An Investigation of Different Factors On Durability of Metakaolin Geopolymer Cement Mortar

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Abstract

In the preliminary part of this research, orthogonal experiments were conducted to identify metakaolin admixture and activator content as the two main influencing factors through four factors that mainly affected the basic properties of metakaolin geopolymer specimens. It is crucial to study the durability of construction materials under harsh environments. Therefore, this experiment mainly tested the anti-permeability, sulfate corrosion resistance, and freezing-thawing resistance of metakaolin geopolymer pastes with different ratio of metakaolin admixture and alkali activators content. The results indicated that with the changes of metakaolin and activator content, both of two anti-permeability curves increased firstly and then decreased. The sulfate corrosion resistance showed that both weight loss curves firstly decreased and then increased trend. Corrosion resistance curves increased first and then decreased in different curing time. Freezing-thawing resistance tests indicated that weight loss rate and strength loss rate declined and then ascended. Moreover, the microscopic SEM and FT-IR experiments were used to more directly reflect the patterns of durability changes.

Introduction

As industrialization acceleration and AI intelligence development, more and more varieties of construction materials are being used. In 2019, the output value of China's construction industry was nearly 25 trillion yuan, and the number of employed people reached 55 million. Construction industry output value accounted for the national GDP ratio rose year by year, employment accounted for nearly 7% of total employment. It is clear that the construction industry is the pillar industry of China's industry or all over the world. As a major component of civil structures, test studies are essential, which affects the improvement of people's living standards. Research on cement-based building materials focuses on the durability, high strength, and fast-setting properties of cement-based materials.

But the frequent use of ordinary portland cement (OPC) has been bringing obvious problems of large amount of greenhouse gases emission, and environmental problems have been becoming more and more critical[1]. The amount of natural resources is consumed by OPC production[25]. Thermal and electrical energy was consumed more than 2.72 GJ/ton and 65 kWh/ton, respectively[25, 26]. In addition, the deterioration of civil infrastructure and structures age shortening made of cement and concrete due to durability problems are inevitable global problems despite the presence of some prevention technologies[2–4]. So new materials are sought to meet not only the strength requirements needed for basic building performance, but also higher requirements for durability. Low carbon materials geopolymers were first proposed by Davidovits in 1978[5, 6]. By combining reaction of industrial by-products (generally fly ash, slag, metakaolin, etc.) with alkaline solutions is to create geopolymer cement. The special structure of geopolymers make it sustainable materials with many advantages such as mechanical properties[7] and some durabilities[8].

Metakaolin was divided into the reaction products of low-calcium systems. Due to the N-A-S-H gels with highly crosslinked (mainly Q4) and zeolite-like structure[9], the mechanical properties perform better than

OPC. Unlike cement alone as a cementitious material, the percentage of metakaolin has an impact on the performance. So the content of geopolymer was of great importance to this new systems. Recent studies[3] in durabilities are in acid resistance with different materials. However, the doses of metakaolin and alkali activators are not sure for more information or conclusion in durabilities including anti-permeability, sulfate corrosion resistance, and freezing-thawing resistance. The metakaolin content and alkali activator content still needs to be continued to research on durability of geopolymer systems.

This paper firstly analyzed two factors of four possible factors, including metakaolin, alkali activator, β s and modulus, that had large effects on geopolymer system through orthogonal experiments before durability tests: the amount of metakaolin content and the amount of alkali activator content. Then, the corresponding durability tests were conducted according to the two groups of variables. Combining with the micromorphology to reveal the possible causes affecting durability changes. Based on the development of science and technology, through this experiment also lay the foundation for combining AI mechanization intelligence and engineering use. The use of the durability in the future can more flexible in the actual engineering.

Experimental Method

2.1 Materials

2.1.1 Metakaolin and OPC

Metakaolin, provided by Datong Jinyuan Kaolin Co., Ltd., was applied to part of solid mixture. Metakaolin parameters were in line with GB/T 14563 – 2008 and other relevant specifications. According to experimental materials, the metakaolin powder, used in tests, was amorphous. The relevant physical and chemical properties of metakaolin were shown in Table 1. 42.5R type ordinary Portland cement (OPC), in accordance with Chinese standard GB175-2007 "General purpose portland cement", was supplied by Hubei HuaXin cement Co., Ltd, and OPC was used as a another binder for solid materials in geopolymer system. The properties of Normal Portland cement was shown in Table 2.

Table 1 Chemical and physical components of metakaolin

Compo nents	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Ti O ₂	Ca O	Mg O	K ₂ O	Na ₂ O	White ness	PH value	Water content	Loss on ignition	Average grain size
Conten t	53.4 7%	44.0 2%	0.2 6%	0.4 7%	0.2 6%	0.2 1%	0.1 8%	0.0 8%	93.50 %	6.8	0.20%	0.14%	13 μ m

Table 2 Chemical and physical components of ordinary Portland cement

Compositi ons	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Mg O	SO ₃	Loss on ignition	Initial Time	Final Time
Content	55.30 %	26.04 %	6.61 %	4.32 %	2.44 %	2.33 %	3.12%	130min	170min

2.1.2 Alkali activator

The alkaline activator materials for this experiment were prepared by sodium silicate solution and NaOH particles in a certain ratio. Pour NaOH particles into sodium silicate solution and stirring at the same time to adjust the experimental solution modulus. The sodium silicate compositions were shown in Table 3 and NaOH was supplied by Tianjin Kaitong Chemical Reagent Co., Ltd. and the analytical purity was over 96.0%. The solution was sealed and kept in the environment for 24 hours before synthesis.

Table 3 Components of sodium silicate

Components	SiO ₂	Na ₂ O	°Bé	Modulus
Content	27.20%	8.75%	39	3.2

2.2 Preparation

The experimental formulation was carried out according to Table 4. Two different variants (metakaolin content and activator content) of geopolymer mortar were synthesized for testing. The metakaolin powder and alkali activator were mixed in a planetary mixer. The designated materials were stirred slowly for 1 minute and then at high speed for 1 minute. Then, the mixture specimens were poured into standard molds for the different experiments, vibrated and troweled flat. The freezing-thawing resistance experiment and sulfate corrosion resistance experiment both used apparatus with dimensions of 70.7mm × 70.7mm × 70.7mm. Six cubic test blocks were prepared for each group of experiment, and the average value was used for the results. Curing time was adjusted according to different experiments and curing chamber was in temperature (20°C) and relative humidity 90%-95%. Factor 1 was the content of metakaolin, the metakaolin to cement ratio was from 0.8 to 0.5. Factor 2 was the content of activator, the activator to solid was from 0.1 to 0.4. Figure 1 showed the completely experiment process.

Table 4 Experiment compositions

Test Label	Cement(g)	Water(g)	Metakaolin(g)	Modulus	Activator(g)	Standard sand(g)
GS1	160	320	640	1.5	200	1600
GS2	200	320	600	1.5	200	1600
GS3	240	320	560	1.5	200	1600
GS4	280	320	520	1.5	200	1600
GS5	320	320	480	1.5	200	1600
GS6	360	320	440	1.5	200	1600
GS7	400	320	400	1.5	200	1600
GA1	320	320	480	1.5	80	1600
GA2	320	320	480	1.5	120	1600
GA3	320	320	480	1.5	160	1600
GA4	320	320	480	1.5	200	1600
GA5	320	320	480	1.5	240	1600
GA6	320	320	480	1.5	280	1600
GA7	320	320	480	1.5	320	1600

2.3 Characterization

2.3.1 Anti-permeability

Anti-permeability not only characterized the ability of cement and concrete to resist the passage of water, but also affected the performance of cement and concrete against other durability e.g. chloride ion penetration, etc. Under constant water pressure, the amount of water permeation on surface of materials after a certain period of time was used to measure anti-permeability. The higher the value of water pressure, the better the anti-permeability of geopolymer pastes and the higher the compactness of specimens. Experiment used SS-15 type digital display mortar anti permeability apparatus. Before the experiment started, each group of six mortar specimens were numbered, the sides of the specimens were evenly coated with glass cement and installed into the mortar anti-permeability apparatus for fixing. Then sealed around with glass cement, and the test could be conducted after 24 hours of complete solidification of glass cement. The initial water pressure set 0.2MPa, and the maximum water pressure set 4.2MPa. Mortar anti-permeability apparatus automatically rose up 0.1MPa every 1h and specimens were observed every 30min. Anti-permeability test values were determined using Eq. 1:

$$P = H - 0.1 \quad (1)$$

where:

P = geopolymer specimen pastes anti-permeability average value, MPa;

H = specimen pressure values when permeability occurred in three of the six specimens, MPa.

2.3.2 Sulfate corrosion resistance

Most environments such as oceans, salt lakes, and groundwater contained sulfate ions. Sulfate corrosion is a slow process. In order to accelerate the process of erosion in the test, high concentration sulfate solutions were needed to attain corrosion effect. The sulfate corrosion resistance experiment was measured according to the National Standard GB/T 749–2008[10]. The maintained GS and GA specimens were placed in plastic tanks filled with sulfate solution for 28d and 56d soaking. After soaking certain days, the specimens were weighed and tested for compressive strength. Sulfate corrosion resistance was measured by weight loss rate and compressive strength corrosion resistance coefficient. The sulfate corrosion resistance coefficients were determined using Eq. 2 and Eq. 3:

$$\Delta M_s = \frac{M_0 - M_1}{M_0} \times 100\% \quad (2)$$

where:

ΔM_s = geopolymer paste weight loss rate, %;

M_0 = weight of geopolymer paste before soaking, g;

M_1 = weight of geopolymer paste after 28d/56d soaking, g.

$$R_f = \frac{R_c}{R} \times 100\% \quad (3)$$

where:

R_f = geopolymer paste compressive strength corrosion resistance coefficient, %;

R_c = geopolymer paste average compressive strength after 28d/56d of soaking, MPa;

R = geopolymer paste average compressive strength after 28d/56d without soaking, MPa.

2.3.3 Freezing-thawing resistance

When outdoor temperature was low, building materials were often damaged by freezing-thawing cycles. Freezing damage alone would not cause large cracks in cement and concrete buildings. When the building materials were repeatedly subjected to freezing-thawing damage, small cracks would gradually expand and the safety of structures was greatly threatened. Freezing-thawing resistance test was tested in accordance with National Standard JGJ/T70-2009[11]. Specimens were tested in a freeze-thaw testing machine for 50, 100 times at a temperature range of -15°C to $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Freezing-thawing resistance tests were reflected by weight loss rate and compressive strength loss rate shown in Eq. 4 and Eq. 5:

$$\Delta M = \frac{M_{fz0} - M_{fz1}}{M_{fz0}} \times 100\% \quad (4)$$

where:

▲M = weight loss rate after 50/100 times freezing-thawing test,%;

M_{fz0} = weight of specimens before freezing-thawing test,g;

M_{fz1} = weight of specimens after 50/100 times freezing-thawing test,g.

$$Cs = \frac{C0 - C1}{C0} \times 100\% \quad (5)$$

where:

Cs = compressive strength loss rate,%;

C0 = control specimens average compressive strength,MPa;

C1 = experiment specimens of 50/100 times average compressive strength,MPa.

2.3.4 Scanning electron microscopy

S-3000N type scanning electron microscope, made by HITACHI, was used to measure specimen pastes SEM images. Resolution was up to 5nm. Meanwhile, E-1045 type gold spraying instrument was used to experimental research. Figure 2 showed SEM apparatus. During the experiment, specimen pastes would be cured to 28 days to take small pieces of pastes at the center of specimens and placed in constant temperature drying oven, dried at 70°C for 2 days. The dried specimens were put into the gold spraying instrument for surface gold spraying, and then experimental specimens were shown in Fig. 3. After the specimen was processed, they were put into scanning electron microscope for micro-structure observation.

2.3.5 Fourier transform-infrared spectroscopy

Nicolet Avatar 330 type Spectrum, made by Thermo Fisher Technology Co., was used to measure FT-IR. In the experiment, small pieces of specimen from the center of geopolymer cement pastes were put into laboratory bowl and ground to powders. Then small amounts of specimen were mixed with KBr powder in the ratio of 1:100 and two powders were evenly mixed. The KBr powder should be kept in the infrared drying oven to keep dry. The FT-IR scanning ranges from 400 cm^{-1} to 4000 cm^{-1} . Figure 4 showed the FT-IR apparatus.

Results And Discussion

3.1 Anti-permeability

Figure 5 reflected the images of cement mortar and metakaolin geopolymer pastes under anti-permeability experiments for two different significant influencing factors. The permeability pressure values of the control group was at 3.3 MPa. Under the two different groups of influencing factors, permeability pressure values of formed specimens increased and then decreased as the dosing of metakaolin and alkali activator increased. At GA5/GS5, the maximum permeation pressure values were

reached for both groups of specimens. At the same time, it could also be observed that the activator doses had a greater effect on the anti-permeability than the metakaolin doses before GA5/GS5. The change in activator contents after GA/5GS5 also had a greater impact on the anti-permeability than the change in geopolymer contents. The water absorption of metakaolin was relatively high, and for the same mass of metakaolin and cement, the density of metakaolin was smaller than that of cement. Thus the volume of metakaolin was larger and the porosity was higher when more metakaolin powders were mixed to form specimens. The interior had more connected pores, due to high water absorption of metakaolin, which caused water to permeate into the interior first during the experiment and the anti-permeability pressure value was small. When the mixture of metakaolin was gradually reduced and filled with cement, the original previously connected pores gradually changed to closed pores, and experimental water needed to pass through the closed pores to reach the inside of specimens during the experiment, and the closed pores were more difficult for water to permeate than the connected pores, so the anti-permeability improved. Metakaolin contents changed smaller like GS7 specimens, the slurry formed by geopolymer cement mortar approximated the slurry formed by cement mortar, and the anti-permeability resistance pressure gradually converged to the value of the anti-permeability resistance pressure of cement mortar.

On the other hand, the increase in the amounts of activator were a significant influence on the anti-permeability. The amounts of activator were directly related to the dense degree of geopolymer mortar specimens. When the activator contents were relatively small, geopolymer in the reaction process, metakaolin didn't have enough alkaline environment, only part of materials occurred geopolymerization reaction, the reaction generated material was silicate oligomer, materials in the mortar structure of the arrangement were not dense enough. There were more pores between these substances and these pores were connected to each other. Water permeated through the pores into the interior of specimens, so the permeation pressure of geopolymer mortars were smaller when activator contents were smaller. More activator contents would adequately react to form structural units in silicate aggregates. Highly dense structure made it difficult for water to penetrate inside the structures [12]. This effectively enhanced anti-permeability. Excess excitation caused hardening of the retained Na_2SO_3 in the internal structures. At the junction of sodium silicate hardened part and geopolymer structure, remained sodium silicate would also wrap part of geopolymer structures, water entered geopolymer mortar specimens to reduce permeation pressure, resulting in poor anti-permeability.

3.2 Sulfate corrosion resistance

The sulfate corrosion resistance test was measured based on 28 days and 56 days weight loss rate and compressive strength corrosion resistance coefficients. The results were shown in Fig. 6 and Fig. 7. From Fig. 6, cement pastes sulfate corrosion resistance was also shown. The weight loss rate of cement mortar was 0.76% and 1.52% after 28 days and 56 days of immersion in 5% sodium sulfate solution. It could be observed in Fig. 6 that regardless of changing the amount of metakaolin or the amount of activator, the weight loss rates showed a trend of decreasing first and then increasing after 28 days and 56 days solution immersion. Meanwhile, two influences had similar or better weight loss rates than cement mortar at GA4/GS4 to GA6/GS6. Metakaolin contents played a major role in sulfate corrosion

experiments.Reducing a certain amount of metakaolin and increasing certain activator contents strengthened sulfate corrosion resistance of geopolymer cement mortars.And reduction of metakaolin dosages had a greater effect on increasing changes of activator in sulfate corrosion resistance. Figure 7 showed compressive strength corrosion resistance coefficients of two influencing factors.Same as Fig. 6,in GA/GS4 to GA/GS6 content,two factors had positive influence.When choosing metakaolin as the main variable,strength loss was caused by the breakage of the molecular structure[13]. the formation of crystalline compounds promoted the loss of strength[14].The geopolymer reacted with Ca^{2+} generated by the cement hydration products during the process.High metakaolin made it different to enhance geopolymerization reaction.Due to the low Ca^{2+} contents, the geopolymerization reaction rate was not uniform, resulting in some metakaolin separated from alkaline activator before the reaction occurred.The internal structure of geopolymer was not dense and more internal pores were easy to let sulfate ions in. The weight changes may had caused by highly porosity[15].With the increasing of cement,more Ca^{2+} were involved in reaction.The generated $[\text{SiO}_4]$ and $[\text{AlO}_4]$ combined with cement hydration products to improve internal structural compactness of specimens.The amount of metakaolin gradually decreased and the amount of $\text{Ca}(\text{OH})_2$ generated by the reaction increased, which could react with SO_4^{2-} and thereby reduced corrosion resistance.

When choosing alkali activator as the main variable,with less activator contents, the amount of precursor generated during geopolymerization of metakaolin in an alkaline environment was less, resulting in less calcium silicate gels.The internal structure of oligoaluminate structure formed by insufficient reaction wasn't dense enough. Sulfate ions were easy to enter inner space of specimens.Activator contents continued to increase,the amount of precursor generated gradually increased, the calcium silicate gels also gradually increased.Both $[\text{SiO}_4]$ and $[\text{AlO}_4]$ produced condensation polymerization reactions with silicate oligomers and then produced aluminosilicate polymorphs[16].This improved the dense degree of the geopolymer mortar, and sulfate ions didn't easily access the interior of the mortar specimens.Due to own alkalinity, excessive doping of the activators affected the reaction process in the system and inhibited to prevent the sulfate ions from leaching into the internal structure,at the same time led to more pores space and both strength and weight decreased.

3.3 Freezing-thawing resistance and appearance

Figure 8 and Fig. 9 illustrated weight loss rates and compressive strength loss rates of freezing-thawing resistance experiment after 50 times and 100 times.The appearance of test specimens was shown in Fig. 10 to Fig. 14.

For appearance test,it could be seen in Fig. 10 and Fig. 13 to Fig. 14 that high metakaolin content specimens appeared more pores and cracks. Figure 13 showed that some edges of samples were peeling off, which led to weight loss rate increase.Metakaolin content decreased 40%,as GS5,only a small number of pores appeared on the surface of the test specimen, and the surface was relatively smooth. Figure 11 and Fig. 12 depicted different activator contents of test specimens.The surface of geopolymer mortar test blocks with small activator had damage and more pores, the surface of geopolymer mortar

test blocks with activator to solid ratio of 0.3 had no obvious damage and only a little slurry loss occurred. The above comparative analysis showed that the freezing-thawing resistance of geopolymer mortar was influenced by metakaolin content and activator content. Appropriate content of two factors could help to improve the freezing-thawing resistance of geopolymer system.

Regarding to Fig. 8 and Fig. 9, in Fig. 8, with the increase in the times of freezing-thawing cycles, it would cause an increase in the weight loss rate of the test specimens, while the change in metakaolin content and activator content led to an increase in the weight loss of geopolymer mortar after reduction first. When test proportion attained GA5/GS5, the freezing-thawing weight loss was lower than cement specimen. The most important cause for the weight loss was the water expansion in the mortar which was permeable while freezing occurred [17]. Freezing occurred led to the water volume increases by approximately 9% and hydraulic pressure was created due to this effect [18]. High metakaolin content had high water absorption, which made more H_2O filled with inner structures and hydraulic pressure was more than less metakaolin content specimens. The frozen ice force exceeded the strength of the mortar, the formation of micro-cracks started and weight loss deterioration occurred. Thus Fig. 9 compressive strength decreased. A. Allahverdi [19] and L. Basheer [20] had verified this explain and made similar conclusions.

Figure 9 could be also made other conclusions that with the metakaolin content decreased, the freezing-thawing resistance of specimens improved. There was a large amount of calcium ions in cements, which generated hydrated calcium silicate during the hydration reaction. Metakaolin was essentially an amorphous silica-alumina compound, which underwent progressive chain-end degradation catalyzed by alkali-activator cementitious materials, and hydrated calcium silicate underwent condensation reactions with $[SiO_4]$ and $[AlO_4]$. The hydrated calcium silicate generated by hydration fully reacted with $[SiO_4]$ and $[AlO_4]$ in geopolymerization and interaction, improving the internal structure of the geopolymer mortar. However, more cement affected the geopolymerization reaction.

Another factor in Fig. 9 showed that the magnitude of activator content directly determined the degree of reaction of the metakaolin in the preparation of geopolymer. When the activator content was small, the reaction between metakaolin and alkali activator formed the oligoaluminate structure, which had low compactness. On the other hand, due to the small activator, the mixture of metakaolin and alkali activator was incomplete, which led to the existence of excess metakaolin in the test specimen. During the freezing-thawing cycles, the residual metakaolin was easy to adsorb water molecules, resulting in the poor freezing-thawing resistance of geopolymer mortar. With the increase of content, metakaolin reacted fully with alkali activator, and the connection between these reaction products was mainly based on covalent and ionic bonds, forming a multi-polymer aluminosilicate structure, which improved the compactness of geopolymer mortar and thus the freezing-thawing resistance of geopolymer mortar enhanced. Excess sodium silicate interfered with the reaction proceed. During the test curing process, the sodium silicate directly hardened into solid form, reducing the compactness of geopolymer, and water entered the test specimen along the gaps between the sodium silicate and the geopolymer mortar as the freezing-thawing cycle test proceeded.

3.4 Scanning electron microscopy

The Two influencing factors topography of metakaolin geopolymer cement pastes durability and control cement paste topography were shown in Fig. 15 to Fig. 21. As seen in Fig. 15, $\text{Ca}(\text{OH})_2$, C-S-H and Aft were the mainly hydration products. From large number of literature[21–24], the hexagonal version was calcium hydroxide, the calcium alumina was elongated and acicular, and C-S-H was fluffy and fibrous. Figure 16 to Fig. 18 images were different activator contents geopolymer pastes. In the case of less activator content, the internal structure of the geopolymer was not homogeneous, and the massive material was distributed in clusters. At meanwhile, it could be found that a large number of metakaolin particles were wrapped without participating in the reaction, and the structural morphology at the tomographic section was different and the structure was porous. This affected the performance of the biased metakaolin geopolymer in terms of durability. The porous structure made it easier for water to flow inside and leaked out during the permeability tests, and it was evident in Fig. 5 that the anti-permeability resistance was worse at this time. Meanwhile, for freezing-thawing cycles and sulfate corrosion resistance, water act as a carrier to carry harmful ions into the interior to destroy the structures due to the loose internal structures. And the volume expansion when the water freezes made the test specimens caused cracks. Figure 17 showed the structure of the geopolymer was evenly distributed and densely arranged, and the metakaolin basically reacted completely. The pores disappeared gradually and the gel material wrapped around the particles, making the internal contact areas larger, each material connected with each other to improve the structural compactness. Polymetallic silicate attached to the internal structure to make properties of geopolymer durability better. With more activator contents mixed, as shown in Fig. 18, sodium silicate was not strong after becoming hardened body. Inner structures comes with more pores at the same time. Thus durability of geopolymer pastes became worse again.

Figure 19 to Fig. 21 images showed another factor: metakaolin content. Because of high water absorption of metakaolin, there were many flaky metakaolin particles on the surface of the gel materials that were not involved in the reaction, and these particles were loosely packed. Same as the low activator, the appearance of durability of geopolymer behaved bad. More pores and unreacted materials were performed in Fig. 19. Metakaolin content decreased and the specimens image was shown in Fig. 20 to Fig. 21. The suitable cements involved in the geopolymerization reaction, which improved the setting time of geopolymer and achieved the requirement of rapid hardening, and the hydrated $\text{Ca}(\text{OH})_2$ was interwoven with the aluminosilicate in the geopolymer in a network structure, which made the geopolymer more dense. It could be seen in Fig. 21 that cement and metakaolin were mixed together, and part of the structure was very closely arranged.

3.5 Fourier transform-infrared spectroscopy

The FT-IR images of two influencing factors of geopolymer were shown in Fig. 22 to Fig. 23. In Fig. 23, with the change of metakaolin content, the peak area of 3449.44 cm^{-1} band gradually became slower, where the absorption peak area of O-H gradually decreased, the $\text{Ca}(\text{OH})_2$ produced by hydration participated in geopolymerization reaction during depolymerization and condensation, and the reaction consumed a

large amount of $\text{Ca}(\text{OH})_2$ and crystalline water. In the 1097.97 cm^{-1} band, the absorption peak gradually shift from high to low wave number band. Si-O bond was replaced by Al-O bond. The newly generated geopolymer structure produced Si-O-Al bonds. On the other hand, Fig. 22 image was different activator contents. The shape of 795.70 cm^{-1} absorption peak gradually narrowed and tended to become steeper as the activator content increased. The reaction of the geopolymer to form a polyaluminosilicate structure steepened the peak here. The absorption peak in the 1097 cm^{-1} band gradually shifted to the lower wavenumber band, and different tetrahedra structures were combined to form new substance.

By compare with Fig. 22 and Fig. 23 images, the FT-IR showed similar chemistry bonds. This indicated that the two influencing factors had similar effects on the chemical bonds formed internally. These two influencing factors changed the geopolymer in terms of durability through different actions. Microscopic experiments also provided further proofs for macroscopic experiments.

Conclusion

In this research, metakaolin and activator contents, two mainly factors, were involved in the geopolymer experiments to study the durability. According to anti-permeability test, sulfate corrosion resistance test and freezing-thawing resistance test, macroscopic experiments were made to identify visual impact. What's more, microscopic experiments including SEM and FT-IR were shown to justify and explain the macroscopic appearances. The following conclusions could be shown:

- We tested the durability including anti-permeability, sulfate corrosion resistance and freezing-thawing resistance. Using maximum pressure values, weight loss rates, compressive strength corrosion resistance coefficients to measure the properties. We found that with the decrease of metakaolin content, the anti-permeability of geopolymer behaved increased first and then decreased. The change of increasing activator content was the same as the metakaolin curve. Sulfate corrosion resistance curve showed that incorporated appropriate proportion of both factors facilitated resistance to sulfate attack. Freezing-thawing resistance and appearance also showed the same influences as sulfate resistance tests.
- SEM and FT-IR images were needed to aid in understanding changes in macroscopic phenomena. From the micrograph, it was possible to see more clearly the effect of different factors changes on the internal structure of the forming geopolymer, so that the reasons affecting the durability changes could be reasonably analyzed.
- Experiments indicated that GS5 and GA5 were the best groups of durability of metakaolin geopolymer cement mortars. In future practical use, the ratio can be used as a reference for durability improvement in practical engineering applications, while more relevant experimental tests are needed to improve the development of the geopolymer system.

Declarations

Disclosure statement

No potential conflict of interest was reported by the authors.

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Figures

Figure 1

Geopolymer paste durability experiments process

Figure 2

SEM apparatus

Figure 3

Specimens after process

Figure 4

Nicolet Avatar 330 type Spectrum

Figure 5

Maximum pressure values of GS/GA and cement control pastes

Figure 6

Weight loss rates of GS/GA and cement control pastes

Figure 7

Compressive strength corrosion resistance coefficients of GS/GA and cement control pastes

Figure 8

Weight loss rates after 50/100 times of GS/GA and cement control pastes

Figure 9

Compressive strength loss rates after 50/100 times of GS/GA and cement control pastes

Figure 10

Original cement specimen and after 100 times test specimen

Figure 11

GA1 geopolymer and after 100 times test GA1 geopolymer

Figure 12

GA5 geopolymer and after 100 times test GA5 geopolymer

Figure 13

GS1 geopolymer and after 100 times test GS1 geopolymer

Figure 14

GS5 geopolymer and after 100 times test GS5 geopolymer

Figure 15

Cement paste SEM in 5000k image

Figure 16

GA1 paste SEM in 5000k image

Figure 17

GA5 paste SEM in 5000k image

Figure 18

GA7 paste SEM in 5000k image

Figure 19

GS1 paste SEM in 5000k image

Figure 20

GS5 paste SEM in 5000k image

Figure 21

GS7 paste SEM in 5000k image

Figure 22

GA paste FT-IR image

Figure 23

GS paste FT-IR image