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# Curing time and heating conditions for piezoelectric properties of cement-based composites containing PZT

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#### HIGHLIGHTS

#### GRAPHICAL ABSTRACT

- A double preheated technique that enhance piezoelectric properties of piezoelectric cement was uncovered.
- Piezoelectric cement with preheat treatments at 140 °C and cured for 1 day provides notable  $d_{33}$  and  $\varepsilon_r$  values.
- Piezoelectric cement cured for more than 3 days prior to the polarization shows minor effects on piezoelectric properties.
- 50% PZT/cement composites with d<sub>33</sub> = 110.9 pC/N has been fabricated as piezoelectric cement sensors potentially used in SHM.

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#### 1. Introduction

Piezoelectric sensors and actuators have been applied to structural health monitoring (SHM) in civil engineering in products such as tunnel linings, bridges, side slopes, rigid pavements, and some concrete structures over the last two decades [1–5]. Many studies



#### ABSTRACT

Samples of piezoelectric cement, consisting of equal volumes of lead zirconate titanate and type I Portland cement, were treated through a heating technique and cured for controlled lengths of time to produce notable piezoelectric properties. Piezoelectric properties were measured by considering curing times of 1, 3, and 7 days, and applying 23 °C or 140 °C treatments on the piezoelectric cement samples. Curing time showed minor effects on the piezoelectric strain factor  $d_{33}$ , relative dielectric constant  $\varepsilon_r$ , and electromechanical coupling coefficient  $K_t$  at late ages for specimens that were cured for more than 3 days. Pretreatment with higher temperatures on piezoelectric cement is expected to obtain higher  $d_{33}$  and  $\varepsilon_r$  values than posttreatment would obtain. Two heat treatments at 140 °C and 1 day of curing for piezoelectric cement provide superior  $d_{33}$  and  $\varepsilon_r$  values, with  $d_{33} = 110.9$  pC/N and  $\varepsilon_r = 756$  at 100 days. However, it is necessary to enhance the  $K_t$  of piezoelectric cement at least 3 days' curing.

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have discussed the use of piezoelectric sensors and actuators instead of strain gauges for health monitoring and damage detection [6–14]. Piezoelectric sensors can be affixed on surfaces and embedded in structures or materials to monitor, detect, and estimate mechanical behavior according to the reactions of output voltages or impedance. For instance, Xu et al. [6] examined the impedance spectra of lead zirconate titanate (PZT) piezoelectric sensors near their resonance frequency to detect structural crack damage. Meng and Yan [7] installed piezoelectric ceramic sensors

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in eccentric compression concrete columns for online crack monitoring. Jabir and Gupta [8] affixed thick-film ceramic strain gauge sensors to beams to measure strain change, and compared the results with those of metal foil strain gauges. According to the electromechanical impedance of a single PZT sensor and the wave propagation technique for multiple PZT sensors, smart aggregates (with embedded PZT transducers) were applied to study both the local and overall conditions of a structure by comparing the loading, output voltage, and cracking [9]. Recently, many smart aggregates have been reported in connection with structural damage detection [12], strength gain in concrete [13], and temperature and loading effects of concrete structures [14].

Most conventional piezoelectric sensors and smart aggregates made with piezoelectric ceramics and piezoelectric polymers [4,9,12–14] exhibit superior properties of electrical sensitivity for structural health monitoring. Cement-based piezoelectric composites have been developed since 2002 to eliminate mismatches in volume compatibility and acoustic impedance for concrete structures and conventional piezoelectric sensors [15–17]. For similar purposes, the 0–3 type cement-based piezoelectric composite (named piezoelectric cement) has been presented [18–24], particularly for piezoelectric cement containing 50 vol.% PZT inclusions with acoustic impedance near 9 or  $10 \times 10^{-6}$  kg-m<sup>-2</sup> s<sup>-1</sup>, which approaches the acoustic impedance of concrete [16]. This type of piezoelectric cement, which has both piezoelectric inclusions and a cement matrix, shows piezoelectric properties after it has been polarized through applying outer electric voltages.

Piezoelectric cement is a two-phase composite with PZT inclusions uniformly distributed in a cement matrix. Potentially, piezoelectric cement can be used as piezoelectric sensors and actuators for SHM in concrete structures. Nevertheless, low values of the piezoelectric strain factor  $(d_{33})$  in piezoelectric cement constitute a typical weakness for piezoelectric cement applications in comparison with the superior values attained by piezoelectric PZT ceramic. For the past decade, many studies have attempted to promote the piezoelectric properties of piezoelectric cement by considering the forming pressure of specimens [25–27], curing temperature [27–29], poling voltage and temperature [28–30], poling time [28,30–33], cement type and piezoelectric inclusion type [24,25], particle size and amount of PZT content [18,21,22,28,30,34,35], and admixtures [22,23,36-43]. In those studies, piezoelectric cement samples that contained higher PZT content or were subjected to higher poling temperatures and poling voltages always displayed higher piezoelectric properties. Higher poling efficiency was observed when piezoelectric cement was subjected to poling times ranging from 40 to 45 min [23,28,33]. For most piezoelectric cement with 50–70% PZT particles, the highest piezoelectric strain factor  $d_{33}$  and relative dielectric constant  $\varepsilon_r$  values reported are approximately 60 pC/N and 300, respectively.

As previously mentioned, 50 vol% PZT in piezoelectric cement suffices to lower the mismatched acoustic impedance between piezoelectric cement and concrete structures in SHM. However, the piezoelectric properties of most piezoelectric cement containing 50% PZT are  $d_{33} = 35-55$  pC/N and  $\varepsilon_r = 60-300$  [16–18,24,25, 37–39], depending on the manufacturing process, poling conditions, and constituents. In addition, piezoelectric cement sensors can be used in SHM without any supplementary charge amplifier when the  $d_{33}$  value exceeds 70 pC/N [40]. Research aimed at producing substantial piezoelectric properties for piezoelectric cement in concrete structures is ongoing.

A recently proposed heating technique achieved superior piezoelectric properties in piezoelectric cement containing 50 vol% PZT [44], yielding  $d_{33}$  = 106.3 pC/N and  $\varepsilon_r$  = 477 when heat treatment was applied at 150 °C. Higher temperature treatments on specimens prior to polarization cause lower dielectric losses and higher poling efficiency. This heat treatment is a more effective method for enhancing the  $d_{33}$  and  $\varepsilon_r$  values for 0–3 type PZT/cement composites without adding admixtures in comparison with previous reports [15–43]. To consider the effects of heating on cementbased piezoelectric composites, in the present research, combinations of heating conditions and curing times were investigated for their effects on the piezoelectric properties of PZT/cement composites with 50 vol% PZT. The heating technique reported in this article differs slightly from that of a previous study [44].

#### 2. Experiments

#### 2.1. Materials and specimens

Piezoelectric cement consists of type I Portland cement as the matrix and PZT ceramic as the inclusion. Fresh cement is required with 349 m<sup>2</sup>/kg fineness and specific gravity of 3.15. The properties of the PZT ceramic used in this research, which were measured by Eleceram Technology (Taiwan), are as follows: density =  $7.9 \times 10^3$  kg/m<sup>3</sup>;  $d_{33} = 470$  pC/N;  $\varepsilon_r = 2100$ ; and piezoelectric voltage factor  $g_{33} = 24 \times 10^{-3}$  V-m/N. The particle size of the PZT ceramic, shown in Fig. 1, was in the range of 75–150 µm (#100–#200 ASTM sieve). In the specimen, 50% of the volume was PZT and the other 50% was cement. This type of piezoelectric cement is denoted as "PP material" in this article.

Cement and PZT particles were prepared first, and then mixed without additional water by using a solar-planetary blender for 5 min to ensure that the constituents were uniformly dispersed with each other. The mixture was divided into three portions, each of which was put into a 15-mm-diameter cylindrical steel mold, forming layers. Each layer of the mixture in the mold was peened with a rubber hammer to expel any air that could have been introduced by the mixing process. Subsequently, 80 MPa compression was applied to the mixture for 5 min, forming a disc-like specimen. Specimens were cured in a controlled chamber at 90 °C and 100% relative humidity during the first curing day to ensure that hydration would produce suitable strength; when necessary, the specimens were placed in 90 °C water for longer curing. Before polarization, specimens of three PP materials were cured for 1 day (PP1), 3 days (PP3), and 7 days (PP7).

When a specimen's final curing day was reached, that specimen was polished to a thickness of  $2 \pm 0.05$  mm. The pores of the specimens were observed and measured through optical microscopy (OM) at 350x magnification. The porosity was then determined through an image analysis with pixel threshold criteria. Nine positions on each specimen were measured to determine the average porosity of the corresponding specimen.



Fig. 1. The PZT ceramic (left) was granulated to 75-150 µm particles (right).

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