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Effect of zeolite on waste based alkali-activated inorganic binder efflorescence

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HIGHLIGHTS

• 5A zeolite enhances the compressive strength and bending strength.

• 5A zeolite decreases the pH value and bicarbonate ion concentration.

• A conformational mechanism for inhibiting efflorescence is proposed.

• The mechanism relies on the special ion-exchange properties of 5A zeolite.

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ABSTRACT

Alkali-activated inorganic binder has been a research hot spot in the field of building materials owing to its wide range of raw materials, low energy consumption, less pollution and superior performances. The bicarbonate salt weathering is one of prime cause of deterioration of porous materials. To inhibit the efflorescence of waste based alkali-activated inorganic binder, 5A zeolite powder was blended and the efflorescence inhibition mechanism was analyzed. Effect of 5A zeolite content on the mechanical properties, bicarbonate ions concentration, pH value and pore size distribution of the obtained alkali-activated inorganic binder were investigated. The results showed that the compressive strength and bending strength of the waste based alkali-activated inorganic binder are improved with adding moderate content of 5A zeolite powder, and the pH value of specimens decreases with increasing amount of 5A zeolite powder content, the average pore size of samples decreases with adding moderate content of 5A zeolite powder due to its micro-aggregate filling effects. The efflorescence of waste based alkali-activated inorganic binder is effectively reduced with the adding of 5A zeolite powder, and the inhibition mechanisms mainly are that the special ion-exchange properties and micro-aggregate filling effects.

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1. Introduction

Research on effectively use of industrial waste in alkaliactivated technology, which were initiated reported by Davidovits [1], has been a hot spot in recent years in building materials field. Alkali-activated inorganic binder can be synthesized from aluminosilicate waste materials such as steel slag [2-4], blast furnace slag [5–7] and fly ash [8–10] under strong alkaline condition. Compared with cements, ceramics and metals, alkali-activated inorganic binder have many advantages, such as early high strength [11–13], low permeability [14–16] and good fire resistance behavior [17,18]. However, there exist some disadvantages which cannot be ignored such as efflorescence [23]. It is well known that alkaliactivated materials were activated with alkali such as sodium hvdroxide (NaOH), potassium hvdroxide (KOH), sodium silicate (Na2SiO3), so soluble alkali can be dissolved out from alkaliactivated material surface and reacted with carbon dioxide in the air to form bicarbonate, which hindered the practical application of alkali-activated materials. Efflorescence is a very important problem that should be reduced in development of alkaliactivated materials.

Some studies of the efflorescence of alkali-activated materials were reported, however, most of them were based on fly ashbased alkali-activated material. Najafi et al. reduced efflorescence in alkali-activated inorganic binder by adding Al-rich mineral admixtures and cured at elevated temperatures [19]. Minfang Han et al. found that the efflorescence extent of fly ash-based





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alkali-activated binder specimen decreased with addition of 5A zeolite and lead to the lesser pore volume of macropores [20]. Zuhua Zhang [21] et al. discussed the relationship between composition, pore structure and efflorescence of fly ash-based alkali-activated binder, and found that the efflorescence rate depended on the activation conditions, slag addition and curing temperature.

In this work, the study aims to investigate compressive strength, flexural strength, bicarbonate ions concentration, pH value and pore size distribution of alkali-activated inorganic binder prepared using steel slag and blast furnace slag as resource material and activated by sodium hydroxide after cured for1, 3 and 60 days when 5A zeolite powder was partially replaced with waste powder at levels ranging from 0% to 25% with an interval of 5%.

2. Material and methods

2.1. Raw materials

The source of aluminosilicate to produce alkali-activated inorganic binder was steel slag and blast furnace slag, produced in Shandong steel group, its specific surface area were 376 m²/kg and 436 m²/kg, respectively. The specific surface area of steel slag and blast furnace slag and 5A zeolite were studied using Brurauer Emmerr Teller (BET) Procedure. 5A zeolite chosen in this experiment is a kind A type zeolite with 5A aperture, which was made from the Ca²⁺ ions exchange with Na⁺ in the 4A zeolite, it can absorb the particles or molecular less than 5A [22]. The 5A zeolite powder was from Wufeng ceramic company of China and its specific surface area was 1145 m²/kg. The morphological features of 5A zeolite powder was tested by scanning electron microscope and presented in Fig. 1. The sodium hydroxide was used as activator to prepare the alkali-activated inorganic binder specimens and its content was 5%. The chemical composition of raw materials was determined by X-ray fluorescence spectroscopy and was shown in Table 1. The particles diameter of 5A zeolite, blast furnace slag and steel slag were tested by Beckman Coulter LS 13 320 and were illustrated in Fig. 2, Fig. 3 and Fig. 4, respectively.

The particles size distribution of 5A zeolite powder (Fig. 2) suggested that the average particle size (Xav) was 4.460 μ m and the median particle size (X50) was 4.270 μ m. The particles size distribution of blast furnace slag (Fig. 3) suggested that the average particle size (Xav) was 11.78 μ m and the median particle size (X50) was 9.892 μ m. The particles size distribution of steel slag (Fig. 4) suggested that the average particle size (Xav) was 9.847 μ m and the median particle size (X50) was 3.857 μ m.

2.2. Preparation of the specimens

5A zeolite powder was used as substitute of waste powder such as steel slag and blast furnace slag at levels ranging from 0% to 20% with an interval of 5% in this experiment. The proportion of three ingredients (steel slag, slag and sand) was

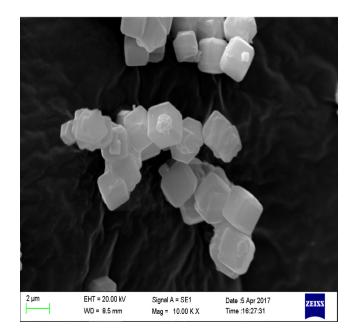


Fig. 1. The SEM photo of 5A zeolite powder.

1:1:1. The activator solution should be prepared before producing the alkaliactivated inorganic binder mortar. Analytically pure NaOH were added to water and stirred continuously for 15 min until the sodium hydroxide was total dissolved, then the solution was cooled to 20 °C. The steel slag, blast furnace slag and 5A zeolite powder were weighed and mixed for 24 h according to solid materials ratio presented in Table 2. In order to obtain homogeneous alkali-activated inorganic binder mortar, the mixed powder was added into mixing pan and the activating solution was poured continuously during the mixing. Then the alkali-activated inorganic mortar was molded and the mold size used in the experiments was $40 \times 40 \times$ 160 mm. One solid cast iron with size of $40 \times 40 \times 160$ mm was placed above the mold and pressed gradually until 20 MPa and kept the pressure on for 10 min. The alkali-activated inorganic mortar with water to solid ratio of 0.1 was cured for 60 days. All specimens were cured in standard curing chamber (20 °C and 95% R.H.) for 24 h and demolded, after that the specimens were placed under the same conditions cured for 60 days. The raw material ratio of alkali-activated inorganic binder mortar adding 5A zeolite powder was illustrated in Table 2.

2.3. Tests procedures

The efflorescence are products of reactions between CO_2 in the air and OH^- ions in specimen evaporated with water from inside to surface [19,21,23]. The mechanism of efflorescence of formation can be sketched as followings:

 $CO_2(g) + 20H^-(aq) \to CO_3{}^{2-}(aq) + H_2O$

 $\text{CO}_3^{2-}(\text{aq}) + \text{CO}_2(\text{g}) + \text{H}_2\text{O} \rightarrow 2\text{HCO}_3^{-}(\text{aq})$

 $Na^+(aq) + HCO_3^-(aq) + nH_2O \rightarrow NaHCO_3 \cdot nH_2O(s)$

So the efflorescence can be characterised by measuring the concentration of bicarbonate ion of alkali-activated inorganic binder mortar specimens leaching liquid which cured for 60 days. The procedures were as follows, the specimen was placed in 250 ml distilled water for 48 h in order to make the efflorescence products which mainly was bicarbonate dissolve in water completely. Then, 10 ml leaching liquid was taken and diluted to 100 ml which can be determined by hydrochloric acid standard solution titration [21]. The efflorescence inhibition mechanism was analyzed by measuring the pore size distribution and pH value. Scanning electron microscopy was used to characterize the alkali-activated inorganic binder and to investigate the micro-structure of the alkali-activated inorganic binder specimens with different 5A zeolite powder content. The mineralogical compositions of alkali-activated inorganic binder specimens for 60 days were assessed by X-ray powder diffraction (XRD) with Cu Ka radiation using a BRUKER D8 ADVANCE diffractometer equipped with a copper tube operated at 40 kV and 40 mA, applying an angular step of 0.02 for 2 s with 2 h spanning from 10° to 60° (20).

3. Results and discussion

3.1. Compressive strength

The results shown in Fig. 5 were the mean strength of three specimens. It can be seen from Fig. 5 that the compressive strength increases with 5A zeolite powder content, and its value is largest when the dosage of 5A zeolite powder is 15 wt%. However, compressive strengths could be decreased when a threshold (15%) is surpassed; the compressive strength of the waste based alkaliactivated inorganic binder decreases with increasing content of 5A zeolite powder. The reason may be that, on one hand, the 5A zeolite powder are fine particles with high specific surface area of 1145 m²/kg, it can be acted as micro aggregates and evenly distributed in the samples to achieve tight accumulation condition and make the samples compact. On the other hand, the 5A zeolite powder are rich in Ca²⁺ ions, which are benefit to improve mechanical properties of samples according to previous studies [24,25]. Temuujin et al. [26] found that rich calcium materials could improve the density degree when adding into alkali-activated inorganic binder, the reason is that rich calcium materials to form C-S-H gel in the end under strong alkaline conditions. Yip [27] drew similar conclusion, namely the Ca²⁺ ions in the alkali-activated inorganic binder system would generate Ca(OH)₂ at first and then form C-S-H gel, which quickened the reaction process because that provide more nucleation position for the formation of the alkaliactivated inorganic binder. Thus, the compressive strength of the alkali-activated inorganic binder is improved by adding a certain

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