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# Electrochemical impedance spectroscopy (EIS) of hydration process and drying shrinkage for cement paste with W/C of 0.25 affected by high range water reducer



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#### HIGHLIGHTS

- Cement paste with lower water-cement ratio of 0.25.
- The electrochemical spectroscopy (EIS) used for assessing the evolution of the rheological properties of fresh cement paste.
- Effect of high range water reducing admixture on EIS.
- The parameter of Rct in ElS closely related to the fluidity of cement paste tested by mini cone.

#### ARTICLE INFO

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#### ABSTRACT

The objective of this paper is to examine the hydration behaviour, fluidity and drying shrinkage of cement paste with lower water-cement ratio (W/C) of 0.25. The electrochemical technique is utilized to assess the evolution of the hydration process of fresh cement pastes with different amount of high range water reducing admixture (HRWR), as well as the fluidity measured by the mini cone method. The experimental results show that HRWR can effectively influence the hydration behaviour of paste. The parameter  $R_{ct}$  obtained by Nyquist curves varies as the dosage of HRWR increase from 0.5% to 1.4%, indicating that HRWR indeed has obvious effect on the ability of paste transferring charge, further influence the structure of fresh cement paste. Meanwhile, the parameter of  $R_{ct}$  is closely related to the fluidity of fresh cement paste measured by mini cone. Additionally, the drying shrinkage of cement paste increase when the dosage of HRWR changes from 0.8% to 1.4%. The cement paste with 0.8% HRWR has the highest increasement of drying shrinkage at the first 10 days. However, the drying shrinkage of cement pastes with 0.8% HRWR has the lowest value at 90 days.

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#### 1. Introduction

As the most important construction materials, cement is widely used in the world and dominate the built environment. The cement paste is the most definitive component of mortar and concrete. Therefore, the fluidity properties of cement paste are of greatest importance in making quality concrete products, as well as in conducting efficient construction works [1].

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When Portland cement is mixed with water, a series of chemical reactions begin to take place. The reactions of cement with water proceed at different rates for the various mineral phases and involve both hydrolysis and hydration processes [2]. Hydration is a chemical process leading to the formation of hydrates and has thermodynamic, kinetic, and structural features, which depend on both chemical and physical parameters [3]. Setting is a definite time event that corresponds to the transition of the paste from soft to hard. The changes in the physical state of water and ionic concentrations within the gauging water were reflected by the change of cement paste electrical conductivity, which can be measured by many technologies [4–6].

The hydration behaviour of fresh cement pastes is very interesting because of their influence on the consistency, workability and

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setting characteristics of the cement. Understanding how to control the hydration behaviour of the fresh cement paste is very important for the economical proportioning of concrete and proper mixing and placement methods. In this paper, the hydration behaviour of fresh cement paste with the water-cement ratio (W/C) of 0.25 are studied by the methods electrochemical impedance system, and the fluidity is measured by the mini cone, respectively. The purpose of this paper is to examine the hydration behaviour and fluidity of fresh cement paste with different amounts of high range water reducing admixture, and the drying shrinkage of hardened cement paste.

#### 2. Experimental

The materials used in the preparation of cement paste are Portland cement 42.5, a kind of high range water reducing admixtures (HRWR) named by polycarboxylated-based superplasticizer produced by Yucai in China, and normal tap water. The chemical composition of cement and the properties of high range water reducing admixture are listed in Tables 1 and 2.

In this paper, the water-cement ratio (W/C) is kept at 0.25. In order to study the effect of HRWR on hydration behaviour and fluidity of cement pastes, five different groups of fresh cement paste are used, the dosage of HRWR is 0.5%, 0.8%, 1.0%, 1.2% and 1.4% of cement mass (wt.%), respectively, which are remarked by G1, G2, G3, G4 and G5, respectively.

In this paper, the mini cone is applied to investigate the fluidity of cement paste, which is similar to the slump cone (ASTM C-143) used for concrete [7]. The dimensional shape of the mini cone is shown in Fig. 1(a). The top and bottom of the mini cone is

**Table 1**Chemical compositions of Portland cement.

SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	R <sub>2</sub> O	SO <sub>3</sub>
21.08	5.47	3.96	62.28	1.73	0.50	2.63

**Table 2**Properties of high range water reducing admixtures (HRWR).

State	Color	pH value	Density	Water solubility
Liquid	Brown	7.5	1.02-1.06	Good solubility

36 mm and 60 mm, respectively, and the height of the cone is 60 mm. When the mini cone is used for the spread measurement of fresh mixture, the truncated cone mould is firstly placed on a glass plate, and then filled with fresh paste. The resulting final diameter of the fresh paste sample is the mean of two measurements made in two perpendicular directions (Fig. 1(b)). In addition, for each cement mixture, the spread measurement is conducted for 10 min, 20 min, 30 min, 60 min, 90 min and 120 min, respectively. The setting time is calculated from the cement and water mixing together.

The viscosity of all cement paste with different HRWR, is used to describe the internal friction of cement paste and a measurement of resistance to flow, tested by a coaxial rotating cylinder viscometer that determined apparent viscosity of the cement paste, referred to the method provided in the literatures [8.9]. The tests were taken under rotor speed control mode with cylinder spindle at 60 rpm. In this mode, the shear rate was increased with the rotor speed increase. After mixing, the as-prepared slurry was immediately poured into the grout cup. The apparent viscosity value was taken when the needle in the viscometer was stabilized, or 45 s after the rotation of shaft in cases when the needle had not stabilized due to the thixotropy of the cement pastes. The time of viscosity value reading was generally between 10 and 30 s. The entire process totally took below 60 s, which was less than the initial setting time. The apparent viscosity was obtained under constant rotor speed, then the shear rate was almost similar. So the results of viscosity can be taken for comparison.

The electrochemical impedance method is employed to evaluate the structure evolution of hydration behaviour of cement paste. The two-point electrode technique is used for the electrochemical impedance measurement, as shown in Fig. 2. The two electrodes are made by special material which can resist the high alkaline environment. In this paper, in order to minimize the point contact resistance resulting from poor contacts between the electrodes and sample surfaces, the electrodes are directly inserted the fresh mixture. The impedance measurements are carried out using advanced electrochemical system of Parstat 2273. The real part ( $Z_{re}$ ) and imaginary part ( $Z_{im}$ ) of the cell impedance are measured for various frequencies (10,000  $\sim$  0.01 Hz), which can be plotted as Nyquist curves. The electrochemical impedance measurement is conducted from the fresh paste at the first one hour and two hours which are calculated from water mixing cement together.

For drying shrinkage test, three bar specimens measuring  $280 \times 25 \times 25$  mm with two embedded copper heads at the two

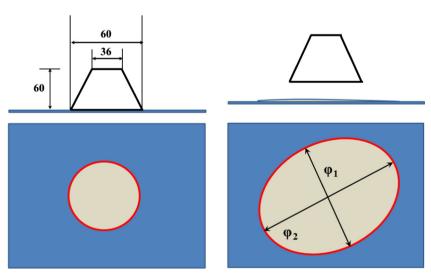


Fig. 1. The spread measurement of cement mortar used by mini cone, right (a) and left (b) (Unit: mm).

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