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THE EFFECTS OF POLYALUMINIUM CHLORIDE (PAC) SLAG ON THE PROPERTIES OF RECYCLED CONCRETE

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In this paper, recycled concrete aggregates and PAC slag were used to prepare concrete. X-ray fluorescence (XRF), X-ray diffraction (XRD) and scanning electron microscope (SEM) were used to analyse the micro-properties of the PAC and the concrete. Furthermore, the effects of the activators on the water absorption, compressive strength and frost resistance of the concrete were studied. The results showed that the effects of the activators on the water absorption were not obvious, but they could improve the activity of the PAC slag, thus, the hydration products in the mortar significantly increased and the micro-structure of the mortar became denser. Furthermore, the compressive strength of the concrete at 28 days increased by 26.5 % when the activator was mixed with 3% water glass and 3 % sodium sulfate. Moreover, the activators could improve the frost resistance of the concrete. However, when the activators were added in the concrete together, negative effects were found on the frost resistance of the concrete, with a mass loss of 20 %.

INTRODUCTION

More and more industrial slags and waste aggregates are being generated, creating excessive waste piles. According to the national green development strategy, the main focus is on eliminating waste materials and promoting energy recycling. Poly Aluminium Chloride (PAC) is a high-quality water purification agent recognised worldwide. Its preparation process mainly uses calcined bauxite, calcium aluminate powder and hydrochloric acid to obtain liquid PAC, and the precipitated solids form a waste slag, whose main components are SiO_2, Al_2O_3, CaO, etc [1-2].

The activation of the slag is beneficial in improving the workability of the concrete and recycling of the slag, helping modern factories to eliminate their dependence on cement and stone. The study [3] has shown that PAC slag will have similar properties to metakaolin after treatment by water washing and low temperature calcination. Several researchers have conducted deep and long-term experiments on alkaline slag and slag concrete, focusing on the workability to the hydration products of concrete. The results show that the addition of alkali slag and slag effectively enhances the performance of the concrete [4-6]. Yang [7] found that the 28-d compressive strength of recycled concrete treated with 5 % water glass was enhanced by 29 %. The study by Wu [8] found that the change in the compressive strength of ordinary concrete and recycled plastic concrete eroded at 3 % and 5 % concentrations was less than that of ordinary concrete. Fang [9] found that alkali activated fly ash-slag formed a compact and dense microstructure in the interfacial transition zone (ITZ) at 28 days, which would benefit the long-term performance of the concrete. The existing research has amply demonstrated that the combination of activators, such as water glass and slag, can improve the workability of the concrete, but for weaker aggregates, there is still research to be undertaken.

The combination of recycled aggregates and industrial slag can be a solution to the problem of waste aggregate affecting the environmental impact. Several scholars have analysed the alkali-active slag concrete containing recycled coarse aggregate from slag morphology, different external activators, long-term work performance, etc., and concluded that the recycled aggregate has a particular effect on the slag concrete, but the excessive addition of recycled aggregate will have an adverse effect on it [10-12]. Farhan and Nanayakkara [13,14] found that when the compressive strength of concrete prepared by combining an alkali-activated slag and a rebiotic after activator treatment was comparable, where the tensile strength was higher than that of plain concrete, but the internal structure and homogeneity were poorer. Wang[15] used recycled concrete powder to replace blast furnace slag in the preparation of an alkali-activated slag mortar. The results showed that the addition of the recycled concrete powder increased the compressive strength of the mortar, but it showed a negative effect on the 28-day strength. Berndt[16] found that slag was particularly beneficial for concrete containing recycled aggregates, reducing the strength loss. Yang [17] found that the tensile strength of the new concrete was mainly dependent on the tensile strength of the alkali-excited slurry and the recycled coarse aggregate. Cui [18] found that the essence of the freeze-thaw damage of waste-doped composite powder concrete is the reason that there is a tendency for the microstructure to lose stability causing a physical change in the process of the gradual deterioration of the pore structure, which is the process of accumulating damage caused by cracks, pores and other defects inside the specimen, expanding until destruction.

The current research shows that the excitation of the slag activity significantly enhances the workability of slag concrete, but the combination of a new slag and recycled concrete is yet to be studied. In this paper, two methods of single and multiple doping of excitants have been designed, a PAC slag was used to replace 15 % of the cement and recycled aggregates were used to replace 50 % of the natural aggregates to produce an externally blended PAC slag recycled concrete. The macroscopic properties of the modified PAC slag recycled concrete were studied, combined with microscopic test data to analyse the performance change mechanism. The results of the study provide a basis for analysing the workability and mechanical properties of PAC slag recycled concrete.

a)

b)

Figure 1. (a) recycled aggregate and (b) Polyaluminium chloride slag.

EXPERIMENTAL

Materials

The raw materials of the concrete in this test include cement, fly ash, standard sand, PAC waste residue, a polycarboxylate superplasticiser and an activator. The cement is P.O 42.5 ordinary silicate cement, and the properties of the cement are shown in Table 1. The recycled coarse aggregate was mechanically crushed and washed to remove any debris, where the crushed stone, in a range of 5 mm to 20 mm in particle size, was screened for use as recycled aggregate, with a saturation density of 2.729 g·cm^{-3}, water absorption of 4.1 % and crushing value of 11.4 %, as shown in Figure 1. The PAC slag comes from polyaluminium chloride slag pre-treated by Henan Gongyi Xiangji Assembly components Co., Ltd., as shown in Figure 1. The test activator is sodium sulfate and water glass.

The PAC slag was dried in an oven for 5 hours at a constant temperature of 95 °C. Because the PAC slag contains gravel and some debris, it needs to be crushed into a particle size range of 0 – 0.075 mm. The main components of the PAC slag are shown in Table 2.

Specimen mixes

In order to study the effect of the activator on the recycled concrete from the PAC slag, this paper designed two methods of single and compound mixing of the activator at three different ratios. The PAC slag with a particle size below 0.075 mm was selected to replace 15 % of the cement and the recycled coarse

Table 1. Physical and mechanical properties of the cement.

	Density (g·cm^{-3})	PSA (m^2·kg^{-1})	Coagulation time (min)		Compressive strength (MPa)		Flexural strength (MPa)	
			Initial set	Final set	3d	28d	3d	28d
P.O 42.5	3.1	412	167	233	23	51.6	4.9	7.3

Table 2. X-ray fluorescence test results.

Number	Chemical formula (%)							
	SiO_2	Al_2O_3	CaO	TiO_2	Fe_2O_3	MgO	Cl^-	Other
PAC	45.72	27.80	5.68	3.86	3.42	1.34	2.25	10.40

Table 3. Mixing ratio (kg·m^{-3}).

Number	Cement	Sand	Naturals aggregates		Recycled aggregates		PAC	Sodium sulfate	Water glass	Water Reducer	Water
			5 – 10	10 – 20	5 – 10	10 – 20					
H1	376.25	655.64	405.87	811.74	-	-	0	0	0	0.5 %	150.5
H2	338.63	655.64	202.94	405.87	202.94	405.87	15 %	0	0	0.5 %	150.5
H3	338.63	655.64	202.94	405.87	202.94	405.87	15 %	0	4 %	0.5 %	150.5
H4	338.63	655.64	202.94	405.87	202.94	405.87	15 %	5 %	0	0.5 %	150.5
H5	338.63	655.64	202.94	405.87	202.94	405.87	15 %	3 %	3 %	0.5 %	150.5

aggregate was used to replace the natural aggregate to prepare the PAC slag recycled concrete. H1 without any external activators and recycled aggregates was used as the base group, the proportion of the PAC slag replacing the cement was 15 % and the proportion of the recycled coarse aggregates replacing the natural aggregates was 50 % in all the control groups. The proportions are shown in Table 3.

Test Method

This experiment uses three test methods to test the specimens, water absorption, compressive strength and freeze-thaw cycle. These tests used 100 mm × 100 mm × 100 mm cubes where the macro-mechanical properties of the specimens were tested. We also use X-ray diffraction (XRD) and Scanning electron microscope (SEM) tests to microscopically test the specimens, so that we can better analyse the PAC slag recycled concrete.

The water absorption rate: the dried specimen was soaked in water at room temperature (25 °C). The water level was 20 to 30 mm above the sample. 24 hours later, the samples were removed, then wiped dry and immediately weighed, using the equation as below:

$$W = \frac{m_1 - m_0}{m_0} \times 100 \quad (1)$$

where W is water absorption of the specimen (%), m_0 is drying mass of the specimen (g) and m_1 is mass of the specimen after 24 h of water absorption (g).

Freeze-thaw cycle: The specimens were placed in a freeze-thaw damage chamber. The specimens were subjected to D25, D50 and D75 cycles to observe the appearance of the specimens and to test the rate of mass loss per the equation as below:

$$M_S = \frac{M_0 - M_t}{M_0} \times 100 \quad (2)$$

where M_S is mass loss rate of the specimen (%), M_0 is initial mass of the specimen with an accuracy of 0.1 g and M_t is mass of the specimen at the time with an accuracy of 0.1 g.

SEM and XRD were carried out using Field Emission Environmental Scanning Electron Microscopy and a Bruker X-ray diffractometer, respectively. The SEM specimens were removed from the central area of the damaged cement mortar for test observations. The XRD specimens were ground into a powder and then pressed into the machine through a glass sheet.

RESULTS AND DISCUSSION

Water absorption rate of the PAC slag recycled concrete

As can be seen in Figure 2, the water absorption of the concrete is below 6.0 % for all the mix ratios, which meets the specification requirements for a superior product (≤ 6 %). The water absorption of the baseline group H1 was 3.5 % and there was a small decrease in the water absorption with the incorporation of the PAC waste and recycled aggregates, with a water absorption of 3.3 %. The magnitude of the water absorption in the concrete is mainly related to the porosity of the components. When the PAC slag powder is substituted for the cement in mortar, the PAC slag has a low activity. A reduction in the amount of hydration products within the mortar and a reduction in the amount of C-S-H and AFt filling the pores, the reduced structural compactness and increased porosity of the mortar will also lead to increased shrinkage of the mortar. However, the recycled coarse aggregate has a high water absorption rate and the external water is absorbed by the recycled aggregate through the capillary pores within the concrete, so the reduction in the water absorption rate is not significant.

The water absorption rates of H3, H4 and H5 all decreased, but the decrease was not significant compared to H2, indicating that the activator had

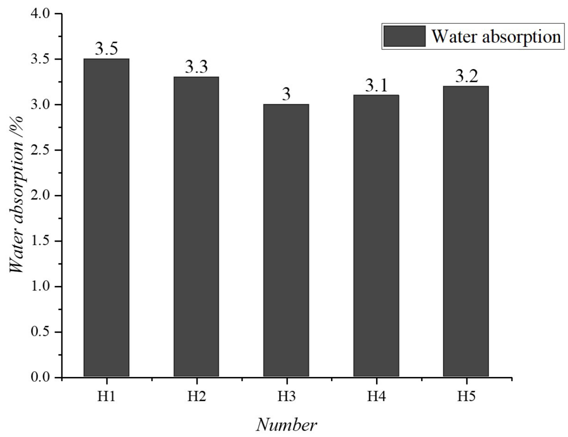


Figure 2. Water absorption.

a small effect on the water absorption rate of the PAC slag recycled concrete. The decrease in the water absorption is mainly due to the internal pore space being improved by the PAC slag in the presence of water glass and sodium sulfate.

Table 4. Test results of the curb stone concrete.

Number	PAC slag dosage (%)	Sodium sulphate (%)	Water glass (%)	Water absorption (%)
H1	0	0	0	3.5
H2	15	0	0	3.3
H3	15	0	4	3.0
H4	15	5	0	3.1
H5	15	3	3	3.2

Compression strength of the PAC slag recycled concrete

The water absorption and compressive strength of the concrete at the different mix ratios are shown in Table 4.

Comparing the 7 d and 28 d compressive strengths revealed that the water glass and sodium sulfate significantly increased the mechanical properties of the PAC slag recycled concrete. The 7 d and 28 d compressive strengths of H1 are 40.5 MPa and 44.9 MPa, respectively, meeting the strength requirement of C35. The 7 d and 28 d compression strength of H2 were 34.1 MPa and 37.9 MPa, respectively, showing a significant decrease in the compression strength. However, the minimum requirement of a specimen with a 28-d average strength greater than 35.0 MPa is met. Every index of the recycled coarse aggregate is lower than that of the natural coarse aggregate, and the weak section of the residual old mortar and the new concrete mortar on the surface of the recycled coarse aggregate leads to the deterioration in the compressive strength. The 7 d and 28 d compression strength of H3 were 37.4 MPa and 40.6 MPa, respectively, the 7 d and 28 d compression strength of H4 were 38.7 MPa and 41.6 MPa, respectively, and the 7 d and 28 d compression strength of H5 were 39.6 MPa and 43.9 MPa, respectively. The compressive strength of H3 ~ H5 shows an increasing trend, where the incorporation of the activator has a significant effect on the compressive strength of the PAC slag recycled concrete, due to the effect of the activator on the PAC slag material and the mortar.

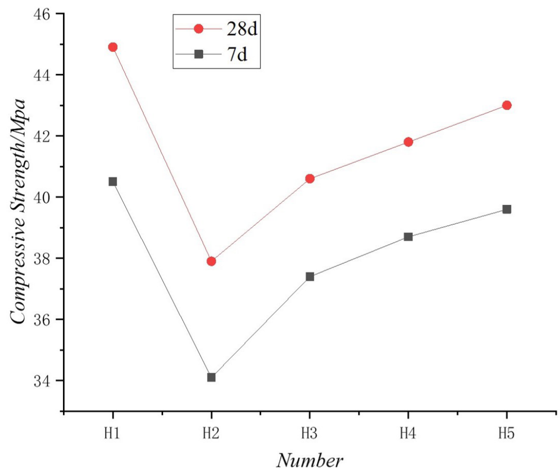


Figure 3. Water absorption.

As can be seen in Figure 3, when the water glass was mixed at 4 %, the 7-d compressive strength of H3 increased by 12.9 % compared to H2, while the 28-d compressive strength increased by 19.4 % compared to H2. When sodium sulfate was mixed at 5 %, the 7-d compressive strength of H4 increased by 16.9 % compared with H2, while the 28-d compressive strength increased by 22.9 % compared with H2. When water glass was compounded with sodium sulfate at 3 % each, the 7-d compressive strength of H5 was increased by 19.6 % compared to H2, while the 28-d compressive strength was increased by 26.5 % compared to H2. The effect of the activator compound on the concrete is significantly better than that of the single activator, which can raise the strength of the PAC waste recycled concrete to essentially 96 % of the strength grade of the natural aggregate.

It has been proven through research that the incorporation of an activator can significantly increase the activity of PAC slag materials, the hydration products in the cement slurry are significantly increased and the slurry structure is dense and continuous, which is the main reason for the increase in the compressive strength. Water glass, as a strong alkaline activator, accelerates the process of hydration in the cement in the mortar, producing a large amount of C-S-H gel, and the rapid reaction of the active substances in the PAC slag with water glass, which leads to the formation of new hydration nucleation sites, resulting in the faster development of the early strength.

However, too rapid a hydration reaction can also reverse the dispersion of the hydration products, with the surface of the PAC slag particles being wrapped in hydration products and their internal structural organisation not being sufficiently dense. When sodium sulfate is added into the concrete mortar as the activator, when an alkaline environment exists, active substances, such as Ca^{2+} in the slag powder, will dissolve rapidly as the pH rises, and the presence of Na_2SO_4 will cause Ca^{2+} to polymerise with silicates and aluminates in the mortar to form substances such as AFt.

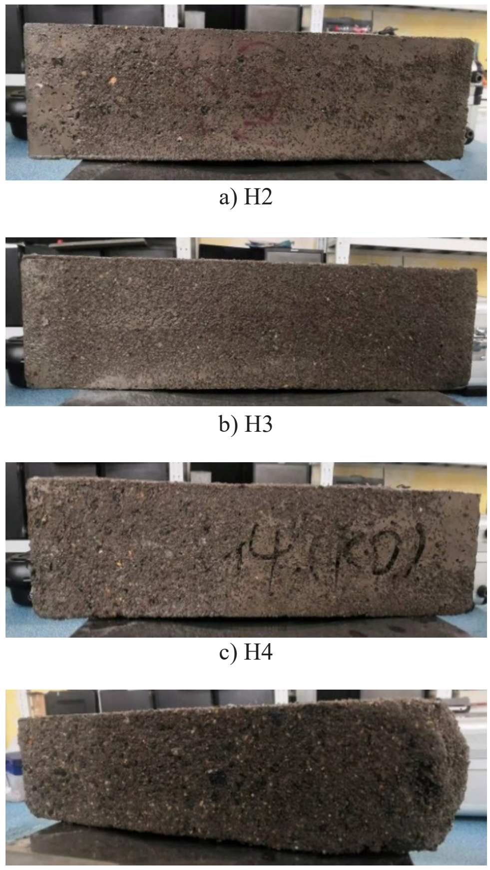
When the recycled coarse aggregate is added instead of the natural aggregate, the strength decreases due to the change in the internal skeleton supporting structure of the concrete, but the strength growth effect of the PAC slag and activator mixed into the concrete is greater than that of the recycled coarse aggregate, and it has an obvious effect on the early and later strength of the concrete.

Freeze-thaw of the PAC slag recycled concrete

The H3, H4 and H5 groups of specimens were damaged by freeze-thaw cycles, the appearance of the specimens changed significantly, the outer skin of the specimens was completely damaged and peeled off, and the area damaged and peeled off increased with the number of freeze-thaw cycles. The quality loss of the H5 specimen after D50 times was close to the upper limit of the specification, with the complete bare leakage of the aggregate, the loose and porous structure of the specimen, and a strength loss rate of 20.0% after D75 times, in which the external integrity of the specimen was completely destroyed.

According to the freeze-thaw cycle, the D50 mass loss is given in Table 5, the quality loss of H1 – H4 all met the specification requirement of less than 3.0 %, and the freeze-thaw cycle D50 quality loss of H2 – H5 are all higher than those of the reference group H1.

As can be seen in Figure 5, all the curves show an increasing trend, but the mass loss of the re-doped specimens of H5 is higher than that of the other groups at the different numbers of cycles. This shows that the combination of compound activators has a large effect on the freeze-thaw performance of the concrete, probably because the concrete absorbed a large amount



a) H2

b) H3

c) H4

d) H5

Figure 4. The D75 specimen.

of the solution leading to the internal collapse, resulting in a sharp deterioration in the frost resistance. After D50, H3 and H4 both showed signs of a delayed trend and both had a lower quality loss than H2, indicating that the water glass and sodium sulfate mixture had some inhibiting effect on the freeze-thaw damage of the PAC slag recycled concrete.

As can be seen from Figure 6, D50 shows a decreasing, then increasing and then decreasing trend. The first drop indicates that the addition of recycled aggregate has a negative effect on the frost resistance of the concrete, mainly because the recycled aggregate consists of crushed aggregate and already cured concrete mortar with a porosity greater than that of the natural material, and during the freeze-thaw cycle, the internal interface of the recycled coarse aggregate has a relatively

Table 5. Rate of the mass loss and strength loss.

Number	Mass loss (%)			28 d Compression strength (MPa)		rate of strength loss (%)
	D25	D50	D75	D0	D50	
H1	0	0.6	0.8	44.9	44.0	2.0%
H2	0	0.8	1.0	37.9	36.0	5.0%
H3	0.1	0.2	0.5	40.6	36.6	9.8%
H4	0.2	0.3	0.7	41.8	37.6	10.0%
H5	1.8	2.9	5.2	43.0	34.4	20.0%

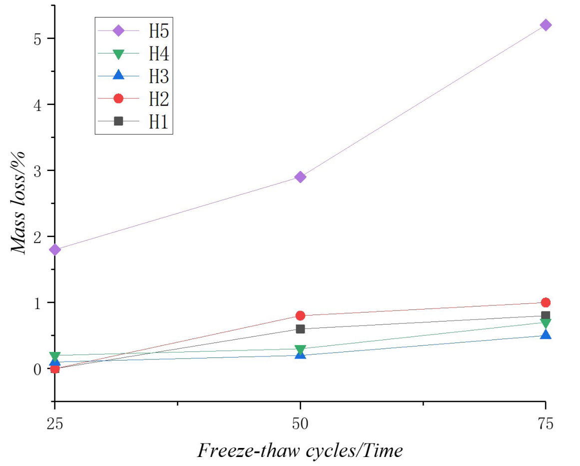


Figure 5. Mass loss.

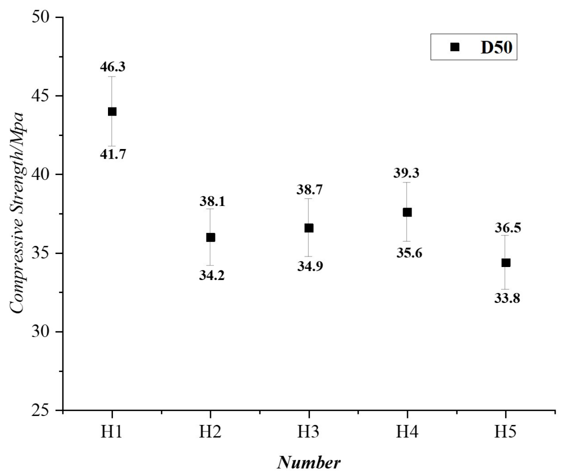


Figure 6. Compressive strength of the freeze-thaw tests.

weak bond between the old and new mortar. The action of water glass and sodium sulfate on the hydration products allows the hydration products to more fully fill the pores in the recycled aggregate, improving the pore structure of the recycled aggregate, leading to an increase in the denseness of the aggregate and enhancing the frost resistance of the concrete. The other drop indicates that the mixed activators have a negative impact on the frost resistance of the concrete.

XRD of the PAC slag recycled concrete

XRD tests were carried out on the experimental group to analyse their main mineral composition, with specimens aged 3 d and 28 d. The results of these tests are shown in Figure 7.

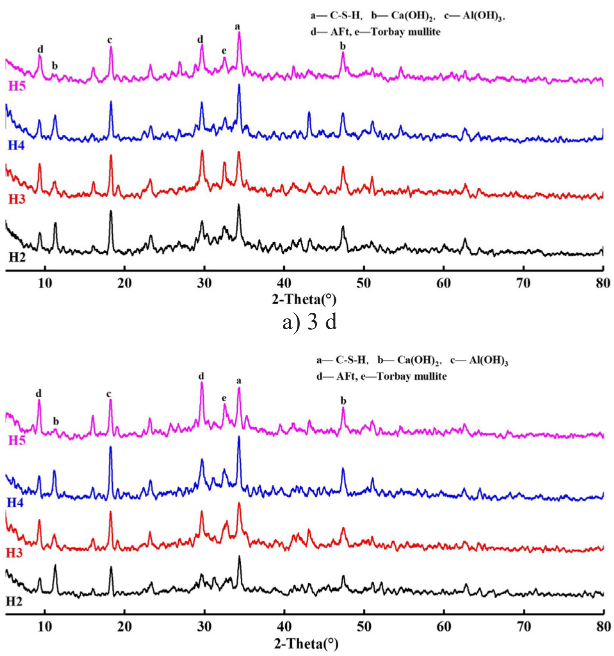
As can be seen in Figure 7a, the main hydration products of the mortar are hydrated calcium silicate gel (C-S-H) and calcium hydroxide ($Ca(OH)_2$), with significant amounts of aluminium hydroxide ($Al(OH)_3$). Comparing H2 with H3, H4 and H5, it is obvious that the wave peaks of $Ca(OH)_2$ decrease significantly after doping with the exciter, while the number and height of the C-S-H wave peaks increase significantly. This indicates that, under the influence of the exciter, the increase in the pH value of the slurry caused the precipitation of the active substances (SiO_2, Al_2O_3) to react with the $Ca(OH)_2$ generated by the hydration of the silicate cement, generating more hydration products and promoting the hydration of the cementitious materials in the mortar [19], which can enhance the mechanical properties. Due to the destruction of the covalent bonds in the stable glass phases (e.g., Si-O-Si, Al-O-Al) in the PAC slag, thus, transforming the product into an unstable state, the purpose of increasing the activity of the PAC slag is achieved [20]:

$$\equiv Si-O-Si\equiv +3OH^- \rightarrow [SiO(OH)_3]^-$$

$$\equiv Si-O-Al\equiv +7OH^- \rightarrow [SiO(OH)_3]^- + [Al(OH)_4]^-$$

$$2[Al(OH)_4]^- + 3SO_4^{2-} + 6Ca^{2+} + 4OH^- + 26H_2O \rightarrow 3CaO\cdot Al_2O_3\cdot 3CaSO_4\cdot 32H_2O$$

The water glass, as a strong alkaline exciter, accelerates the hydration reaction, generating more C-S-H gels and also converting the high sulfur type of hydrated calcium sulfate to the monosulfur type, which causes a conversion that results in a small reduction in material strength [20].



a) 3 d

b) 28 d

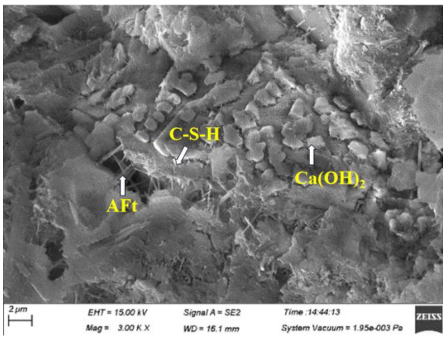
Figure 7. Results of the XRD tests.

As seen in Figure 7b, at this point in the mortar, the main hydration products are calcium alumina (AFt) and C-S-H gel. In the early stages, these two hydration products increase with the amount of incorporated exciter, and the AFt and C-S-H gels fill the microscopic pores of the mortar matrix species, improving the structural density and strength. However, some studies [20-22] showed that the $Ca(OH)_2$ generated after increasing the amount of the sodium sulfate admixture caused microcracking and swelling damage inside the mortar. Because AFt has certain swelling properties as a hydration product [20-22], it can fill into the pores inside the PAC waste-cement cementitious sand specimens and improve the mechanical properties of the material. However, too much AFt filling the pores can cause destructive stresses due to the volume expansion, resulting in a reduction in the strength of the specimen. The incorporation of sodium sulfate results in an increase in the quantity of C-S-H, the main hydration product of the mortar, which has a significant effect on the early and later strength of the mortar.

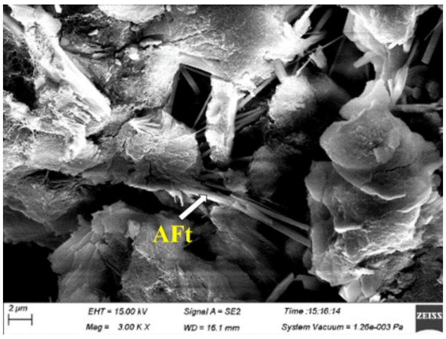
SEM of the PAC slag recycled concrete

As can be seen in Figure 8, the pores in the H2 matrix structure are filled by a small amount of fibrous C-S-H gel, with a small amount of flocculated C-S-H and bulk $Ca(OH)_2$ attached to the matrix bulk structure, while the hydration is still in progress. Comparing the microscopic morphology of H2 and H3, it was found that the fibrous C-S-H increased in H3, filling the pores of the concrete mortar, and the smaller bulk $Ca(OH)_2$ disappeared, indicating that the water glass activator promoted the hydration process of the mortar. H3 shows that the surface of PAC slag particles is attached by the hydration products, and a small amount of rod-like AFt penetrates through the particles, indicating that the active substances SiO_2 and Al_2O_3 are released when the water glass is incorporated into the PAC slag. Compared with H4, it was found that the hydration products are flocculent C-S-H gel, attached to the elementary body, and layered $Ca(OH)_2$. The increased number of fibrous gels in H4 filled the pores of the elementary body and a higher number of grape-like C-S-H gels were found wrapped around the PAC slag particles in H4, indicating that the activity of the PAC slag material was enhanced by the sodium sulfate at this time. A large number of small pieces of $Ca(OH)_2$ appear in H5, and this $Ca(OH)_2$ is then attached to a large number of flocculent C-S-H gels, and no pores or cracks appear in the elementary body, so that the mechanical properties of the material are at an optimum.

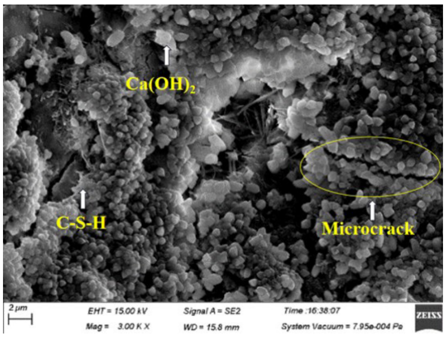
As can be seen in Figure 8, the elementary body of H2 is loose and porous, with flocculent C-S-H gels filling the pores and the bulk hydration product being $Ca(OH)_2$. Observing H3 revealed an increase in the denseness of the mortar elementary body, which is the main reason for the improved mechanical properties of the concrete; with the addition of water glass, the mortar elementary body is well encapsulated by the hydration products. Observing H4 revealed that the degree of denseness of the mortar elementary body gradually increased with the addition of sodium sulfate and that the hydration products were well aggregated. No obvious PAC slag particles were observed, only a small number of microcracks, and $Ca(OH)_2$ was observed dispersed as a whole on the surface of the matrix, indicating that the mixing of the PAC



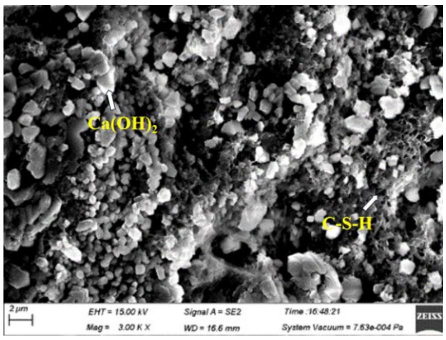
a) H2 – 3 d



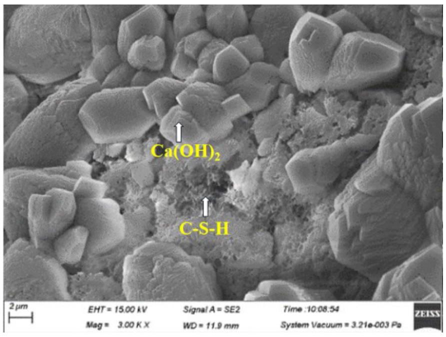
b) H3 – 3 d



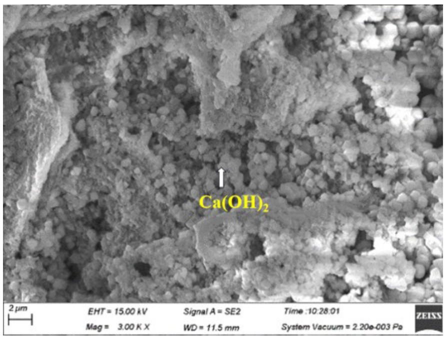
c) H4 – 3 d



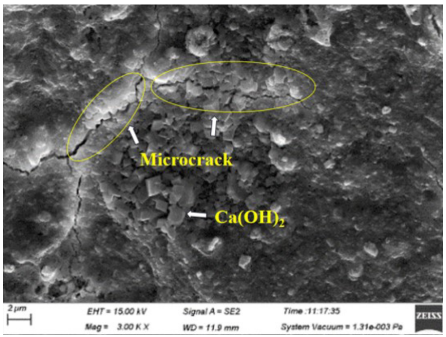
d) H5 – 3 d



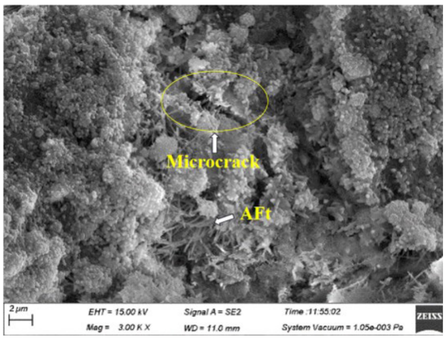
e) H2 – 28 d



f) H3 – 28 d



g) H4 – 28 d



h) H5 – 28 d

Figure 8. SEM test results.

slag with the cement slurry as a continuous and dense whole under the excitation of 5 % sodium sulfate, with a good degree of crystallisation of the hydration products, was the main reason for the improved mechanical properties of the concrete mortar. Observing H5 shows that the hydration process is extremely adequate and the hydration products are abundant and dense, but there are more microcracks, which contribute to the reduced frost resistance.

CONCLUSIONS

In this paper, the compressive strength, water absorption and freeze-thaw cycles were tested by designing different proportions of PAC waste slag recycled concrete, and the following conclusions were obtained:

(1) The specimens with 3% water glass and 3 % sodium sulfate were stronger than all the other specimens, but not as strong as the natural aggregate concrete. Recycled coarse aggregates replaced the natural aggregates and the internal skeletal structure of the concrete changes leading to a reduction in strength. When mixing PAC slag into the recycled concrete, through the excitation of water glass and sodium sulfate, there is a significant enhancement of the internal pore structure and an increase in the early and later strength of the concrete.

(2) In the freeze-thaw cycle experiments, the specimens mixed with 3 % water glass and 3 % sodium sulfate had the lowest strength and the highest mass loss. The mixture of water glass and sodium sulfate negatively affected the frost resistance of the PAC slag recycled concrete. However, the results from the single admixture showed that the single admixture of 4 % water glass and 5 % sodium sulfate had an inhibitory effect on the freeze-thaw damage.

(3) The microscopic tests show that water glass and sodium sulfate promote the formation of C-S-H and AFt, which has an effect on the internal pores and results in a dense matrix structure. Moreover, it also stimulates the hydration process between the PAC slag and the concrete, so that the PAC slag is mixed with the cement slurry as a continuous dense whole.

Acknowledgments

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