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Effect of activators, admixtures and temperature on the early hydration performance of desulfurization ash

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HIGHLIGHTS

- Different activators on the properties of the desulfurization ash are explained.
- Pozzolanic reactivity and mechanical property of desulfurization ash is studied.
- Different hydration products are detected by testing methods.

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ABSTRACT

The methods of activators, active mineral admixtures and temperature were used to activate the pozzolanic and self-cementitious properties of desulfurization ash, as well as to compare and analyze the 7 days and 28 days compressive strengths of desulfurization ash mortar. Meanwhile, the early hydration process and hydrated products of desulfurization ash were analyzed with X-ray diffraction analysis (XRD), FT-IR spectra (FT-IR) and scanning electron microscopy (SEM). The research results show that different kinds of activators and active mineral admixtures can activate the early active of desulfurization ash, as follows, the first is quicklime, then sodium silicate, slag powder, triethanolamine, Na_2SO_4 and the last is silica. By the combination of organic chemical activators and inorganic mineral admixtures mentioned above, the 7 days and 28 days compressive strengths of desulfurization ash mortar can be up to 20 MPa and 22.3 MPa, respectively. The strength can still be raised by enhancing the proper curing temperature.

1. Introduction

The desulfurization ash has a relatively high pozzolanic activity because of containing a certain of active SiO_2 and Al_2O_3. At the same time, the desulfurization ash has self-cementitious property [1–5] which can be used as a new cementing material, and it also contains f-CaO which can form CaO—Al_2O_3—SO_3 system. Qian et al. [2] and Song et al. [3], have explored the characteristics and self-cementitious mechanism of desulfurization ash. The self-cementitious strength of CFBC (Circulating Fluidized Bed Combustion) fly ash is affected by its fineness and chemical compositions, especially its f-CaO and SO_3 contents. Higher f-CaO and SO_3 contents is more beneficial to the development of self-cementitious strength [4]. The mechanism of self-cementing is based on two reactions: generation of calcium silicate hydrate (C—S—H) by the reaction of active SiO_2 with CaO; generation of ettringite (AFt) by the reaction of active Al_2O_3 with gypsum and CaO [4,5]. Up till now, few studies have been done on the activity excitation of desulfurization ash's cementitious system. Different things have an impact on the early mortar strength and hydration process of the desulfurization ash's cementitious system in this study; such as, chemical activators, active mineral admixtures (quicklime, silica, slag powder, Na_2SO_4, triethanolamine and sodium silicate) and temperature.

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2. Experiment study

2.1. Materials

Desulfurization ash was provided by a power plant in Nanchong City (China); silica and slag powder were provided by a company in Chengdu Iron & Steel Co., Ltd (China). The chemical composition of them are presented in Table 1. X-ray diffraction pattern of the desulfurization ash is shown in Fig. 1.

Quicklime Jiangyou Tian mining Co., Ltd, (China); containing 82% of activity CaO, digestion temperature was 90 °C, digestion time was 3 min, belonging to the fast digestion lime.

Na_2SO_4 analytical reagent, content ⩾99.0%.

Triethanolamine (TEA) analytical reagent, content ⩾99.0%.

Sodium silicate modulus was 1.03, Na_2O content was 19.3–22.8% and SiO_2 content was 19–22.1% (mass percentage).

2.2. Mix proportions and test methods

2.2.1. Mix proportions

The amounts of desulfurization ash, quicklime, silica, slag powder, Na_2SO_4, triethanolamine, sodium silicate were used in all mortar mixtures to explore the optimum content of their own. A total of 23 mortar mixtures were used in this experimental presenting in Tables 2 and 3.

2.2.2. Test methods

The ratio of mortar specimen sand to cement was 3.0, and the ratio of water to cement was 0.60. Specimen size was 40 mm × 40 mm × 160 mm. De-molding was conducted after maintained in a curing box under (20 ± 1) °C and 95% relative humidity for (24 ± 2) h, and then specimens were placed in the box to the testing ages. Specimen molding and compressive strength test were operated ISO-679:1989.

The paste's water to cement ratio of specimen is 0.53. Specimen size was 20 mm × 20 mm × 20 mm. De-molding was conducted after maintained in a curing box under (20 ± 1) °C and 95% relative humidity for (24 ± 2) h, and then specimens were placed in the box to the testing ages. After paste type specimen was broken, it was soaked with anhydrous ethanol, then it was fine ground until it was recognized with 200 mesh sieves. Finally put some powder in dry dish to spare and the others were measured by XRD, SEM and FT-IR respectively.

2.2.3. X-ray diffraction analysis (XRD)

The crystalline minerals of desulfurization ash and the products of the hydrated desulfurization ash were identified, using X-ray powder diffraction (XRD) measurement in a X'Pert PRO diffractometer (PANalytical) with Cu Ka radiation and a position sensitive detector. The accelerating voltage was 35 kV and the current was 60 mA. Diffraction peaks on the XRD spectrum were detected by software package X'Pert HighScore and Plus MDI Jade 5.0.

2.2.4. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) test was carried out using ASTEREO SCAN440 (Leica Cambridge Ltd) to investigate the morphology of desulfurization ash particles and the hydration products of the desulfurization ash. Specimens were mounted into aluminum stubs using double-sided adhesive carbon disks and sputter coated with gold. To ensure that electrical charge was efficiently carried away from specimen surfaces, a line of silver paint was applied connecting the specimen sides to the stub.

2.2.5. FT-IR spectra (FT-IR)

Fourier transform infrared (FT-IR) spectroscopy was carried out using a Spectrum One FT-IR (PE companies of United States). Powder samples were mixed with KBr. The resulting powder was then pressed at 2000 psi for 5 min to produce a pellet for analysis. The analysis was carried out in the frequency range of 400–4000 $cm^{-1}$with 0.5 cm^{-1} resolution.

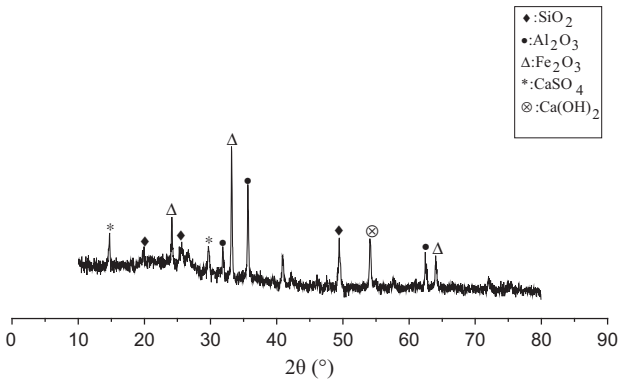

Fig. 1. Desulfurization ash's X-ray diffraction pattern.

3. Experimental results and discussion

3.1. Influence of alkali excitation agent on desulfurization ash mortar strength

The influences of quicklime and sodium silicate on desulfurization ash mortar strength are shown in Figs. 2 and 3.

According to Fig. 2 we can observe that the compressive strength of desulfurization ash mortar increases with the adding of quicklime dosage. When the quicklime content is 20%, the compressive strengths of 7 days and 28 days are up to 5.8 MPa and 18.5 MPa, respectively, which increases by 625% and 1750% compared with the mortar strength of undoped activator's desulfurization ash. It indicates that quicklime can promote the growth of mortar strength significantly in a certain dosage range. It is mainly because that a low degree of polymerization's [SiO_4] and [AlO_6] can absorb OH^-, which leads to the production of the negative charges of the desulfurization ash particles. However, the quicklime colloid particles are positively charged through digestion. Then the positive and negative charges attract each other so that the hydration calcium silicate, hydration calcium aluminate and ettringite are generated quickly [1]. In addition, the lime improves the alkalinity of the system and activates the activity of desulfurization ash, leading to the corrosion of the densified outer layer of desulfurization ash particles and leaving more active cores exposed for reacting forming additional hydration products [6,7]. It also accelerates the generation of AFt, C—S—H and gypsum, therefore it promotes the development of early strength [8].

According to Fig. 3, the mortar strength of desulfurization ash increases with the adding of sodium silicate's dosage when its alkalinity is relatively low. As the sodium silicate content is up to 2.0%, the compressive strengths of 7 days and 28 days are the highest, i.e. 2.0 MPa and 4.9 MPa, respectively, increase by 150% and 390% respectively compared with the mortar strength of undoped activator's desulfurization ash. If the sodium silicate's content is more than 2.0%, the mortar strength of desulfurization ash will decrease with the increasing of dosage. The reason is that the sodium silicate is a kind of soluble alkali, which hydrolyzes and forms NaOH, making the system's OH^- concentration increase [9], and the Si—O and Al—O bond on the surface of desulfurization ash

Table 1
Chemical composition % by mass.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	TiO_2	SO_3	R_2O	f-CaO
Desulfurization ash	38.63	30.83	16.77	3.59	1.45	5.46	1.05	3.66
Silica fume	90.36	0.58	2.53	1.22	–	0.92	2.54	–
Slag powder	32.46	16.37	1.82	35.38	0.88	2.44	0.78	–