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Quantitative invalidation characterization of Portland cement based on BSE and EDS analysis

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HIGHLIGHTS

- A quantitative invalidation characterization method of Portland cement is established.
- Crystal structures with lengths greater than 3.0 μm appear around cement particles can be regarded as complete invalidation.
- The relationship between the content of oxide containing Al and the degree of invalidation of Portland cement is proposed.

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ABSTRACT

The degradation of mechanical properties of Portland cement caused by the invalidation of Portland cement is a serious problem that affects the quality of concrete nowadays. However, an effective invalidation characterization method of Portland cement has not been set up. Back scattering electron (BSE) and energy-dispersive spectrometry (EDS) were adopted to quantitatively characterize the invalidation of Portland cement in this study. The EDS results demonstrated that the acicular crystals generated near cement particles in BSE images are ettringites. Therefore the invalidation of Portland cement is mainly caused by the expansive ettringite generated from the hydration of oxides containing Aluminium, and crystallisation corrosion is the main invalidation mechanism. When the microstructure of acicular morphology with a length longer than 3.0 μm is observed from BSE images of Portland cement samples, the cement can be quantified as exhibiting complete invalidation. Meanwhile, the content changing of element in the area of ettringite formation acquired with EDS analysis can also be used to characterize the invalidation of Portland cement. When the content of oxide containing aluminium is not more than 5.0%, the cement is not judged as invalidated; the invalidation of cement will occur once the content of oxide containing aluminium exceeded 5.0%; when the content of oxide containing aluminium exceeded 10.0%, the cement is considered completely invalidated.

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1. Introduction

The quality of Portland cement significantly influences the performance of concrete [1–3]. Existing standards do not specify the shelf life of Portland cement. However, invalidation of Portland cement is likely to occur due to the influence of environmental conditions. While the compressive strength of Portland cement is 5 MPa lower than the standard compressive strength, mechanical performance of cement will not be up to standard [4]. As a result, the compressive strength of concrete by using invalidated cement cannot be guaranteed, and quality-related safety accidents could frequently occur [5–7]. Therefore, invalidation characterization of Portland cement is necessary.

In the research on the invalidation characterization of Portland cement, visual observation and strength test methods have been mainly used. The visual observation method is used to detect severe caking on cement surface according to a phenomenological empirical experience. Chen [8] classified agglomeration during cement storage classified into three categories: warehouse caking, pile caking and bag caking. Furthermore, they provided suggestions for preventing agglomeration of Portland cement by controlling the content of C_3A in the clinker. However, no quantitative parameters of the corresponding relationship between the content of C_3A and invalidation of Portland cement were presented. The

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compressive strength test method is used to determine cement invalidation by testing the compressive strength of 28-day cement mortar specimens and examining whether the strength of the cement meets the specifications. However, this method exhibits strong hysteresis. Zou [9] utilised energy-dispersive spectrometry (EDS) to determine the types and contents of elements in mineral constituents in montmorillonite powder to determine which varieties of montmorillonite drugs fail. He [10] utilised scanning electron microscopy (SEM), EDS and X-ray diffraction to analyse and characterize the corrosion invalidation products on the steel structures of a substation. The results showed that the corrosion invalidation of steel structures was caused by the electrochemical corrosion of carbon steel in humid atmosphere. Inspired by the above two research in different fields and so on, we think that the microscopic detection is very useful for the quantitative invalidation characterization of Portland Cement. Therefore, a quick and accurate method of quantitative invalidation characterization of Portland cement was proposed in this study by using microscopic analysis methods, such as BSE and EDS, to investigate the microstructure's morphological change and the invalidation essence of Portland cement.

2. Materials and methods

2.1. Materials

In this experiment, P·II 52.5 Portland cement produced by Jiangnan-Onoda Company (Nanjing, China) was used. Physical properties and chemical composition of the Portland cement are shown in Tables 1 and 2, respectively. One kind of fine sand for making cement mortar was standard quartz sand. The kind of sand were used as aggregate, i.e., fine sand with 98% SiO_2 and an apparent density of 2640 kg/m^3, a fineness modulus of 1.8 and a maximum diameter of 2.35 mm. Cement mortar moulds (40 mm × 40 mm × 160 mm), a constant-temperature curing box and a low-speed cutting machine were utilised for the experiments. The materials used for cement micro-sample preparation included sandpaper, diamond paste, clean wiper, anhydrous ethanol and silastic moulds.

2.2. Experimental method

2.2.1. Sample preparation

The main factors that affect the invalidation of Portland cement are humidity and storage period. Therefore, the cement invalidation simulation test mainly investigated the effect of relative humidity and age on the invalidation of Portland cement. The invalidation process of Portland cement under three different humidity conditions, i.e. 75.0%, 85.0% and 95.0, was tested in three humidity environments. Considering the advantages of saturated solutions, such as favourable chemical stability, small temperature coefficient, weak volatility and low toxicity, Organisation Internationale de Metrologie Legale selected 11 types of saturated salt solutions to form a salt solution standard relative humidity table [11]. In this experiment, three types of cheap and commonly available salt, namely, sodium chloride (NaCl), potassium chloride (KCl) and potassium sulphate (K_2SO_4) (as shown in Table 3) were selected to compound the corresponding saturated saline solutions. The humidity source was formed of the constant relative humidity (humidity fixed point) which was maintained in a confined space above the saturated saline solution. At room temperature of 25.0 °C, the saline solutions of the three relative humidity levels were compounded in glass bottles as salt cellars. The cement was stored above the salt solution for 7, 14, 30 and 90 day in a closed environment to observe the invalidation process. The design of the sample preparation is shown in Table 4. The sample cured for 7 day in an environment with a relative humidity of approximately 75.3% was labelled as A7. The other samples were labelled as A14, A30, A90, B7, B14, B30, B90, C7, C14, C30 and C90. Twelve groups of samples from A7 to C90 were tested. Appropriate amounts of cement were obtained from each group to prepare microscopic cement samples with different ages for SEM/EDS detection. Each group was subjected to a mortar compression test.

2.2.2. SEM/EDS experiment

A SEM/EDS experiment was conducted to determine the microscopic morphologies of the cement samples under different humidity environments and ages. Because different laboratories will have different scanning electron microscopy (SEM) equipment, only generalities can be presented in this portion of the documentation. Beginning with material preparation, the cement powder of samples approximately 25 g are mixed with a low viscosity epoxy resin to form an almost dry paste [12]. The epoxy is subsequently cured at 80 °C for 12 h and the specimen was then cut into 1 cm^3 cubes with the cutting machine, ground and polished to achieve 7 mm-thick samples with parallel surfaces. The samples were washed with absolute ethyl alcohol and utilised in the SEM/EDS experiment. SEM was used to observe the change of microstructure morphology near the cement particles. In the meanwhile, EDS was used to detect the contents of mainly existing elements distributed in a certain range of the area which indicated microstructure changes in SEM image. In addition, the contents of elements were equivalently converted to the corresponding oxides in the results of EDS, and the corresponding oxides are major oxide composition of Portland cement. For viewing cement samples, typical settings are an accelerating voltage of 12 kV of and probe currents of 2 nA and 10 nA for the backscattered electron and X-ray imaging, respectively.

2.2.3. Compressive strength test

The cement stored for certain periods under each humidity condition were mixed with sand and water at certain proportions and then moulded [13]. Cement mortar specimens were prepared with cement to-sand ratio of 1:3 and water to-binder ratio of 0.5. Six samples (40 mm × 40 mm × 160 mm) were moulded in each batch. The cement mortar specimens were demoulded after 24 h and maintained in a standard curing room. The curing temperature was 20 ± 1 °C, the relative humidity was maintained at 95% and the curing period was 28 day. Compressive strength tests were performed on a pressure tester at a loading rate of 1.5 kN/s.

3. Results and discussion

3.1. SEM/EDS results

After the cement samples were stored for 7d above the closed NaCl, KCl and K_2SO_4 solutions, the surfaces of the cement samples did not cake. The BSE images are shown from Figs. 1–3. No suspected needle crystals appeared near complete particles. X-ray energy spectrum analysis was conducted to determine the contents of oxides corresponding to the BSE image areas, as shown in Table 5. The contents of oxides containing Al in A7, B7 and C7 were 3.3%, 3.5% and 3.2%, respectively, which did not exceed 5.0%.

After the cement samples were stored for 14 day above the closed NaCl solution, the surfaces of the cement samples did not change. The colour of cement powder deepened slightly after being stored for 14 day above the closed KCl solution.

After the cement samples were stored above the closed K_2SO_4 solution for 14 day, the colour of the cement powder surface deepened significantly, and the cement powder exhibited slight agglomeration. The BSE images of the samples stored for 14 day in the same SEM/EDS operation are shown from Figs. 4–6. Needle crystals with lengths of approximately 1.0 μm appeared, as shown in Figs. 5 and 6. However, the needle structures had a narrow distribution range. Furthermore, X-ray energy spectrum analysis was conducted to determine the contents of oxides containing Al and K (as shown in Table 5). The contents of oxides containing Al in A14, B14 and C14 were 4.9%, 6.9% and 8.1%, respectively, which do not exceed 10.0%.

After the cement samples were stored above the closed NaCl solution for 30 d, the colour of the cement powder deepened slightly. The hardness of the cement powder increased, and slight agglomeration was observed after the cement samples were stored above the closed KCl solution for 30 day. After being stored above the closed K_2SO_4 solution for 30 d, the cement powder caked

Table 1
Physical properties of Portland cement.

Test projects	Specific surface area (m^2/kg)	Water demand of normal consistency (%)	Initial setting (min)	Final setting (min)	Stability
Measured value	373.0	27.9	174.0	229.0	Qualified

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