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Controlling and increasing the inherent voltage in cement paste

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An inherent voltage occurs in cement paste, making the material an electret. Both poling (up to 225 V/m DC, causing long-range ion movement) during the 24 h setting and the use of sodium silicate liquid (which provides Na^+ ions) as an admixture were found to increase the inherent voltage, in addition to making the voltage more stable and better controlled. Without poling and sodium silicate, the inherent voltage after curing varied in sign and magnitude in an uncontrolled fashion among specimens poured from the same mix; for the same specimen, it varied significantly with the curing time and asystematically during the first 10 days of curing and stabilised thereafter. With both sodium silicate and poling, the voltage was positive (same polarity as the poling voltage), decreased with time throughout curing, and levelled off at a voltage that increased with increasing sodium silicate content, with the highest value attained in the present study being 0·35 V. The time constant for depoling during curing ranged from 2 to 7 days in the initial depoling period (up to 11 days), and ranged from 74 to 150 days in the subsequent period.

Introduction

The inherent voltage in a material refers to the stable voltage that is present in the material in the absence of an applied electric field. A material that exhibits an inherent voltage is an electret.

The electret behaviour originates from a built-in voltage, which is stable and conventionally exists due to some form of prior poling or excitation. The poling involves the application of a voltage, which causes electric polarisation; that is, the separation of the positive and negative charge centres. This separation is due to the difference in the spatial distribution of the positive and negative charges in the material. As the negative ions move toward the positive end of the applied electric field and the positive ions move toward the negative end of the applied electric field, the electric field resulting from the polarisation opposes the applied electric field. The special aspect of the electret effect is that the charges associated with the polarisation relax and form a core, which then induces surface charges of opposite sign.[1] It is the surface charges that are responsible for the electret effect. As the surface charge is opposite in sign from that associated with the polarisation and the electret voltage is measured at the surface, the electret voltage polarity is opposite to the polarisation voltage polarity and is the same as the poling voltage polarity (Fig. 1).

Electrets employed in desalination and air filters use their permanent charge polarisation to attract and trap the charged particles.[2] The ones used in γ-radiation dosimeters measure the reduction in the charge of an electret upon the neutralisation of surface charges due to the air ionised by the γ-radiation. The amount of loss in charge is directly proportional to the radiation present.[3] In addition, electrets are used in microphones, which make use of the effect of mechanical strain on the inherent voltage. Other applications of electrets include dust anchoring,[3] blood platelet adhesion[4] and memories.[5]

Electrets are in the form of dielectric materials, namely polymers and ceramics.[6–17] Examples are silicon dioxide (SiO_2), $Pb(Ti,Zr)O_3$, $CaTiO_3$, MgO-CaO-SiO_2-Al_2O_3 and hydroxyapatite, polyvinylydene fluoride (PVDF), polymethyl methacrylate (PMMA), polyethylene terephthalate (PET), polyethylene (PE) and polypropylene (PP). Electrets are commonly made by excitation, such as corona charging[18] and electron beam bombardment.[18,19]

Cement-based materials such as concrete are important construction materials. Cement-based electrets were first reported by Panchapakesan,[20] who observed an inherent voltage in cement paste prepared conventionally by mixing cement and water and subsequent

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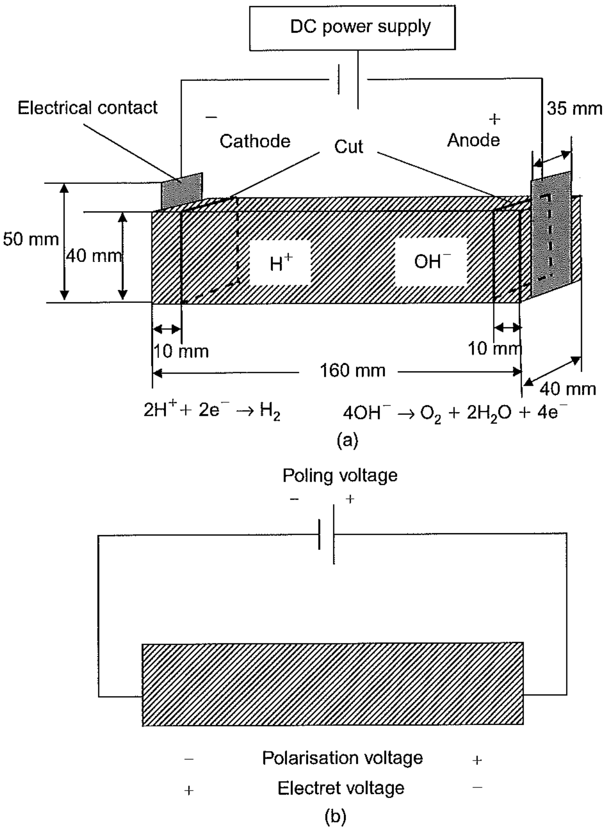

Fig. 1. (a) Specimen configuration for poling; (b) illustration of the polarisation and electret voltage polarity

curing (without electrical excitation). The inherent voltage can be positive or negative (with the linear voltage–current characteristic intersecting the voltage axis at a positive or negative value), with uncontrolled variability in both sign and magnitude among specimens poured from the same mix or from different mixes,[20] due to the origin of the inherent voltage being related to the slightly inhomogeneous distribution of ions. That the cement paste is an electret has been confirmed by the observed classical effect of a current pulse on the voltage and the associated skin-core electret formation.[20] Cement-based electrets are potentially attractive for attaining multifunctionality in structures, such as an environmentally friendly concrete chimneys that can suck particles from the effluent and a concrete wall that can detect high energy electromagnetic radiation.

Panchapakesan[20] observed the inherent voltage in cement paste after curing and did not monitor the voltage during curing. Study of the voltage during curing is expected to shed light on the process of electret formation. Therefore, the present study addressed for the first time the change of the inherent voltage during curing. In addition, it investigated the use of electrical excitation (namely, electric poling) during the setting of cement paste and the use of an ionic admixture to control and increase the inherent voltage.

The objectives of this study were: (*a*) to investigate the process of electret formation in cement; (*b*) to better control the inherent voltage in cement; and (*c*) to increase the inherent voltage in cement.

Research approach

Electric poling by using a DC electric field is well known in the case of ferroelectric materials, in which ferroelectric domains are aligned. In the present study, poling was optionally conducted on cement-based materials during setting for the purpose of enhancing the inherent voltage. After the setting and during the subsequent curing, which was in the absence of an applied electric field, the voltage across the specimen was monitored as a function of time. This allowed the process of natural depoling during curing to be investigated.

Due to the electrochemical reactions that accompany the poling process and the consequent generation of gases at the electrodes, the end regions of each specimen in the vicinity of the electrodes were porous and were removed by cutting prior to testing. This porosity was undesirable for practical applications and furthermore, poling during setting was inconvenient in the field. Despite the fact that poling was associated with serious disadvantages its effect was addressed in the present study.

Another method used in this study to enhance the inherent voltage involved increasing the ion content. For this purpose, an ionic compound is dissolved in the water that is used in a cement mix. Ionic compounds such as chlorides and sulfates are undesirable, however, due to their negative effects on the durability of cement-based materials, particularly those that contain steel reinforcing bars. Therefore, in the present study sodium silicate (also known as water glass) was chosen as a liquid admixture to enhance the ion content. Sodium silicate has long been used as an admixture in cement for underground construction,[21] grout,[22–24] waste solidification,[25] acid-resistant cement,[26] crack diminution admixture[27] and cement sealer;[28] it has not, however, been previously explored for use in cement for enhancing the dielectric behaviour.

Experimental methods

All of the cement-based materials studied were cement pastes. In other words, no aggregate (fine or coarse) was used. The cement used was Portland cement (Type I; Lafarge, Southfield, MI, USA). Sodium silicate (Na_2SiO_3, liquid, Grade 50; Occidental Chemical Corporation, Dallas, TX, USA) was optionally used as an admixture for the provision of Na^+ ions. The water–cement ratio was 0·35. In the mixtures in which sodium silicate was used, a high-range water-reducing agent (Glenium 3000 NS; Degussa Admixture, Cleveland, OH, USA) was used, the amount being 1·0% by mass of cement. A rotary mixer with a flat beater was used for mixing. In the absence of sodium silicate, no water-reducing agent was used.

Silica fume exists in the form of fine particles of size around 0·1 μm. Its presence as an admixture in cement is known to decrease the liquid permeability.[29] The liquid permeability relates to ion permeability. In order to investigate the effect of liquid or ion permeability on the poling behaviour, the present study included a limited investigation of the effect of silica fume. In the absence of sodium silicate, silica fume (microsilica, EMS 965; Elkem Materials Inc., Pittsburgh, PA, USA) was optionally used, the amount being 15% by mass of cement. The water–cement ratio was 0·35. A high-range water-reducing agent (Glenium 3000 NS) was used, the amount being 1·0% by mass of cement.

Cement paste specimens with different proportions of sodium silicate were prepared by mixing and subsequent setting (24 h) and then curing (27 days), with the setting step performed optionally in the presence of an applied electric field of either 31 or 225 V/m for the purpose of poling. Demoulding occurred immediately after the 24 h setting. After pouring into moulds, an external vibrator was used to facilitate compaction and decrease the number of air bubbles. Immediately after the pouring of the cement mix into the mould, a constant voltage (36 V, unless stated otherwise, corresponding to an electric field of 225 V/m) was optionally applied across the 160 mm length of the specimen during the 24 h setting for the purpose of poling (Fig. 1). The DC power supply used was a TO36-10M Regatran Semiconductor Power Supply (The Electronic Measurements Co., Eatontown, NJ, USA). After the 24 h period of setting and optional simultaneous poling, the specimen was demoulded and placed in a moisture chamber for 27 days of curing, while the voltage between the two electrical contacts was measured intermittently using a multimeter (30XR; Meterman Test Tools, Everett, WA, USA). Both setting and curing were performed in a moisture chamber at a relative humidity of nearly 100%.

The poling electric field was applied through electrical contacts that were located at the end surfaces of the specimen; namely between a specimen end and the proximate polyethylene mould surface. Polyethylene was chosen due to its electrical insulation ability. The mould cavity was 160 mm × 40 mm × 40 mm. The end surfaces were perpendicular to the direction of the electric field, which was along the length of the specimen. Each electrical contact covered most of the area of an end surface, so the electric field was essentially uniform throughout the cross-section of the specimen.

The electrical contacts were in the form of carbon fibre mat that had been coated with silver paint, which was fabricated using the following procedure. Carbon was chosen due to its electrical conductivity and chemical inertness. (The use of copper or steel in place of carbon led to undesirable reactions that resembled corrosion at the copper or steel.) A piece of carbon fibre mat (one grammage; 34 g/m^2) was sandwiched between two pieces of carbon fibre mat (two grammage; 8 g/m^2). These mats were wet-laid, non-woven carbon veil, with fibres of length 6 and 12 mm and diameter 7 μm, and binder in the form of polyvinyl alcohol (Grade 20301A, Optimat Carbon Fiber Veil; Technical Fiber Products Inc, Newburgh, NY, USA). Silver paint was manually applied between the mats to form the sandwich. In addition, silver paint was applied on the opposite surfaces of the sandwich. Each electrical contact was of size 50 mm × 35 mm and thickness 0·328 mm before application of silver paint at the outer surfaces and 0·38 ± 0·02 mm after application of silver paint at the outer surfaces. The 50 mm length of each electrical contact was along the direction perpendicular to the base of the mould cavity.

The electrochemical reactions that occurred while the cement was setting generated gases (probably O_2 at the anode and H_2 at the cathode), thus causing the formation of large voids around the electrical contacts. The fact that gas evolution occurred at the electrodes during poling suggests that the drift of ions in response to the applied electric field was long range rather than short range.

A possible reaction at the anode was the following, although it has not been ascertained:

$$4OH^- \rightarrow O_2 + 2H_2O + 4e^- \qquad (1)$$

The OH^- ions are inherently present in cement and this reaction generates oxygen gas. A possible reaction at the cathode was the following:

$$2H^+ + 2e^- \rightarrow H_2 \qquad (2)$$

The H^+ ions are present in cement. Reaction (2) generates hydrogen gas.

The gas generation reactions (1) and (2) above do not involve carbon, the electrical contact material. In other words, carbon, which merely serves as an electrical conductor, is not responsible for the gas generation.

The commercial sodium silicate product was 100% liquid. The solid content was 44·45 wt.%; that is, the proportion of the solid component after heating the product to 1000°F (540°C). This water amount was included in the total water amount used in the cement mix, so that the water–cement ratio was maintained at 0·35. The composition of each of three cement mixes used is shown in Table 1.

Results and discussion

The voltage was measured across the specimen by using the same electrical contacts as in poling. The poling was carried out only during setting (24 h) – not

Table 1. Ingredients in each of three cement mixes. In each case, the total water amount corresponds to a water–cement ratio of 0·35

Admixture	Water excluding that in the sodium silicate product: % by mass of cement	Sodium silicate product: % by mass of cement	Water-reducing agent: % by mass of cement
None	35	0	0
Na^+ ions* (0·5 mol/l†)	33	3·63	0
Na^+ ions* (1·0 mol/l†)	31	7·26	1·0

*From sodium silicate admixture.
†Concentration of Na^+ ions in the water used in the mix.

during the subsequent curing. After poling, the voltage was monitored in the absence of an electric field up to 27 days of curing for the purpose of investigating the stability of the electret formed by the poling.

Four specimens containing silica fume (without sodium silicate) were poled at 36 V (an electric field of 225 V/m) during the 24 h setting. Every specimen fractured during the setting (at setting times as low as 4 h), such that the crack occurred about 2 cm from the cathode. One specimen containing silica fume was poled at 24 V during the 24 h setting. It also fractured, with the crack occurring about 6 cm from the cathode. The fracture during poling of all specimens containing silica fume was attributed to the low permeability of the cement containing silica fume[29] and the consequent difficulty of drift of the ions in response to the applied electric field. The fact that the fracture occurred near one of the electrodes suggests that the drift of ions due to the electric field was long range rather than short range.

Immediately after removal of the poling voltage at the end of the 24 h setting period, the specimen was demoulded and measurement of the voltage across the specimen was immediately started. Table 2 shows the voltage immediately after setting and that after both setting and the subsequent 27 day curing. Each entry in Table 2 is for one specimen.

The measured voltage was positive for all compositions when poling was conducted, as shown in Table 2. This means that the measured voltage had the same polarity as the poling voltage, as expected for electret formation (Fig. 1(b)).

Without poling, the measured voltage was either positive or negative, although always small in magnitude (Table 2). This means that, without poling, the sign of the voltage could not be controlled, whenever the voltage was measured. In addition to the three specimens without poling listed in Table 2, numerous other specimens made from various batches (mixes) without poling were also studied. Among the specimens poured from the same mix, some had various positive values, whereas others had various negative values of the inherent voltage. This uncontrolled variability in both sign and magnitude among specimens supports

Table 2. Voltage across the specimen, measured during curing with the electric field off. The voltage is in the same polarity as the poling voltage

Na^+ concentration: mol/l	Poling electric field (V/m)	Measured voltage (V)		Voltage at 28 days divided by that at 1 day
		1 day*	28 days†	
0	0	0·007	0·003	0·429
0	0	−0·001	0·003	−3·22
0	0	−0·001	−0·004	3·82
0	31	0·90	0·04	0·044
0	31	0·89	0·02	0·022
0	225	1·39	−0·16	–
0	225	1·40	−0·40	–
0	225	1·20	−0·10	–
0·5	225	0·74	0·18	0·243
0·5	225	0·90	0·18	0·200
0·5	225	0·90	0·22	0·244
1·0	225	0·67	0·31	0·463
1·0	225	0·86	0·35	0·407
1·0	225	0·86	0·33	0·384

*Immediately after 1 day of setting.
†After 1 day of setting, followed by 27 days of curing.

the notion that the inherent voltage was due to inhomogeneous distribution of the ions. In spite of the conventional thorough mixing, slight inhomogeneity is possible. The slight inhomogeneity does not affect the mechanical properties, as shown by the absence of significant variation in the mechanical properties of various specimens made from the same cement mix in an earlier study by Chung[29] and many other researchers on materials that were similarly prepared. The electrical behaviour was more sensitive to slight inhomogeneity than the mechanical behaviour, since the inhomogeneity was in terms of electrical charges.

The variation of the inherent voltage during curing was also studied. The first three entries (without poling or sodium silicate) in Table 2 correspond to the three curves in Fig. 2 (labelled 1, 2 and 3 respectively). These figures show that the voltage varied significantly during the first 10 days (after demoulding), such that it decreased and increased (in a fashion that was not systematic in terms of the sign of the voltage) with time during curing for the same specimen and the highest voltage reaching values up to 0·015 V; after the first 10 days, the voltage became relatively stable. The large voltage variation during the first 10 days was attributed to: (*a*) the high fluidity of the mix and the consequent ease of ion movement, (*b*) the scatter of the ions as the ion movement occurred, and (*c*) the high rate of the hydration reaction, which involved ions. As curing occurred, the fluidity and the hydration reaction rate decreased, thus reducing the extent of ion movement and lessening the ion scattering. As a result, the voltage became more stable as curing took place.

In specimens for which poling had been conducted during setting, the voltage decreased with time during curing, such that the deviation from monotonic decreasing behaviour was quite small, as shown in Figs 3 and 4. The decrease of voltage with time was an indication of the gradual reduction of the electret effect after the removal of the applied electric field. However, this reduction slowed with increasing curing time, due to the stiffening of the material as curing took place. That the deviation from monotonic decrease was small in specimens for which poling had been conducted was due to the large values of the voltage, which caused the ion scattering to have relatively little effect.

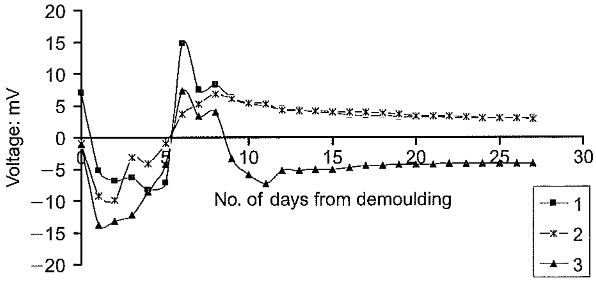

Fig. 2. Variation of the measured voltage with the curing age in days in the absence of sodium silicate and without poling for specimens 1, 2 and 3, which correspond, respectively, to the first three entries in Table 2

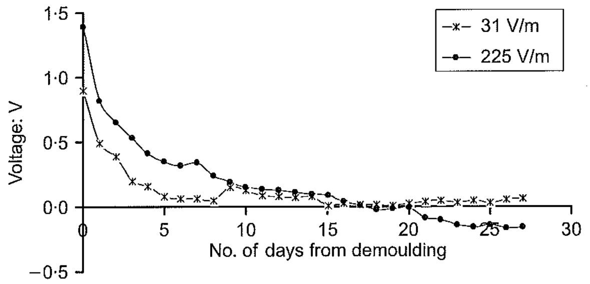

Fig. 3. Variation of the measured voltage with the curing age in days in the absence of sodium silicate after poling at 31 and 225 V/m.

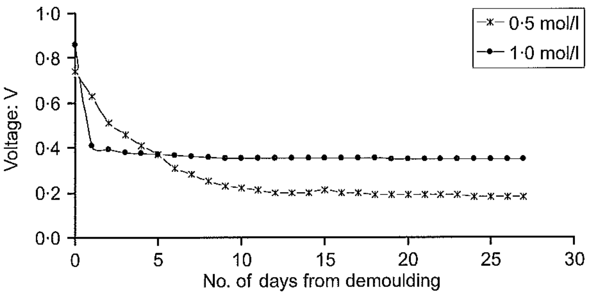

Fig. 4. Variation of the measured voltage with the curing age in days in cement with 0·5 and 1·0 mol/l sodium ions after poling at 225 V/m

The electret stability differed among the various compositions and the various poling conditions. In the absence of sodium silicate, poling with a voltage of 5·0 V (electric field of 31 V/m) led to an increased electret effect (Table 2 and Fig. 3). However, an increase in the poling voltage from 5 to 36 V (electric field increased from 31 to 225 V/m) caused the polarisation effect to overshadow the electret effect, so that the measured voltage was negative at 28 days of curing (Table 2 and Fig. 3). For a poling voltage of 36 V (electric field of 225 V/m), an increase of the sodium silicate concentration from 0 to 1·0 mol/l increased the electret stability, as shown by an increased value of the ratio of the voltage at 28 days to that at 1 day (Table 2 and Fig. 4). Thus, both poling and sodium silicate helped the formation of a stable electret.

The time constant β of the depoling process can be obtained by using the equation:

$$V = V_0 \exp(-t/\beta) \tag{3}$$

where V is the voltage at time t and V_0 is the voltage at $t = 0$. This equation means that log (V/V_0) is linearly related to t, with a negative slope that relates to the reciprocal of β. Such semi-logarithmic plots using the data of Fig. 4 are shown in Figs 5 and 6, respectively. The plots in Figs 5 and 6 are not linear, but each plot may be approximated as two linear segments of two different slopes, as shown by the straight lines in Figs 5 and 6. The β values obtained from these slopes are shown in Table 3. The segment at earlier times is referred to as period 1; that at later times is referred to as period 2. For each of the specimen types, the β value for period 1 is lower than that of the corresponding period 2. This difference between the two periods is due to the relatively low degree of cure in period 1 compared to period 2.

The time constant (β) values calculated from the slopes in Figs 5 and 6 are shown in Table 3. In period 1, a higher Na^+ concentration (1·0 mol/l) gives a lower value of β, probably due to a higher driving force for homogenisation for the higher Na^+ concentration. In period 2, the difference in β between the two Na^+ concentrations cannot be discerned, due to the data scatter.

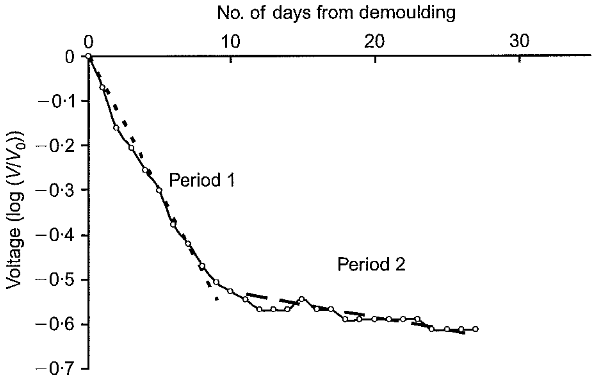

Fig. 5. Log(V/V$_0$) plotted against time corresponding to the data for 0·5 mol/l in Fig. 4. The slopes for two portions of each curve are shown and labelled 1 and 2

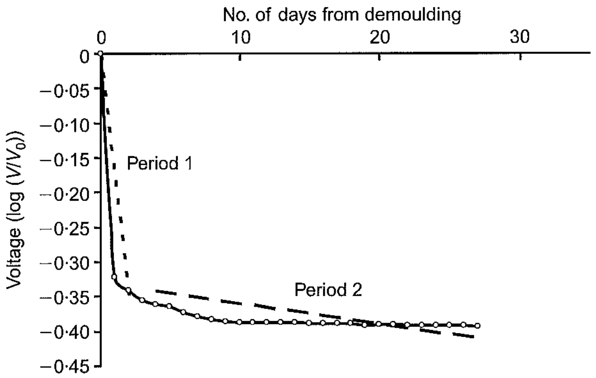

Fig. 6. Log(V/V$_0$) plotted against time corresponding to the data for 1 mol/l in Fig. 4. The slopes for two portions of each curve are shown and labelled 1 and 2.

Table 3. Time constant (days) for depoling. The poling immediately prior to the depoling was conducted at an electric field of 225 V/m

Na^+ concentration: mol/l	Poling electric field: V/m	Period 1	Period 2
0·5	225	7·1 ± 2·1	74 ± 54
1·0	225	2·5 ± 1·1	150 ± 80

Conclusion

Both poling (e.g., with a poling electric field of 225 V/m) during the 24 h setting of the cement paste and the use of sodium silicate liquid (e.g., 1·0 mol/l Na^+ ions in the water used in the cement mix) as an admixture were found to increase the inherent voltage, in addition to making the voltage more stable and more controlled. Without poling and sodium silicate, the inherent voltage after curing varied in an uncontrolled fashion among specimens poured from the same mix, taking positive and negative values (as first reported by Panchapakesan[20]); for the same specimens, it varied with the curing time significantly and asystematically during the first 10 days of curing and stabilised afterward, due to the association of the electret formation with ion movement, which diminished with curing. With both sodium silicate and poling, the voltage was positive (same polarity as the poling voltage), decreased with time throughout curing, and levelled off at a voltage that increased with increasing sodium silicate content, with the highest value attained in this work at 0·35 V.

Due to the long-range ion movement during poling in the setting period, the presence of silica fume as an admixture caused fracture in the specimen near the cathode during poling. No fracture occurred in the absence of silica fume.

The time constant for depoling during curing ranged from 2 to 7 days in the initial depoling period (up to 11 days), and ranged from 74 to 150 days in the subsequent period. The time constant for depoling was higher for 0·5 mol/l than 1·0 mol/lM Na^+ ions in the initial period.

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Discussion contributions on this paper should reach the editor by 31 July 2009