

# Electrochemical investigations on the corrosion behaviour of reinforcing steel in diatomite- and zeolite-containing concrete exposed to sulphuric acid



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## HIGHLIGHTS

- We have identified corrosion behaviour of reinforcing steel in concrete exposed to sulphuric acid.
- We found that porosity plays a very important role in reinforcing concrete.
- The steel reinforcement in the zeolite was less corroded by the H<sub>2</sub>SO<sub>4</sub> solution than the reference and the diatomite samples.

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## ABSTRACT

Corrosion is a major concern for most structural applications. Its detrimental effect significantly reduces the life of metallic components. This paper presents the results of an experimental investigation of corrosion in the steel reinforcement of concrete samples having three different substituents: 20% diatomite, 20% zeolite, and a reference without zeolite or diatomite. All concrete specimens were subjected to a solution of 0.5 M H<sub>2</sub>SO<sub>4</sub> for 160 days, and every 15 days electrochemical impedance spectroscopy (EIS) measurements were performed. The results indicated that porosity plays a very important role in reinforcing concrete. The steel reinforcement in the zeolite was less corroded by the H<sub>2</sub>SO<sub>4</sub> solution than the reference and the diatomite samples.

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## 1. Introduction

Reinforced concrete is an excellent construction material due to its technical, economical and ecological advantages. However, corrosion of the reinforcing steel in concrete has become a major problem in construction [1,2]. The main causes of reinforcing steel corrosion are reaction with various aggressive agents, such as atmospheric carbon dioxide and chloride ions, and chemical attack throughout the service life of the concrete [3]. In ordinary Portland cement, these harmful effects can be reduced by substitute pozzolans [4]. Under a corrosive environment, concrete properties can be improved by using pozzolans such as zeolite and diatomite [5–7].

Zeolite is an important pozzolan. There are numerous publications on the properties and use of zeolite [8–13]. Zeolites form a large family of crystalline aluminosilicates which have been widely used as additives in construction since ancient times. Zeolites are microporous crystalline solids with a well-defined structure consisting of a three-dimensional network of silicon–oxygen (SiO<sub>4</sub>) and aluminum–oxygen (AlO<sub>4</sub>) tetrahedra and offering large, internal and external surface areas, [14]. The high surface area is the basis of the high reactivity. Previous works on the pozzolanic reactivity of zeolites have shown that zeolitic minerals are able to react with lime, produce cementitious phases and improve concrete properties [15].

Diatomite (kieselguhr) is a pozzolanic material containing high amounts of natural amorphous silica (silicon dioxide, SiO<sub>2</sub>). Diatomite is a sedimentary rock composed of the fossilized skeletons of unicellular fresh-water plants known as diatoms. Diatoms are honeycomb silica structures that give diatomite useful characteristics

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such as high absorptive capacity, high surface area, high permeability, small particle size, chemical stability, excellent grindability, low thermal conductivity and low bulk density [16–18]. Due to these properties, diatomite can be used as a pozzolanic material for partial replacement of cement in the production of concrete.

Corrosion was a significant phenomenon when the August 17 and November 12, 1999, Duzce earthquakes occurred in Turkey. Extensive research was carried out by Uygur [19] on the results of this earthquake damage. Building collapse was reported to be as high as 80% due to the low mechanical properties of the concrete and steel bars. It was shown that even 1 year of atmospheric pollution could cause serious corrosion production on steel bars, decreasing their area and damaging the bar-concrete surface. Corroded steel bars lose their ductility and can easily fail in a brittle manner. Moreover, it was concluded that using corroded steel bars in a structure could result in vital failures during dynamic loads such as earthquakes.

In the present study, the prepared samples were immersed in a solution of 0.5 M  $H_2SO_4$  in order to investigate the reactions which take place during acid rain damage to concrete. Several structural elements of concrete are susceptible to chemical attack by sulphuric acid, including foundations (due to groundwater containing sulphuric acid formed by the oxidization of pyrite in backfill), industrial floors of chemical plants, basement walls of buildings near chemical plants, superstructures (due to acid rain), etc. [20].

Therefore, the purpose of this study was to use electrochemical impedance spectroscopy (EIS) to assess the corrosion resistance of reinforcing steel from the chemical attack of 0.5 M  $H_2SO_4$  when the concrete was blended with zeolite and diatomite.

## 2. Experimental study

### 2.1. Materials and specimen preparation procedures

The chemical composition and some physical properties of the materials used for the concrete preparation are given in Table 1. Table 2 shows the percentage contributions of substituents of the three sample types. A schematic illustration of the embedded steel in the concrete samples is presented in Fig. 1 [21]. The total area of each sample was equal to 94.2 cm<sup>2</sup>.

The test specimens were produced according to TS EN 197-1 (EN 197-1, 2002) [22], and CEM I 42,5 R [23] Portland cement was used as a concrete binder. The diatomite was obtained from the Kutahya region and the zeolite was provided from the Balikesir region of Turkey. Well water obtained from the Duzce region was used for mixing. The water/cement ratio was 0.5.

The porosity values were determined according to the ASTM C642 [24] standards and presented in Fig. 2. The zeolite sample exhibited the smallest porosity, equal to 0.85, the reference 2.05 and the diatomite 4.67. The first measurements were carried out 15 days after immersing the samples in solution.

### 2.2. Test method: electrochemical impedance spectroscopy (EIS)

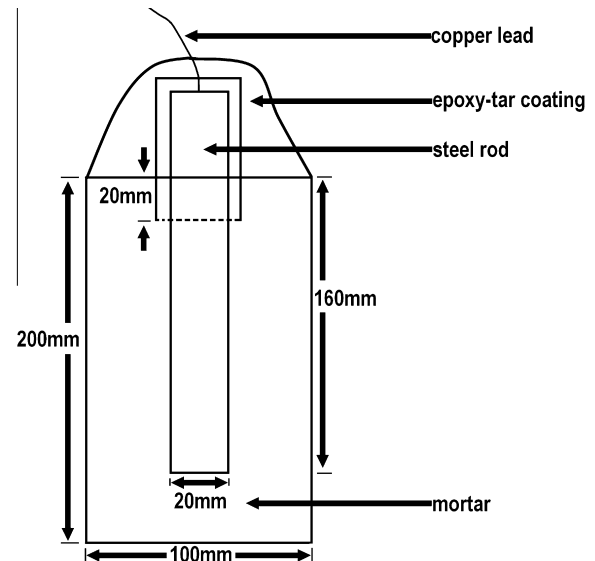
Electrochemical methods based on alternating currents can be used to obtain insights into corrosion mechanisms and to establish the effectiveness of corrosion control methods. The use of electrical impedance spectroscopy (EIS) as a

**Table 1**  
Chemical composition and physical properties of three types of concrete constituents.

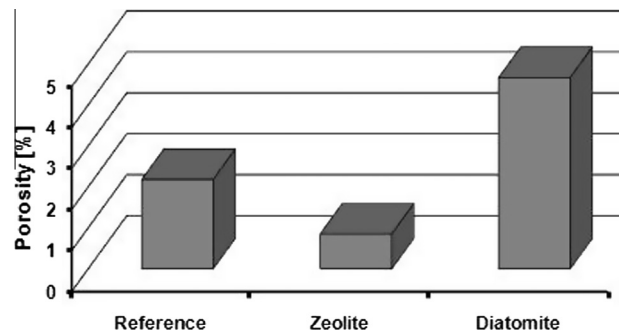
| Chemical components%           | Reference |                |              | Physical properties                  | Reference |                |              |
|--------------------------------|-----------|----------------|--------------|--------------------------------------|-----------|----------------|--------------|
|                                | Reference | Pure diatomite | Pure zeolite |                                      | Reference | Pure diatomite | Pure zeolite |
| SiO <sub>2</sub>               | 18.68     | 79.56          | 68.85        | Blaine size (cm <sup>2</sup> /g)     | 4249      | 13.640         | 5740         |
| Al <sub>2</sub> O <sub>3</sub> | 4.67      | 6.54           | 11.71        | Specific weight (g/cm <sup>3</sup> ) | 3.17      | 2.28           | 2.18         |
| Fe <sub>2</sub> O <sub>3</sub> | 3.53      | 2.76           | 1.29         | 90 μm Sieve analyses (%)             | 4.08      | 9.80           | 17.60        |
| CaO                            | 64.56     | 2.45           | 3.97         | 45 μm Sieve analyses (%)             | –         | 28.60          | 35.80        |
| MgO                            | 0.98      | 0.79           | 1.06         |                                      |           |                |              |
| SO <sub>3</sub>                | 3.00      | 0.48           | 0.18         |                                      |           |                |              |
| Na <sub>2</sub> O              | 0.14      | 2.63           | 0.29         |                                      |           |                |              |
| K <sub>2</sub> O               | 0.73      | 0.69           | 2.19         |                                      |           |                |              |
| Loss of ignition               | 3.92      | 3.88           | 10.00        |                                      |           |                |              |
| Free lime                      | 1.74      | –              | –            |                                      |           |                |              |
| Insoluble residue              | 0.50      | 75.98          | 37.32        |                                      |           |                |              |

**Table 2**  
Composition percentages of tested materials.

| Name of the sample | Reference      | Diatomite                           | Zeolite                           |
|--------------------|----------------|-------------------------------------|-----------------------------------|
| Constituents       | 100% Reference | 80% Reference<br>20% Pure diatomite | 80% Reference<br>20% Pure zeolite |



**Fig. 1.** Shape of working electrode.



**Fig. 2.** Porosity of concrete of zeolite, reference and diatomite samples.

characterisation tool in the study of cementation materials has been well documented [25–29]. Electrochemical measurements were carried out in a three-electrode type cell with separate compartments for the reference electrode (Ag/AgCl),

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