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Long term mechanical properties of alkali activated slag

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Abstract. This article reports a study on the microstructural and long-term mechanical properties of the alkali activated slag up to 180 days, and cement paste is studied as the comparison. The mechanical properties including compressive strength, flexural strength, axis tensile strength and splitting tensile strength are analyzed. The results showed that the alkali activated slag had higher compressive and tensile strength, Slag is activated by potassium silicate (K_2SiO_3) and sodium hydroxide (NaOH) solutions for attaining silicate modulus of 1 using 12 potassium silicate and 5.35%sodium hydroxide. The volume dosage of water is 35% and 42%. The results indicate that alkali activated slag is a kind of rapid hardening and early strength cementitious material with excellent long-term mechanical properties. Single row of holes block compressive strength, single-hole block compressive strength and standard solid brick compressive strength basically meet engineering requirements. The microstructures of alkali activated slag are studied by X-ray diffraction (XRD). The hydration products of alkali-activated slag are assured as hydrated calcium silicate and hydrated calcium aluminate.

1. Introduction

Alkali activated slag is a type of cementitious material through the chemical reaction obtaining [1-3], with slag and alkaline activators such as sodium silicate, NaOH as the main raw materials, with or without a certain cold heat history of aluminate natural minerals containing silicon or industrial waste residue (e.g metakaolin, phosphorus slag, steel slag, coal gangue, fly ash, red mud, etc) and alkaline activators (such as alkali metal hydroxide, alkali metal carbonate, alkali metal sulfate, etc). It uses industrial waste slag as the raw material and has the advantage of high temperature resistance, corrosion resistance, energy conservation and environmental protection, and has aroused the wide concern of scholars both at home and abroad [4-6]. Since the 1980s in China, alkali activated slag has begun to be studied and also made a lot of research results [7-10]. But its long-term mechanical properties still hasn't been published, and the guide specifications and standardsis on alkali activated slag are rarely seen at home and abroad. Moreover, the lack of detailed and reliable technical indicators greatly hinders its popularization and application. Therefore, this essay studies on the long-term mechanical properties of the alkali-activated slag. The results indicate that alkali activated slag is a kind of rapid hardening and early strength cementitious material with excellent long-term mechanical properties. The microstructures of alkali activated slag and cement paste are studied by X-ray diffraction (XRD). The hydration products of alkali-activated slag are assured as hydrated calcium silicate and hydrated calcium aluminate.

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2. Materials and Formulation

2.1. Raw materials and mix proportion

S95 slag is proposed by mass fraction of 36.9% SiO_2, mass fraction of 15.66% Al_2O_3, mass fraction of 37.57% CaO, mass fraction of 9.3% MgO, mass fraction of 0.36% Fe_2O_3.The activity indexes of slag are mainly: the quality coefficient 1.69, the alkaline coefficient 0.97, and the activity coefficient 0.42.The main parameters of potassium silicate solution are shown in table 1.The mass fraction of sodium hydroxide is not less than 96.0%.

Table 1. Parameters of potassium silicate solution.

Baume degree	Density	Modulus	mass fraction, %	
			K_2O	SiO_2
46.3	1.465	2.76	15.98	28.15

[a] Note: Baume degree for potassium silicate is the unit of density for liquids measured by hydrometer.

P.O42.5 ordinary Portland cement with mass fraction of 94.5% SiO_2 is used to prepare cement paste (OPC), which is analyzed as counterpart. Test uses two relatively optimal mixing ratio (mixing ratio is various mass fractions of raw materials) W35, W42 of alkali activated slag, and main parameters are shown in table 2.

Table 2. The proportion parameters used for text.

Number	Slag	Cement	Modulus of potassium silicate	potassium silicate amount, %	Sodium hydroxide,%	Water amount %
W35	1	0	1.0	12	5.35	35
W42	1	0	1.0	12	5.35	42
OPC	0	1	0	0	0	25

2.2. Production and maintenance of specimens

The samples were prepared by mixing raw materials--slag and alkaline activators in a 5 liter pan mixer. The sequence of mixing was important to homogenize the fresh paste. Thus, the raw materials were firstly mixed for about 30 s. the alkaline activators and water were then poured into the pan, and stirred at low speed for approximately 6 min until the mixture was glossy and well combined. The fresh paste was cast in various shapes of moulds for measurement of strength. The OPC control samples with a water/binder ratio of 0.25 were machined to the same size for comparison. They were maintained in a humidistat at 20 ± 2℃ and 95% relative humidity. The samples were demolded after 24 h of humidity maintaining and then stored in a humidistat at 20 ± 2℃ and 50% relative humidity until testing commenced.

3. The strength varies with time

3.1. Flexural strength

Figure 1 shows the test results of flexural strength of paste with time, it can be seen that with the increase of the age, the flexural strength of alkali activated slag is increasing. Before 14d, the flexural strength developed rapidly, and after 28d, the flexural strength developed gradually. The flexural strength of the W35 specimen was significantly higher than that of W42 and OPC. The damage process of alkali-activated slag specimen is significant. With the increase of load, a clear crack is appeared in the edge of the test piece. When the ultimate load is reached, the specimens are damaged.

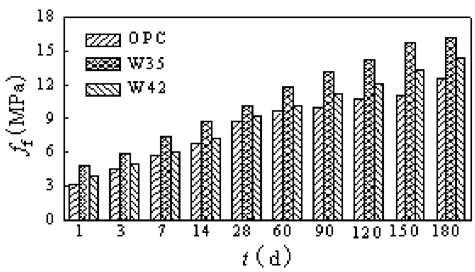


Figure 1. Flexural strength of cementing materials.

3.2. Compressive strength of cementing materials

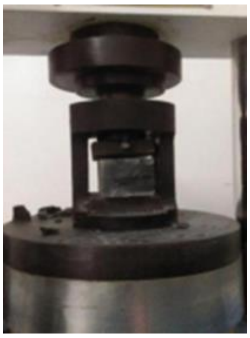
Table 3 and table 4 show the compressive strength of W35 and W42. Figure 2 shows test apparatus and variation curves of compressive strength of cementing materials with time. It can be seen from figure 2 that at 1d ~ 3d, the development of compressive strength of alkali-activated slag is fastest. Compared with the compressive strength of 28d, the compressive strength of 1d and 3d can reach 54% and 72%, respectively, and its compressive strength at 3d ~ 7d was slow, and its compressive strength of 7d was 88%. After 7d, its compressive strength of was slower, but the strength continued to increase with time. In addition, its strength tended to stabilize until 120d ~ 180d. Under the same curing condition, the compressive strength of alkali-activated slag is significantly better than that of OPC. Test proves that alkali-activated slag is a kind of cementitious material with fast hardening speed and high early strength.

Table 3. Compressive strength of W35.

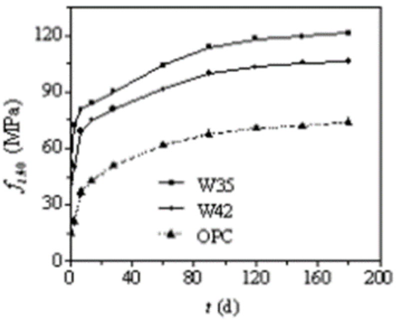
Age （d）	Compressive strength of W35 （MPa）						$f_{l.40}$ （MPa）
	A01	A02	B01	B02	C01	C02	
1	50.42	52.90	54.25	52.59	51.38	52.66	52.37
3	67.06	67.31	72.82	77.35	73.63	77.88	72.67
7	77.25	79.31	80.25	82.56	82.13	82.65	80.69
14	82.55	84.63	81.75	83.54	85.67	84.98	83.85
28	85.50	90.48	89.87	90.91	92.82	91.38	90.16
60	101.40	103.81	105.12	104.38	104.82	105.49	104.17
90	111.75	113.86	112.89	114.03	115.64	114.80	113.82
120	116.39	118.57	118.88	117.81	119.36	118.63	118.27
150	118.14	119.23	120.75	121.62	118.77	119.84	119.73
180	120.59	122.65	119.44	120.86	122.24	121.31	121.18

Table 4. Compressive strength of W42.

Age （d）	Compressive strength of W42（MPa）						$f_{l.40}$ （MPa）
	D01	D02	E01	E02	F01	F02	
1	35.86	34.33	35.61	32.35	35.02	38.24	35.23
3	49.41	42.35	48.84	48.59	51.89	50.05	48.52
7	66.60	69.78	68.48	67.03	71.87	69.32	68.84
14	76.25	75.12	80.38	80.36	78.09	76.64	77.80
28	80.81	79.88	79.56	78.94	85.97	80.13	80.88
60	89.96	90.52	92.04	93.27	91.17	90.62	91.26
90	98.77	100.31	99.84	100.19	98.90	99.64	99.61
120	101.22	103.79	102.27	104.38	103.95	102.48	103.02
150	104.56	105.25	107.57	106.15	105.56	104.31	105.57
180	105.84	106.08	107.27	106.13	104.75	106.15	106.04



(a) Test apparatus



(b) Curves of compressive strength

Figure 2. Compressive strength of cementing materials.

3.3. Compressive strength of cube

Table 5 shows the test results of compressive strength of cube with time. From table 3, it can be seen that the cube compression strength of W35 is slightly higher than that of W42. During the determination of the compressive strength of the cube specimens, the test phenomenon is that when the ultimate load is near the limit load, the specimen will make a large, brittle sound and crack. alkali-activated slag is similar to the ordinary concrete compressive test pieces, and the destruction form of alkali-activated slag specimen has two opposite cones, and the details are shown in figure 3. Curing age for 180d, the highest compressive strength of cubic specimens is 96.43MPa of W35 specimens.

Table 5. Test results of compressive strength of cube.

Age （d）	Compressive strength of W35			$f_{cu.70.7}$ (MPa)	Compressive strength of W42			$f_{cu.70.7}$ (MPa)
	G0	H0	I0		J0	K0	L0	
1	40.68	42.55	41.79	41.67	27.37	28.87	28.31	28.18
3	57.92	58.23	57.33	57.83	38.61	39.16	38.68	38.82
7	63.65	64.69	64.29	64.21	54.01	53.95	55.08	54.35
14	65.91	66.42	67.84	66.72	61.17	62.69	61.85	61.91
28	71.69	72.24	71.31	71.75	63.73	65.24	64.58	64.53
60	81.92	82.75	84.01	82.89	75.95	76.40	75.53	73.01
90	89.56	90.21	91.95	90.57	78.22	81.01	79.83	79.69
120	93.76	94.52	94.07	94.12	81.97	82.70	82.58	82.43
150	94.52	95.76	95.55	95.28	83.25	85.39	84.73	84.46
180	96.53	95.11	97.65	96.43	84.07	85.27	85.16	84.83

(a) Before the test

(b) After the test

Figure 3. Compressive strength of cube.

3.4. Splitting tensile strength

The splitting tensile strengthes of the two kinds of ratio W35 and W42 are shown in table 6.

Table 6. Test results of splitting strength of cube.

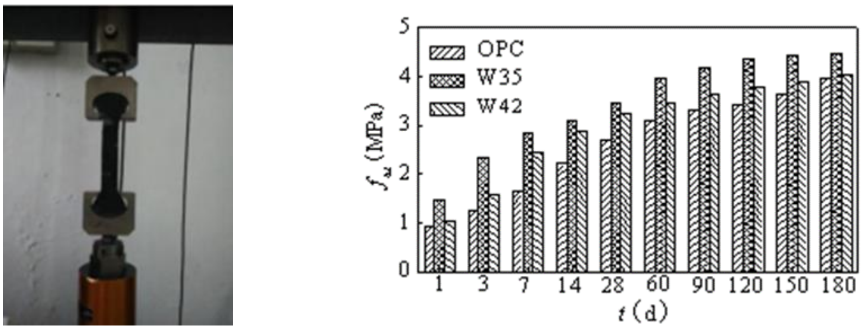
Age (d)	Splitting strength of W35			f_{ts} (MPa)	Splitting strength of W42			f_{ts} (MPa)
	PA0	PB0	PC0		PD0	PE0	PF0	
1	1.47	1.28	1.36	1.37	0.89	0.92	1.10	0.97
3	2.12	2.26	2.18	2.19	1.43	1.48	1.51	1.47
7	2.63	2.75	2.59	2.66	2.25	2.18	2.37	2.27
14	2.97	2.84	2.90	2.91	2.63	2.75	2.71	2.70
28	3.41	3.25	3.04	3.23	2.96	3.12	2.99	3.02
60	3.65	3.59	3.84	3.69	3.27	3.35	3.10	3.24
90	3.91	4.03	3.73	3.89	3.36	3.47	3.39	3.41
120	4.02	3.97	4.15	4.06	3.51	3.49	3.58	3.53
150	4.08	4.16	4,.14	4.13	3.55	3.59	3.78	3.64
180	4.22	4.19	4.04	4.15	3.64	3.81	3.80	3.75

The fracture form of the alkali-activated slag specimen is shown in figure 4. Along the middle line, the crack emerges, and the fracture interface is clear and plain. By comparing the axial tensile strength and splitting tensile strength of the test pieces of W35 and W42, these conversion coefficient of the two are basically the same, and the average value is 0.933.

Figure 4. Fracture form of dumbbell type specimen.

3.5. Axial tensile strength

The axial tensile strengthes of the two kinds of ratio W35 and W42 are shown in figure 5.



(a) Test apparatus (b) Curves of axial tensile strength

Figure 5. Axial tensile strength of dumbbell type specimen.

It can be seen from figure 5 that the development of tensile strength of alkali-activated slag is similar to compressive strength and flexural strength, which increase with the increase of age. The axial tensile strength of W35 specimen is highest, the second of W42 and the lowest of OPC. The tensile strength of the alkali-activated slag specimens 1d, 3d and 7d at normal temperature can reach 33%, 51% and 78% of tensile strength at 28d. After 60d, the development of tensile strength of alkali-activated slag slowed down. At 180d, the tensile strength of alkali-activated slag increased by 126%

compared with 28d. By comparison, the 28 d alkali-activated slag flexural strength is about 1/23 ~ 1/9 of compressive strength of cementing materials, and axis tensile strength is about 1/34 ~ 1/25 of compressive strength of cementing materials, which show that with the increase of age, the growth of axis tensile strength is slow, and its strength is lower than compressive and flexural strength.

4. Compressive strength of block

The block compressive strength of the ratio W35 are shown in table 7. The fracture forms of the single row of holes block compressive strength, single-hole block compressive strength and standard solid brick compressive strength are shown in figure 6. The results showed that the compressive strength of single row of holes block of alkali activated slag is equivalent to that of MU10 (*MU means m*asonry unit, and 10 means the compression strength of block is 10MPa) concrete block. Moreover, the compressive strength of standard solid brick of alkali activated slag is in the middle of that of MU20 and MU25 (*MU means m*asonry unit, and 10 means the compression strength of block is 20MPa and 25MPa) concrete block. Which basically meet engineering requirements.

Table 7. Test results of compressive strength of block.

Block type	1	2	3	4	5	Average, MPa
Single row of holes block	8.88	10.97	10.3	10.11	10.46	10.46
single-hole block	14.78	14.67	13.98	15.24	15.17	14.75
standard solid brick	23.13	23.04	23.45	23.67	22.98	23.25

(a) Single row of holes block (b) Single-hole block (c) Standard solid brick

Figure 6. Fracture forms of block.

5. XRD analysis

The standard maintenance 3d, 7d, 28d alkali-activated slag cementing materials and standard maintenance of 28d cement paste are scanned, and the diffraction pattern is shown in figure 7.

Through analyzing figure 7, it can be seen that for the OPC control sample, the XRD pattern shows some sharper reflection peaks corresponding to ettringite $[Ca_6[Al(OH)_6]_2(SO_4)_3 \cdot 26H_2O]$, portlandite $[Ca(OH)_2]$, calcite ($CaCO_3$), and also to calcium silicate hydrate (CSH) gel $[(CaO)_x \cdot SiO_2 \cdot (H_2O)_y]$. The XRD patterns of alkali-activated slag samples have been detected the presence of CSH-type hydration products using a peak of ~30° 2θ, which overlapped the amorphous hump of the slag. Peaks of a hydrotalcite-type phase similar to $Mg_6Al_2CO_3(OH)_{16} \cdot 4H_2O$ were also identified. Peaks of tetracalcium aluminate hydrate $(C,M)_4AH_{13}$ were also observed, and some of them overlapping those of hydrotalcite. Some carbonation seemed to have taken place, because all sample preparation was performed in the open laboratory. However, the peaks were weak and overlapped with other phases. It has also reported that the cement paste 28d hydration products have a large amount of calcium hydroxide, calcium alum and calcium carbonate and other crystal substances, Which is obviously different from the hydration product of alkali-activated slag with hydrated calcium silicate and hydrated calcium aluminate. In addition to the hump in the region between 17 :35 degree which reflect the amorphous composition of the geopolymer structure, where this hump increased in broadness with time reflecting the increased content of the formed geopolymer structure with time.

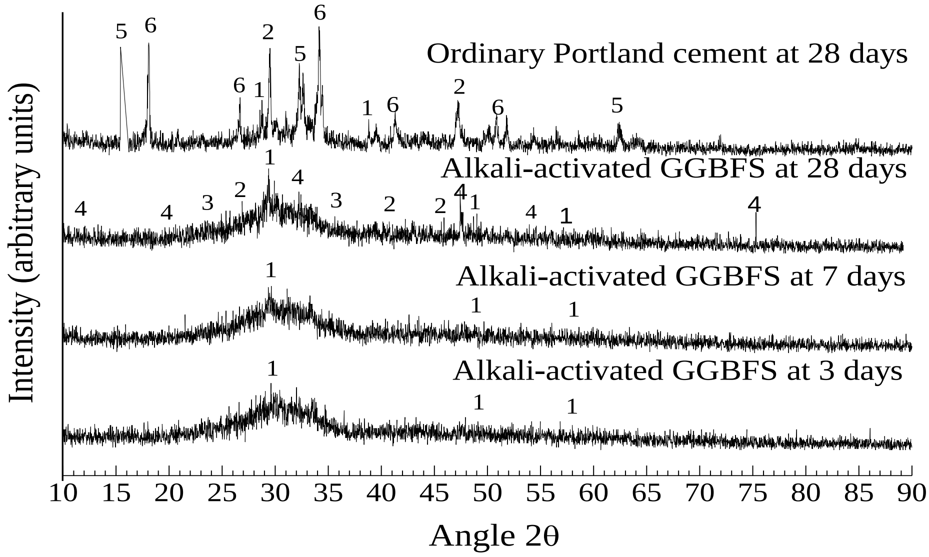


Figure 7. XRD patterns at room temperature.

6. Conclusions

(1)Test proves that alkali-activated slag has excellent long-term mechanical properties and is a kind of rapid hardening and early strength cementitious material.

(2) Single row of holes block compressive strength, single-hole block compressive strength and standard solid brick compressive strength basically meet engineering requirements.

(3) The hydration products of alkali-activated slag are assured as hydrated calcium silicate and hydrated calcium aluminate by XRD analysis, which is obviously different from the hydration products of OPC.

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