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The mechanical properties and electrochemical behavior of cement paste containing nano-MgO at different curing temperature

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HIGHLIGHTS

- Nano-MgO was used as admixture to improve the mechanical properties and electrochemical behavior.
- The flexural and compressive strength of cement paste containing nano-MgO cured at 60 °C was higher than that of 20 °C.
- Hydration feature of cement paste containing nano-MgO cured at different temperature was studied by EIS test.
- A suitable electrochemical equivalent model was applied to fit the measured electrochemical impedance data of cement paste.

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ABSTRACT

The purpose of the research work is to investigate the mechanical properties and electrochemical behavior of cement paste containing nano-MgO (NM) which cured at different temperature. The measurements of flexural strength, compressive strength and electrochemical impedance spectroscopy were conducted. A suitable electrochemical equivalent mode was applied to fit the measured electrochemical impedance data (Nyquist curve), therefore the electrochemical property for cement paste containing NM can be quantitatively obtained. Results demonstrated that the flexural and compressive strength gradually increased with the increasing of NM contents ranged from 0 wt% to 9 wt% in cement paste. Moreover, the strength of the cement paste containing NM was increased gradually when the curing temperature rising from 20 °C to 60 °C. In addition, with the increasing of NM content and the rising of curing temperature, the electrolyte solution resistance R_s and the ion transport processes resistance R_{ct1} showed increasing tendency. And XRD, TG-DSC and SEM were used to characterize the phases, mass/heat changes and microstructure of the hardened cement pastes, which provided some evidence on these research findings.

1. Introduction

It is generally accepted that the durability of concrete is the major factor affecting the service life of engineering structures, and crack is one of the main reasons which causes the premature deterioration of concrete [1]. In recent years, the durability of cement-based composites has become more and more prominent [2–5]. Durability and service life of concrete structure will be greatly reduced with the generating of cracks, which will cause possible huge economic losses, even loss of human lives. It is an effective measure to prevent shrinkage and cracking of concrete by using the volume expansion caused by the swelling component in the hydration process to compensate for shrinkage, such as magnesium oxide (MgO) [6]. It was reported that the carefully calcined and sized MgO powders had the potential of being used as expansive agents for compensating thermal shrinkage and thus preventing cracks in mass concrete due to thermal stresses [7,8]. However the presence of excessive amounts of MgO in hardened cement-based materials may lead to expansion and crack formation, which will lead to the reduction of mechanical performances. In order to avoid the possible deleterious effects, most specifications have

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Table 1
Chemical compositions (wt%) of ordinary Portland cement.

Composition	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O	K_2O	LOI*
Portland cement	64.65	21.71	5.06	4.33	0.94	1.07	0.20	0.54	0.86

* LOI: loss on ignition.

placed a limit on the content of MgO that can be present in cement, and the MgO content does not exceed 6% generally [9–12].

In recent years, the technology of nano-structured material is developing astonishing rapidly and will be applied extensively to many materials in the future. Although cement is a kind of common building material, its main hydrate C-S-H gel is a natural nano-structured material [9]. The use of nanoparticles in concrete materials significantly modifies their behavior not only in the fresh but also in the hardened conditions, as well as the mechanical and microstructure development. Moradpour [13] studied the effects of nanoscale expansive agents on the mechanical properties of non-shrink cement-based composites, the results indicated that the mechanical strength of the composites mixed with the nano-MgO were higher than those of a plain composite and the autoclave expansions of Portland cement paste containing 3% and 5% nano-MgO were 0.10% and 0.12%, respectively. The researches indicated that the thermal conductivity of cement paste containing NM is higher than that of pure cement paste after heated [14]. Riza [15] reported the effects of different percentages of pre-saturated expanded perlite aggregate micro and nano size MgO on autogenous shrinkage of the mortars, and the results showed NM has appeared to be more effective than micro size MgO. A research results also indicated that the addition of the micro and nano MgO particles to poly methyl methacrylate have improved the quality of bone-cement union [16]. Ye [9] reported the maximum content of NM added into ordinary Portland cement reaches up to 8% for soundness, and the shrinkages of dam concrete with NM and light burnt MgO may be completely compensated in safety.

In order to more effectively analyze microstructural changes include the hydration process and the durability experiment, electrochemical impedance spectroscopy (EIS) has been used to estimate the properties of concrete [17,18]. The cement-based material is a complex nonuniform multiphase system which consists of the solid phase, gas phase and liquid phase. In principle, the solid phase is generally considered to be an insulator, and the conductivity of a cement system is mainly contributed by the ions in the solution of continuous microspore network such as Ca^{2+} and OH^- [19–22]. With the action of a variable frequency electric field, these phases in the cement-based material are interconnected to constitute an integral circuit. Therefore, it was possible to estimate the composition and intrinsic microstructure of the cement paste by EIS.

Based on the researches of the use of NM in cement-based composites, few studies have been reported on the mechanical performances and electrochemical behavior in assessment of cement-based composites containing NM at different curing temperature, but the effect of temperature on concrete such as dam concrete is very huge in practical engineering. In this paper, we propose to add NM into the ordinary Portland cement, and take different curing temperature (20 °C, 40 °C, 60 °C) for the cement samples, then the mechanical strength, electrochemical impedance spectroscopy and equivalent electrochemical circuit simulation are provided in the paper. In addition, the hydration phases, the mass/heat change and the microstructure of the hardened cement pastes are characterized by X-ray powder diffraction (XRD), Thermogravimetry-Differential Scanning Calorimeter (TG-DSC) and Scanning Electron Microscopy (SEM), respectively.

2. Materials and methods

2.1. Raw materials

A commercial ordinary Portland cement (42.5 grade, Blaine specific surface area 310 m^2/kg complying with Chinese standard (GB 175–2007) was used, its compressive and flexural strengths at the age of 28 days were 46.4 and 7.2 MPa, respectively. The chemical compositions of the cement are given in Table 1. Nano-MgO (NM) particles were supplied by Hangzhou Wanjing New Material Co., Ltd. in China, with specific surface area 25 m^2/g, average diameter 50 nm and bulk density 0.25 g/ml. Its mineral composition is presented in Fig. 1.

2.2. Preparation of cement paste

For the cement paste, cement with NM was fully mixed under dry conditions beforehand. The cement paste was prepared using a planetary mixer (ISO 9597). A cement (containing NM) to water ratio 0.5 was used, together with different NM content of 0%, 3%, 5%, 7% and 9%, the mixture proportions of specimens are presented in Table 2. The fresh paste was cast into prismatic moulds 40 mm $\times$ 40 mm $\times$ 160 mm on a jolting table. The paste samples which were cured at 20 ± 2°C and about 95% RH moisture were demoulded at 24 h and then stored in saturated limewater at 20 °C, 40 °C and 60 °C until required for testing.

2.3. Strength test for cement containing nano-MgO

The flexural and compressive strengths were obtained by Microcomputer electro- hydraulic servo pressure testing machine (HG-HY-300F). The span for flexural strengt was 100 mm and the area for compressive strength was 40 $\times$ 40 mm^2. Three prisms were tested for each sample at the designated ages. The flexural strength test was carried out by a group of three samples, and then

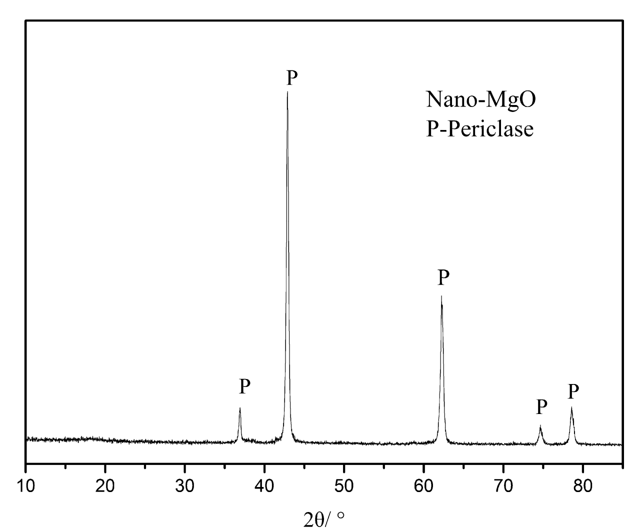

Fig. 1. XRD patterns of nano-MgO.