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# Evaluation of alkali reactivity of concrete aggregates via AC impedance spectroscopy

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## HIGHLIGHTS

- The alkali reactivity of concrete aggregates was evaluated by AC impedance spectroscopy.
- Results of AC impedance spectroscopy are compared with linear expansion ratio and relative dynamic elastic modulus.
- AC impedance spectroscopy can be used to test and evaluate the alkaline reactivity of concrete aggregates.

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## ABSTRACT

The alkali reactivity of concrete aggregates was evaluated by AC impedance spectroscopy as the main method along with linear expansion ratio, relative dynamic elastic modulus, and mercury intrusion porosimetry tests. The electrochemical parameters of AC impedance spectra showed a close relationship with the hydration reaction and microstructure of cement-based materials. The results obtained by the linear expansion ratio, the relative dynamic elastic modulus, and the pore structure characteristics were basically consistent with those obtained by the impedance spectra. Therefore, the ASR trend shown by the resistance in AC impedance spectra, Rs, and the phase angle θ, can be considered as basically reliable. The intensity of the ASR of concrete changed with different contents of alkali aggregates. The test showed that in the ASR at an early age, the ratio between the active and inactive aggregates had the worst point. As the content of active alkali aggregates was at the worst point, the ASR led to the maximum expansion ratio and caused the most severe harm to the concrete specimens.



## 1. Introduction

Alkali–silica reaction (ASR), a common durability problem of concrete, was first discovered in the 1940 s [1–3] and has since then remained a hot topic of engineering research in the subsequent decades. In ASR, $Na^+$, $K^+$, and $OH^-$ in the pore solution of concrete react with the active siliceous components of aggregates and cause the cracking of concrete through the water swelling of reaction products [4–6]. Unlike the destruction induced by external media such as carbonation and erosion by salt solutions, ASRs occur entirely inside the concrete, thus causing severe damage to the entire concrete structure. The studies on ASR involve many aspects; the determination of the alkali reactivity of aggregates is still one of the most important aspects [7,8]. The existing methods for the determination of the alkali reactivity of aggregates include the lithofacies method, chemical method, and mortar bar method (MBM); the most commonly used method is MBM. However, all of them have some drawbacks such as poor accuracy, susceptibility to misjudgment, and other issues.

AC impedance spectroscopy is an electrochemical method used to study the microstructure and properties of materials. By inputting some small-amplitude sinusoidal AC voltage signals with different frequencies, the output response can be measured, and the properties of materials can be studied [9–12]. A concrete system can be considered as an electrochemical system because concrete is a porous medium, and the solution in pores is an electrolyte. Since the 1980 s, a series of AC impedance spectroscopic studies on concrete material have been carried out including the hydration process of cement, structure of hardened paste, durability of concrete, and corrosion problem of rebar in concrete [13–19]; however, impedance spectroscopic studies on the ASR of concrete are

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rare. AC impedance spectroscopy was used as the main method to study the alkali reactivity of concrete aggregates. Moreover, the linear expansion ratio, relative dynamic elastic modulus, and pore structure characteristics of specimens for ASR were tested. The linear expansion ratio provides the basic expansion characteristics of concrete specimen under ASR; the relative dynamic elastic modulus provides the deterioration of the mechanical properties of specimen after water swelling; the pore structure characteristics provide the basic variation in the internal porosity and pore size distribution of concrete. By comparing these parameters with AC impedance spectra, we determined the reliability of AC impedance spectroscopy in evaluating the alkali reactivity of concrete aggregates.

## 2. Raw materials and test methods

### 2.1. Raw materials for test

The cement used in these tests was pure Portland cement made of Portland cement clinker and 5% gypsum; its mineral and chemical compositions are shown in Tables 1 and 2. The alkali content in this cement is 0.53%. As we know the alkali-aggregate reaction is very slow in normal state. However, it should be accelerated in laboratory. As Portland cement was used in these tests, a certain amount of NaOH was added to adjust the alkali content to 1.25%. The alkali content of cement was expressed as $Na_2O_{eq}$, where $Na_2O_{eq}\% = (Na_2O + 0.658K_2O)$ wt% [8,20,21].

The aggregates involved in these tests included active and inactive aggregates. The main active component in the active aggregate was amorphous $SiO_2$; its chemical composition is shown in Table 3. The main inactive aggregate was quartz sand. These two types of aggregates were obtained by sieving with a 4.75 mm square hole sieve to control the particle size between 1.18 mm and 4.75 mm.

### 2.2. Preparation and curing of specimens

The specimens used in these tests were prism specimens divided into two types, i.e., type A and type B. The A-type specimen had a molded dimension of (30 × 30 × 300) mm; it was used for the linear expansion ratio and relative dynamic modulus tests. The B-type specimens had a molded dimension of (30 × 30 × 30) mm, it was used for AC impedance spectroscopy and pore structure test. The cement/aggregate/water ratio of the specimens was 1:2:0.3. The aggregate contained active and inactive aggregates of different proportions. According to the different mass percentages of active aggregate in overall aggregates, 0%, 20%, 40%, 60%, 80%, and 100%, the corresponding numbers of specimens were A0, A20, A40, A60, A80, A100 and B0, B20, B40, B60, B80, B100, respectively. The detailed proportions are shown in Table 4. After the specimens were molded, they were kept in the curing chamber under standard curing conditions (temperature: 20 ± 2 °C, relative humidity ≥95%) for 24 h, released, and then placed in a reaction chamber at 60 °C. The linear expansion ratio, relative dynamic elastic modulus, AC impedance spectroscopic response, and pore structure characteristics of the specimens were measured periodically.

## 3. Results and discussion

### 3.1. AC impedance spectra of specimens

The schematic diagram of AC impedance spectroscopy is show in Fig. 1. Literatures [22,23] show that AC impedance spectroscopy parameters can better reflect some microstructural characteristics of concrete material. For example, $R_s$, the electrical resistance of an

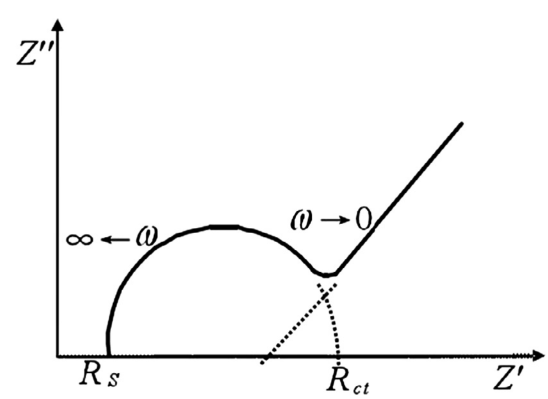


**Fig. 1.** Schematic diagram of AC impedance spectroscopy.

**Table 1**
Mineral compositions of cement (wt%).

| $C_3S$ | $C_2S$ | $C_3A$ | $C_4AF$ | Gypsum |
|---|---|---|---|---|
| 57.65 | 19.95 | 6.85 | 10.55 | 5 |

**Table 2**
Chemical compositions of cement (wt%).

| CaO | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | MgO | $SO_3$ | $K_2O$ | $Na_2O$ |
|---|---|---|---|---|---|---|---|
| 63.87 | 21.79 | 4.77 | 3.38 | 3.14 | 2.29 | 0.55 | 0.21 |

**Table 3**
Chemical compositions of active aggregate (wt%).

| $SiO_2$ | $Al_2O_3$ | CaO | MgO | $Fe_2O_3$ | $K_2O$ | $Na_2O$ | $SO_3$ |
|---|---|---|---|---|---|---|---|
| 58.51 | 14.94 | 12.89 | 4.78 | 4.37 | 2.66 | 1.64 | 0.21 |

**Table 4**
Mixing proportions of concrete samples (wt%).

| | **A0, B0** | **A20, B20** | **A40, B40** | **A60, B60** | **A80, B80** | **A100, B100** |
|---|---|---|---|---|---|---|
| **Cement** | 1 | 1 | 1 | 1 | 1 | 1 |
| **Reactive Aggregate** | 0 | 0.4 | 0.8 | 1.2 | 1.6 | 2 |
| **Non-reactive aggregate** | 2 | 1.6 | 1.2 | 0.8 | 0.4 | 0 |
| **Water** | 0.3 | 0.3 | 0.3 | 0.3 | 0.3 | 0.3 |