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Effects of lithium carbonate on performances of sulphoaluminate cement-based dual liquid high water material and its mechanisms

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HIGHLIGHTS

- A novel sulphoaluminate cement-based dual liquid high water material was prepared.
- The LC has a large effect on its performance and hydration.
- The compressive strength increases continuously as the L increases.
- The setting time becomes shorter and bleeding becomes lower as the LC increases.

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ABSTRACT

Sulphoaluminate cement-based dual liquid high water material has some defects, such as longer setting time, higher bleeding capacity and lower strength and so on. In order to improve its defects, the effects of lithium carbonate on those properties were studied, and its mechanisms were analyzed through micro testing, including hydration heat tests, X-ray diffraction, scanning electron microscopy, and thermogravimetric analysis. The results show that the setting time of high water material becomes shorter, its bleeding capacity becomes lower and its compressive strength becomes higher when the content of lithium carbonate increases from 0%, 0.1%, 0.2%, 0.3% to 0.5%. Lithium carbonate helps to accelerate the hydration process of high water material at early stage and to increase the amount of the accumulated heat of hydration heat. The testing results of the XRD, the DTA-TG, and SEM show that the ettringite is the main hydration product, and the amount, the shape and the microstructure of ettringite are all different as the lithium carbonate increases.

1. Introduction

High water material slurry has a water-cement ratio with higher than 3.0. According to Feng Guangming's studies [1], the material contains over 95 vol% of water (the water-cement ratio is equal to or higher than 6.3) is called super high water material, and the material that contains less than 95 vol% of water is called common high water material. Its water-cement ratio is 3–12 times higher than the common Portland cement slurry. It has many advantages, such as better fluidity, shorter setting time and less consumption per unit volume and all these make it suitable for filling mining in coal mine and metal mine, and solution-cavity filling, etc [2–3] As the most commonly used cementitious material, common portland cement can only be used in such projects which have a lower water-cement ratio (0.5–1:1). Therefore, it is not suitable for making high water material.

Sulphoaluminate cement (CSA cement) is a special kind of cement invented by China in 1970, and It has a shorter setting time and higher early strength [4,5]. The main hydration product of CSA cement is AFt (cf. Eq. (1)).

$$3CaO \cdot Al_2O_3 \cdot 6H_2O + 3(CaSO_4 \cdot 2H_2O) + 19H_2O = 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31H_2O \quad (1)$$

According to Eq. (1), the generation process of AFt needs a lot of water, and it is possible to make high water material with CSA cement. In addition, the existence of calcium sulfate and calcium hydroxide helps to accelerate the generation and to generate more AFt. The more AFt it generates, the more water it needs [6,7].

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Therefore, calcium sulfate and calcium hydroxide can be added to CSA cement clinker to make high water material. The setting time of high water material's slurry is shorter, so in order to ensure that it has good construction characteristics, dual liquid is adopted. We used CSA cement clinker as A, the mixture of anhydrite and quicklime as B. The research [8] shows the material has the best property when anhydrite and quicklime are mixed with the proportion of 4:1by weight. Mix A with B at the proportion of 1:1 in mass to make high water material. The reaction equations are shown as Eq. (2)–(4) [9–11].

$$C_4A_3\bar{S} + 2CSH_2 + 34H \longrightarrow AFt + 2AH_3\ (\text{gel}) \tag{2}$$

$$C_2S + 2H \longrightarrow CSH(I) + CH \tag{3}$$

$$AH_3(\text{gel}) + 3CH + 3CSH_2 + 20H \longrightarrow AFt \tag{4}$$

Slurry A and slurry B, which are mixed separately with water, can last for 30 h–40 h without solidification. When they are mixed at the proportion of 1:1 in mass, they quickly hydrate and solidify. The compressive strength can be adjusted by adjusting the water cement ratio and adding admixture, and the setting time can be adjusted from 8 min to 90 min as needed.

As a new type of material, high water material has been researched by many researchers. For example, Feng Guangming researched the application of super high water material in goaf filling of coal mine. Wang Xufeng and his workmates tested the properties of cemented backfill with super high water material [12,13]. With a higher water-cement ratio, high water material is easy to bleed and it takes a long time to set and its compressive strength is low. So how to improve its property under the condition of a higher water-cement ratio is always the focus in the field.

As a CSA cement accelerator, lithium carbonate has been researched by many researchers. The results of the previous researches show that lithium carbonate can significantly shorten the setting time of the CSA cement and enhance its early compressive strength and flexural strength but weaken its later compressive strength and flexural strength [14–16].

Lithium carbonate can promote the hydration of CSA cement and speed up the generation of ettringite. But if it is suitable for making high water material and can improve its property, these have not been researched. Therefore the effects of lithium carbonate on properties and hydration process of sulphoaluminate cement-based dual liquid high water material are researched in this paper by analyzing the properties of the high water material from its bleeding capacity, setting time and uniaxial compressive strength and analyzing its hydration process, hydration products and microstructures through hydration heat tests, XRD tests, DTA-TG analysis and SEM-DES tests. Through the research, to provide theoretical basis and guidance for the application of sulphoaluminate cement-based high water material in engineering.

2. Materials and test methods

2.1. Materials

The materials of high water material are mainly CSA cement clinker, anhydrite and quicklime. Mixed anhydrite and quicklime together at the proportion of 4:1 and ground to 0.075 mm. The effective calcium sulfate in anhydrite is 83.7 wt%, and calcium oxide in quicklime is 75 wt% and the effective component of lithium carbonate is 99.99 wt%. The chemical composition of CSA cement clinker cf. Table 1 and its mineral composition cf. Table 2 (Table 3).

2.2. Test methods

2.2.1. High water material property tests

(1) Bleeding capacity and setting time tests

Dual liquid high water material includes A and B. Added lithium carbonate to B. The ratio cf. Table 4. Added water to A and B respectively and stirred for 3 min and then tested their setting time and bleeding capacity. The setting time test method cf. *Test method for setting time of water filling materials* MT/T 420-1995 (2005).

(2) Compressive strength tests

The compressive strength was tested cf. *Test method for strength of cement mortar* (GB/T 17671-1999) and used the mold of 70.7 mm * 70.7 mm * 70.7 mm. Mixed the materials according to the proportion shown in Table 4, and then poured the mixed grout into the mold to make standard samples. When the samples are molded, maintained them under standard condition (20 °C, 95 wt.%RH). Unloaded the mold after 4 h of molding, and then tested the compressive strength at the curing ages of 1 day, 3 days, 7 days and 28 days, respectively.

2.2.2. Hydration process and microstructure tests

(1) Hydration heat tests

Sieved A and B using 0.075 mm sieve and evenly mixed them respectively. Then tested them using SHR-650 II cement hydration heat test instrument. The test methods cf. *Cement hydration heat determination method* (GB/T 12959-2008).

(2) DTA-TG tests

Sealed the samples in alcohol and dried them in the vacuum drying oven at the temperature of 35 °C and under the pressure of 0.08 MPa one day before testing. Ground the dried samples to 0.045 mm and used Braker D8 Advance polycrystalline X-ray diffractometer to scan continuously at the speed of 0.020/s to test the mineral composition of the hydration products.

(3) XRD and SEM analysis tests

Used permanent thermal effect equipment to test the thermogravimetric curve under air. The heating rate is 10 °C/min. Dried the unground samples in the vacuum drying oven and then used 250FEG SEM to observe the shape and microstructure of the hydration products.

3. Results and discussion

3.1. Bleeding capacity and setting time tests analysis

The results of the high water material's bleeding capacity when added lithium carbonate of different proportion cf. Fig. 1. The results show that, with the increase of the lithium carbonate content, the bleeding capacity decreases gradually. When the lithium carbonate content is 0 wt%, the mixed grout's bleeding capacity is 20 vol% after 1 h. With the increase of the age, the bleeding capacity decreases gradually. After 24 h, the bleeding capacity is 5 vol%. When the lithium carbonate content is 0.2 wt%, the bleeding capacity is 5 vol% after 1 h and 1 vol% after 8 h. After 24 h, there's no bleeding. When the lithium carbonate content is between 0.3 wt% and 0.5 wt%, the bleeding capacity decreases significantly. The bleeding capacity is only 1 vol% after 1 h and after 4 h, there's no bleeding. When the lithium carbonate content is over 3 wt%, it has little effect on the bleeding capacity.