Self-sensing Of Portland Limestone Cement Pastes Without Functional Fillers

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Received: Dec. 09, 2025; Accepted: Feb. 09, 2026

Portland Limestone Cement (PLC) has been recently widely accepted in the construction industry as a replacement to the Ordinary Portland Cement (OPC). PLC was found to exhibit comparable mechanical strength to the OPC, and its production reduces the emissions of greenhouse gasses. However, the intrinsic self-sensing capability of PLC pastes without conductive fillers remains poorly understood. Self-sensing is the correlation between mechanical loading and changes in intrinsic electrical properties of the cement paste. This work investigates the piezopermittivity and piezoresistivity of the PLC paste and compares it with OPC paste. For this study, 3 specimens of PLC paste were prepared by mixing water and cement with a ratio of 0.35 and with dimensions of 60×60×10 mm. The specimens were tested under low-level cyclically-increasing compressive stress. Surface-mounted aluminum electrodes connected to an LCR meter were employed to monitor the change in the capacitance and resistivity of the specimens. The results reveal that PLC paste exhibit moderate yet unstable electrical sensitivity to the mechanical loading. It also shows irreversible electrical evolution under cyclic loading which suggests an accumulative microstructural changes contrary to its OPC counterparts. Under 2 kPa of compressive stress, the PLC specimens show an average fractional change of -0.053%, and 0.23% for capacitance and resistance, respectively. These findings suggest that while PLC demonstrates sensitivity to low-level mechanical loading in the elastic range. However, its long-term self-sensing stability may be limited due to the accumulation of the microstructural changes.

Keywords: Structural health monitoring; Damage detection; Capacitance-based sensing; Piezopermittivity.

http://dx.doi.org/10.6180/jase.202608_31.057

1. Introduction:

Reducing the emissions of greenhouse gases is the predominant motive for studies that aim at cleaner environment. Sustainable and low-emissions production processes are highly sought-after to control the climate change. Production of the Ordinary Portland Cement (OPC) consumes massive amount of energy and produces around 5% of the global CO_2 emissions [1, 2]. Thus, there are continuous efforts to reduce the energy consumption and CO_2 emissions of the Portland cement production industry. Reducing the clinker content of the OPC is an effective way to reduce the emissions of the greenhouse gases. This can be achieved by incorporating Supplementary Cementitious Materials (SCM) such as: limestone. OPC that contains more than 5% of limestone is referred to as Portland Limestone Cement (PLC). A reduction close to 10% of the total greenhouse (CO_2, N_2O, and CH_4) emissions can be achieved by incorporating 15% of limestone in the OPC[3, 4]. Several European countries have pioneered the use of PLC for decades, however, the countries of North America remained reluctant until 2004. The use of PLC was approved as part of the specifications AASHTO M240, and ASTM C595 [5, 6] in 2012, which permit a content of limestone in OPC up to 15%. The European standards allow a marginal ratio of up to 35% of limestone content [5, 7]. Since the adoption of the PLC in the construction industry in the countries of north America, most of the Portland cement manufacturer around the world started to produce and use it as well.

Generally, there are 2 ways to produce PLC, namely: interground and additive. Since the limestone is softer than the clinker, usually the interground PLC contains finer limestone particles [8]. The PLC produced using both ways has been investigated for their compressive strength, modulus of elasticity, flexural strength, shrinkage behavior, and other physical properties. The goal of these investigations was to compare the performance of the PLC with the OPC. In general, incorporating the limestone in OPC was found to induce minimal changes in the overall behavior of the OPC from the physical strength perspective. However, these changes might have noticeable influence on the PLC behavior from the self-sensing point of view. It was reported that PLC mixtures are slightly less dense from its OPC mixtures counterparts, which results in a decrease of its modulus of elasticity up to 7% [6]. In addition, increased fineness of the limestone content promotes early-age cracking and reduces the flexural strength of the mixture [6, 9]. The flexural strength experiences a decrease of up to 12% when interground PLC is used (i.e. fine limestone content) [6]. This decrease in the strength is attributed to the dilution effect, since the limestone is an inert filler and the water to cement ratio is subsequently increased [10, 11]. The fine interground limestone in PLC mixtures increases the autogenous shrinkage and the capillary stresses, which could promote cracking development [8].

Structural Health Monitoring (SHM) is of extreme importance for old and new structures to ensure safety of the users. Failing to track the condition of the materials and the structural integrity of infrastructures could result in catastrophic failures, financial losses, and fatalities [12]. Continuous SHM is usually performed by utilizing external sensors to measure the physical state of the structure, such as: strain gages, longitudinal variable transducers (LVDT), and others. However, installing these sensors poses many challenges including: cost, technical expertise, and location-specific sensing. Thus, self-sensing started to gain ground in the field of SHM. Selfsensing is defined as the ability of the material to sense its physical status without external sensing devices [13–15]. Self-sensing is mostly achieved by utilizing the electrical properties of the material. Any change in the electrical properties of the materials could indicate a change in its physical properties [16]. In conductive materials such as metals the resistance of the material is considered a good sensing indicator for self-sensing purposes. However, materials with poor electrical conductivity such as: cement pastes, and concrete might not show the same level of sensitivity in resistance to qualify it for sensing. Thus, conductive functional fillers are usually utilized to enhance the electrical properties. These functional fillers includes but not limited to: steel fibers, graphite, and carbon nanotubes [17, 18]. Enhancing the electrical properties of the cementitious materials not only contribute to self-sensing, but has applications in energy harvesting and regulation of pavement's temperature [18, 19].

The approach of adding conductive fillers to the concrete limits the application of the selfsensing techniques to newly constructed structures, and might raise its cost. Investigating the electrical-based self-sensing of cement materials without conductive filler has received little attention. Moreover, there are no studies that investigates the self-sensing properties of the PLC pastes to best knowledge of the author. A new self-sensing indicator was recently studied and showed superior performance when compared to the resistance, which is capacitance [15]. Since capacitance is basically a measure of an insulator to store the electrical charge, it is reasonable to use it with cementitious materials. However, the electrodes need special preparation where the material is sandwiched by two electrically conductive sheets. The preparation of the electrodes will be discussed further in the methods section. Capacitance is directly related to the material property of permittivity. The change in permittivity of the material due to mechanical excitation is referred to as piezopermittivity. Similarly, the change in resistance due to mechanical excitations is referred to as piezoresistivity. These variations in resistance and capacitance upon loading of the cement paste specimens serve as indicators of microstructural evolution, forming the basis of selfsensing behavior.

The study aims at investigating the intrinsic self-sensing behavior of PLC paste without conductive fillers by examining changes in electrical resistance and capacitance under cyclic compressive loading. Specifically, this work aims at (i) characterizing the piezoresistive and piezopermittive responses of PLC paste, (ii) assessing the reversibility and stability of these electrical signals under repeated loading, and (iii) comparing the observed behavior with previously published results for OPC paste tested under identical experimental conditions. Low stress level was selected to capture early microstructural changes under elastic strains rather than simulate service loads.

2. Methods

To test the self-sensing properties of cement pastes that contains higher limestone content, a PLC type I was used. 3

PLC cement paste specimens were prepared, the specimens are identical replicates prepared, cured, and tested under the same conditions to assess repeatability. The specimens in this work were identical replicates in geometry, curing conditions, electrode configuration, and testing parameters of the ones that were prepared by OPC and reported in the work of the author's team [15]. Thus, no variability between the specimens in both studies except for the cement type.

2.1. Materials

The mix was prepared by a flat-beater planetary mixer according to the ASTM C 305-06 standard [20]. Only cement and water were used for preparing the paste with a water/cement ratio of 0.35 . The cement powder is Portland cement Type 1L (the cement has a content of 13wt% interground limestone, ASTM C595) [21] provided by LaFarge Corp, Southfield, MI.

Three Special wooden molds were fabricated with dimensions of 60 mm $\times$ 60 mm $\times$ 10 mm to pour the cement paste in. The molds were lightly coated with a thin layer of commercial mineral oil to facilitate demolding. The oil was applied only to the mold surfaces and did not come into contact with the interior bulk of the cement paste. After pouring the mix in the oiled molds, it was thoroughly vibrated to take the shape of the molds, and remove any air. The specimens were covered to allow for self-curing at a controlled room temperature of 24°C and 65% humidity for 24 hours. After which the specimens were demolded and its surfaces were visually inspected and gently cleaned to remove any residual oil prior to curing.and For curing, the specimens were submerged in a water bath for 28 days at the same room conditions.

2.2. Testing methods

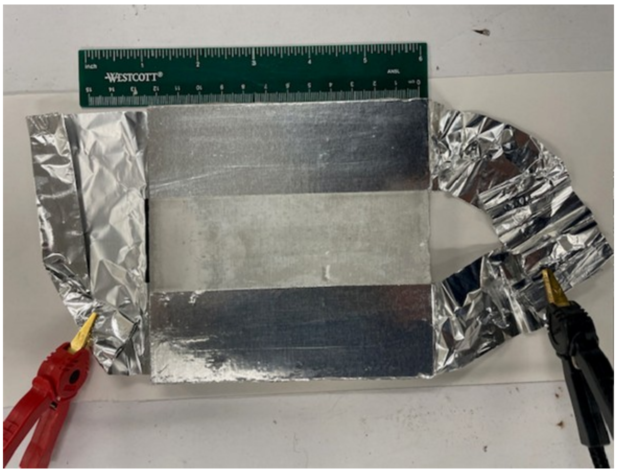
The specimens were air dried under 24°C room temperature for 3 days before the testing for its electrical properties. The electrodes were fabricated from aluminum foil (16μ m thick) for each specimen. Then the electrodes were attached to the specimen with one layer of double-sided adhesive tape (79μ m thick) with the same area as the electrodes. The electrodes were attached to both surfaces of the specimen on the opposite sides. A central area with dimensions of 20 mm$\times$ 60 mm on both faces of the specimen was not covered with the aluminum foil electrode as shown in Fig. 1 (a)(b).

Aluminum foil electrodes attached using double-sided adhesive tape were employed in this study to enable simultaneous resistance and capacitance measurements in a simple and costeffective manner. Compared to conductive coatings such as silver paint, aluminum foil electrodes offer a substantially lower material cost while allowing consistent electrode geometry across specimens [22, 23]. The efficacy of this electrode configuration has been demonstrated in previous studies, where stable and repeatable electrical measurements were obtained using similar setups [15]. The LCR meter used in this study is not designed to directly measure the capacitance of a bare conductor; therefore, the non-conductive adhesive layer was intentionally introduced to ensure a stable electrode-specimen interface during capacitance measurements. Despite the insulating nature of the adhesive tape, the measured electrical resistivity values were found to be close to previously reported direct-current (DC) resistivity values for comparable cementitious materials [15, 22]. This agreement indicates that the aluminum foil-adhesive electrode system provides adequate electrical contact quality for both resistance-based and capacitance-based measurements, and that any interfacial effects are not dominant in the present results. Nevertheless, the influence of electrode configuration remains an important consideration for future refinement of self-sensing measurement techniques.

The platforms shown in the same figure are made of nonconductive materials (i.e. wood). The lower platform was resting on the testing table, where the bottom of the specimen only touches it. Porcelain plates with known and equal weight were utilized to apply the force on the specimen through the upper platform. Each of the two platforms has width equal to that of the central area and covers the entire area. Each of the two electrodes sandwiching the specimen protrudes out of the specimen area to serve as an electrical lead for connection to the measurement meter by electrical clipping. The aluminum foil leads were folded several times to ensure firm clipping and electrical connection stability. The aluminum-foil electrodes were connected to an LCR meter (Hioki IM3536, Japan) to measure the resistance and capacitance of the specimen. The LCR meter was set at 5 V , and 100 kHz , these settings were selected since it provides high stability, magnifies the sensing capabilities, and microstructural damage detection [15]. As a reference, the resistance and capacitance of the specimen under no loads were also recorded. All the testing and electrical measurements were conducted in a temperature and humidity controlled room of 24°C and 65%, respectively.

2.3. Loading pattern

A cyclically-increasing loading pattern was applied on the specimen with each cycle starts from zero load level. The loading cycles were incrementally increased at each time of application. The stress of the specimen was calculated as



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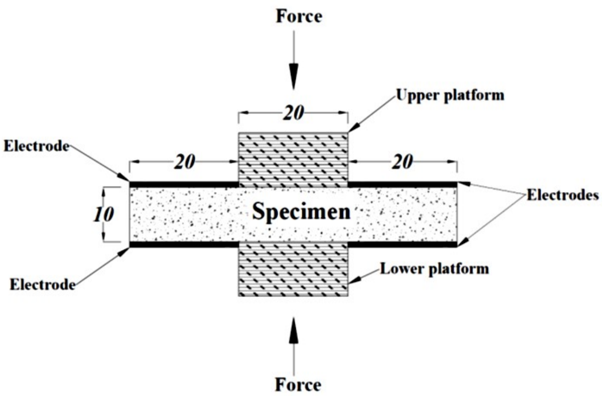


(b)

Fig. 1. (a) Photograph of the PLC paste specimen showing the aluminum foil electrodes connected to the LCR meter. (b) Schematic of specimen geometry and electrode configuration (all dimensions in mm). The central region has 2 opposite platforms to ensures uniform stress and electric field distribution and minimizes edge effects during measurements.

the load of each cycle divided by the area of the platform (Fig. 2). The maximum applied stress was 2.0 kPa , which is well below the compressive strength of the cement pastes. This low stress level is supposed to induce the elastic deformations only on the specimens. The loading pattern is designed to test the reversibility of the capacitance and resistance of the specimen. In total, 9 cycles of loading and unloading were applied on each specimen. The electrodes were attached to the LCR meter during these loading cycles to record the change in the resistance and capacitance of the specimen.

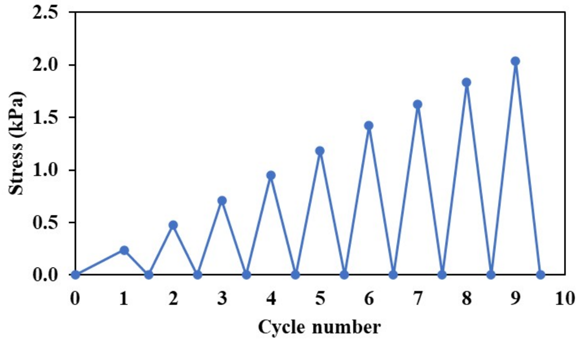


Fig. 2. Cyclic loading protocol applied to the specimens, showing the incrementally increasing compressive stress level with cycle number. Each cycle consists of loading and complete unloading to zero stress.

3. Results and discussions

The results of the PLC paste specimens are presented in the following sub-sections. In addition, a comparison between the behavior of the PLC paste and OPC paste specimens is performed.

3.1. Results of the PLC specimens

The initial capacitance, and resistance values were different for each PLC specimens as shown in Fig. 3 (a)(b), respectively. This behavior is expected since cement paste is nonhomogeneous. However, the discrepancy of the initial values of the PLC paste is more noticeable when compared to the OPC paste as will be further discussed in the next subsection. This indicates that limestone content promote irregularity in the microstructure probably due to the increase in the nucleation sites [9, 24]. The change in the capacitance and resistance with respect to the cycle number is not apparent due to the coarseness of the vertical scale. Thus, in order to visualize the variation in the results of the PLC specimens, the values were normalized with reference to the initial values (i.e. the current value - the initial value at zero stress level) as shown in Fig. 3 (c)(d). The error bars show an increasing trend with each cycle which indicates a divergence in both the capacitance and resistance values as the stress increases.

The fractional change in the capacitance and resistance corresponding to the cyclic loading pattern is shown in Fig. 4 (a)(b), respectively. The fractional change was calculated with respect to the initial value according to Eq. (1). Similar to the capacitance and resistance values, the fractional change in the capacitance and resistance is showing an increasing pattern when the stress is gradually increased. This behavior is not expected especially because the stress

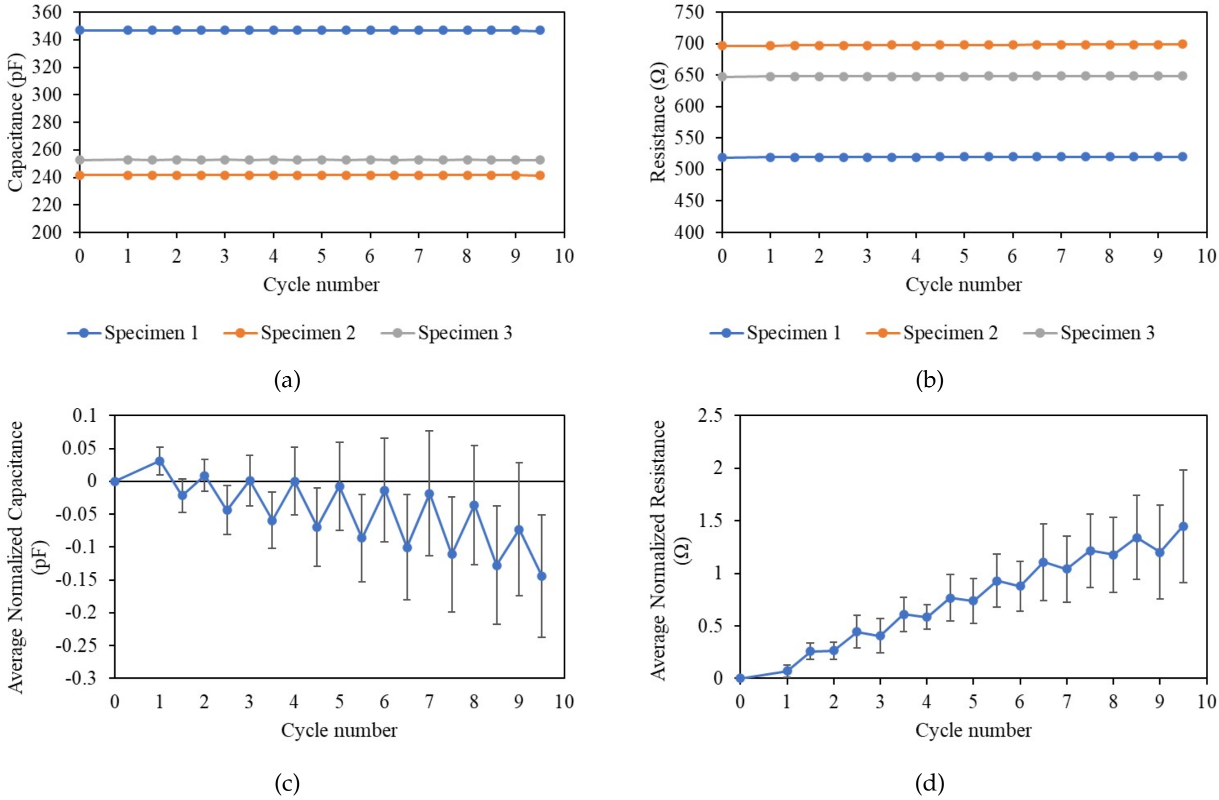


Fig. 3. Electrical response of PLC paste specimens under cyclic compressive loading: (a) measured capacitance and (b) measured resistance as a function of cycle number; (c) normalized capacitance ($C - C_0$) and (d) normalized resistance ($R - R_0$). Error bars represent standard deviation obtained from three replicate specimens.

is kept well within the elastic capacity of the cement paste.

$$\text{Fractional change (\%)} = \frac{\text{Value at } n \text{ cycle} - \text{Value at initial cycle}}{\text{Value at initial cycle}} \times 100\% \quad (1)$$

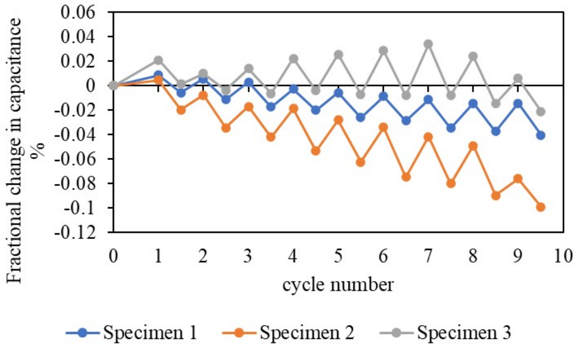
Fractional change values were calculated relative to the initial unloaded state.

The results show a completely irreversible behavior of both the capacitance and resistance of the PLC specimens. This is apparent when examining the base values at unloaded states, where the capacitance and resistance do not return to its previous values (Fig. 3). This behavior suggests that a change in the electrical and/or the physical properties of the tested specimens. Since the electrical contacts and the humidity of the specimens were not altered, the change in the electrical properties of the testing apparatus was eliminated. Thus, a change in the physical properties of the tested specimen is the more likely scenario.

In addition, a trend of decreasing capacitance and increasing resistance at the loading state of the consecutive cycles can be attributed to a decrease in the inner connectivity of the tested material (i.e. cement paste). The electrical conductivity of the cement pastes is predominantly governed by the mobility of ions such as ($Ca^{2+}, Na^{+}, K^{+}, OH^{-}, SO_4{}^{2-}$, etc.) in the pore solution [25]. If the pore structure is changed or interrupted in a plastic fashion (i.e. irreversible damage), it alters the base electrical properties of the cement paste. Considering that the conditions of the testing of the specimens were not altered, the previous behavior suggests that micro-cracking was the responsible factor. Micro-cracking of PLC at early age was previously reported in the literature, and it was found to correlate with the fineness of the limestone filler [8, 9]. This microcracking is a natural result of the increased autogenous deformation of the cement pastes that contain limestone [24].

3.2. Comparison with the OPC specimens

To evaluate the effect of using limestone in OPC on the self-sensing properties of cement pastes, the reference state is considered the OPC paste. The results of a same specimens



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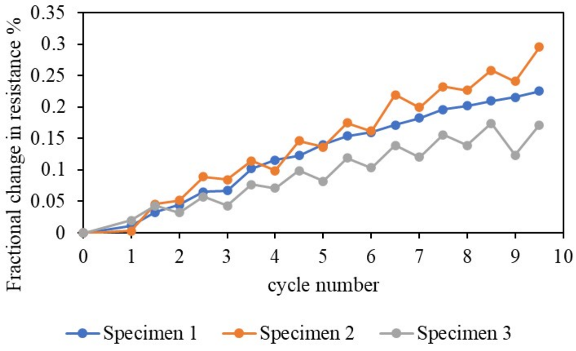
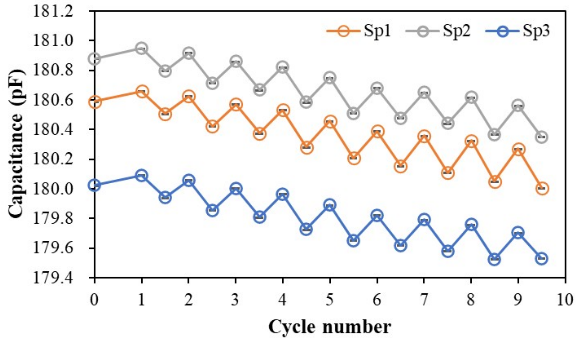


(b)

Fig. 4. Fractional change in (a) capacitance and (b) resistance of PLC paste specimens.



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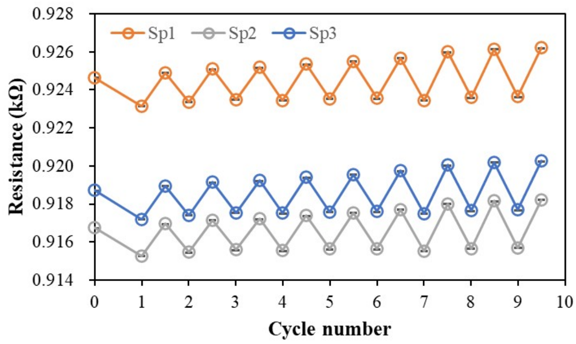


(b)

Fig. 5. Electrical response of OPC paste specimens under cyclic compressive loading: (a) capacitance and (b) resistance. Data reproduced from [15], obtained using the same specimen geometry, electrode configuration, curing conditions, and loading protocol as the present PLC experiments.

that were cast using OPC are presented in Fig. 5, which shows the effect of the cyclic compressive stress on the capacitance and resistance of the OPC specimens in Fig. 5 (a)(b) , respectively. It is worth mentioning that the stress level at each cycle is identical for the PLC paste and OPC paste specimens. The comparison between the results of OPC paste and PLC paste reveals that the capacitance of OPC paste follows a similar but weaker overall trend of increasing after each cycle of loading. While the resistance of the OPC paste shows a reversible pattern when unloaded contrary to the PLC paste.

The average initial capacitance of the PLC specimen is around 280.5 ± 57.8pF while its value for the OPC is around 180.5 ± 0.44pF, this higher initial value could indicate higher dielectric permittivity. The capacitance is directly proportional to the permittivity (ε) of the material, and follows Eq. 2:

$$C = \frac{\varepsilon A}{d} \tag{2}$$

where A : is the cross-sectional area, and d : is the distance between the electrodes of the specimen. A higher ε of the PLC may indicates an increased ionic mobility within the pore solution and/or a greater polarizability of hydration products. Limestone particles are often finer than clinker and act as nucleation sites [9], which accelerates hydration and leads to a refined and more packed particles and higher ionic concentration in early-age pore solution. In addition, more electrical double layers forming at solid-liquid interfaces. Thus, higher initial capacitance of the PLC can be a result of a higher moisture or ionic content in the pores and increased interfacial polarization. To test the hypothesis, the resistivity was measured at different values of frequency where the electric potential was set at 1 V , the results are shown in Fig. 6. The figure demonstrates that the specimens exhibit frequency-dependent electrical

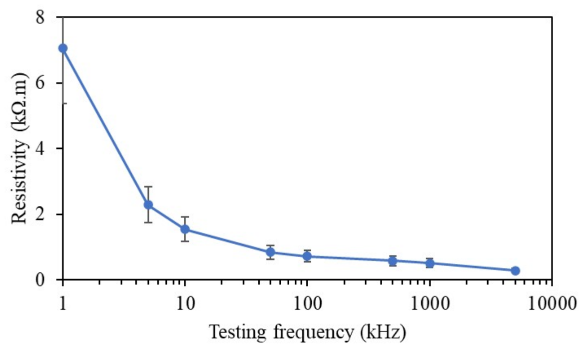


Fig. 6. Effect of testing frequency on the resistivity of the PLC specimens. The error bars represent the standard deviation. The frequency values were plotted on a logarithmic scale at the x-axis.

behavior, with resistivity decreasing significantly as frequency increases. At low frequencies, charge carriers may be trapped or influenced by interfaces (grain boundaries, voids, or phase boundaries), leading to high apparent resistivity. At higher frequencies, these barriers are bypassed or polarization effects cannot follow the fast-alternating field, resulting in lower measured resistivity [26]. This is typical of cement pastes, where charge movement becomes easier at higher frequencies [27]. Although this behavior was reported for the OPC specimens[15], the resistance of the PLC specimens increased when the compression stress increased, this indicates a negative piezoresistivity contrary to the OPC specimens.

The decreasing trend of the cyclic capacitance is more apparent in the PLC paste. The OPC paste shows minor fluctuation and more stable capacitance with smaller drops per each cycle of loading when compared to the OPC paste. The fractional change in capacitance, gives insight into the rate of degradation or sensitivity to cyclic loading. The capacitance is more responsive to damage or microstructural evolution in the PLC than in the OPC paste, which suggests that PLC might be more prone to cyclic fatigue effects in the microstructure. Thus, PLC pastes may have more porous or weaker C-S-H carboaluminates phases, explaining higher sensitivity. However, it is more prone to dielectric degradation and suffers a higher degradation rate under loading.

While the significant cyclic degradation of the capacitance in the PLC paste can be attributed to several factors as previously discussed, the measurement of the resistance of the specimens can help with a deeper understanding of the self-sensing behavior. The noticeable increase in the base resistance of the PLC specimens (see Fig. 3 (d)) is not shown in its OPC counterparts (Fig. 5 (b)).

At the end of the 9th loading cycle, the resistivity of the PLC pastes almost tripled and surpassed the OPC paste. This increase indicates a cumulative conductivity degradation upon increase in the compressive stress. The degradation in resistance highlights the possibility of structural damage, such as: microcrack formation, debonding at interfaces, redistribution of ionic conductivity, and closure of micro pores and conduction pathways which aligns with the literature [9]. On the other hand, OPC paste exhibit a narrow band of resistance fluctuation between cycles (around $\pm 0.0015 k\Omega$), which indicates a negligible progressive microstructural damage. The near-reversible base resistance of the OPC paste suggests an elastic behavior, while the irreversible base resistance of the PLC paste suggests a plastic physical behavior. Comparing the average initial resistance at the same LCR settings (i.e. 5 V and 100 kHz) of the PLC specimens is $621.4 \pm 91.7\Omega$, and for the OPC specimens is $920.3 \pm 4.0\Omega$, which gives resistivity values of $0.149 \pm 0.022 k\Omega.m$, and $0.221 \pm 0.000 k\Omega.m$. The higher standard deviation of the PLC paste specimen further highlights its greater heterogeneity from electrical point of view. The electrical resistivity of cement pastes was reported to decrease when the limestone content of the cement is increased [28]. The lower initial resistivity of the PLC paste specimens indicates higher ionic conductivity which can be a result of more connected pores that allow the movement of the dissolved ions in the pore solution more freely. While a larger and more connected pores enhances the conductivity, it has an adverse effect on the stability of the microstructure of the PLC paste and make its self-sensing properties more susceptible to moisture and microcracking effects.

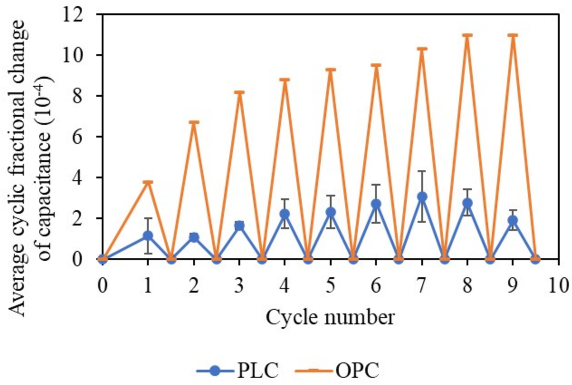
The relatively high standard deviation observed in the electrical response of the PLC specimens is attributed to the accumulation of the variations of the PLC microstructure. The heterogeneity of PLC pastes is expected to influence pore connectivity and electrical transport pathways, leading to increased specimen-to-specimen variability. Such inherent microstructural heterogeneity stems from limestone incorporation, including variations in limestone particle size, spatial distribution, and the resulting pore structure. The irreversible electrical behavior observed under cyclic loading is therefore more appropriately associated with early-stage microstructural rearrangement, localized damage development, and microcracking. The effect of polarization on the measured electrical properties is expected to be minimal since the test was conducted using high-frequency alternative current. Microstructural characterization (e.g., particle size distribution, porosity analysis, or microscopy) is outside the scope of this work, However, Future work incorporating detailed microstructural investigations is recommended to more directly correlate limestone distribution, pore structure evolution, and electrical response in PLC-based self-sensing systems.

In comparison with the previous work on the OPC pastes [15], the average cyclic fractional change in capacitance, and resistance are shown in Fig. 6, respectively. The cyclic fractional change is calculated for each cycle with reference to the value at the unloaded state immediately before that cycle. The cyclic fractional changes in the capacitance and resistance of the OPC paste specimens are significantly larger (up to $10-11\%$ and -25% to -30%, respectively), reflecting a strong and reversible piezopermittivity and piezoresistivity. In contrast, PLC paste specimens display smaller cyclic changes ($1-3\%$ in capacitance and $\pm1\%$ in resistance), suggesting moderate and irreversible piezopermittivity and piezoresistivity. The visible error bars (which represents the standard deviation) in the PLC data, compared to the absence of error bars in OPC data, indicate that PLC specimens exhibit greater variability in their electrical response due to microstructural heterogeneity due to the limestone filler distribution. On the other hand, OPC shows highly reproducible behavior across all tested specimens. Thus, PLC pastes could detect the early-age and low-stresses damage accumulation. This suggests that the intrinsic electrical response of PLC paste may exhibit reduced stability under prolonged cyclic loading, which could pose challenges for long-term self-sensing applications. Thus, PLC has a potential short-term or early-age sensing applications, such as damage initiation detection, construction-stage monitoring, or quality control during curing.

For self-sensing applications, the variability observed among the PLC paste specimens is a potential limitation. In practical structural applications, such inconsistent behavior may complicate signal interpretation and hinder the ability to provide precise diagnostic information regarding the structural condition. Calibrating each individual component of a structure is neither practical nor feasible. Nevertheless, if a structure is constructed using PLC, its self-sensing capability could still be valuable for detecting microstructural damage and tracking crack propagation.

4. Conclusions and recommendations

PLC is now widely accepted in the construction industry because of its environmental advantages and similar mechanical strength to OPC. The self-sensing potential of PLC paste was investigated in this work and compared to the OPC paste from a previous study. The PLC paste is more



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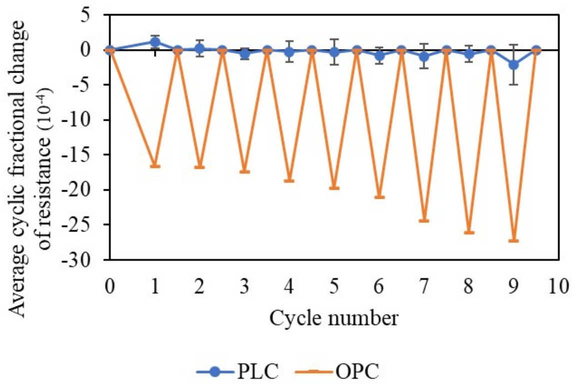

(b)

Fig. 7. Comparison of average cyclic fractional change in (a) capacitance and (b) resistance for PLC and OPC paste specimens. Fractional changes were calculated with respect to the unloaded state immediately preceding each loading cycle. Error bars indicate one standard deviation for PLC specimens.

prone to early-age damage and accumulative structural changes under cyclic loading even for very low stress levels. With continued cyclic loading, PLC specimens showed irreversible electrical evolution, characterized by residual changes of 0.02% in resistance and -0.08% in capacitance after unloading at most.

The PLC paste shows 3 times less piezopermittivity and approximately $25-30$ times less piezoresistivity than OPC paste, which suggests lower sensitivity. Thus, the long-term sensing capabilities of the PLC paste can be compromised due to microstructural damage. However, the proposed self-sensing approach, based on the simultaneous monitoring of capacitance and resistance, remains a viable technique for detecting damage and tracking crack propagation.

Future research directions include: (i) microstructural investigation of PLC paste using imaging and damage-detection techniques to directly link electrical response with internal damage mechanisms, and (ii) evaluation of self-sensing performance under higher compressive stress levels representative of practical structural health monitoring applications.

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