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Research and Application of Slag Activity Stimulation at Low Temperature

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Abstract

Aiming at the problem that slag has low hydration rate at low temperature, physical and chemical stimulation to slag are studied. The results show that, physical stimulation increases the slag reaction contact area, and effectively improves the slag hydration reaction rate. Chemical activator JFJ-1 can significantly promote the activity of slag and enhance the slag's compressive strength. The optimum adding amount of JFJ-1 is 5%. Under the condition of 10°C, the compressive strength of low density slag cement slurry develops much more fast for the use of JFJ-1 and the strength can reach 6.8MPa in 24h, 16.4MPa in 48h. Laboratory tests show that the low density slurries has no free fluid and good rheology.

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Keywords: slag; low temperature; physical and chemical stimulation; mechanism of hydration ;slag cement slurry

1. Introduction

With the progress of science and technology and the enhancement of people's environmental protection consciousness, the high energy-consumption materials, such as portland cement, cannot meet the requirement of society development. Research of industrial waste (slag, fly ash and so on) as the production of building materials has been paid more and more attention. Slag is produced when smelting pig iron, with the advantages of fast solidification and early strength, high compressive strength, resistance to acid and alkali corrosion, low density, high temperature resistant, low price, a wide range of

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sources and so on, which can be used for cementing engineering, civil engineering, traffic engineering and other fields. So slag is expected to become the new cementitious materials of green environmental protection, which has become one of the hot research in recent years^[1-3].

The chemical composition of slag is similar to cement, with potential reaction activity. So it needs activator to stimulate slag reaction activity. In the previous studies, the test temperature of slag stimulation is generally above room temperature, and researches at low temperature are rare^[4-6]. This paper attempts to use the physical and chemical stimulation to improve the slag activity under the condition of low temperature, and broaden the usable range of slag .

2. Experiment materials and methods

2.1. Experiment materials

Experiment materials include: slag, provided by Jinan Lu Xin Building Materials Company of Shandong. The main chemical composition of slag is shown in Table 1; activator JFJ-1, laboratory-made solid-state alkaline activator; floating beads, produced by Liaocheng; micro-silicon, provided by Shengli Oilfield cementing companies.

Table 1. The chemical composition of slag

chemical composition	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	Loss
%	33.34	37.55	16.06	0.73	9.89	0.34	0.21

2.2. Experiment methods

Use the German Polysis company's RMS vertical mill to grind the slag. the slag powder is dried in drying box and naturally cooled in air. Then the slag powder is put into a dry bag as experimental raw materials for use. The slag powder particle sizes are tested by Ji Nan Run Zhi company's Rise-2000 laser particle size analyzer. According to the API10B-3-2004 standard, prepare slag slurry with water cement ratio of 0.75, and put the slurry into the 50mm×50mm×50mm copper mold, then maintenance the mold within SL-B type multifunctional maintenance vessel. At last use the NYL-300 type press to test compressive strength of test block at different ages.

3. Results and discussion

3.1. Physical stimulation effects on slag activity

Grind the slag with RMS vertical mill and measure the powder's particle size distribution before and after grinding. The results of particle size analysis are as shown in Figure 1. The main features of slag before and after grinding are shown in Table 2 .

Table 2 The main features of slag before and after grinding

Slag samples	D10/um	D25/um	D50/um	D75/um	D90/um	Dav/um	S/V(m ² /cm ³)
Slag S1 before grinding	5.05	6.62	9.19	12.12	15.92	10.02	0.718
Slag S2 after grinding	1.18	1.59	2.24	3.51	5.60	2.90	2.181

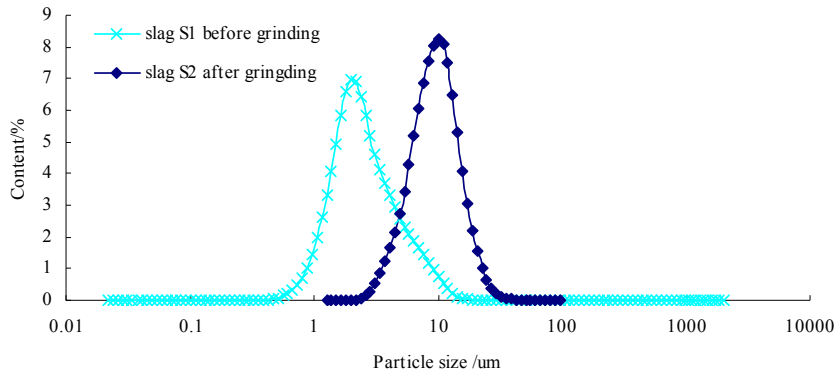


Fig.1. the slag powder's particle size distribution before and after grinding

According to Fig.1. and Table 2, after the grinding process the average particle size D_{av} of slag reduces from 10.2um to 2.90um, about 0.3 times (30% of) the pre-grinding, and surface area increases from 0.718 m^2/cm^3 to 2.181 m^2/cm^3 , about 3 times the pre-grinding, which greatly increased the contact area of slag hydration reaction. Moreover, the grinding process increases the lattice defects on the slag, which can improve the reactivity of slag.

Testing the 24h compressive strength of slag samples S1 and S2, and the experimental formula are as the followings:

1#: 400g slag S1 +300 g water +12 gJFJ-1, the curing temperature: 25°C

2#: 400g slag S1 +300 g water +12 gJFJ-1, the curing temperature: 10°C

3#: 400g slag S2 +300 g water +12 gJFJ-1, the curing temperature: 25°C

4#: 400g slag S2 +300 g water +12 gJFJ-1, the curing temperature: 10°C

Experiment results: for the 1# and 2# cement stones, the 24h compressive strength is 0; for the 3#, the 24h compressive strength is 22MPa, for the 4#, the 24h compressive strength is 3MPa. Compared 1# and 3#, 2# and 4#, they both show that the physical grinding can improve the reactivity of slag. And the Comparison between 3# and 4# indicates that temperature is an important factor affecting the activity of slag.

3.2. Chemical stimulation effects on slag activity

Keep water-cement ratio (0.75) invariable and add different amount of laboratory-made solid-state alkaline activator JFJ-1 to the slag S2, then respectively measure the effects of JFJ-1 on slag activity to identify the optimal dosage of JFJ-1.

Experiment conditions: curing temperature 10 °C, W/C = 0.75, JFJ-1 dosage relative to the amount of water quality .The results are as shown in Fig. 2.

Fig.2. shows, when the JFJ-1 plus under 3%, the compressive strength of slag increase slowly with the dosage of JFJ-1, when the JFJ-1 plus above 3%, the compressive strength of slag increase quickly and reached the maximum at 5%. when the JFJ-1 plus increases more than 5%, the compressive strength of slag decrease slowly with the dosage of JFJ-1. It can be seen the optimal amount of JFJ-1 dosage is about 5%. Considering the excess dosage of JFJ-1 will increase the consistency of paste, which is not conducive to the construction site, the proposed optimal dosage of JFJ-1 is 4.5% -5%. When the dosage of JFJ-1 is

5%, slag has high strength at 10 °C, and the 24h strength is 8.8MPa, 48h strength is 21.6MPa, which shows JFJ-1 can effectively stimulate slag activity at low temperatures .

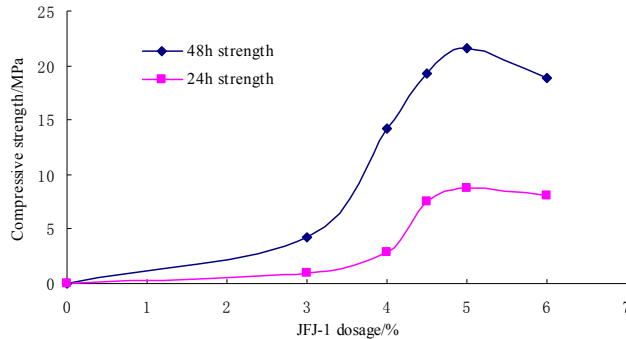


Fig.2. the effect of JFJ-1 dosage on the compressive strength of slag S2 at 10°C

Mechanism analysis: slag is a polymer composed by glass phase and crystalline phase. The glass phase is the active ingredient, which mainly contains calcium-rich phase and silicon-rich phase^[4]. Hydration characteristics of slag in alkaline activator is: after the decomposition of OH^- in alkaline activator, $-\text{O}-\text{Si}-\text{O}-\text{Al}-\text{O}-$ of vitreous structure have depolymerization reaction, which produces $[\text{SiO}_4]^{4-}$ tetrahedra and $[\text{AlO}_4]^{5-}$ tetrahedron. Then polycondensation reaction produce a new $-\text{O}-\text{Si}-\text{O}-\text{Al}-\text{O}-$ network structure of inorganic polymer, which is essentially different from the hardened body of inorganic ion composition in Portland cement. Therefore, the nature of slag hydration can be put in the following way: in neutral and weak alkaline environments, a small amount of OH^- cannot provide sufficient energy to overcome the $-\text{O}-\text{Si}-\text{O}-\text{Al}-\text{O}-$ decomposition activation, so the slag shows lower activity in neutral and weak alkaline environments. While in alkaline and strongly alkaline environment, OH^- concentration is high enough to overcome the $-\text{O}-\text{Si}-\text{O}-\text{Al}-\text{O}-$ chain decomposition activation energy, making the phase of calcium-rich to hydrate and disintegrate rapidly, then polycondensation reaction and generate a new polymer, strengthen the structure of hydration products, which showing a steady increase of the strength of slag^[7-15].

4. Application of Slag

According to the research above, the slag S2 and JFJ-1 are applied to the development of slag cement slurry. On the basis of theory of close packing^[16-19], choose hollow microsphere, slag and microsilica as the raw materials. Their particle size distribution curves are shown in Fig.3.

As Fig.3.shows, hollow microsphere, slag and microsilica built-up a ternary size distribution. As the coarse texture, hollow microsphere formed skeleton of the test block, while the slag fill in the gap of hollow microsphere as a smaller particle, and microsilica, as the least particle, fill in the gap which formed by hollow microsphere and slag. They form a closely packed system, reducing the porosity between particles, making slag the cement system more dense.

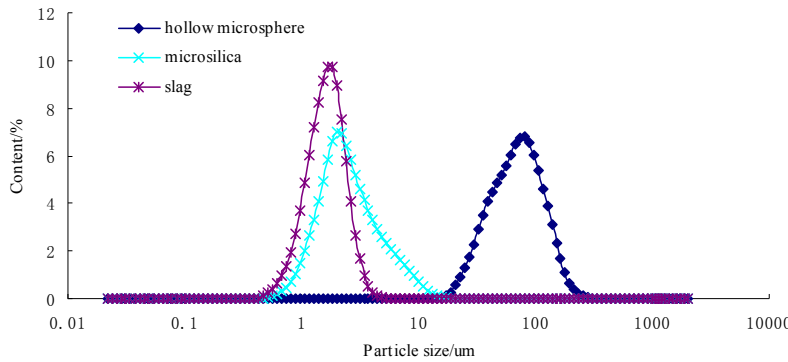


Fig.3. the particle size distribution curve

The basic slag cement slurry formulation is: slag +33% hollow microsphere +5% microsilica+4.5% (relative to the mass fraction of water) JFJ-1, $W / C = 0.75$. Basic properties of the neogenic cement system are shown in Table 3:

Table 3:Basic properties of the slag cement system

Curing temperature /°C	Density /g/cm ³	24h Strength /MPa	48h Strength /MPa	n	K /Pa.sn	Free water /ml
10	1.32	6.8	16.4	0.932	134.4	0

Table 3 shows that: Under the condition of low temperature at 10°C, the low density cement slurry system has very good early strength properties, 24h strength reaches 6.8 MPa, 48h strength reaches 16.4MPa, which well meet the low density cement slurry cementing strength requirements. Slag flow index $n=0.93$, consistency coefficient $K=134.4$, which means prepared slag system has good rheological properties. We suggest develop additives (filtration reducing agent, dispersing agent, accelerating agent and so on) suitable for slag cement system and form slag cement slurry system with good properties. We are surely convinced that this slurry will have a good application prospect in deepwater cementing operation because of its early strength and low price.

5. Conclusion

(1) Physical grinding can make the lattice defects and the surface area increased, which significantly increase the contact area of the hydration reaction of slag, thereby improve the slag reaction activity.

(2) JFJ-1 can effectively promote the activity of slag; when $W/C = 0.75$ and dosage of JFJ-1 is 5% of the water quality, the strength of the slag achieves the best in different ages.

(3) Low density cement slurry system prepared with pulverized slag and activator JFJ-1, of which 24h strength reaches 6.8 MPa, 48h strength reaches 16.4MPa, can be adequate to well meet the low density cementing strength requirements under the low temperature of 10°C.

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