Mechanical Properties and Micromechanism of Geopolymers to Replace Cement Stabilized Crushed Stone

Yongxiang Li, Lijuan Yang, Xiao Li, Yongfei Li, Qiang Zhang, and Shoude Pang

Abstract In order to realize the resource utilization of solid waste, the principle of alkali excitation is used to prepare geopolymers with fly ash, mineral powder and wet carbide slag as the main materials to replace part of the cement as the cementing material for the pavement base. Geopolymer-stabilized crushed stone was prepared by compounding cement and aggregate with geopolymer, and the unconfined compression strength, indirect tensile strength, compression rebound modulus, scour resistance and microscopic X-ray diffraction (XRD) and scanning electron microscopy (SEM) tests were carried out to study the effect of the change of geopolymer content on the mechanical properties of geopolymer-stabilized crushed stone and its mechanism. The test results show that when adding 30% geopolymer, the mechanical properties similar to those of cement can be obtained to a certain extent. XRD and SEM analysis showed that the geopolymer provided appropriate amount of silico-alumina and calcareous components to form calcium silicate hydrate (C–S–H) and calcium silicate (aluminum) hydrate (C–(A) –S–H) condensation. The glue can form a dense structure and increase the strength of the mixture.

Keywords Geopolymer · Stabilized crushed stone · Mechanical properties · Micromechanis

1 Introduction

Fly ash, mineral powder and wet carbide slag are industrial solid wastes, and improper treatment will cause environmental pollution. At present, the harmless treatment of industrial solid waste has become one of the problems that need to be solved when China implements "double carbon" measures. If it can be prepared into pavement base

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material, it can not only effectively solve the environmental pollution and ecological damage caused by a large number of solid wastes, but also provide a large number of raw materials for highway construction, and make solid wastes play a greater utilization value.

The comprehensive utilization of solid waste materials in road engineering is a research hotspot at present [1]. In 1980s, fly ash-coal gangue mixture was used in road base and subbase in the United States, and its application effect was confirmed by testing. Since the twenty-first century, the United States has mixed iron tailings crushed stone into building materials as the subbase and subgrade of asphalt concrete pavement, and achieved good application results [2]. In 2014, an airstrip was built with geopolymer concrete at West Wellcamp Airport in Brisbane, Australia [3]. In recent years, Shen [4] obtained the best mix ratio of steel slag, fly ash and phosphogypsum as road base materials, and found that its early strength was higher than that of lime-fly ash and lime-soil pavement base materials, and its long-term strength was much higher than that of cement stabilized aggregate, which could meet the relevant requirements of pavement base materials. Arulrajah [5] found that fly ash, slag and calcium carbide residue can be used to stabilize recycled building materials as road base or subbase through unconfined compression strength and compression rebound modulus tests. Hu [6] discussed the possibility of using fly ash and red mud geopolymer as the cementing material of crushed stone aggregate through unconfined compression strength, failure strain and drying shrinkage tests. The results showed that geopolymer stabilized aggregate had a stable application prospect in the application of flexible pavement. In China, the research on resource utilization of industrial solid waste started late. In 1990s, Maanshan Mine Research Institute applied iron tailings to pavement materials [7]. Chang'an University mixes lime and coal gangue with soil and uses it for road base [8]. In 1997, China built the first steel slag asphalt pavement test section [9]. In recent years, Beijing Research Institute of Mining and Metallurgy and Guangxi Pingguo Aluminum Company jointly developed a new type of red mud road base with good road performance using lime, fly ash, red mud and some additives as raw materials, which filled a domestic gap [10–12]. Qin and Liang [13] have also carried out the research on replacing part of cement with red mud and applied it in cement stabilized macadam base, which has certain reference value for the development of new materials for road base. Wang [14] through the tests of mechanical properties and crack resistance of cement stabilized macadam with three types of aggregate gradation, three cement dosages and fly ash, the results show that after adding fly ash, the early unconfined compression strength, flexural strength and splitting strength of cement stabilized macadam decrease, which helps to reduce early cracks. Xu [15] found that adding fly ash and slag to cement stabilized macadam can improve the mechanical properties and shrinkage characteristics of cement stabilized macadam through unconfined compression strength, indirect tensile strength. Chu [16] comprehensively analyzed the road performance of cement stabilized macadam mixture with iron tailings, and found that the addition of iron tailings can improve the unconfined compression strength, indirect tensile strength, water stability and frost resistance of cement stabilized macadam mixture. Liu [17] found that the mechanical properties of slag-based polymer and fly ash stabilized macadam are better than those of cement stabilized macadam in unconfined compression strength, splitting strength, compression rebound modulus.

Although some scholars and engineering units have carried out research and engineering demonstration on the preparation of pavement base materials from solid wastes, there are few technical researches on the preparation of pavement base materials from solid wastes at home and abroad based on the principle of geopolymer. In this paper, geopolymer cementitious materials prepared from fly ash, mineral powder and wet carbide slag by alkali excitation are used to replace part of cement stabilized macadam. The mechanical properties are evaluated by unconfined compression strength, indirect tensile strength, compression rebound modulus and scour resistance. The mineral composition of raw materials and microstructure changes of samples at different ages are analyzed by XRD analysis and SEM test, and the formation mechanism of geopolymer strength is analyzed, which provides a new method for comprehensive utilization of solid waste materials.

2 Experiment

2.1 Experiment Material

The raw materials used in the experiment are geopolymer cementing material, cement, aggregate, alkali activator and water.

Geopolymer cementitious materials include fly ash, mineral powder and wet carbide slag, and the main chemical compositions of effective minerals of the materials are shown in Table 1. The fly ash is low-calcium F-type fly ash with a median particle size of 12.72 μm; The mineral powder is S95 grade mineral powder mixed with 7% limestone, 15% fly ash and 78% slag. The moisture content of wet carbide slag is 33%.

Cement is taken from Jidong Cement Company, Hohhot, Inner Mongolia, and its strength grade is P.O42.5.The physical and mechanical properties of cement are tested according to the specification "Test Methods of Cement and Concrete for Highway

Table 1 Chemical compositions of materials

Composition	Fly ash	Mineral powder	WET carbide slag
SiO_2	44.9	29.9	3.20
Al_2O_3	42.7	18.6	1.34
CaO	4.74	34.6	93.4
Fe_2O_3	3.16	1.36	0.388
TiO_2	1.80	3.07	–
SO_3	–	2.73	0.498
MgO	–	6.92	–

Engineering" (JTG E30-2005), and all meet the requirements of the specification. See Table 2 for the test results.

Aggregates are divided into four grades, and the particle sizes of each grade are: 19–26.5 mm, 9.5–19 mm, 4.75–9.5 mm, 0–4.75 mm.

The alkali excited material is sodium hydroxide produced by Tianjin Zhiyuan Chemical Co., Ltd., which is a white translucent granular crystal.

2.2 Mix Proportion Design of Geopolymer Cementitious Materials

This study uses sodium hydroxide to stimulate fly ash, mineral powderand wet carbide slag to mix with water to prepare geopolymer cementitious materials. The total amount of materials is 2.58% with alkali, and the water-cement ratio is 0.5. The mix design is shown in Table 3. The setting time, fluidity and soundness indexes were measured, and the optimum formula was obtained by combining the compressive strength and drying shrinkage of geopolymer in 3d, 7d and 28d.

2.3 Performance Test of Geopolymer Cement Stabilized Crushed Stone

Mix proportion of Geopolymer Cement. It is necessary to control the cement dosage within a reasonable range to ensure certain mechanical properties and frost resistance and improve its crack resistance [18]. According to the recommended cement dosage value of cement stabilized material mixture ratio test in "Construction Guidelines for Highway Base and Subbase" (JTGT-F20-2015), 5% of total cement-geopolymer material dosage is proposed. Finally, the proportion of total cementitious materials is set at 5% and four groups of mixing ratios (Table 4) are determined for performance test.

Manufacture and testing of test pieces. Add a certain amount of water to the aggregate, mix well, and put it in a closed plastic bag for soaking. After soaking for four hours and within one hour before the specimen is formed, add geopolymer and cement materials with different mixing ratios, and stir well, and make the specimen within one hour; The model is made by Tiantong Tongda hydraulic part demoulding machine, and the mixture is poured into the test mold for three times and tamped evenly, and demoulded within 2–6 h. Immediately after weighing, put it in an airless plastic bag, seal it, tie the bag mouth tightly, and move it to a standard curing room with a temperature of 20 ± 2 °C and a relative humidity of over 95%; The specimen is placed on the iron frame, and the spacing is more than 10 cm. On the last day of the curing period, take out the specimen, observe whether the corners of the specimen

Table 2 Physical and mechanical properties of cement

	Fineness/%	Initial setting time/min	Final setting time/min	Soundness/mm	Rupture strength/MPa		Compressive strength/MPa	
					3 d	28 d	3 d	28 d
Test value	2.5	180	255	2.8	5.4	7.5	21.8	45.8
Specification value	≤ 10	≥ 45	≤ 600	≤ 5	≥ 3.5	≥ 6.5	≥ 17	≥ 42.5

Table 3 Geopolymer mix design

Sample number	Fly ash/%	Mineral powder/%	Wet carbide slag/%
A1	10	85	5
A2	0	88	12
A3	0	95	5
A4	20	75	5

Table 4 Design results of cement-geopolymer stabilized crushed stone mix proportions

Sample number	Proportion of geopolymer to cementitious materials (%)	Proportion of cement to cementitious materials (%)	Grading of aggregate
B1	0	100	0–4.75 mm 4.75–9.5 mm 9.5–19 mm 19–31.5 mm = 41%:14%:24%:21%
B2	30	70	
B3	60	40	
B4	90	10	

are worn or missing, and measure the quality. Then, soak the specimen in water at about 20 °C so that the water surface is about 2.5 cm above the top of the specimen.

In this paper, the unconfined compression strength, indirect tensile strength, compression rebound modulus and scour resistance tests are carried out according to "Test Methods of Materials Stabilized with Inorganic Binders for Highway Engineering" (JTG E51-2009). Cylindrical specimens with a height of 150 mm and a diameter of 150 mm are prepared, and the number of matched reference specimens in each group is 9. In the unconfined compressive strength test, the specimen should be cured for 7 days, 14 days and 28 days. During the test, the specimen should be immersed in water for one day in advance, and the universal testing machine should be used for the test. Indirect tensile strength and compressive resilience modulus test shall be conducted according to "Construction Guidelines for Highway Base and Subbase" (JTGT-F20-2015) after the specimen is cured to 28 days, 60 days and 90 days. The curing age of the anti-erosion test is 28 days, after which it is soaked for 24 h. Using the anti-erosion tester, the peak impact force is 0.5 MPa, the scouring frequency is 10 Hz, and the scouring time is 30 min.

2.4 Microscopic Analysis

X-ray diffraction (XRD) was used to analyze the phase composition of the four raw materials. The parameters were: 40 kV, 100 mA, Cu target, scanning speed 5°/min. The scanning range is 10–90; The instruments used for SEM are Hitachi S-4800 Field Emission Scanning Electron Microscope in Japan and BRUKER Energy

Spectrometer in Germany. The microscopic area of the sample is scanned by line and plane, and the distribution of elements, the types of elements, their atomic ratio and weight ratio are analyzed.

3 Results and Discussion

3.1 Mix Proportion Design of Geopolymer Cementitious Materials

The performance indexes of geopolymer cementitious materials with different mixing ratios are shown in Fig. 1. For cementitious materials, the setting time has an important influence on the setting degree and the strength of the mixture. The initial setting time should not be too early and the final setting time should not be too late. The fluidity affects the plasticity of cementitious materials, and the drying shrinkage and compressive strength are important indexes to examine the crack resistance and mechanical properties. As can be seen from Fig. 1, sample A1 has moderate initial setting time and final setting time, good fluidity and stability, low drying shrinkage coefficient and high compressive strength at 3 d, 7 d and 14 d; The initial setting time of sample A2 is relatively long, the final setting time is short, and the stability is poor. The initial setting time of sample A3 is short and its stability is poor. A4 sample has a long final setting time, poor fluidity and shrinkage. Considering all performance factors, sample A1 is selected as the mixture ratio of geopolymer cementing material.

3.2 Performance Test of Geopolymer Cement Stabilized Crushed Stone

Unconfined Compression Strength. The influence of geopolymer content change on the compressive strength of materials is shown in Fig. 2; It can be seen from Fig. 2 that, with the increase of geopolymer content, the change law of the compressive strength of the mixture at different ages is basically the same, with a slight increase at first and then a decrease. When the geopolymer content is 30%, the unconfined compression strength reaches the maximum value, and the unconfined compression strength at 7 d, 14 d, and 28 d is increased by 3.8%, 2.51%, and 1.46% compared with that at zero. When the content of geopolymer increased to 60%, the unconfined compression strength decreased sharply, and reached the minimum when the content of geopolymer was 90%, and the unconfined compression strength was only 2.40 MPa at 7 days. According to the provisions of the 7-day-old unconfined compressive strength standard of cement stabilized materials in “Technical Guidelines for Construction of Highway Roadbases” (JTG/T F20—2015), when the dosage is 30%

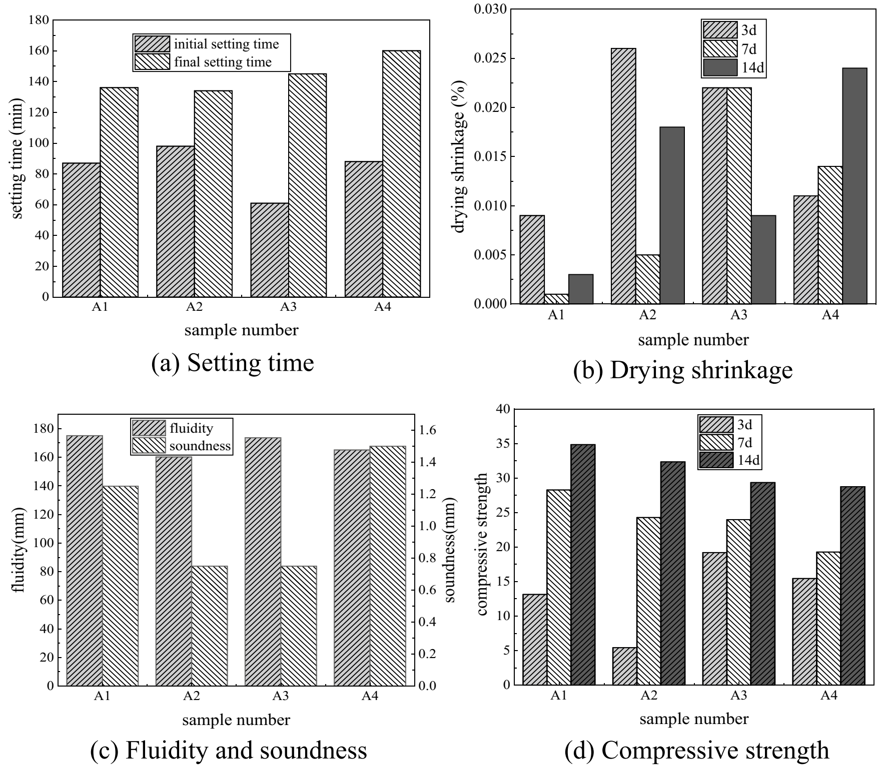


Fig. 1 Performance index of geopolymer cementitious materials with different mixing proportions

and 60%, it can meet the technical requirements of the heavy traffic volume of the second grade and below.

It can be seen from Table 1 that geopolymer materials mainly provide silicon, aluminum and calcium. Si can promote the strength of geopolymer, and Al can promote the polymerization rate. Proper Si/Al ratio can increase the strength of geopolymer, and a proper amount of calcium component can play a better role in strengthening, thus generating C–S–H gel in the system, providing more nucleation sites for the formation of geopolymer, and making the silicon and aluminum components in raw materials better dissolve out, thus accelerating the gelation process [19]. It shows that 30% geopolymer can provide high-quality Si/Al ratio, and ensure that the mineral components in cement can be fully hydrated, coagulated and hardened. With appropriate dosage of alkali activator, it can fully ensure that geopolymer can give full play to the potential activity of calcium component and silicon-aluminum component in the strength process. However, the Si/Al ratio of geopolymer is out of balance with the increase of content, and the calcium component and silicon-aluminum component can't give full play to their potential activity, which leads to a significant decrease in the strength of the mixture.

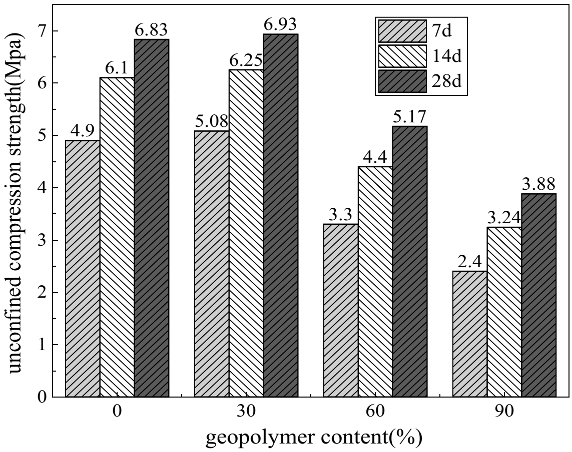


Fig. 2 Effect of geopolymer content change on the unconfined compression strength of materials

Indirect Tensile Strength. The influence of geopolymer content on the indirect tensile strength of materials is shown in Fig. 3; It can be seen from Fig. 3 that, with the increase of geopolymer ratio and the decrease of cement ratio, the change law of tensile strength at different ages is the same, showing a decreasing trend. When the content of geopolymer is 30%, the indirect tensile strength of the mixture of 28 d, 60 d and 90 d is only 0.4%, 0.8% and 1.8% lower than that of 0%. When the content of geopolymer is increased to 60% and 90%, the indirect tensile strength is obviously reduced. According to the requirements of "Specifications for Design of Highway Asphalt Pavement" (JTG D50-2017) for traffic grade, cumulative equivalent axle number Ne, and tensile strength structural coefficient, when the local polymer content is 30%, it meets the requirements of heavy traffic of grade II and below highways for pavement base.

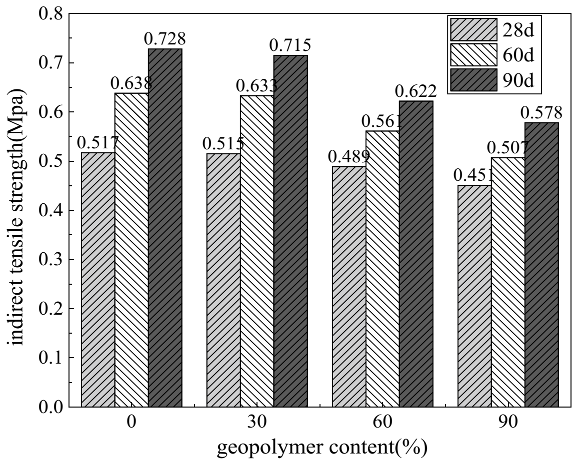


Fig. 3 Effect of geopolymer content change on indirect tensile strength of materials

The indirect tensile strength of the mixture with 30% content is approximately equal to that of the mixture with 0% content of geopolymer, because calcium oxide (CaO), silica (SiO_2) and other components in geopolymer react to form hydrated calcium aluminosilicate (C–(A)–S–H) gel [20], which can replace dicalcium silicate (C_2S) and tricalcium silicate in some cement. When the content of geopolymer increases, the calcium-based compounds in cement, mainly dicalcium silicate (C_2S), which has significant influence on the strength formation, decrease obviously, and their strength under the maximum pressure can be resisted.

Compression Rebound Modulus. The influence of geopolymer content on the compression rebound modulus of materials is shown in Fig. 4. It can be seen from Fig. 4 that at different ages, the compression rebound modulus of the mixture shows the same trend, and decreases with the increase of geopolymer content. When the content of geopolymer increases from 0 to 30%, the compression rebound modulus of the mixture decreases by 7.6%, 7.9% and 5.5% at 28 d, 60 d and 90 d, respectively, but it can be seen from Fig. 2 that the unconfined compression strength of the mixture increases by 2.8% at 28 d. This shows that adding a certain proportion of geopolymer can improve the compressive strength of the mixture, at the same time, the ability to resist vertical deformation and the crack resistance of cement stabilized macadam. However, on the premise of ensuring the strength and cementation, the elastic modulus of hydration products produced by polymer and cement is lower than that of cement hydration products. When geopolymer with low elastic modulus is added, the strength and flexibility of geopolymer mixture are improved, the rigidity is decreased, and the shrinkage deformation ability is improved. In this paper, referring to the experimental data of a large number of scholars and the calculation and analysis of Qu [21] optimum value range of compression rebound modulus of semi-rigid materials, when adding 30% geopolymer, the results are in line with the optimum value range of compression rebound modulus of semi-rigid base materials in laboratory test, and it is suitable for the performance requirements of heavy traffic volume of second grade and below.

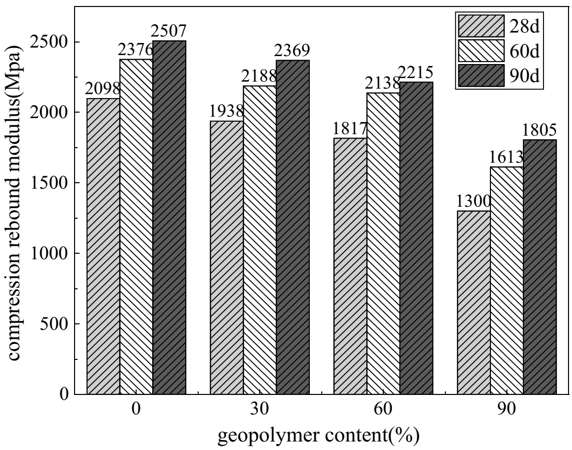


Fig. 4 Effect of geopolymer content change on compressive resilience modulus of materials

Scour Resistance. The influence of geopolymer content on scour resistance of materials is shown in Fig. 5. From Fig. 5, it can be seen that the cumulative erosion amount and erosion loss of the specimen increase gradually with the increase of geopolymer content, and the scour resistance of the specimen decreases gradually. Compared with 0% content, the erosion loss of 30% content only increases by 0.2%, and when the content increases to 90%, the erosion quality loss is 2.3 times that of 0% content. This shows that when the content of cement and geopolymer cementitious materials is increased to 30%, the difference between the cohesiveness and mechanical strength of cement and geopolymer cementitious materials is very small. When the scouring is carried out to a certain extent, the fine particles on the surface are gradually lost, and the skeleton composed of coarse particles plays a major role in bearing capacity. However, with the increase of the content of geopolymer to 90%, the mechanical properties of the mixture are greatly reduced, and the cohesiveness of geopolymer cementitious materials is insufficient, which leads to the double increase of erosion loss. According to Xiong and Gao [22] and others' research on the control index of anti-erosion performance of cement stabilized macadam base, it is found that when the content of geopolymer is 30%, it meets the requirements of heavy traffic of grade II and below highways for pavement base.

Through the unconfined compression strength test, it can be seen that the strength is the highest when the geopolymer content is 30%, and the tensile strength, compression rebound modulus and scour resistance test are similar to the mechanical properties of pure cement, and all the properties meet the requirements of heavy traffic of grade II and below highways for pavement base. Therefore, it is feasible to use geopolymer to replace part of cement, and comprehensive utilization of resources can be achieved.

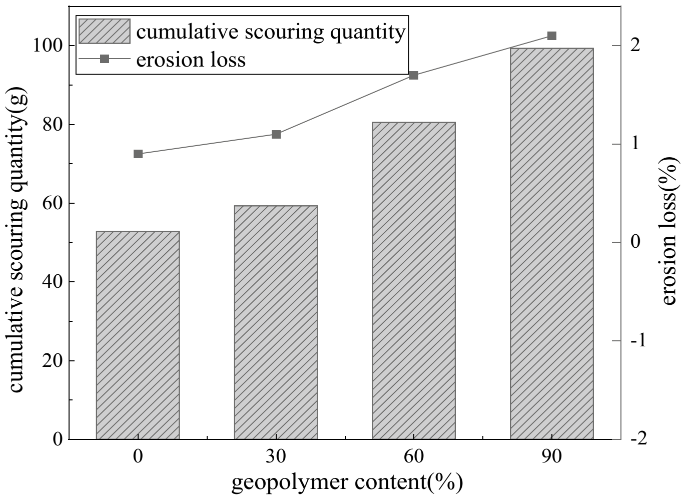


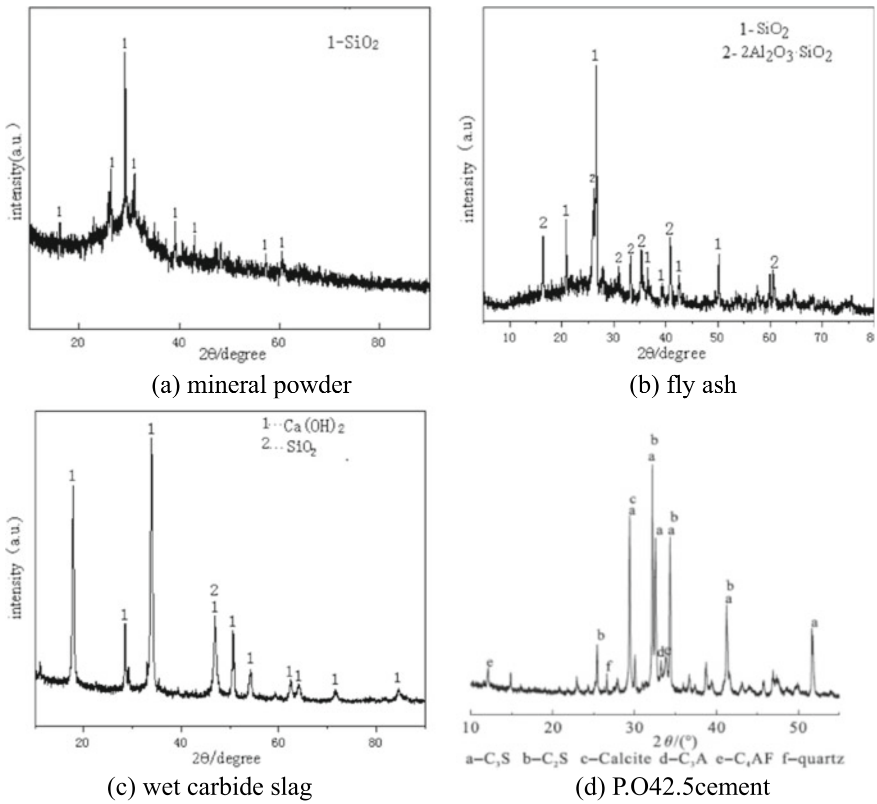
Fig. 5 Effect of geopolymer dosage change on the erosion resistance of the material

3.3 Formation Mechanism of Strength

Material Composition Analysis. The X-ray diffraction patterns of mineral powder, fly ash, wet carbide slag and cement are shown in Fig. 6.

It can be seen from Fig. 6a that the diffraction peaks at $2^{\theta} = 16.16°$, 26.38°, 29.18°, 31.08°, 39.18°, 43.04°, 57.26°, 60.38° are mainly quartz (SiO_2) crystals, and their diffraction peaks are high in intensity.

It can be seen from Fig. 6b that $2^{\theta} = 16.42°$, 25.93°, 33.19°, 35.24°, 36.52, 50.14°, 60.36° are characteristic peaks of mullite, and it can be known that the main component dried at 115 °C is mullite. Mullite mainly comes from the decomposition products of kaolin, illite and other clay minerals in coal, especially illite, which is a typical clay mineral rich in iron, potassium, sodium and magnesium. When the temperature is slightly higher, it begins to decompose into aluminosilicate. There are obvious Si diffraction peaks at $2^{\theta} = 27.97°$, 20.87°, 50.15°, and a wide diffraction characteristic peak at 20–35°, which indicates that there is vitreous material in fly ash,



(a) mineral powder (b) fly ash

(c) wet carbide slag (d) P.O42.5cement

Fig. 6 X-ray diffraction patterns of raw materials which can form a three-dimensional network structure with stable structure when it reacts with alkali activator.

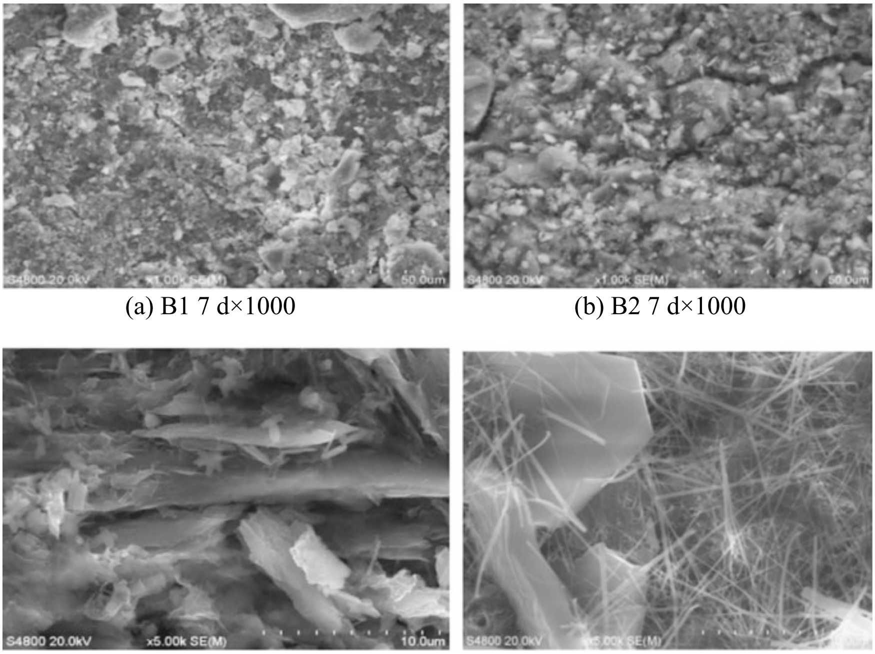
It can be seen from Fig. 6c that the phase represented by the diffraction peaks at $2^{\theta} = 17.82°, 28.48°, 33.9°, 46.9°, 50.56°, 54.16°, 62.38°$ is mainly $Ca(OH)_2$, and a small amount of quartz is contained at $2^{\theta} = 46.9°$. (SiO_2) phase crystal.

It can be seen from Fig. 6d that the main mineral components of P.O42.5 cement are tricalcium aluminate (C_3A), tetracalcium ferrate (C_4AF), dicalcium silicate (C_2S) and tricalcium silicate (C_3S).

Microcosmic Appearance. SEM *photos are* shown in Fig. 7. Figure 7a, b are SEM photos of samples with 0% and 30% content after curing for 7 days, respectively, and the magnification is 1000 times. It can be seen that there are large and small pores on the surface of the mixture, which can be better combined with the binder, and the embedding capacity among the particles will also be improved, which is conducive to increasing the strength of the mixture. Figure 7c, d are SEM photos of 30% doped samples at 7 d and 28 d, respectively, with a magnification of 5000 times. It can be seen that there are strip-shaped particles on the surface and the surface is smooth. This is because at the initial stage of hydration reaction, after cement is mixed with water, C_3A reacts quickly, and a large number of flaky calcium aluminate hydrate are quickly generated, which are connected with each other to form a structure with early strength. With the increase of age, the slow dicalcium silicate (C_2S) in the hydration reaction of cement also began to participate in the reaction. At the same time, the hydrated calcium silicate (C–S–H) and hydrated calcium aluminosilicate (C–(A)–S–H) gels produced by the geopolymer reaction wrapped the aggregate particles completely, and no exposed aggregate particles were seen.

4 Conclusion

(1) Through comprehensive analysis of fluidity, soundness, setting time and drying shrinkage of geopolymer cementitious materials with different proportions, sample A1 (85% mineral powder, 10% fly ash and 5% wet carbide slag) is the best proportion of geopolymer cementitious materials.
(2) When 30% geopolymer is used instead of cement, the unconfined compression strength of the mixture can be appropriately improved, and its compression rebound modulus can be reduced to a certain extent; And can obtain a mixture similar to the indirect tensile strength and scour resistance of cement stabilized macadam.
(3) A proper amount of silica-alumina components and calcium components in the geopolymer can generate hydrated calcium silicate (C–S–H) and hydrated calcium silicate (C–(A)–S–H) gel under the action of alkali excitation, thus creating conditions for accelerating coagulation and improving the strength of the mixture.
(4) When proper amount of geopolymer is added, the mechanical properties of the mixture can meet the anti-scour ability, meet the requirements of heavy traffic



(a) B1 7 d×1000 (b) B2 7 d×1000

(c) B2 7 d×5000 (d) B2 28 d×5000

Fig. 7 SEM pictures of sample B1 and sample B2

of grade II and below highways for pavement base and have good anti-cracking, which can provide some technical support for engineering practice.

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