Advances in Electrical Characterization Techniques for Smart Construction Materials in SHM Applications for Masonry Buildings

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ABSTRACT

It has been shown that incorporating an adequate amount of electrically conductive fibers into structural mortars and clay bricks, near the percolation threshold, improves their ability to monitor local load redistribution, leading to damage-sensing capabilities. Moreover, reducing the fiber concentration allows for the detection of piezo properties, which may include piezoresistive, piezocapacitive, or piezoelectric behaviors. This topic is of specific interest in this work, where we examine different electromechanical characterization techniques to study various types of fillers for composite mortars and bricks, including carbon-based materials (carbon nanotubes, microfibers, graphene), steel fibers, and bioinspired structures. The construction materials incorporated in our research consist of cement pastes, mortars, and clay. All those mixes can be classified into percolated and non-percolated materials. The first type of material (percolated) has demonstrated capabilities for damage sensing, while the second material (non-percolated) exhibits a focus on electromechanical properties. In both cases, the key insight is the sensitivity of the construction material. To find out those interesting capabilities, it has been necessary to incorporate different electrical approaches, such as biphasic square, open circuit potentiometry and electrical impedance spectroscopy (EIS). Furthermore, the composition, geometry and number of electrodes, as well as the acquisition system have several implications on the electrical response. Electrical and mechanical measurements must be interpolated based on their respective timestamps, and then correlated and analyzed across both elastic and plastic regions. Additionally, compression and bending mechanisms were applied using ramp and square cyclic loading signals, with frequencies below 5 Hz, depending on the type of characterization performed. Following this logic, we have developed experimental methodologies to perform electromechanical characterizations and constructed models to physically explain the parameters emerging from these electro-mechanical correlations.

Keywords: Smart masonry, smart cement mortars, smart bricks, carbon-based fibers, bioinspired polymers, piezoelectricity, structural health monitoring.

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INTRODUCTION

Sensing composites capable of self-monitoring their state of strain through piezoresistivity, piezocapacitance and piezoelectricity hence by providing measurable variations in their electrical outputs when mechanically strained, are produced by mixing traditional construction materials with suitable electrically conductive inclusions [1–4]. One way to achieve piezoresistive and piezoelectric properties is to use nanostructured materials with electrical conductivity, such as graphene. However, even though 20 years have passed since Konstantin Novoselov, Andre Geim, and colleagues published the paper titled "Electric field effect in atomically thin carbon films" in Science [5], and despite a Nobel Prize in Physics and billions of dollars of investment, graphene (along with other two-dimensional (2D) materials, such as hexagonal boron nitride, without which much of the research on graphene would be impossible) remains one of the most exciting research fields.

Graphene-based cement composite materials have shown excellent electrical conductivity and piezo- properties [6, 7]. Recently, a case study was proposed where the synthesis of graphene brick nanocomposites with graphene oxide was reported [8]. Considering the fabrication of fired bricks involves high-temperature annealing, it was assumed that this process could transform GO to graphene when it can be embedded in a clay matrix before firing. In any case, analysis of the graphene market suggests it could reach a value of US\$1.5 billion by 2027, but much of the research on 2D materials remains confined to laboratories.

In this context, bioinspired alternatives to carbon-based fibers are emerging. A recent study reported the discovery of a new 2D crystalline phase formed by the self-assembly of proteins on van der Waals (vdW) solids, exhibiting an epitaxial relationship with the underlying lattice [9]. This structure consists of fully ordered monolayers of β-sheet lamellae, closely mimicking the architecture of traditional 2D nanomaterials and opening possibilities for bio-derived alternatives to other conductive fibers. Moreover, certain β-sheet-rich proteins, such as Nephila clavata (spider silk) and Bombyx mori (silkworm silk), can undergo thermal treatment without complete decomposition, resulting in the formation of carbonaceous structures. These bioinspired structures may serve as electrical contributors, whether damage-sensing materials or piezo- construction composites [10, 11].

In the field of cement-based composites, electrical methods are used to monitor electrical resistivity or conductivity and their changes in response to applied forces. These techniques are based on the application of an electrical current, which can be either direct (DC) or alternating (AC), to measure the corresponding response. This response can be either an electrical voltage or an electrical current. AC methods, such as electrical impedance spectroscopy (EIS) are commonly used in monitoring the hydration process [12] including the pore size distribution of the cementitious structure [13], while DC methods are used for measuring reversible changes in electrical resistance [14]. Nevertheless, challenges remain regarding the piezoresistive behavior and, more broadly, the electrical properties of cement-based composites, particularly in addressing polarization effects and the influence of electrode number and quality [15]. To address some of these issues, this work explores a biphasic approach, in which an alternating square wave signal at 1 Hz (low frequency) switches between positive and negative cycles to minimize polarization effects in the electrical response [16]. In addition, the biphasic method is employed to investigate damage sensing in over-percolated cement-based mortars, as well as piezoresistive and piezocapacitive behaviors in under-percolated smart construction materials [11].

Unlike most prior studies, this work also applies electrical impedance spectroscopy (EIS) and open circuit potential (OCP) acquisition to explore piezo-impedance and piezoelectricity, respectively, in clay-based composites. Finally, EIS is used to assess whether certain electrical parameters may exhibit immunity to environmental variations, making them more reliable for sensing applications.

METHODS

Materials Used in Testing

The tested materials measured through the biphasic approach included two types of cement mortar specimens: (i) 4 cm cubic samples incorporating 0.1 wt% graphene, and (ii) beams measuring 4 cm × 4 cm × 16 cm, with some doped with 0.5 wt% carbon fibers and others with 1.0 wt% carbon nanotubes. The cement mortar cubes were selected for piezo -resistive/-capacitive detection, whereas the beams were designated for damage detection. The materials selected for the piezo-impedance approach were 4.5 cm × 4.5 cm × 6.5 cm clay-based bricks containing 0.5 wt% steel fibers. Cement paste cubes with sides of 5 cm were embedded in a reinforced concrete plate (RCP) exposed to outdoor environmental conditions. Subsequently, the temperature of the RCP was monitored in parallel with EIS measurements to assess impedance parameters that may exhibit immunity to temperature influence. Furthermore, to explore piezoelectric properties, bricks doped with silk fibroin at a concentration of 0.1% by volume were studied.

Electrical Approaches

The electrical methodologies used in this research include alternating current (AC) signal excitations, specifically biphasic and impedance-based techniques, as well as open circuit potential (OCP) measurements, as shown in Figure 1. Biphasic excitation involves a 1 Hz square-wave input signal with an amplitude of ±10 V. This input is delivered as an electrical current regulated by a gain resistor placed in series with the construction material. To minimize signal loss and avoid distortion in the measured electrical resistance, the gain resistor should be of the same order of magnitude as the electrical resistance of the construction material [11, 17]. Based on the applied current and the resulting voltage drop across the material, electrical resistance and capacitance are calculated, as shown in Figure 1(a). This biphasic approach is implemented using the Smart Materials Electrometer (SME) [18], which operates in a three-electrode configuration consisting of: (i) a current-sensing electrode, (ii) a voltage-sensing electrode, and (iii) a reference electrode.

Figure 1(b) illustrates the experimental setup used to acquire electrical impedance data using a four-electrode configuration. A PalmSens4 Potentiostat / Galvanostat applies a sinusoidal waveform with an amplitude of 250 mV. Due to the low voltage amplitude, the material exhibits a linear response characterized by a phase-shifted current.

The frequency range typically explored spans from 0.1 Hz to 1 MHz. This range is selected because higher frequency values can introduce magnetic induction effects into the electrical response, which may interfere with the accurate characterization of material properties, particularly in systems where capacitive and resistive behaviors dominate at lower frequencies [19].

Finally, the OCP was measured using an instrumentation amplifier integrated into the SME. Additionally, the potentiostat/galvanostat featured an open-circuit potentiometry mode, allowing simultaneous acquisition of voltage and electrical current, as shown in Figure 1(c). The sampling rate was set to 100 Hz, and the measurement cables were connected directly to the smart construction material. To shield the piezoelectric measurements from surrounding electrical noise, the entire system was enclosed in a Faraday cage.

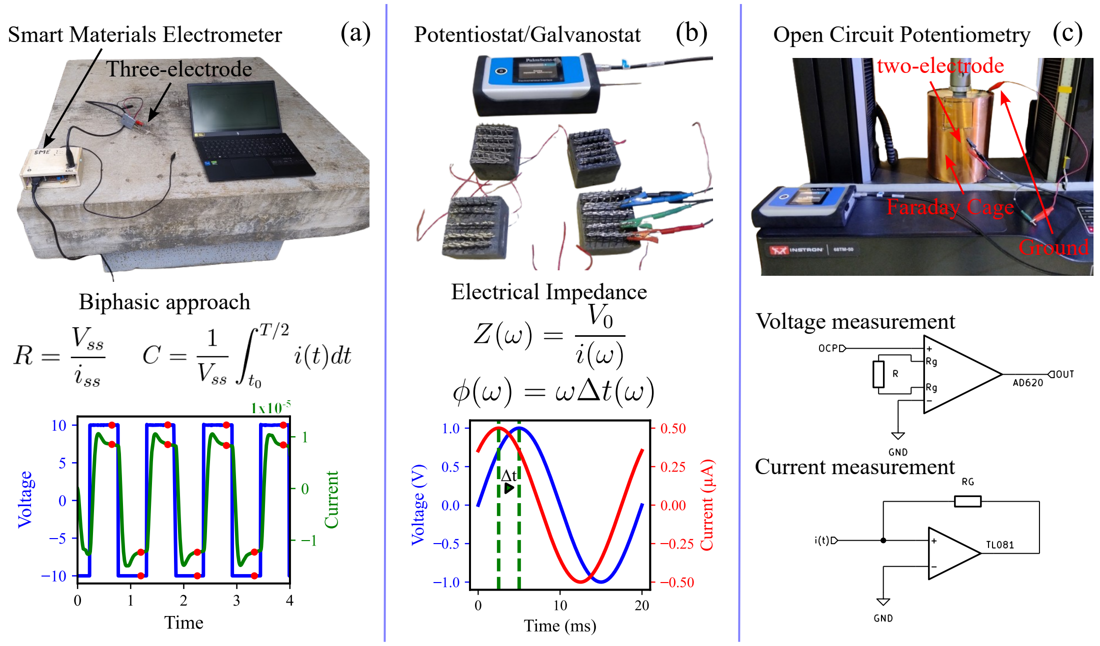

Figure 1. Electrical techniques in structural health monitoring. (a) biphasic approach, (b) electrical impedance spectroscopy, and (c) open circuit potentiometry.

Mechanical Measurements

Mechanical measurements were performed using a Universal Testing Machine (Instron 6850), as presented in Figure 2(a). What is more, the specimens' strain fields were recorded using a Digital Image Correlation System (DIC). Electromechanical characterization included: (i) cyclic compression tests with a triangular waveform, (ii) stepwise (stair-shaped) compression tests, and (iii) bending tests with a progressively increasing triangular load profile, as illustrated in Figure 2(b). The bending tests were used for damage sensing and were performed under displacement control at a rate of 0.5 mm/min, with a maximum displacement of 1.5 mm. The primary objective of these measurements was to obtain non-elastic loading cycles, which were later compared to the material's elastic modulus. This methodology enables the assessment of mechanical property degradation while simultaneously recording variations in electrical resistance using the biphasic approach. In contrast, the compression tests for piezoresistive and piezocapacitive approaches were conducted under force control at a loading rate of 200 N/s, whereas piezoelectric measurements were performed at a loading rate of 1500 N/s. Both tests were carried out until a maximum force of 5 kN. On the other hand, the stepwise compression tests were applied for piezo-impedance sensing, with each loading step lasting 4 minutes during which impedance measurements were recorded. The applied force was increased in 1 kN increments up to a maximum of 8 kN.

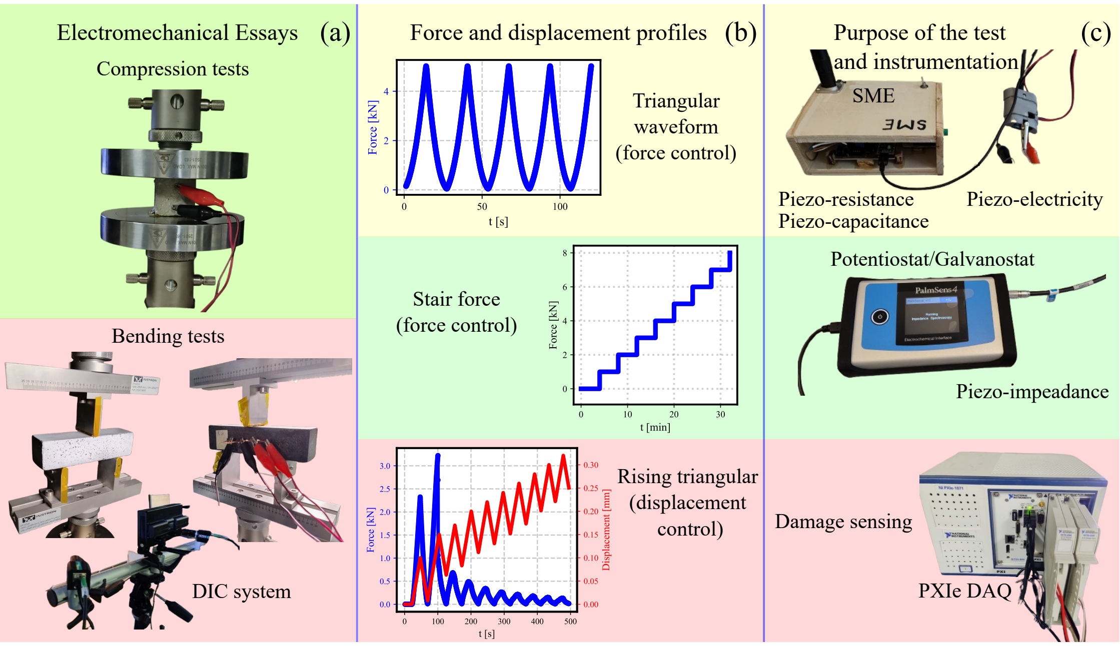

Figure 2. (a) Experimental setup for piezoelectric and damage sensing, (b) mechanical profiling, and (c) electromechanical characterization purposes including their respective acquisition system.

During mechanical testing, electrical measurements were conducted using the SME, the Potentiostat/Galvanostat, and the PXIe data acquisition system from National Instruments. The SME was employed for piezoresistive, piezocapacitive, and piezoelectric characterization, while the Potentiostat/Galvanostat was used for impedance measurements and also for piezoelectric characterization. Finally, the PXIe system was involved in resistive measurements for the damage-sensing approach, as these tests required long acquisition times and higher resolution in analog-to-digital conversion, which the SME could not provide.

RESULTS

Piezo-resistance, Piezo-capacitance, Piezo-impedance, and Piezoelectric Properties

The resistance and capacitance of graphene/cement-based composites under cyclic loading exhibited periodic variations that reproduced the triangular mechanical profile. Specifically, electrical resistance decreased as the mechanical force reached its peak, while the capacitance followed the opposite trend, as shown in Figure 3(a). The fractional change in resistance demonstrated greater linearity for strains greater than 0.0002 ϵ, with a regression coefficient of 0.973, compared to capacitance, which had a coefficient of 0.485. Despite its lower linearity, the fractional change in capacitance exceeded 100%, while the change in electrical resistance was approximately 30%. Consequently, the gauge factor derived from the capacitance (9158.424) was significantly higher than that obtained from resistance variation (2312.350).

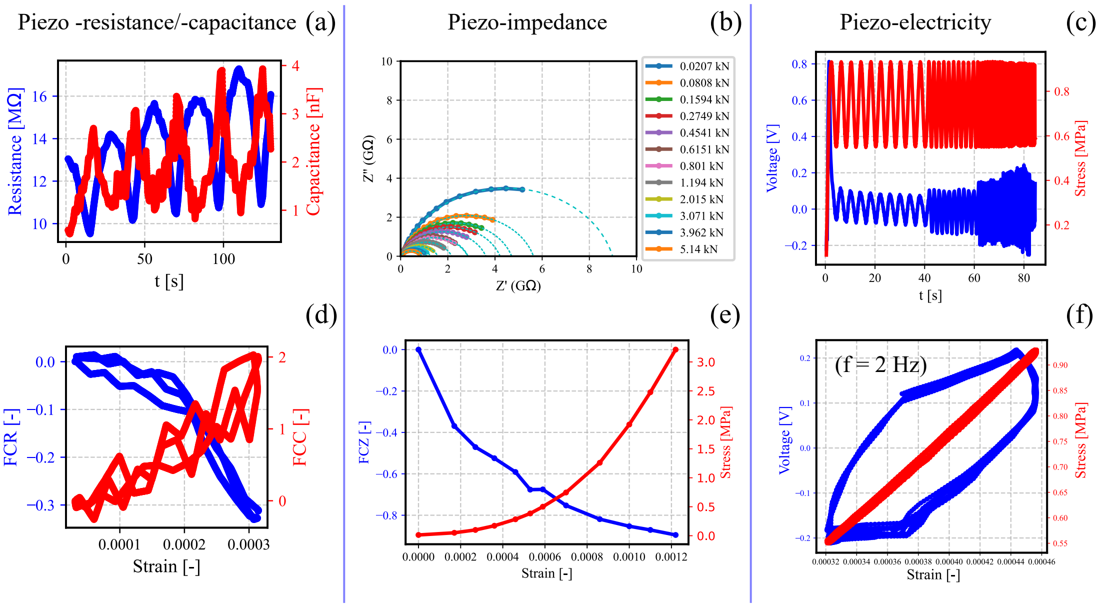

Figure 3. (a, d) Piezoresistance and piezocapacitance of cement mortars with 0.1 %wt of graphene; (b, e) Piezo-impedance in smart bricks with 0.5 %wt of steel fibers; and (c, f) piezoelectric response of smart bricks with 0.1 (% weighted by volume) of pyroprotein based on silk fibroin.

Figures 3(b) and 3(e) show the results of the electromechanical characterization, combining electrochemical impedance spectroscopy (EIS) with compressive loading under displacement control. The charge transfer resistance was calculated for each force. Notably, Figure 3(b) illustrates a clear decrease in electrical impedance following an increase in compressive force. However, this change is not linear with respect to strain, following the nonlinear relationship observed between stress and strain. For strains beyond 0.0002 ϵ the linearity expressed a coefficient R^2 of 0.944 and the gauge factor was 449.834.

The piezoelectricity of the smart brick with silk fibroin increases when the frequency increases from 0.25 to 4 Hz, as presented in Figure 3(c). However, this enhancement comes together with a lag in time or phase (28.8° at 2 Hz) between the voltage and the applied stress, as illustrated in Figure 3(f). Then, the alignment of electric dipoles in response to mechanical stress is not immediate, especially as the frequency increases. The dipoles cannot reorient quickly enough to coincide with the changing stress, resulting in this delay. This phenomenon is further supported by the memcapacitive behavior previously observed in silk fibroin/cement-based composites [17, 20]. Then, there is the need to study new formulations of smart bricks to find out the influence of silk fibroin concentration on the electrical response lag.

Damage Sensing

When the fiber content exceeds the percolation threshold, cement-based composites can exhibit damage-sensing capabilities, as illustrated in Figure 4(a) for mortar doped with 0.5% carbon fibers. For strains within the linear range (below 0.0052), the electrical resistance remained stable, staying below 3 Ω. However, the resistance more than doubled in the phase leading up to crack formation. Although this increase may appear excessive, even higher values would be expected after the onset of damage. Therefore, the electrical resistance surging is mitigated by the concentration and length of carbon fibers (CF), which produce a bridging mechanism that prevents the resistance from rising toward infinity relative to the undamaged state.

The entire development of the damage state in both reference mortar-based composites and those containing carbonaceous fillers is presented in Figure 4(b). The model used to describe the damage behavior under bending setup is detailed in reference [10]. Although the electrical conductivity for the undamaged state of cement mortar doped with CNTs (0.00063 S/m) remains higher than that of unmodified cement mortar (0.00038 S/m), plain cement mortar presents a more significant change in electrical property (expressed as G/G_0) during the damage process compared to CNTs/cement-based mortars. The evolution of damage had little effect on the conductance ratio of CNT/cement-based mortars, likely because the high concentration of CNTs weakens the cement matrix compared to the reference cement mortars [21]. On the other hand, CF/cement-based mortars exhibited the lowest conductivity among the three formulations in the undamaged state (0.814 S/m). Moreover, they showed greater sensitivity to damage beyond 40% compared to the reference mortars.

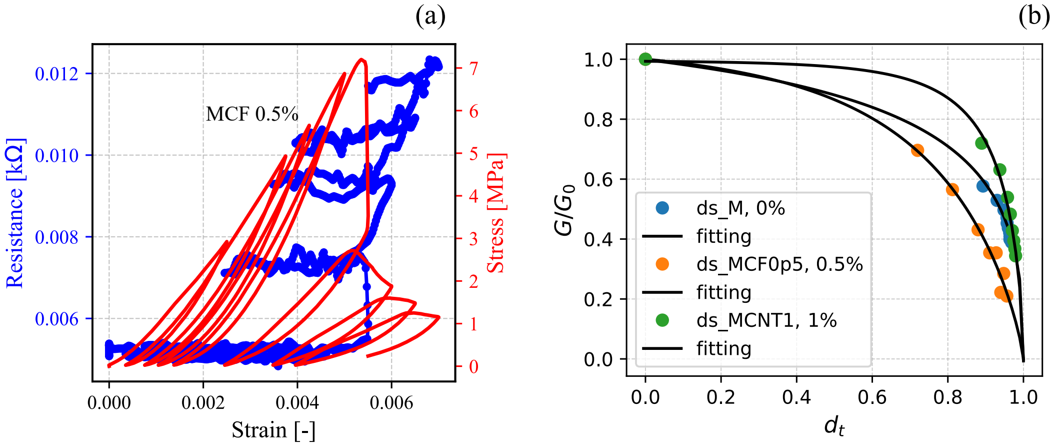

Figure 4. Damage degradation in cement mortars doped with carbon fibers “ds_MCF0p5” and carbon nanotubes “ds_MCNT1”. (a) Variation of electrical resistance under tensile stress. (b) Damage state under tension, represented as the ratio of conductance in the current state G to that in the undamaged state G_0.

Measurements in Real Environments

The RCP element exhibited a temperature variation of 7.9 °C (11.4 °C to 19.3 °C). The charge transfer resistance of the RCP element showed a moderate negative correlation with temperature (Pearson coefficient = –0.873), but the relationship was not statistically significant (p = 0.127). In contrast, pseudo-admittance Q_1 of the RCP element displayed moderate positive correlations with temperature within the 11.4 °C to 24.1 °C range. However, these correlations were not statistically significant, suggesting that the variations in Q_1 may be influenced by external factors unrelated to temperature. This relative insensitivity to temperature implies that Q_1 could serve as a robust parameter for strain sensing under seasonal conditions.

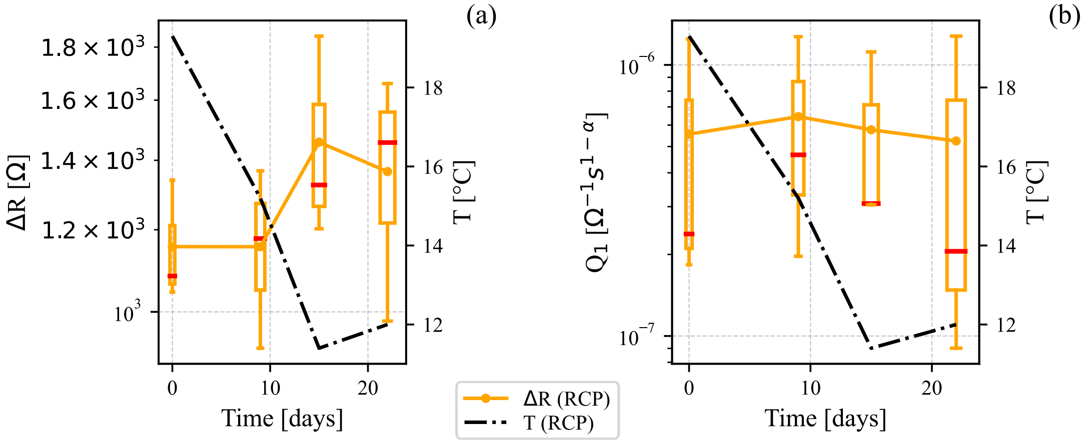

Figure 5. Environmental effects on the AC electrical properties of cement pastes doped with carbon nanotubes embedded in a reinforced concrete plate (RCP).

CONCLUDING REMARKS

This study has explored the integration of conductive carbon-based and bioinspired fillers into traditional construction materials to develop smart mortars and bricks for use in structural health monitoring applications. Through advanced electrical characterization techniques, such as biphasic excitation, open circuit potentiometry, and electrical impedance spectroscopy, the research has demonstrated that materials below the percolation threshold exhibit strong piezoresistive, piezocapacitive, and piezoelectric responses. Graphene-enhanced mortars showed high sensitivity in terms of electrical resistance variations, while silk fibroin-doped bricks revealed promising piezoelectric behavior. Additionally, the study confirmed that carbon fiber effectively detects damage progression, with electrical resistance changes correlating to crack development. Environmental testing showed that certain electrical parameters, like pseudo-admittance, remain stable under temperature variations, making them reliable for outdoor SHM applications. The research underscores the importance of optimizing filler type, concentration, and electrode configuration to enhance sensing performance, and it opens new avenues for sustainable, high-performance materials using bioinspired components like pyrolyzed silk proteins.

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