



In-situ and continuous monitoring of pore evolution of calcium sulfoaluminate cement at early age by electrical impedance measurement



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HIGHLIGHTS

- Pore evolution of calcium sulfoaluminate cement is evaluated.
- Hydration features associated with microstructure evolution are identified.
- Initial formation and evolution of pore structure are analyzed.

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ABSTRACT

This paper investigates the microstructure evolution of CSA cement pastes with different water to cement ratios in various hydration stages. Microstructure information, such as porosity, cumulative and incremental pore volume of cement pastes, is revealed by the innovative non-contact electrical impedance measurement (NCEIM), low temperature differential scanning calorimetry (LT-DSC), nitrogen adsorption test and mercury intrusion porosimetry (MIP). The hydration features associated with microstructure evolution are identified by NCEIM, Micro-Raman spectra and heat evolution test. Through the overall analysis of experimental results, initial formation and evolution of microstructure of CSA cement pastes are identified.

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1. Introduction

Ordinary Portland cement (OPC)-based materials, i.e., traditional concrete materials are the most widely-used artificial materials due to their reliable mechanical properties [1]. These materials are in desperate need of revitalization during several decades: the global production of concrete is expanding rapidly at the rate of approximately 2 billion tons per year, mainly on account of the booming construction industries in China, Brazil and India [2]. By 2050, concrete use is predicted to reach four times the 1990 level [2]. Of particular note is that, on one hand, OPC production is considered as a highly energy intensive process and responsible for huge emission of carbon dioxide [2,3], on the other hand, the rehabilitation and maintenance of deteriorated concrete

structure caused by certain durability issues are bound to aggravate the economic burden of society [4,5]. It is reported that at least US\$1.6 trillion is required to eliminate the deficiencies of infrastructure systems in US [5]. Therefore, the development of alternative, green and sustainable cements is extremely essential and urgent to address concerns of growing durability, maintenance and environmental protection. Calcium sulfoaluminate (CSA) cement is considered as one kind of promising alternative green sustainable cement-based materials in the near future [6]. CSA cement-based materials with low carbon footprint have outstanding tailored properties (shrinkage compensation, large resistance to freeze-thaw/chemical attack) and exhibit comparable or even better durability performance than traditional OPC-based ones [6]. The study of CSA cement-based materials is particularly appealing as their microstructure is very different from that of OPC-based ones [6]. The distinguished overall performance of CSA cement-based materials is primarily attributed to the dense

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pore structure inside and gradual formation of hydrated products [7].

However, until now, the characterization of the microstructure, especially pore structure, of cement-based materials, let alone CSA-based ones, is still extremely complicated by the fact that pores have different shapes, sizes and connectivity [8]. Cement-based materials with the same total pore volume can even show very different performances depending on their different pore size distributions or on peculiar pore network and connectivity [7,8]. It is also important to note that most of techniques require complicated sample pretreatments which can modify, or even damage the microstructure of the sample, especially at early hydration stages. Generally speaking, it is reported that as the hydration time continues, the amount and distribution of gel and capillary pores remarkably change in cement-based materials [7–11].

A wide variety of techniques has been used for the characterization of the pore structure of cement pastes. The frequently-used microstructure characterization technique for the study of cement pastes is mercury intrusion porosimetry (MIP) that allows the measurement of pore radii in a wide range, such as from 2.5 nm to 360 μm . However, the presence of intrinsic ink-bottle effect somewhat misleads the detailed information of pore structure although some active corrections have been adopted [8,12,13]. Nitrogen adsorption/desorption test is taken as a valid alternative technique for the study of cement paste microstructure [12]. It is emphasized that many data analysis methods and equations in nitrogen adsorption/desorption test are dependent on different assumptions used [8].

The application of non-destructive, in-situ and real-time techniques for microstructure characterization in cement-based materials, especially CSA-based ones is still limited to few academic examples [7]. In this work, an applicative utilization of the innovative non-contact electrical impedance measurement (NCEIM), low temperature differential scanning calorimetry (LT-DSC) and other techniques concerning the study of the microstructural evolution of hydrating CSA cement pastes with different water to cement ratios is preliminarily presented. The combined utilization of these techniques allows similarities and differences in the hydration mechanism and microstructure development of four different cementing systems to be highlighted.

2. Materials and analytical methods

2.1. Raw materials

CSA cement used in this study is manufactured by Tangshan Polar bear Special Cement CO., Ltd. The amount of gypsum of CSA raw cement is taken up 16% by weight. The chemical compositions of CSA cement are shown in Table 1 and cumulative particle percentage of raw CSA cement is illustrated in Fig. 1. BET surface area of CSA cement is 1.787 m^2/g by nitrogen adsorption test. Water used in this study is demineralised.

2.2. Analytical methods

2.2.1. Electrical impedance measurement

The non-contact electrical impedance measurement (NCEIM) is an innovative technique to non-destructively monitor the evolution of hydration, pore structure, permeability and ion diffusivity of cement pastes in real time [9–11,14]. The detailed working mechanism of NCEIM can be found in Ref. [9]; metallic measuring electrodes in traditional electrical impedance measurement are replaced by two inductive components (transformer and leakage current meter). Considering that the chemical water demand for complete hydration of a typical CSA approximately ranges up to 0.62 [11], four CSA cement pastes with different water to cement ratios

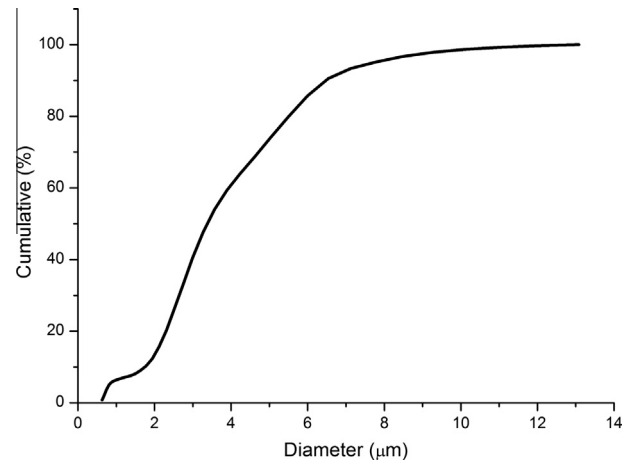


Fig. 1. Cumulative particle percentage of raw CSA cement.

0.4, 0.5, 0.6 and 0.7 were prepared in this study. These fresh pastes were prepared in a planetary-type mixer at 45 rpm for 2 min first and then at 90 rpm for 2 min. The electrical impedance response (modulus and phase) of cement pastes with bulk volume of 1.4 L were recorded continuously for 1 day. The whole test procedure was implemented in an environmental chamber with temperature 20 $^{\circ}\text{C}$ and relative humidity 100%. In the meantime, the temperature development of cement pastes was recorded by a non-contact infrared temperature sensor equipped by NCEIM. With regard to CSA cement pastes, pore structure analysis based on NCEIM is illustrated as follows: porosity (ϕ) is derived from a general effective media model by Eq. (1) [15]:

$$\phi = (0.72F^{-1/2} + 0.18) \times (400^{1/2} - F^{1/2}) / (400^{1/2} - 1) \quad (1)$$

where F is the formation factor and can be determined by impedance response at 1 kHz [15].

While cumulative volume of pores ($V(\leq d)$) whose sizes are less than d , can be determined as [9,10]:

$$V(\leq d) = V_s \times \phi \times \left[1 - (d_{\min}/d)^{D_f} \right] \quad (2)$$

where V_s is the apparent sample volume, approximately 1.4 L, d_{\min} is the minimal pore diameter and predicted as 6.2 nm according to the classic electrical double layers model, D_f is the fractal dimension for pore space and can be calculated from the combination of fractal electrical network and pore structure network [9].

Accordingly, the incremental pore volume of CSA cement pastes is the difference between two adjacent cumulative volumes of pores [9,10].

2.2.2. Heat evolution

It is necessary to analyze the heat evolution of cement pastes since it is identified as a straightforward qualitative indicator of initial formation of microstructure and determination of hydration stages of cement pastes [10,11]. To the study of heat evolution at early age, the isothermal calorimetry test was performed on CSA pastes with different water to cement ratios. The de-ion water was poured into a glass ampoule as a reference, and the investigated fresh CSA paste with the same weight as water was also poured into other glass ampoule of calorimetry using the syringe. The heat evolution of the CSA paste was recorded continuously with hydration time in one day.

2.2.3. Setting time test

The setting time of CSA cement pastes with different water to cement ratios were measured by Vicat instrument according to ASTM C 191-99 [16,17].

2.2.4. Pretreatment for LT-DSC, nitrogen adsorption, MIP and Micro-Raman test

With the purpose of elimination possible discrepancies of results caused by the pretreatment of cement pastes in LT-DSC, nitrogen adsorption, MIP and Micro-Raman tests, according to the pretreatment method recommended by Irigoien et al. [7], the hydrating samples were immersed conformably in ethyl alcohol for 7 days prior to tests in order to replace the pore solution in the pastes with alcohol and stop hydration reactions to greatest extent since the solution was full of Na^+ , K^+ , SO_4^{2-} , Ca^{2+} and OH^- , etc [7,18,19]. The alcohol was renewed regularly every day to guarantee the complete counter-diffusion process [8]. The sample fragments with sizes smaller than 5 mm were then dried in the vacuum oven at 40 $^{\circ}\text{C}$ in a glass reactor for 24 h to remove the remaining free water and alcohol as possible and reserve metastable hydrated phases, Aft/AFm, to greatest degree since Aft may lose some crystallization water and transfer to AFm in some local regions at above 60 $^{\circ}\text{C}$ [11]. In addition, for the cases of LT-DSC and nitrogen adsorption, the characteristic

Table 1

The chemical composition of CSA cement (wt%).

MgO	Al ₂ O ₃	SiO ₂	SO ₃	CaO	TiO ₂	Fe ₂ O ₃
1.7884	36.9257	2.1582	12.8391	43.2713	1.9141	1.0845

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