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

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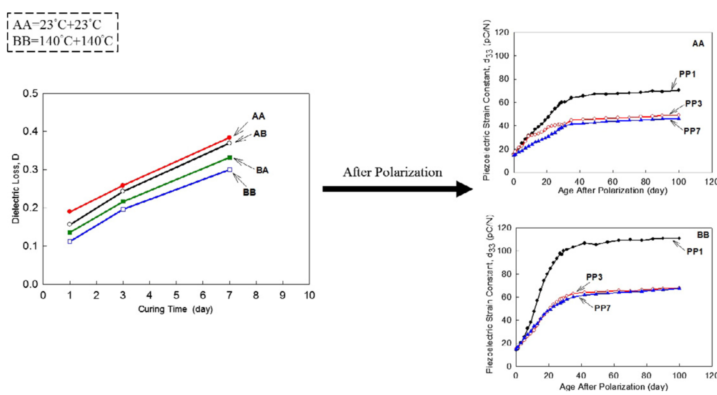
HIGHLIGHTS

- 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 ε_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_{33} = 110.9 pC/N has been fabricated as piezoelectric cement sensors potentially used in SHM.

GRAPHICAL ABSTRACT



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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 ε_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 ε_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 ε_r values, with d_{33} = 110.9 pC/N and ε_r = 756 at 100 days. However, it is necessary to enhance the K_t of piezoelectric cement at least 3 days' curing.

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 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 10×10^{-6} kg-m^{-2} s^{-1}, 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 ε_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 ε_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 ε_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 ε_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 cement-based 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^2/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 = 7.9×10^3 kg/m^3; d_{33} = 470 pC/N; ε_r = 2100; and piezoelectric voltage factor g_{33} = 24×10^{-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 ± 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.

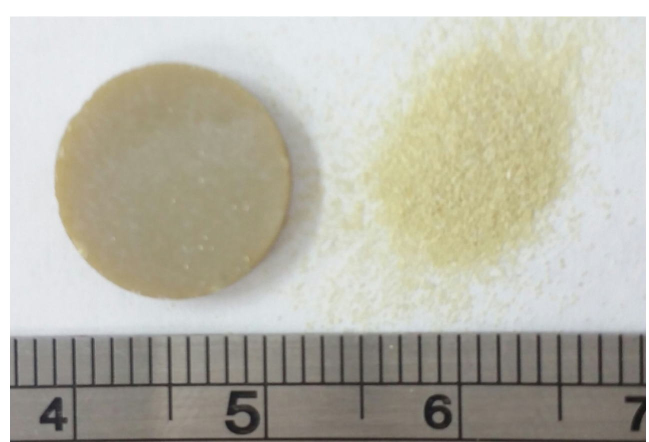

Fig. 1. The PZT ceramic (left) was granulated to 75–150 µm particles (right).

2.2. Temperature treatment

Two levels of temperature were defined as A = 23 °C and B = 140 °C. After the pores of specimens had been observed through OM, each specimen was heated either to Temperature A or B in an electric furnace for 40 min with heating rate of 10 °C per minute. This heating step was defined as the pretreatment of the specimens. The specimens were then cooled at ambient air temperature. Subsequently, both sides of the specimens were coated with SYP-4570 silver paint. The silver paint formed the electrodes, as shown in Fig. 2, and the specimens were then baked in an oven at 150 °C for 30 min to cure the silver and cause each electrode to adhere firmly to the specimen.

Afterward, the specimens were placed in ambient air temperature for 1 day. Specimens were heated again at Temperature A or B for 40 min. This heating step was defined as the posttreatment of the specimens. Therefore, each specimen of PP material was fabricated with some combination of three curing durations and four heating conditions; for example, PP7BA represents the PP material that was cured for 7 days, subjected to 140 °C pretreatment, and then 23 °C posttreatment. After temperature treatment, dielectric loss D and resistivity ρ of each specimen were measured at 23 °C by using an impedance phase analyzer (Model 6520A) at 1 kHz and 1 V prior to polarization.

2.3. Polarization and piezoelectric properties

For the polarization of the PP specimens, each specimen was placed in a 150 °C silicone oil bath and a 1.5 kV/mm poling field was applied for 40 min (poling time) to evoke the piezoelectric properties. In the context of the polarization, trigger time T is a poling duration counted from 0 to a specific poling voltage (here, that voltage is 1.5 kV/mm). Specimens requiring longer trigger times are difficult to polarize because the current breakdown usually occurs on the specimen, as shown in Fig. 3. Poling time is counted from the time when the applied voltage on the specimen reaches 1.5 kV/mm.

Starting 1 h after completion of the polarization process, the piezoelectric properties of the PP material were measured for 100 days. The piezoelectric strain factor d_{33} was directly measured using a d_{33} piezometer (Model P/N 90–2030) with a dynamic force frequency of 110 Hz. The relative dielectric constant ε_r was calculated from the thickness t and the electrode area A of the specimen, and from the permittivity of the free space constant ε_0 and the capacitance C measured at 1 kHz, as follows [30]:

$$\varepsilon_r = \frac{Ct}{A\varepsilon_0} \quad (1)$$

Fig. 2. The PZT/cement composite was coated with silver paint on the surface (left) to form the electrode and without silver paint coated (right).

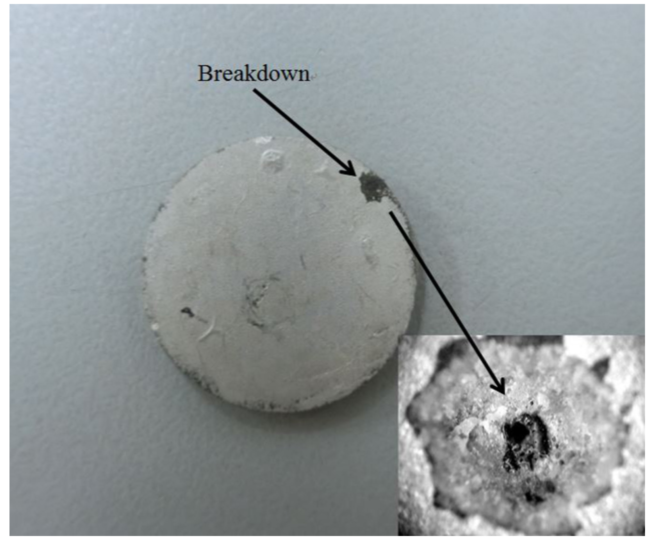

Fig. 3. The PP specimen had a current breakdown with a shock-burned hole.

Impedance–frequency spectra were acquired using an impedance phase analyzer. The thickness electromechanical coupling coefficient K_t was determined using the values of the resonance frequency at the minimum impedance f_m and that at the maximum impedance f_n from the impedance–frequency spectra as follows [32]:

$$K_t^2 = \frac{\pi}{2}\frac{f_m}{f_n}\tan\left(\frac{\pi}{2}\frac{f_n - f_m}{f_n}\right) \quad (2)$$

Each experimental value shown in the results is an average of six specimens measured at controlled conditions of 23 °C ± 1 °C and 50% ± 2% relative humidity and the d_{33} value was measured at nine positions on each specimen.

3. Results and discussion

3.1. Dielectric loss and resistivity before polarization

Specimens of the three PP materials (PP1, PP3 and PP7) were evaluated for porosity. After each specimen had reached its specific

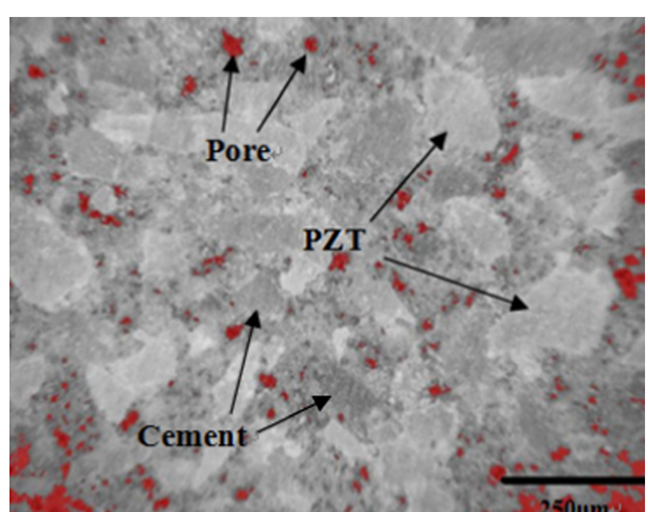

Fig. 4. The OM image of PP material displays the position and the area of pores, cement and PZT particles.

curing time, the pores of that specimen were monitored and located through OM; in Fig. 4, the pores are highlighted with crimson spots. Following the image analysis, a dividing value of 70 was used to distinguish black and white (threshold) in the OM images; the average porosity values for PP1, PP3 and PP7 were calculated respectively to be 3.39%, 3.25%, and 2.85% in these experiments. Porosity tended to decline with the curing time for PP materials; the hydration of ordinary cement continues to develop with the curing time.

To evaluate the feasibility of polarization for the PP materials, dielectric loss D and resistivity ρ were measured after the temperature treatments. Twelve groups of specimens were defined by combinations of three curing durations and four heating conditions. Each specimen had undergone one of these four heating conditions, involving a pretreatment and posttreatment at 23 °C or 140 °C. Fig. 5 shows that the D values increased with the curing time; for instance, for PP1, PP3, and PP7 at the same heating condition AA, the D values were 0.190, 0.259, and 0.384 respectively. Because the volume of cement solids increases with the curing time, longer curing durations tend to decrease the porosity, which affects the dielectric loss of the specimen [44].

As shown in Fig. 5, the PP specimens pretreated at the higher temperature (BA and BB) always showed lower dielectric losses than the specimens pretreated at the lower temperature did (AA and AB). This phenomenon was also observable for posttreatment; for instance, the pair of AA and AB showed lower D values under the AB heating condition than that under the AA heating condition; this trend was observed for the pair BA and BB. Regarding the 23 °C and 140 °C treatments, the PP materials treated at 140 °C before and after the electrodes were completed exhibited lower dielectric losses. Higher temperatures caused more free water to be expelled from these PP specimens, which may explain the lower dielectric losses. For the PP1 material, for instance, the heating conditions of AA, AB, BA, and BB yielded D values of 0.190, 0.157, 0.136, and 0.112 respectively, indicating that the BB condition had the lowest dielectric loss.

For the resistivity ρ of the PP materials measured at 1 V, as shown in Fig. 6, the specimens displayed higher resistivity with longer curing times because more cement solids developed. For instance, the resistivity values of PP1AA, PP3AA, and PP7AA materials were 56.4, 62.4, and 63.8 kΩ-m, respectively. Heating conditions for the same curing duration also showed that the specimens subjected to the higher temperature pretreatment exhibited lower resistivity, possibly because of microstructural changes in

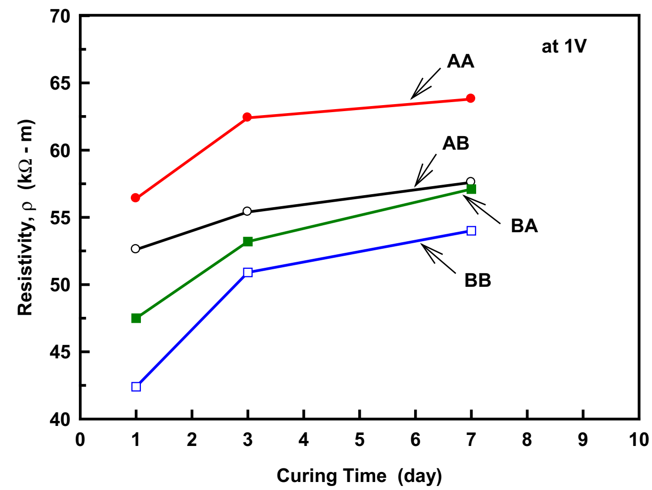

Fig. 6. The resistivity of the PP materials with respect to curing time and heating condition.

the specimens. The PP1BB material attained the lowest resistivity (42.4 kΩ-m).

3.2. Trigger time during polarization

Current breakdown occurring on the specimen during polarization represents polarization failure, and a trigger time of more than 3 h can be categorized as poling difficulty. Specimens can feasibly be poled if the aforementioned situations can be avoided. For PZT/cement composites with 50 vol% PZT, polarization of specimens cured for longer than 28 days has always been difficult [44]. In the present study, the trigger times for PP materials cured for periods ranging from 1 to 7 days were less than 60 min, as shown in Fig. 7, indicating that PP materials cured for longer times have longer trigger times and less porosity.

Apparently, the PP1 material has a short trigger time; these materials required less than 10 min to reach 1.5 kV/mm (poling field), which is shorter than the times for the PP3 and PP7 materials, as shown in Fig. 7. The trigger time for the PP1BB and PP1AA was approximately 3 min 8 s and 9 min 37 s, respectively. Regarding the effect of temperature treatment, trigger time was ranked in the order of BB < BA < AB < AA. This order is similar to the orderings of dielectric loss and resistivity shown in Fig. 5 and Fig. 6; therefore, higher temperatures shorten the trigger times during

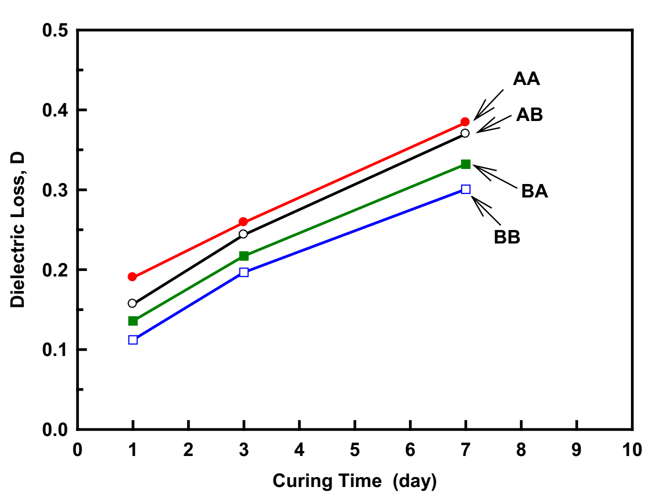

Fig. 5. The dielectric loss D of the PP materials with respect to curing time and heating condition.

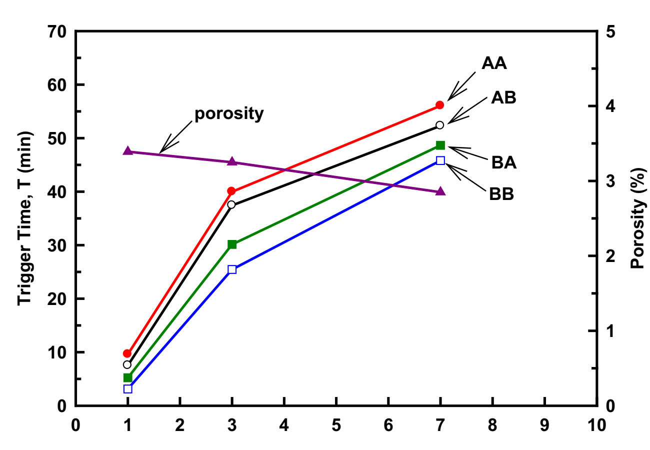

Fig. 7. The trigger time and the porosity of PP materials with respect to curing time and heating condition.

polarization. From Figs. 5–7, the dielectric loss D, resistivity, and trigger time of the PP materials can be generalized as depending strongly on the curing time and heating condition. PZT/cement composites with shorter curing times and higher temperature treatments showed lower dielectric losses and shorter trigger times and were easily poled during polarization.

3.3. Curing time effect on piezoelectric properties

After the specimens had been completely polarized, their piezoelectric properties were measured versus age for 100 days.

3.3.1. Piezoelectric strain factor

The piezoelectric strain factor d_{33} of the PP materials at the AA heating condition, as affected by curing time, is shown in Fig. 8. The d_{33} values for the PP1 material are higher than those for the PP3 and PP7 materials, and variation in the values stabilized after 50 days. It seems that longer curing times retarded the enhancement of the d_{33}, because the d_{33} values of the PP3 and PP7 materials both stabilized after 35 days, which was earlier than the stabilization of the PP1 material. The reason for the d_{33} value of the PP1 material being the highest after 100 days while the PP3 and PP7 materials had lower values is that the PP1 material has the lowest D value shown in Fig. 5, which probably enhanced the polarization efficiency. The effects of curing time on the d_{33} values of the PP materials subjected to AB, BA, and BB heating conditions are shown in Figs. 9–11, respectively. The d_{33} developments shown in these figures resemble that shown in Fig. 8, indicating that, to obtain a superior piezoelectric strain factor of piezoelectric cement, a specimen cured for 1 day is the optimal choice, regardless of the temperature treatment.

In addition, the trends of the d_{33} values for the PP3 and PP7 materials shown in Figs. 8–11 approach each other over time; after 40 days, the initial difference between the PP3 and PP7 materials diminished. Previous studies [39,43] have indicated that curing times of 7, 28, and 56 days for cement-based piezoelectric composites made no difference to the d_{33} values after 50 days of polarization. From Figs. 8–11 and previous reports [39,43], it seems that the curing time has only minor effects on d_{33} for cement-based piezoelectric composites cured for 3 days or longer. However, this finding does not hold for the specimen cured for 1 day because the lower D value of the PP1 material indicated more effective polarization. The d_{33} values for the PP1 materials are far higher than those of the other PP materials, particularly under the BB heating condition, as shown in Fig. 11.

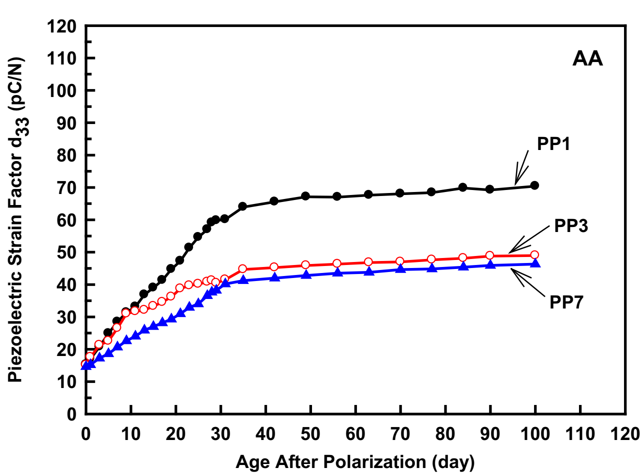

Fig. 8. The piezoelectric strain factor d_{33} of the PP materials affected by curing time at the AA heating condition.

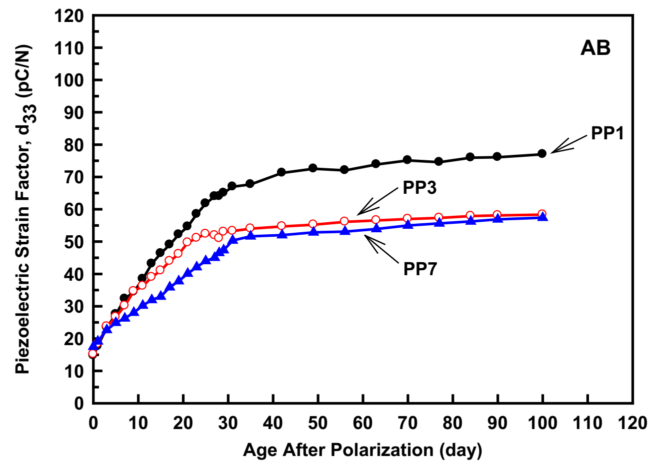

Fig. 9. The piezoelectric strain factor d_{33} of the PP materials affected by curing time at the AB heating condition.

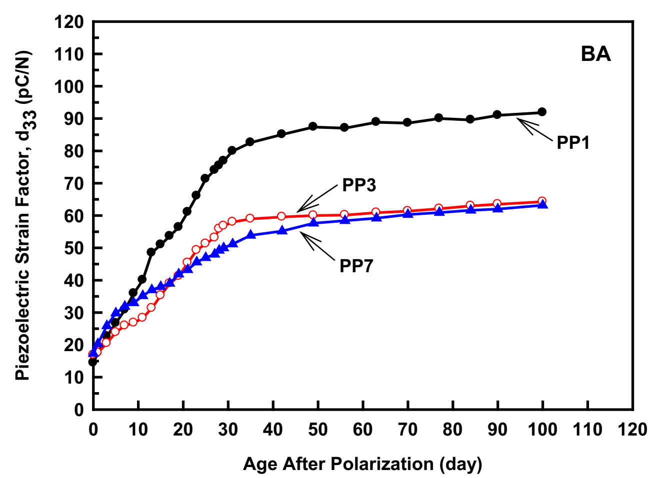

Fig. 10. The piezoelectric strain factor d_{33} of the PP materials affected by curing time at the BA heating condition.

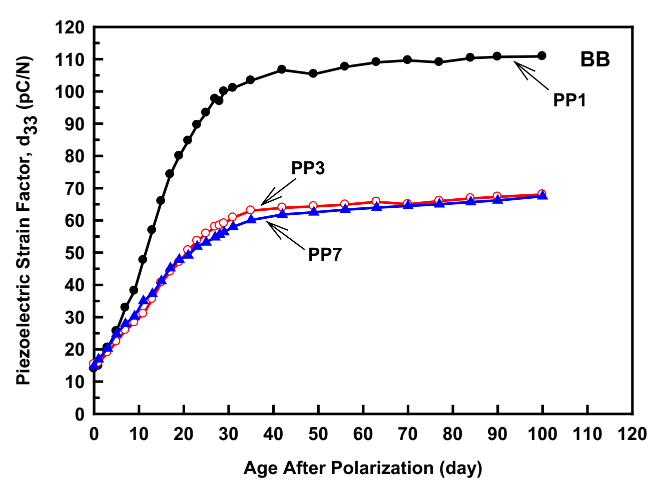

Fig. 11. The piezoelectric strain factor d_{33} of the PP materials affected by curing time at the BB heating condition.

3.3.2. Relative dielectric constant

The relationships between the relative dielectric constant ε_r and curing time for the PP materials subjected to four heating conditions are shown in Figs. 12–15, and the tendency of the ε_r curves, similar to the d_{33} curves shown in Figs. 8–11, is to continue to develop with age, gradually approaching a steady value after approximately 40 days. Under all heating conditions, the specimens cured for 1 day always exhibited higher ε_r values compared with the specimens cured for 3 or 7 days. For instance, under the BB heating condition, the proportion of the dielectric constant for PP1 relative to that for PP3 was 180% at 100 days, as shown in Fig. 15. The ε_r of a specimen cured for 1 day was superior to the corresponding values for the specimens cured for longer.

For the relative proportions of the dielectric constants for the PP3 and PP7 materials measured late in the experiments, in a manner similar to that of the piezoelectric strain factor, there were only a few differences between them, and previous studies have also found this phenomenon for ε_r in PZT/cement composites cured for 28 and 56 days [39,43].

3.3.3. Electromechanical coupling coefficient

Numerous factors can enhance the electromechanical coupling coefficient K_t, such as high PZT content [22,32], low forming pressure of specimens [25], high poling voltage, temperature, and time [29,33], and additional admixtures [39,42,43]. Figs. 16–19 show the effects of curing time on the electromechanical coupling coefficients of PP materials prepared under various heating conditions. The K_t values of the PP3 material increased by 14%–16% compared with the PP1 material. However, the increase in K_t becomes gradually less apparent from 3 to 7 curing days, with only a 2% increase. The K_t values of the PP materials exhibited minor effects from the curing time after 3 days. In addition, the results for PZT/cement composites cured for 7, 28, and 56 days with no temperature treatment showed no significant influence from the curing time [23,39,43]. Increasing the curing time can slightly increase the K_t value, but the effect becomes negligible for PZT/cement composites cured for longer than 3 days.

3.4. Heating condition on piezoelectric properties

When PZT ceramics were subjected to the same poling conditions as the PP materials were, temperature treatment was ineffective in enhancing the d_{33} values of the PZT ceramics [44]. However,

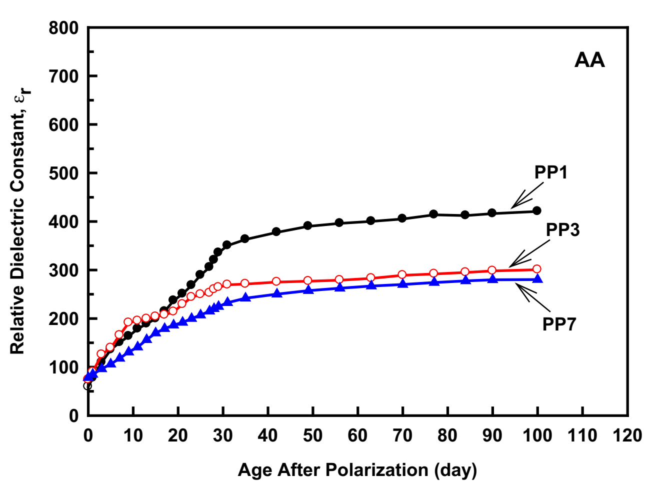

Fig. 12. The relative dielectric constant ε_r of the PP materials affected by curing time at the AA heating condition.

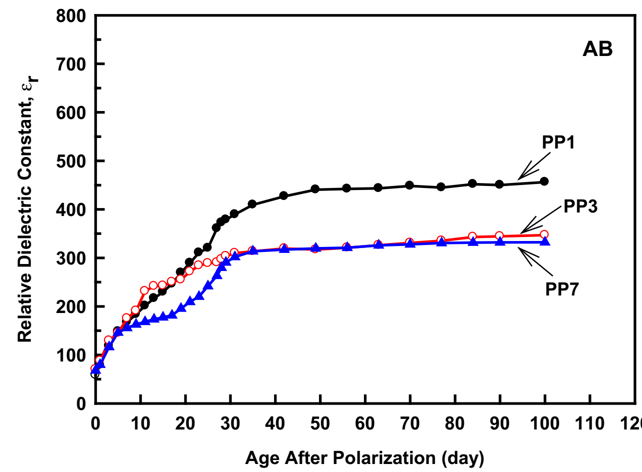

Fig. 13. The relative dielectric constant ε_r of the PP materials affected by curing time at the AB heating condition.

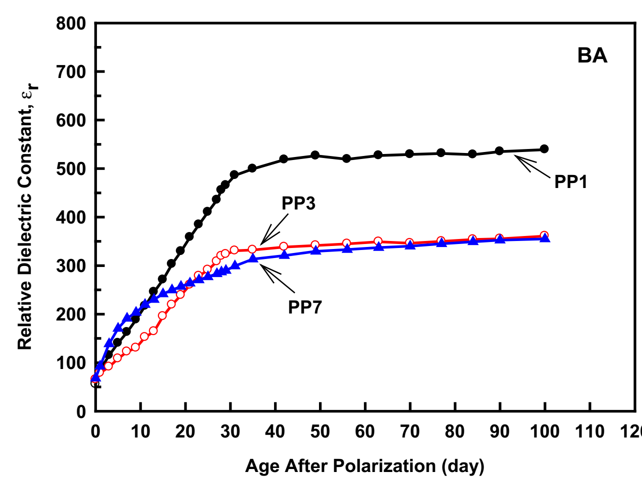

Fig. 14. The relative dielectric constant ε_r of the PP materials affected by curing time at the BA heating condition.

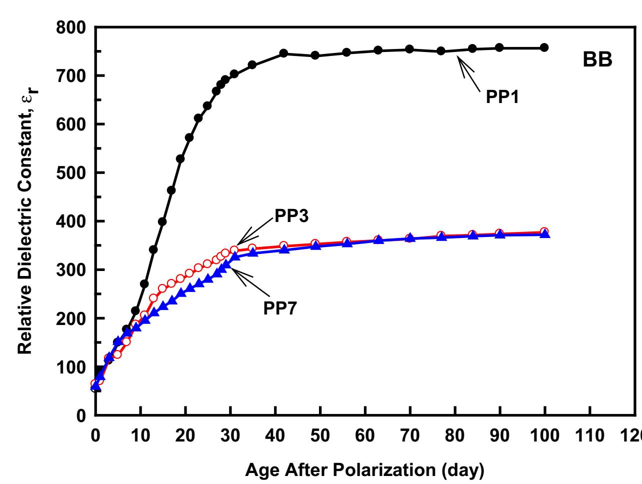

Fig. 15. The relative dielectric constant ε_r of the PP materials affected by curing time at the BB heating condition.

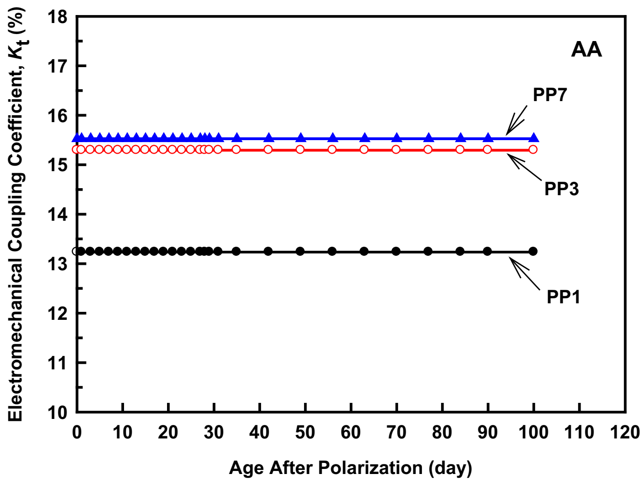

Fig. 16. The effects of curing time on the electromechanical coupling coefficient K_t of PP materials at AA heating condition.

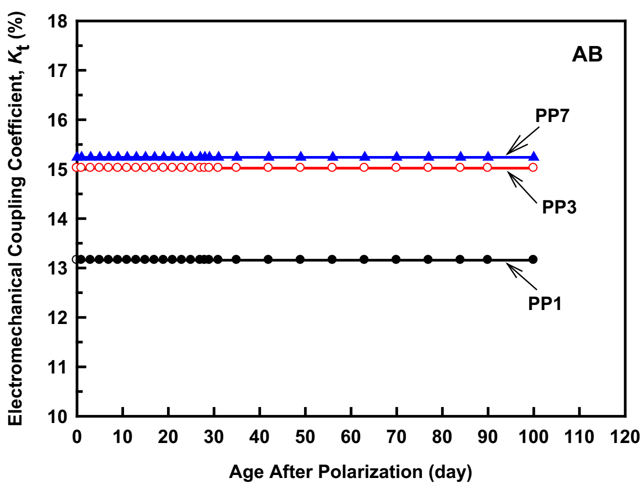

Fig. 17. The effects of curing time on the electromechanical coupling coefficient K_t of PP materials at AB heating condition.

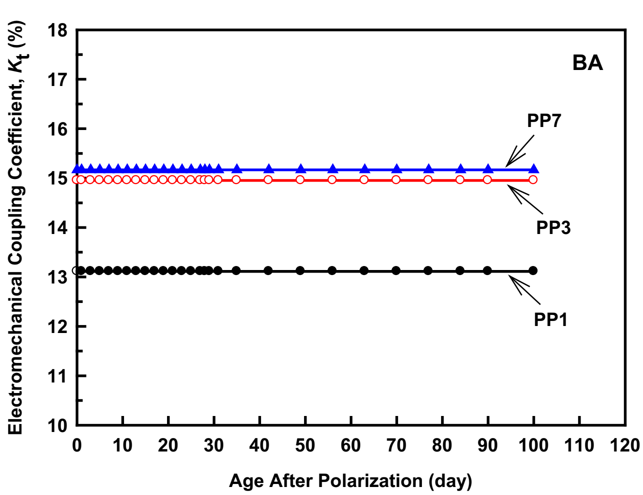

Fig. 18. The effects of curing time on the electromechanical coupling coefficient K_t of PP materials at BA heating condition.

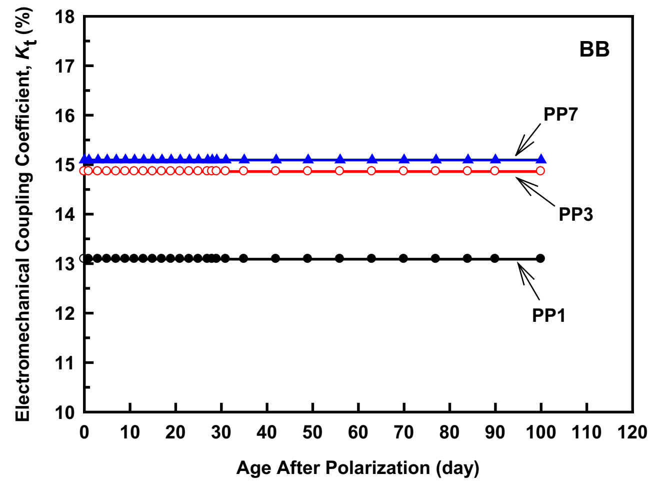

Fig. 19. The effects of curing time on the electromechanical coupling coefficient K_t of PP materials at BB heating condition.

Table 1
Piezoelectric properties of piezoelectric cement with 50% PZT at 100 days.

Heating condition	d_{33} (pC/N)			ε_r			K_t (%)		
	PP1	PP3	PP7	PP1	PP3	PP7	PP1	PP3	PP7
AA	70.4	49.0	46.3	421	301	280	13.23	15.29	15.53
AB	77.0	58.4	57.4	456	347	332	13.15	15.02	15.24
BA	91.8	64.2	63.2	539	361	355	13.11	14.95	15.17
BB	110.9	68.0	67.5	756	377	372	13.09	14.86	15.09

experimental results for the PP specimens heated under the AA, AB, BA, and BB conditions, shown in Figs. 8–19, showed that the piezoelectric properties benefitted from these heating processes. Properties approaching stable values were considered for an analysis of the piezoelectric factors of the PP materials relative to the curing time and heating conditions, as shown in Table 1 (PP materials at 100 days). Regarding the piezoelectric strain factor, the d_{33} values of the PP1 material under the AA, AB, BA, and BB conditions were 70.4, 77.0, 91.8, and 110.9 pC/N, respectively. The 140 °C posttreatment (AB) produced only a 10% increase in d_{33}; compared with the PP1 material without heat treatment (AA condition), 31% and 58% increases were noted corresponding to the PP1 material pretreated at 23 °C and 140 °C. PP materials subjected to pretreatment showed superior d_{33} values. Notably, at longer curing times, the increase in d_{33} from the 140 °C pretreatment will decrease slightly from 58% to 39% for PP1BB and PP3BB because the trigger time increases from 3 min 8 s to 25 min 27 s.

The relative dielectric constant ε_r depends on the temperature treatment, similar to d_{33}; the specimen cured for 1 day was more sensitive to heating conditions and had the highest ε_r values (756 for PP1, 377 for PP3, and 372 for PP7) under the BB condition. PP materials subjected to BB treatment invariably exhibited higher relative dielectric constants. However, temperature treatment slightly decreased the electromechanical coupling coefficient K_t, as shown in Table 1; all such treatments caused only a 1%–3% reduction; this is smaller than the effect of the curing time, which caused a 16%–18% increase.

4. Conclusions

Piezoelectric cement with 50 vol.% PZT subjected to 1.5 kV/mm was tested to examine the effects of curing time and heating conditions on piezoelectric properties. Specimens treated at higher temperatures exhibited lower dielectric losses because they contained less free water. The higher resistivity in the PP materials that had been cured for longer periods was due to the development of cement solids, leading to higher dielectric losses. PZT/cement composites that exhibited lower dielectric losses prior to polarization facilitated poling of the specimens and shortened the trigger times, which led to more effective polarization. PZT/cement composites cured for 1 day always displayed higher d_{33} and ε_r values than did the samples cured for longer, but the K_t trend was opposite. Specimens cured for more than 3 days showed less effect on the d_{33}, ε_r and K_t values at a late age. To enhance the d_{33} and ε_r values, using a higher temperature to pretreat specimens is a more favorable choice than posttreatment. PP materials cured for 1 day and heated at 140 °C before and after the electrodes were made (BB condition) proved ideal for obtaining superior d_{33} and ε_r. At 100 days after polarization, the piezoelectric cement with d_{33} = 110.9 pC/N and ε_r = 756 was measured.

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