# Effect of Free Water on Polarization and Piezoelectric Coefficients of Cement-Based Piezoelectric Composites During Manufacturing Process

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Abstract. Cement-based piezoelectric composites (PECs) consist of calcium aluminate cement (CAC) and lead zirconate titanate (PZT), each accounting for 50 vol.% that can be used for structural health monitoring (SHM) due to their excellent compatibility with cementitious structures. The presence of free water inside the specimen significantly affects the polarization difficulty and piezoelectricity of PEC. Four treatment methods include vacuum drying, ethanol dehydration, non-heat treatment (untreated), and heat treatment to reduce free water in specimens. Experimental results show that reducing the free water content of PEC specimens through vacuum drying, ethanol dehydration, and heat treatment during the manufacturing process can enhance PEC performance. The free water reduction effect of PEC specimens was most with the heat treatment, followed by ethanol dehydration, and least by vacuum drying. The specimen's dielectric loss and relative permittivity before polarization decreased if heat treatment and ethanol dehydration were applied. Heat-treated specimens provide optimal relative permittivity and piezoelectric strain constant after polarization. For the piezoelectric voltage constant, ethanol dehydration of the specimen is better than other treatments. The treatment method affects the resonance frequency value and the electromechanical coupling coefficient of the specimen. Water removal of specimens is not a suitable treatment method to increase the electromechanical coupling coefficient.

# 1. Introduction

The application of piezoelectric sensors, such as lead zirconate titanate (PZT) sensors, in structural health monitoring (SHM) for civil engineering has been implemented for many years [1-4]. When monitoring, PZT sensors face issues such as higher acoustic impedance and poor compatibility compared with concrete structures. To reduce the effect of compatibility between concrete and PZT, cement-based piezoelectric composites (also known as piezoelectric cement) have been developed as a new type of sensor for monitoring concrete structures [5-8]. The microstructure of cement-based piezoelectric composites and their piezoelectric performance have garnered significant attention. Cement-based piezoelectric composites are two-phase composites, with PZT inclusions distributed within the cement matrix. These polarized composites can be piezoelectric sensors for SHM in concrete structures [9-11]. However, compared to PZT, the performance of cement-based piezoelectric composites still needs to improve, enhancing their piezoelectric properties a significant focus of research.

Many studies have comprehensively analyzed the admixtures [12-14], manufacturing process [5,15,16], microstructure [17-19], and performance [7] of cement-based piezoelectric composites. Pan et al. [20] pointed out that specimens subjected to heat treatment at 150°C for 40 min before polarization exhibited lower dielectric losses, making them easier to polarize and producing excellent piezoelectric and dielectric properties. Without mineral admixtures, heat treatment alone increased the piezoelectric strain constant ( $d_{33}$ ) of cement-based piezoelectric composites with 50 vol.% PZT from 55 pC/N to 106 pC/N.

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Cement-based piezoelectric composites are generally prepared by mixing the raw materials (without adding water) and molding them under pressure. After pressure molding, the specimen should be cured through hydration to develop strength; otherwise, it will be damaged during grinding. During the water or steam curing process, part of the water inside the specimen hydrates with the cement to produce crystal water, and the other water exists in the form of free water. The free water within the specimen usually increases the difficulty of polarization. The heat treatment of cement-based piezoelectric composites significantly improves their piezoelectric properties [20]. It is speculated that heat treatment removes free water, thereby enhancing polarization efficiency and yielding better piezoelectric and dielectric performance. Therefore, this study explores other methods of removing free water from the specimens, like vacuum drying and ethanol dehydration, and discusses the effect of treatment methods on the piezoelectric and dielectric properties of cement-based piezoelectric composites to improve the manufacturing process of cement-based piezoelectric composites to improve the manufacturing process of cement-based piezoelectric composites to improve the manufacturing process of cement-based piezoelectric composites to improve the manufacturing process of cement-based piezoelectric composites to improve the manufacturing process of cement-based piezoelectric composites.

# 2. Experimental Overview

2.1 Materials. Fresh calcium aluminate cement (CAC) is the matrix for cement-based piezoelectric composites with a specific gravity and fineness of 2.98 and 4300–4700 cm<sup>2</sup>/g, respectively. Its chemical properties, analyzed according to ASTM C114, are shown in Table 1.

Chamical Composition	Contant (wit %)
Chemical Composition	Content (wt.%)
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> )	40.50
Calcium Oxide (CaO)	37.70
Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> )	13.93
Silicon Dioxide (SiO <sub>2</sub> )	4.40
Titanium Dioxide (TiO <sub>2</sub> )	1.59
Magnesium Oxide (MgO)	1.00
Diphosphorus Pentoxide (P <sub>2</sub> O <sub>5</sub> )	0.14
Potassium Oxide (K <sub>2</sub> O)	0.11
Sulfur Trioxide (SO <sub>3</sub> )	0.10
Sodium Oxide (Na <sub>2</sub> O)	0.07

 Table 1. Chemical composition of cement.

The original dimensions of sintered and non-polarized PZT were  $\varphi 12 \times 1.8$  mm in Figure. 1, and it was pulverized to particles with a size range of 75 – 150 µm shown in Figure. 2 to be used as the inclusions in cement-based piezoelectric composites. The PZT has the specific gravity  $\gamma = 7.6$ , piezoelectric voltage constant  $g_{33} = 23.9$  mV-m/N,  $d_{33} = 650$  pC/N and relative permittivity  $\varepsilon_r = 3400$ . The properties of PZT are shown in Table 2.



Fig. 1. PZT ceramics



Fig. 2. PZT particles

Parameter	Properties
Electromechanical coupling coefficient, $\kappa_t$	0.62
Piezoelectric voltage constant, $g_{33}$ (mV-m/N)	23.9
Piezoelectric strain constant, $d_{33}$ (pC/N)	650
Acoustic impedance $(10^6 \text{ kg/m}^2\text{-s})$	22.42
Mechanical quality factor, $Q_m$	70
Relative permittivity, $\varepsilon_r$	3400
Curie temperature (°C)	220
Velocity, V <sub>d</sub> (m/s)	2950
Specific gravity, γ	7.6

Table 2.	Properti	es of PZT	ceramic	material
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Ethanol is commonly used to stop the hydration reaction of cement in cement-based materials. This study employed it for the dehydration treatment of cement-based piezoelectric materials. The ethanol concentration is above 99.9%, with a specific gravity of 0.789, an autoignition temperature of 363°C, and a rate of evaporation of about 2.4.

#### 2.2 Methodological

2.2.1 Specimen Preparation. Piezoelectric cement (PEC) is a type of 0–3 cement-based piezoelectric composite composed of PZT powders with particle sizes ranging from 75 to 150  $\mu$ m, mixed with cement at a ratio of 50 vol.%. Additionally, 100% plain cement (PC) specimens are prepared for comparison. The mixed powder is compacted using a materials test system (MTS) under a stress of 80 MPa, forming a disk of 15 mm diameter in Figure. 3. The specimens in Figure. 4 are then cured in an environment at 90°C with 100% relative humidity for 12 hours to ensure sufficient strength for grinding. Once the curing is completed, the specimens are polished using a grinding machine, with the designed thickness set at 2 ± 0.05 mm, as shown in Figure. 5.



Fig. 3. Specimens



Fig. 4. Before grinding



Fig. 5. After grinding

2.2.2 Specimen Treatment Methods. The electrodes were fabricated by applying high-temperature conductive silver paste (SYP-4570) to both sides of the polished specimen, followed by curing at 150°C for 30 minutes to form the electrodes. The piezoelectric cement undergoes a two-step treatment process, one after grinding and another before polarization. The electrode fabrication in this study was performed after the first treatment; the properties discussed before polarization refer to the characteristics of the specimens "before polarization," meaning the properties after the second treatment.

There are four treatment methods for specimens: untreated, heat treatment, ethanol dehydration, and vacuum drying. The specimens are placed in a humidity control box  $(23 \pm 1^{\circ}C)$  with 50 % relative humidity for the untreated condition. The heat treatment condition involves heating at 140°C for 60 minutes. For vacuum drying, the specimens are placed in a vacuum environment at  $23 \pm 1^{\circ}C$  for 60 minutes. Ethanol dehydration involves immersing the specimens in 99.9% ethanol, with the first immersion lasting 1 hour, followed by storage in a humidity control box before polarization. The

second treatment involves immersing the specimens for 30 minutes and then placing them in an oven at 90°C for 30 minutes.

The weight of the composites was measured before and after the two treatment processes, as shown in Figure. 6, to calculate the water removal rate, as expressed in Eq. 1. where  $R_W$  is the water removal rate (%),  $W_b$  is the weight before treatment, and  $W_a$  is the weight after treatment.

$$R_W = \frac{W_b - W_a}{W_a} \times 100\% \tag{1}$$



Fig.6. The weight of the composites was measured before and after the two treatments

2.2.3 Specimen polarization. Polarization is the process of applying a high DC voltage to the specimen while maintaining a specific temperature and duration, allowing the dipoles of the specimen to align in the direction of the electric field to acquire piezoelectric properties. In this study, specimens can be polarized after being second-treated. The polarization setup involves immersing the specimen in a 150°C silicon heat transfer fluids bath for 10 minutes, followed by polarization with an electric field of 1.5 kV/mm for 40 minutes (poling time).

2.2.4 Properties measurement and calculation. After the second treatment of the specimen (before polarization), an impedance analyzer (Model 6520) is used to measure the capacitance (C), resistance (R), and dielectric loss (D) of the specimens. The measurement conditions are set to a voltage of 1 V and a frequency of 1 kHz. The same applies to the specimen after polarization, with the additional measurement of the piezoelectric strain constant ( $d_{33}$ ) using a  $d_{33}$  piezometer (Model P/N 90–2030) at a frequency of 110 Hz. The measurement environment is at room temperature of 23±1°C and a relative humidity of 50±2 %.

The capacitance is converted to the relative permittivity ( $\varepsilon_r$ ) using Eq. 2, where t is the thickness of the specimen, A is the area of the specimen, and  $\varepsilon_0$  is the permittivity of the vacuum environment (8.854×10<sup>-12</sup> F/m). The resistance is converted to resistivity ( $\rho$ ) using Eq. 3. The  $d_{33}$  is also converted to the piezoelectric voltage constant ( $g_{33}$ ) using Eq. 4.

$$\varepsilon_r = \frac{C \cdot t}{A \cdot \varepsilon_0} \tag{2}$$

$$\rho = \frac{R \cdot A}{t} \tag{3}$$

$$g_{33} = \frac{d_{33}}{\varepsilon_0 \cdot \varepsilon_r} \tag{4}$$

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The impedance analyzer is used to measure the impedance from 0.02 kHz to 2000 kHz with a voltage of 1 V (i.e., impedance spectrum), and the resonant frequency range of the specimen is identified. The electromechanical coupling factor ( $\kappa_t$ ) is calculated using Eq. 5,

$$\kappa_t = \sqrt{\frac{\pi}{2} \frac{f_m}{f_n} \tan(\frac{\pi}{2} \frac{f_n - f_m}{f_n})}$$
(5)

where  $f_m$  is the frequency with the minimum impedance value in the resonant frequency range, and  $f_n$  is the frequency with the maximum impedance value in the resonant frequency range.

## 3. Results and Discussion

3.1 Water removal rate. The water removal rate is shown in Figure. 7. All four treatment methods effectively removed water during the first treatment. As a result, even though the specimens were stored in a humidity-controlled box, they gradually transitioned from a surface-dry state to an air-dry (AD) state. Heat treatment demonstrated the highest water removal rate among the four treatment methods. Heat treatment removes free water from the specimen and eliminates crystalline water, resulting in a significantly higher water removal rate. In contrast, the water removal rate decreased significantly during the second heat treatment. This reduction is attributed to the effective elimination of free water during the first heat treatment, leaving less water during the second heat treatment. During the second treatment, the vacuum drying and untreated method exhibited a slight increase in weight, possibly due to the absorption of moisture from the surrounding air. This phenomenon resulted in a negative water removal rate, as shown in Figure. 7. These similar outcomes suggest that vacuum drying has minimal impact on the water removal rate of cementitious materials. Ethanol dehydration showed the lowest water removal rate during the first treatment. This is because ethanol replaces free water in the specimen as it is expelled, leading to a relatively minor weight loss. However, after the second treatment-where ethanol was volatilized at 90°C-this method effectively reduced the water content in the specimen.



Fig. 7. Water removal rate with different treatment methods

3.2 Relative permittivity ( $\varepsilon_r$ ) before polarization. Figure 8. shows the relative permittivity of PC (black bars) and PEC (mesh bars) before polarization. Heat treatment and ethanol dehydration are less effective in promoting relative permittivity for both PC and PEC. A decrease in relative permittivity can also serve as an indicator of the efficiency of free water removal. This is because, at 25°C, the relative permittivity of air is approximately 1, while that of water is about 78. Heat treatment

and ethanol dehydration have similar effects on the relative permittivity of PEC. Compared with untreated specimens, the relative permittivity decreased by 41–43% on average; in contrast, the impact of vacuum drying was less obvious, with an average decrease of 26%. It can be concluded that heat treatment and ethanol dehydration produced similar water removal results, while vacuum drying was slightly less effective. In addition, PEC's relative permittivity is higher than PC's, indicating that adding PZT effectively increases the relative permittivity of the cement (i.e., increases capacitance).



Fig. 8. Relative permittivity of PC and PEC before polarization

3.3 Resistivity ( $\rho$ ) before polarization. The resistivity of PC and PEC before polarization is shown in Figure. 9. The resistivity of PC under the four treatment methods was around 200 k $\Omega$ -m. After adding PZT, the resistivity of PEC dropped below 100 k $\Omega$ -m, indicating that adding PZT reduces the resistivity. Resistivity values less than 100 k $\Omega$ -m make PEC facilitate polarization [8]. In contrast, the cement (PC) cannot be polarized due to its high resistivity (approximately 200 k $\Omega$ -m). For different treatment methods, heat treatment and vacuum drying only reduced the resistivity of the specimen by about 3 % compared with the untreated method; the difference was insignificant. However, ethanol dehydration showed a more significant difference, with a 17 % increase in resistivity. This may require microstructural observation studies to further explain this characteristic.



Fig. 9. Resistivity of PC and PEC before polarization

3.4 Dielectric loss before polarization. Dielectric loss refers to the phenomenon where the specimen's charge carriers generate a conductive current during an electric field polarization, consuming part of the electrical energy and converting it into heat. This is closely related to the difficulty and efficiency of polarization. Specimens with lower dielectric losses facilitate polarization. The dielectric loss before polarization is shown in Figure. 10, showing that adding PZT significantly reduces the dielectric loss of PEC. For the treatment methods of PEC, the order of dielectric loss value is untreated > vacuum drying > ethanol dehydration > heat treatment. This shows that heat treatment of PEC is the most effective in reducing dielectric loss, achieving better polarization efficiency, and reducing polarization difficulty.



Fig. 10. The dielectric loss of PC and PEC before polarization

3.5 Relative permittivity ( $\varepsilon_r$ ) after polarization. The relationship between the relative permittivity of polarized PEC specimens and the age after polarization is shown in Figure. 11. The relative permittivity of PEC is related to the age after polarization and gradually tends to a plateau. Different from Figure. 7, the relative permittivity of the heat-treated PEC in Figure. 10 is much higher than that of other treatment methods. This may be related to PEC's lower resistivity and dielectric loss before polarization (as shown in Figures. 8-9), resulting in high polarization efficiency. Materials with better polarization efficiency tend to achieve higher relative permittivity.



Fig. 11. The relationship between the relative permittivity of PEC and the age after polarization

*3.6 Resistivity after polarization.* The relationship between the resistivity of polarized PEC and the age after polarization is shown in Figure. 12. The resistivity of PEC continues to increase with age. This may be due to the cement particles within the specimen changing microstructure through electric charges or continuing to hydrate, resulting in a denser structure. As a result, the resistivity of the specimen continues to increase. At the early age (before the 7th day), the effect of residual charge within the specimen is still not fully understood. On the 56<sup>th</sup> day, the resistivity of the untreated specimens is higher than that of the treated specimens. This could be because the treated specimens have less moisture, limiting the hydration that can occur, which in turn leads to lower resistivity. In comparison between Figure. 12 and Figure. 9, the resistivity of the specimens after polarization is lower than before polarization. This is because residual charges are left within the material after polarization, which results in lower resistivity in the polarized specimens.



Fig. 12. Relationship of resistivity and the age after polarization

3.7 Piezoelectric strain constant. The piezoelectric strain constant of polarized PEC related to the age after polarization is shown in Figure. 13. The piezoelectric strain constant increases with age. This can be attributed to the continuous changes in the microstructure such that the piezoelectric strain constantly increases over time. The piezoelectric strain constants of heat-treated and ethanol-dehydrated specimens are higher than those of untreated and vacuum-dried specimens. Lower resistivity and dielectric loss of materials before polarization, as indicated in Figure. 9 and Figure. 10, lead to facilitating polarization. The more uniformly the dipoles within the specimen are aligned, the higher the piezoelectric strain constant. Thus, heat treatment and ethanol dehydration enhance the piezoelectric properties by ensuring better dipole alignment, while untreated and vacuum-dried specimens, with less effective polarization, exhibit lower piezoelectric strain constants.



Fig. 13. Relationship of piezoelectric strain constant and the age after polarization

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3.8 Piezoelectric voltage constant. Figure 14. shows the relationship between the piezoelectric voltage constant and age after polarization. The piezoelectric voltage constant slightly decreases over time. From Figure. 11 and Figure. 13, the relative permittivity and the piezoelectric strain constant grow with age. The relative permittivity of heat-treated specimens is higher than that of ethanol-dehydrated specimens, as shown in Figure. 11. This leads to a lower piezoelectric voltage constant calculated from Eq. 4. for heat-treated specimens by comparing them with ethanol-dehydrated ones. Ethanol dehydration specimens have the optimum piezoelectric voltage constant, indicating that when fabricating cement-based piezoelectric composites, the specific treatment method should be chosen based on the particular needs.



Fig. 14. Relationship of piezoelectric voltage constant and the age after polarization

3.9 Impedance spectrum and electromechanical coupling coefficient. Figures 15–18 show the impedance spectrum of PEC for four treatment methods. Regardless of the treatment method, the impedance at the resonant frequency decreases with age, with no significant shift in peak frequency. Furthermore, the frequency of the resonance peak varies slightly depending on the treatment method. The frequency values of the resonance peaks were 132.6 kHz, 133.8 kHz, 135.1 kHz, and 142.6 kHz for untreated, vacuum drying, heat treatment, and ethanol dehydration specimens, respectively. By substituting the resonant frequencies,  $f_n$  and  $f_m$ , into Eq. 5, the electromechanical coupling coefficient for each treatment method of the specimen can be calculated, as shown in Figure 18. The electromechanical coupling coefficient  $\kappa_t$  remains constant with age, although the impedance at the resonant frequency decreases. The order of the electromechanical coupling coefficients from low to high for the four treatment methods is reversed as the sequence for the frequency values of the resonance peaks. In other words, the order of the peak frequencies is ethanol dehydration > heat treatment > vacuum drying > untreated.



Fig. 15. Impedance spectrum of untreated



Fig. 16. Impedance spectrum of vacuum drying



Fig. 17. Impedance spectrum of ethanol dehydration

130

1000

140

Fig. 18. Impedance spectrum of heat treatment



Fig. 19. Relationship of electromechanical coupling coefficient and the age after polarization

## 4. Conclusion

Four processing methods, heat treatment, vacuum drying, ethanol dehydration, and untreated, are applied to investigate the effect of free water on the electric and piezoelectric properties of cementbased piezoelectric composites. The conclusions drawn are as follows.

80

60

40

20

0

0

Impedance (k $\Omega$ )

1 Day

3 Days

7 Days 30

14 Days

20

28

27

26

25 ∟ 120

500

- 1. Before polarization, adding PZT to the cement can enhance the relative permittivity of cementbased composites and reduce resistivity and dielectric loss, which is beneficial for the polarization of the specimen.
- 2. The free water content in the specimen can be reduced through appropriate treatment methods. Ethanol dehydration and heat treatment are more effective for piezoelectric specimens.
- 3. Heat treatment and ethanol dehydration of the specimen can reduce dielectric loss and decrease relative permittivity before polarization.
- 4. Heat treatment has the optimum effect on the relative permittivity and piezoelectric strain constant after polarization. For the piezoelectric voltage constant, ethanol dehydration is better.
- 5. The treated method will affect the resonance frequency value of the impedance spectrum of the specimen, causing it to shift to the right in a sequence of untreated, vacuum drying, heat treatment, and ethanol dehydration.
- 6. Heat treatment and ethanol dehydration of the specimens are beneficial to piezoelectric properties but reduce the electromechanical coupling coefficient.

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