig. 1. The results show that the compressive strength increases first and then decreases with the increase of Na<sub>2</sub>O equivalent in the case of a given alkali activator modulus, and reaches the peak value at 18 %, with the increase of Na<sub>2</sub>O equivalent to 24 %. The strength of 3 days and 7 days can be observed approximately in the same direction,

but the rate of change is obvious. In the case of low modulus, the compressive strength varies significantly with the change of  $\text{Na}_2\text{O}$  equivalent. When  $\text{Na}_2\text{O}$  equivalence is in the range of 8–12 % and 24–30 %, the intensity change rate is extremely high.



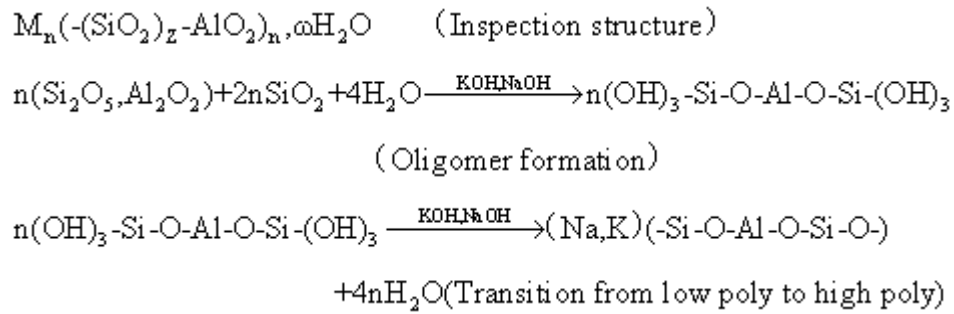
**Figure 1. Relationship between the  $\text{Na}_2\text{O}$  equivalent and strength of MK geopolymer.**

High compressive strength of the metakaolin polymer is due to its dense three-dimensional network shape and state and its stable high polymerization framework, sodium aluminosilicate gel (NASH) [11–15]. Therefore, the compressive strength of metakaolin geopolymer can also reflect the process of metakaolin polymerization. In the same geopolymerization system, the higher the degree of hydration of metakaolin geopolymer, the more complete the geopolymerization, the more complete the NASH structure, the higher the degree of polymerization. By analyzing the effect of different  $\text{Na}_2\text{O}$  equivalent of alkali activator on the compressive strength of the metakaolin polymer, it is known that  $\text{Na}_2\text{O}$  equivalence has an important effect on the hydration degree of the kaolin polymer. When  $\text{Na}_2\text{O}$  equivalence is less than 18 %, the degree of hydration of the metakaolin polymer is increasing with the increase of  $\text{Na}_2\text{O}$  equivalent. It has a positive effect. When  $\text{Na}_2\text{O}$  equivalence is greater than 18 %, with the increase of  $\text{Na}_2\text{O}$  equivalent, the degree of hydration of the metakaolin polymer decreases and has inhibitory effect.

In Fig. 1, the 18 % of the  $\text{Na}_2\text{O}$  equivalent is the optimum activation environment for the hydration reaction of metakaolin. The  $\text{Na}_2\text{O}$  equivalence is less than 18 %. The  $\text{Na}^+$  content in the metakaolin land polymerization system is insufficient to support the geopolymerization of metakaolin. Studies have shown that the three-dimensional structure skeleton of geopolymer is obtained through oxygen bridge linking silicon oxygen and aluminum oxide. The  $\text{Al}^{3+}$  ions in the framework can replace  $\text{Si}^{4+}$ , which leads to the generation of negative charges, and the negative charge needs to be balanced by metal cations in the alkaline activator [16–18]. The concentration of  $\text{Na}^+$  plays an important role in the condensation of second phase silicon oxy tetrahedron with aluminum oxide tetrahedron in the reaction of metakaolin.

The empirical formula of geopolymer is shown in Fig. 2 [19].  $\text{Na}^+$  ion concentration is too low to meet the need of the second stage equilibrium charge, which will result in the termination of the reaction. At the same time, the  $\text{Na}^+$  guiding role is also needed in the third stage of conversion from oligomeric to high polymer state. For example, the concentration of  $\text{Na}^+$  is insufficient, and the polymerization degree of metakaolin polymer decreases, the polymerization chain shortens, and the structure is missing. The content of silicon in activator increases with the increase of  $\text{Na}_2\text{O}$  equivalent, thus increasing the number of available silicon and accelerating the hydration process of metakaolin. As the three stages of the

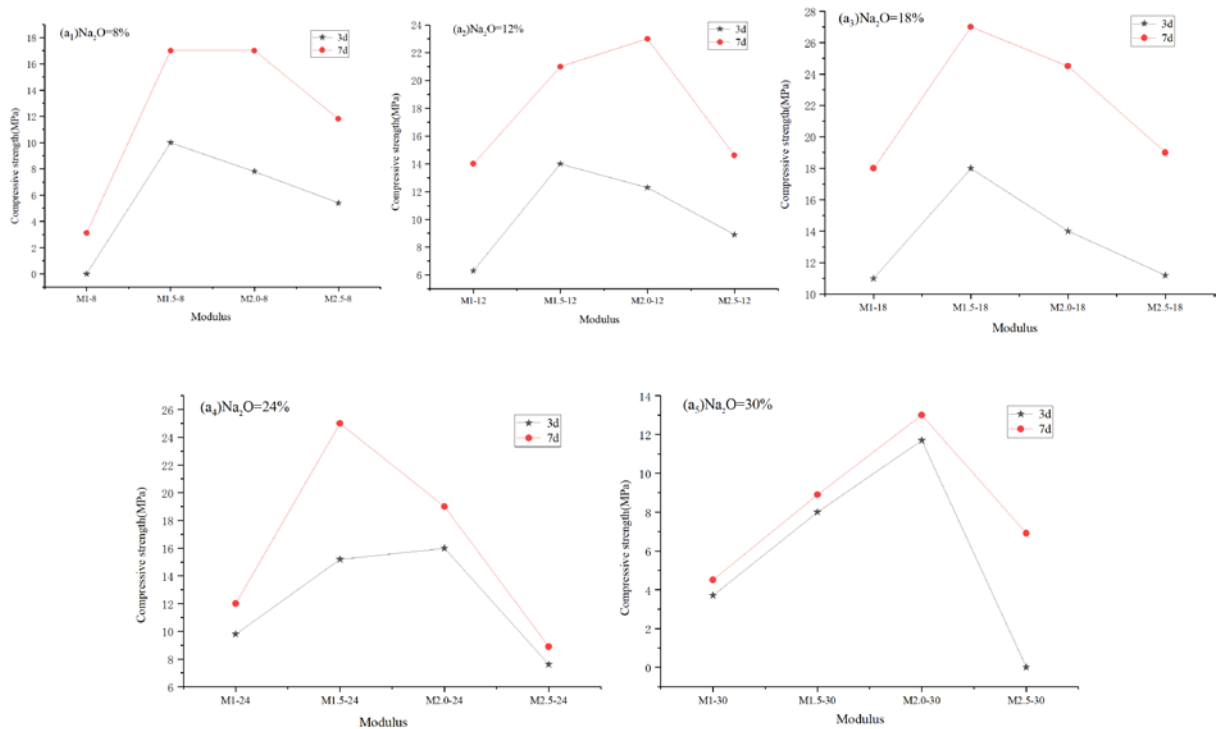
geopolymerization occur at the same time, when the Na<sub>2</sub>O equivalent is greater than 18 %, the reaction in the second and third stages will occur rapidly, and the polymer with low or high polymer will rapidly form on the surface of metakaolin. The metakaolin raw material is wrapped so that it can not be dissolved in contact with the alkaline solution, thus inhibiting the hydration degree of the metakaolin polymer.



**Figure 2** The empirical formula of geopolymer.

### 3.2. Effect of modulus on compressive strength of metakaolin polymer

As shown in Fig. 3, the effect of modulus of alkali activator on the compressive strength of metakaolin polymer is studied. The results show that the compressive strength of the metakaolin polymer increases with the increase of the modulus of the alkali activator at the given Na<sub>2</sub>O equivalent, and reaches the peak value from 1.5 to 2, when the modulus is greater than 2. The strength clearly decreases when the Na<sub>2</sub>O equivalence is at 12 % and 24 % (as shown in a<sub>2</sub> and a<sub>4</sub> in Fig. 3), the inflection point of 3 days and 7 days compression strength curve is not consistent. At 12 %, the compressive strength of 3 days reaches a peak at 1.5 modulus and then decreases; at 24 %, the compressive strength reaches the peak value of 7 days at 2 modulus and then decreases. On the surface of M<sub>1-8</sub>, it is obvious that the water accumulated, and the surface of M<sub>2.5-30</sub> has hardened and formed a layer of sodium silicate shell, but the interior has not yet solidified. It is found that when the modulus is greater than 2, with the increase of modulus, the development of early strength will slow down or even stagnate.



**Figure 3.** Effect of modulus on the aggregation strength of metakaolin land.

By analyzing the effect of different moduli of alkali activator on the compressive strength of metakaolin polymer, the modulus has an important effect on the degree of hydration of the metakaolin polymer. When the activation modulus is less than 1.5, the hydration degree of metakaolin increases with the increase of modulus. When the activation modulus is greater than 2, the hydration degree of metakaolin can be inhibited as the modulus increases.

Studies have shown that the first stage of hydration reaction of metakaolin is aluminosilicate dissolved into silicon aluminum monomer. During this process, when the modulus of the activator is too



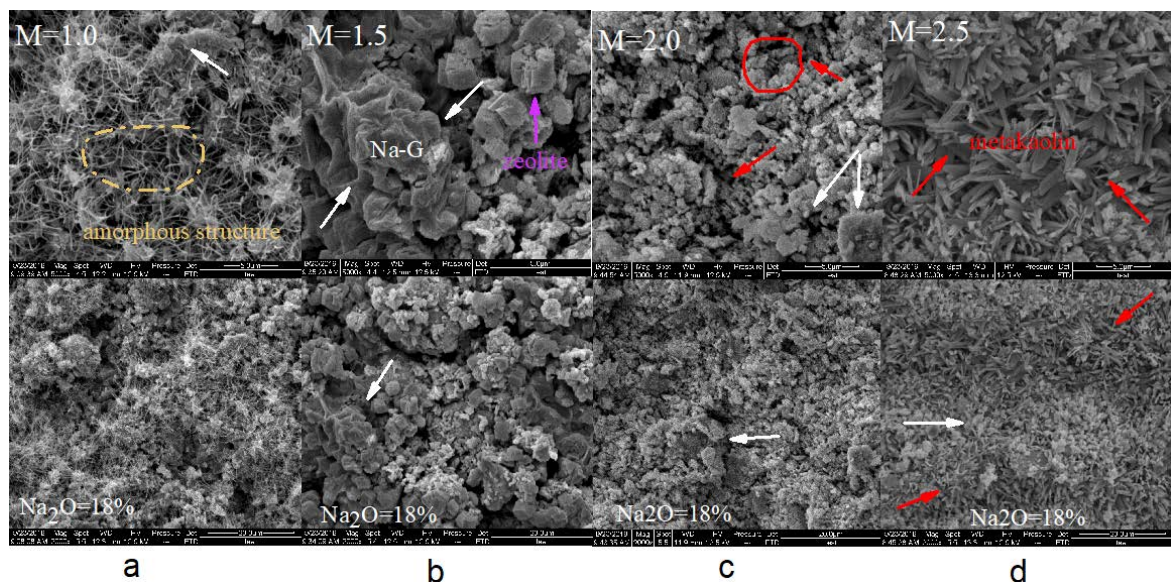
low, less than 1.5, the initial  $\text{Na}^+$  concentration will be very high, which will accelerate the reaction rate of the 2<sup>nd</sup> or 3<sup>rd</sup> stage and inhibit the dissolution of aluminosilicate. The amount of silicon available in the geopolymerization system is reduced. However, when the modulus is greater than 2, the aluminum dissolution from metakaolin will tend to react with  $\text{SiO}_3^{2-}$  in the alkaline activator and hinder the dissolution of silicon in metakaolin [11]. The rate of dissolution of silicon monomer in metakaolin is slowed down [20]. The composition and quantity of Nash gel formed in the process of geopolymerization depend on the degree of reaction of precursors [21]. Therefore, the degree of hydration of metakaolin is high if the precursor material is dissolved sufficiently. On the contrary, the residual amount of precursor material is high, and the degree of hydration is low.

In the first stage of the dissolution of aluminosilicate, the aluminum monomer will first be dissolved, because the bond length of Al-O is 1.75 Å, the bond length of Si-O is 1.61 Å, and the electronegativity of aluminum is small, so the binding force of Al-O is smaller than that of Si-O bond [22]. It inhibited the precipitation of silicon monomer, when the modulus of the alkali activator was greater than 2, the  $\text{Na}^+$  decreased with the increase of the modulus. Therefore, the reaction rate of the 2<sup>nd</sup> or 3<sup>rd</sup> phases would be reduced, resulting in slower consumption of aluminum monomer, restricting the dissolution of silicate and inhibiting the hydration degree of metakaolin [15]. At the same time, due to the increase of modulus, the concentration of  $\text{OH}^-$  in geopolymer system is reduced, so that metakaolin does not completely dissolve, and there will be remnants, resulting in lower degree of hydration of metakaolin.

When alkali activator modulus is too large, due to the low alkalinity in the reaction environment and low content of  $\text{OH}^-$ , the dissolution of aluminosilicate in metakaolin is less, and the formation of Si/Al monomer is insufficient, resulting in lower hydration degree of metakaolin. However, when the activator modulus is too small, the aluminosilicate in the metakaolin is completely dissolved, but the remaining components in the activator fill in the gap. The activator is easily soluble in water and easily destroys the structure of polymer.

### 3.3. SEM analysis

The specimens were scanned simultaneously by scanning electron microscope (SEM) after the compressive strength test. Fig. 4 shows the SEM images of metakaolin geopolymer with different moduli, when the  $\text{Na}_2\text{O}$  equivalent was 18%. By comparing a, b, c, d, we can see that B showed the most complete form of polymer gelation, of which Na-G indicated that the gel was of uniform texture and good morphology of polymerization. The results are consistent with the test results of compressive strength.



**Figure 4. SEM images of metakaolin polymers with different modulus at 18%  $\text{Na}_2\text{O}$  equivalent.**

At the same time, the existence of zeolites was observed in the B diagram, such as the purple arrow. Some studies have shown that the formation of NASH gel will be accompanied by the formation of some by-product zeolite crystals [10], for example, zeolite ZSM-3, zeolite A, zeolite P, sodium carbonate, zeolite 5p and so on. Some studies also suggest that NASH can be transformed into zeolites in time or some other external conditions to form a more stable structure [23].

In Fig. 4a, it could be seen clearly that there are long hairy or silk flocculent amorphous substances on the surface of NASH gel. This phenomenon is observed in three images of b, c and d. However, Fig. 5a<sub>1</sub> and 5a<sub>2</sub> show a similar phenomena, but the number is less than that of the map. The amorphous length of hairy or floc is very short. We think that the amorphous structure is made up of two parts. The first part is

the ionic group formed by ions in the activator remaining in the hydrated system of metakaolin. The second part is the silicon aluminum monomer, which is used in the uncondensed reaction. The dissolution reaction of metakaolin aluminosilicate is quite complete at this time. The concentration of Si/Al monomer is very high, and at this time, because the concentration of  $\text{Na}^+$  is not enough to support such a high concentration of Si/Al monomer to form small geopolymer gels (Si-O-Al-O, Si-O-Al-O-Si-O), the Si/Al monomer will adhere to the surface of geopolymer to form a hairy or silk flocculent amorphous structure.

Fig. 4c and 4d clearly demonstrates that the degree of polymerization of the geopolymer in metakaolin is reduced, the particle size is small, the geopolymer formed is small gel structure, the skeleton is relatively small, the structure is incomplete, the porosity is high, and there are a large number of unreacted kaolin precursors. This conclusion is consistent with the test results of compressive strength. When the concentration of  $\text{Na}_2\text{O}$  is too high, the dissolution of aluminosilicate in metakaolin can be inhibited, the number of unresponsive metakaolin increases, and the degree of hydration of metakaolin decreases.

In Fig. 4, it is obvious that the degree of polymerization of  $a_2$  and  $a_4$  metakaolin polymer is lower than the polymerization degree of  $a_3$ . When  $\text{Na}_2\text{O}$  equivalence is less than 18%, the degree of polymerization of metakaolin polymer is increasing with the increase of  $\text{Na}_2\text{O}$  equivalent, and the texture is more uniform and ordered. When the  $a$  is greater than 18%, the degree of polymerization of metakaolin decreases continuously, from large gel group to small gel particle, and at the same time the unresponsive metakaolin precursor begins to appear. In Fig. 5a<sub>5</sub>, the particles of unresponsive metakaolin can be observed clearly. This conclusion can further confirm the strength test results of the metakaolin polymer.

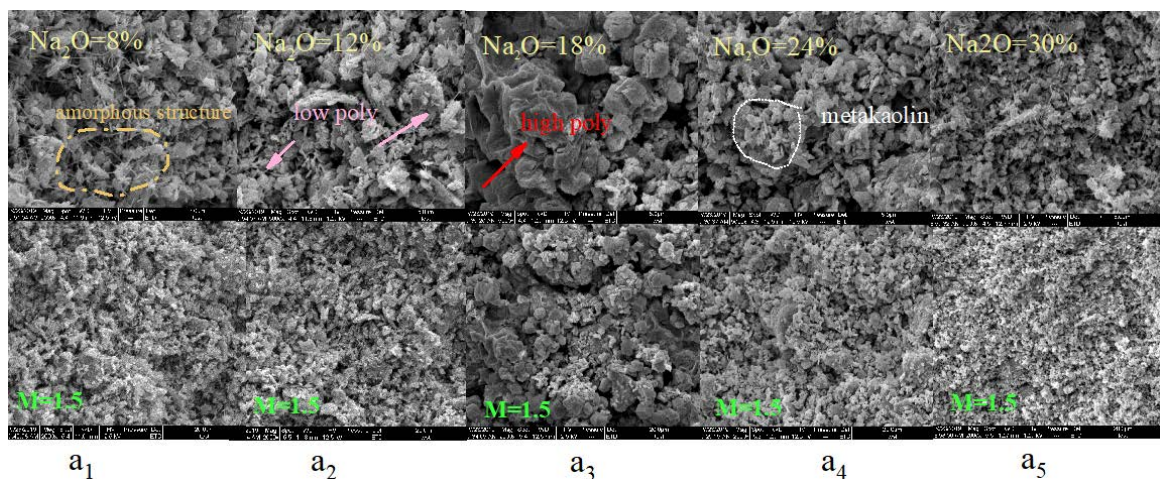


Figure 5. SEM images of metakaolin polymers with different  $\text{Na}_2\text{O}$  equivalents at 1.5 modulus.

### 3.4. FT-IR and XRD analysis

The FTIR spectra of metakaolin and metakaolin polymers are shown in Fig. 6. The FT-IR spectra of metakaolin polymer are selected by 3 days and 7 days of  $M_{1.5-18}$  specimens. In the infrared spectra of metakaolin, the wide peaks between  $1000\text{ cm}^{-1}$  and  $1100\text{ cm}^{-1}$  are considered to be asymmetrical tensile vibrations of silicon tetrahedron and aluminosilicate tetrahedron [24–26]. By comparing the FT-IR spectra of 3 days and 7 days, the spectra of metakaolin polymer can be found to be basically the same, mainly at  $700\text{ cm}^{-1}$ . The FT-IR spectrum of 7 days has a weak absorption peak between  $600\text{ cm}^{-1}$  to  $700\text{ cm}^{-1}$  [24]. 3 days and 7 days of  $M_{1.5-18}$  FT-IR spectra show strong absorption peaks at  $900\text{ cm}^{-1}$  to  $1000\text{ cm}^{-1}$ . Judging by other analyzed references, it seems to be caused by the asymmetric tensile vibration of Si-O-T [26]. Compared with metakaolin, this absorption peak shifts to the left, which we think is the cause of the formation of NASH [26].

The XRD images of metakaolin and metakaolin geopolymer are shown in Fig. 7. The geopolymer test samples are  $M_{1.5-18}$  and  $M_{2.5-30}$ . It is mainly considered that the strength results can be interpreted and verified. Comparing the XRD images of metakaolin precursor materials,  $M_{1.5-18}$  and  $M_{2.5-30}$ , we can see that the width of the peak between diffraction angle ( $20^\circ - 40^\circ$ ) of  $M_{1.5-18}$  specimen is obviously larger than that of  $M_{1.5-18}$  and metakaolin because of the dissolution of aluminosilicate crystals in metakaolin under the action of alkaline activator. Then, the NASH gel was formed by geopolymerization. The gel was a high polymer and an amorphous structure [22]. At the same time,  $M_{2.5-30}$  display the characteristics of quartz and halloysite in this interval. Therefore,  $M_{2.5-30}$  can be considered to have low dissolution rate of aluminosilicate in the process of polymerization, resulting in its early compressive strength being particularly low. In the XRD image of  $M_{1.5-18}$ , the characteristics of Zeolite A were observed, but no phase characteristics of calcite were found. There is also the existence of halloysite and quartz.

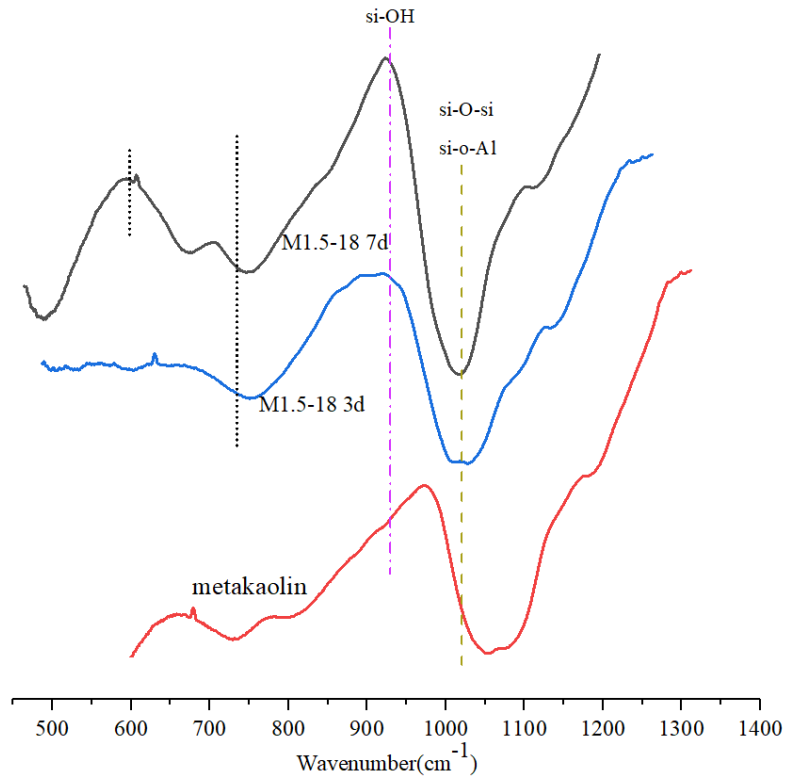


Figure 6. FT-IR spectra of metakaolin and metakaolin land polymer.

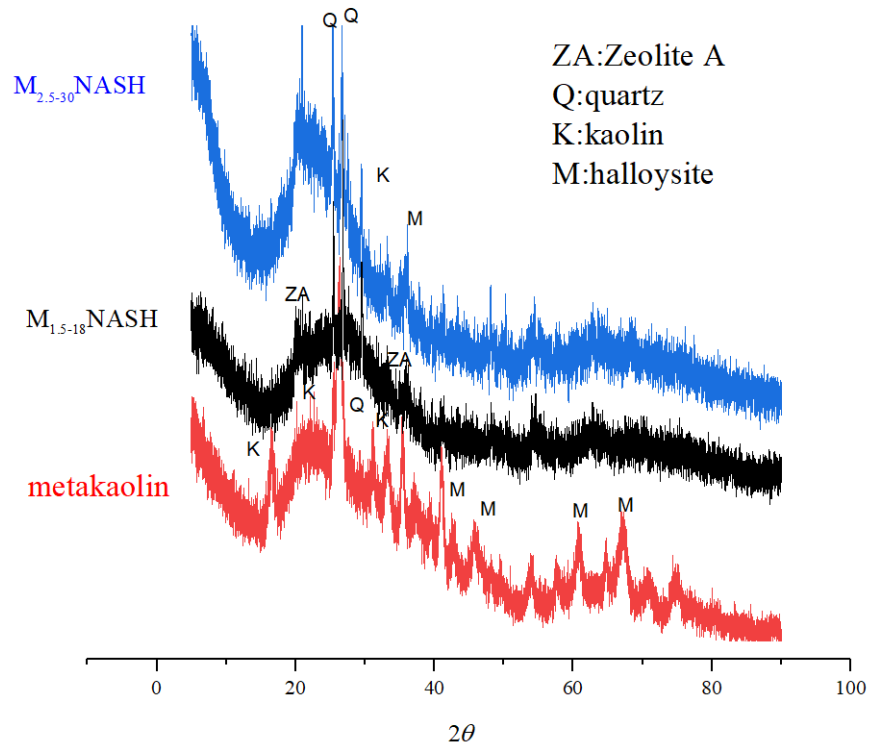


Figure 7. XRD pattern of metakaolin and geopolymer.

#### 4. Conclusion

This paper studied the effect of Na<sup>+</sup> on the hydration degree of alkali activated metakaolin polymer. The effects of Na<sub>2</sub>O equivalent and the modulus of alkali activator on the strength and microstructure of the kaolin polymer were demonstrated. The results of the study led to the following conclusions:



1. 18 % Na<sub>2</sub>O equivalent is the activation environment for the highest degree of hydration of metakaolin geopolymer. When Na<sub>2</sub>O equivalence is less than 18 %, the degree of hydration of metakaolin geopolymer increases with the increase of Na<sub>2</sub>O equivalent. When the Na<sub>2</sub>O content is greater than 18 %, as the Na<sub>2</sub>O equivalent increases, it will inhibit the hydration reaction of the geopolymer reducing its degree of hydration.

2. When the modulus of alkali activator is 1.5, the maximum hydration degree of the geopolymer can be achieved. When the modulus is less than 1.5, the rate of polymer reaction will accelerate and the hydration degree will be accelerated with the increase of the modulus. When the modulus is greater than 2, the reaction of the first stage of the metakaolin hydration process will be inhibited with the increase of modulus, and the hydration degree of metakaolin will be reduced.

3. When Na<sub>2</sub>O equivalence is less than 18 %, the degree of polymerization of metakaolin polymer is increasing with the increase of Na<sub>2</sub>O equivalent, and the texture is more uniform and structured. M<sub>2.5-30</sub> has low dissolution rate of aluminosilicate in the process of polymerization, resulting in its early compressive strength being particularly low.

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