

Smart Cement Binder Concrete Curing and Compressive Piezoresistive Behavior

N. Amani and C. Vipulanandan, Ph.D., P.E.

Center for Innovative Grouting Materials and Technology (CIGMAT)

University of Houston, Houston, Texas 77204-4003.

Email: CVipulanandan@uh.edu; Phone (713)743-4278

Abstract

Smart cement is a highly sensing binder that can be used in multiple infrastructure applications in new constructions and also integrated into in-service infrastructures for real-time monitoring. In new construction the smart cement can be used as the binder in the concrete to make it highly sensing and then used in the construction of onshore and offshore infrastructures. In this study, the concrete with smart cement binder was characterized to identify the most sensitive electrical property for real-time monitoring. Based on the test results electrical resistivity was identified as the most critical electrical property for the concrete. Hence during concrete curing the electrical resistivity was monitored and modeled using Vipulanandan Curing Model. With the compressive loading the resistivity of the concrete increased. The piezoresistive axial strain at peak stress for the 28 days concrete with smart cement was over hundred percent which is 336 times (33,600%) higher compared to the concrete failure strain of 0.3%.

1. Introduction

During the past 200 years cement and concrete have been widely used in many applications and has been well documented. Cement slurries and grouts, based on the water-to-cement ratio, have been used in the construction of shallow and deep oil, gas and water wells both onshore and offshore. Also cement slurries are used to bond the pipes to the formation in horizontal directional drilling. Cement slurries are used to bond the steel casings and pipes to the varying geological formations in the wellbore and also to isolate the formations. In the well application cement has to bond very well with the highly varying natural geological formations with depth and to the human made steel casing and pipes and also has to perform for many decades under varying loading conditions, temperatures, pressures and seismic activities. Hence it is important to monitor the performance of the cement from the time of mixing to the entire service life in-situ (Vipulanandan 2021).

There have been many concrete bridges, highways, dams, buildings, storage facilities, foundations and pipes that have failed over the past hundred years due to loadings, earthquakes, fires and aging. Also dam failures and maintenance are becoming problem around the world and recent failures in Brazil. Failures can result in many types of losses and impact the economy and hence there is a need for real-time monitoring of the changing conditions in the infrastructures.

It is important to eliminate the failures of the highway bridges, pipelines, dams, oil wells and other infrastructures and highly sensing chemo-thermo-piezoresistive smart cement was recently developed to address these issues. Cement can be used in multiple applications because of some of its unique properties, easy to mix with aggregates/additives and also there are several

economical benefits. Concrete is a very popular construction material and has been used for over two thousand years. Concrete with high aggregate content in with a binding agent can be used in the construction of very small to very large structures such as bricks, roads, houses, bridges, pipes, dams, canals, storage, missile silos and nuclear waste containment. To attain the required levels of safety and durability of such structures, mixing proportions and especially aggregate content must be adjusted according to application in order to achieve mechanical requirements which will significantly affect the performance during its life time (Hou et al., 2017). In preparing the concrete and cement slurries, the water-to-cement ratios have been varied from 0.38 to 0.6 based on the mixing method, constituents of the concrete mix and applications (Vipulanandan et al. 2008, 2015a, 2016a, 2018). There are many different testing techniques such as ultrasound, fiber optic, electronic microscopy, X-ray diffraction, thermography and vibro-thermography have been used to study the aging of cement composites and for damage detection (Parvasi et al., 2016). However, many of these methods are difficult to adopt under field conditions where accessibility becomes an issue in deep foundations, buried storage facilities, wells, dams, tunnels and pipes.

Concrete

Concrete is composed of cement, aggregates, water and additives based on the applications. Cement is the most essential constituent in the concrete, which helps in the binding of the aggregates. The additives and water are part of the cement mix to enhance its performance. Immediately after mixing, the concrete quality is determined using the flow cone method for over nine decades. There is a need for better characterization of concrete using material properties which must be easy to adopt in the field.

Smart Cement

Cement is the largest quantity of material manufactured in the world, 4.2 trillion tons in 2017, and is used in many applications. Chemo-thermo-piezoresistive smart cement has been recently developed (U.S. Patent 10,481,143 (2019) Inventor Vipulanandan) which can sense and real-time monitor the many changes happening inside the cement during cementing of wells to concreting of various infrastructure to the entire service life of the structures. In concrete smart cement is the binder which can sense the changes within the concrete. The smart cement can sense the changes in the water-to-cement ratios, different additives, contamination and pressure applied to the cement sheath or concrete in terms of chemo-thermo-piezoresistivity. The failure compressive strain for the smart cement was 0.2% at peak compressive stress and the resistivity change is of the order of several hundred percentage making it over 500 times (50,000%) more sensitive (Vipulanandan et al. 2014-2021).

2. Objective

The overall objective was to highlight the potential use of the highly sensing smart cement integrated with real-time monitoring in new and also in-service infrastructures. The specific objectives are as follows:

- 1) Test the piezoresistive behaviour of concrete with smart cement binder.
- 2) Model the piezoresistive behaviour of the concrete using Vipulanandan p-q stress-piezoresistive strain Model.

3. Materials and Methods

In this study chemo-thermo-piezoresistive smart cement (Vipulanandan et al. 2014-2021; Vipulanandan 2021) was used to develop the concrete. For the curing and compressive behavior studies concrete was cast in plastic cylindrical molds with diameter of 50 mm and a height of 100 mm. Two conductive wires were placed in all of the molds to measure the changing in electrical resistivity. At least three specimens were tested under each condition investigated in this study.

(i). Sample Preparation

In this study table top blenders were used to prepare the cement and concrete specimens.

Smart cement (sensing cement): Cement was mixed with 0.1% carbon fibers to make it piezoresistive material (Vipulanandan et al., 2014a, b; 2015a, b).

Smart Cement Concrete

Smart cement concrete specimens were prepared using smart cement (less than 0.1% carbon fibers) with water-cement ratio of 0.38 (Vipulanandan et al. 2015a). Concrete specimens were prepared using 75% coarse aggregates based on the total volume of concrete. Sieve analysis (ASTM C136) was performed to determine the gradation of aggregate and the gradation. The median diameter (Katzner, 2012), which also represents d_{50} (ASTM) the size of 50% of the particles less than 4.2 mm. After mixing, the concrete were placed in 100 mm height and 50 mm diameter cylindrical molds with two conductive flexible wires 1 mm in diameter (representing the probes) were placed 50 mm apart vertically to measure the electrical resistance. The specimens were cured up to 28 days under relative humidity of 90%. At least three specimens were test under each condition and the average values are presented in the figures, tables and discussion.

(ii). Electrical Resistivity

Two different devices were used to measure the changes in the electrical resistivity of concrete and grout immediately after mixing up to the time they solidify. Both of the electrical resistivity devices were calibrated using the standard solutions of sodium chloride (NaCl).

Conductivity Probe

A commercially available conductivity meter was used to measure the conductivity (inverse of electrical resistivity). The conductivity measuring range was from $0.1\mu\text{S}/\text{cm}$ to $1000\text{ mS}/\text{cm}$, representing a resistivity of $100,000\ \Omega\cdot\text{m}$. to $0.01\ \Omega\cdot\text{m}$. respectively.

Digital Resistivity Meter

The digital resistivity meter measured the resistivity in the range of $0.01\ \Omega\cdot\text{m}$ to $400\ \Omega\cdot\text{m}$.

Electrical Resistance

LCR meter (inductance (L), capacitance (C), and resistance (R)) was used to monitor the electrical resistance of the specimens during the curing time. Two wire method with AC at 300 kHz frequency was used in order to minimize the contact resistances (Vipulanandan et al. 2013). During the initial stage of curing both the electrical resistivity (ρ) electrical resistance (R) were measured to determine the parameters K and G based on the Eqn.1.

$$\rho = \frac{R}{K+GR} \tag{1}$$

In this study, electrical resistance (R) and electrical resistivity (ρ) were measured independently during the initial curing period and the effective calibration factors (K and G) for the materials used in this study (insulators) were determined experimentally. For the smart cement and concrete Parameter G = 0 and Parameter K became stable (constant) in two to three hours. The Parameter K was more than double than the nominal Parameter K_n equal L/A where L is the spacing between the measuring wires and A is the cross section for the specimens tested.

Normalized change in resistivity $\Delta\rho$ with the changing conditions can be represented as follows:

$$\frac{\Delta\rho}{\rho} = \frac{\Delta R}{R} \tag{2}$$

The smart cement material is represented in terms of resistivity (ρ) and the changes due to stress will be quantified to evaluate the sensitivity of the material.

Two Wire Method

The change in resistance was measured using the two probe method with the LCR meter. To minimize the contact resistances, the resistance was measured at 300 kHz using two-wire method. This configuration was first calibrated using the same liquid (cement slurry) to determine the parameter K in Eqn. (1).

(iii). Compression Test (ASTM C39)

The cylindrical specimens (concrete, cement and grout) were capped and tested at a predetermined controlled displacement rate. Tests were performed using the Tinius Olsun machine at a controlling the displacement rate to 0.125 mm per minute. In order to measure the strain, a commercially available extensometer (accuracy of 0.001% strain) was used. During the compression test, the change in resistance was measured continuously using the LCR meter. Two probe method with alternative current (AC) at 300 kHz frequency was used in order to minimize the contact resistances (Vipulanandan and Amani, 2015). The change in resistance was monitored using the two-probe method, and the parameter in Eqn. (2) was used relate the changes in resistivity to the applied stress.

Modeling

Vipulanandan Curing Model

In order to represent the electrical resistivity development of the cement, Vipulanandan Curing model was used (Vipulanandan 2021) and the relationship is as follows:

$$\frac{1}{\rho} = \frac{1}{\rho_{min}} \left[\frac{\left(\frac{t+t_0}{t_{min}+t_0}\right)}{q_1+(1-p_1-q_1)\left(\frac{t+t_0}{t_{min}+t_0}\right)+p_1\left(\frac{t+t_0}{t_{min}+t_0}\right)^{\left(\frac{p_1+q_1}{p_1}\right)}} \right] \tag{3}$$

Where ρ is the electrical resistivity in $\Omega.m$, ρ_{min} is the minimum electrical resistivity in $\Omega.m$, t_{min}

is the time corresponding to the minimum electrical resistivity (ρ_{min}), t represents the curing time, t_0 is the model parameter influenced by the initial resistivity and p_1 and q_1 are time-dependent model parameters.

Vipulanandan Piezoresistivity Model

In order to represent the piezoresistive behavior of the hardened cement, Vipulanandan Piezoresistivity Model (Vipulanandan et al., 2018 a, b, 2021) was used and the relationship is as follows:

$$\sigma = \frac{\sigma_{max} \times \left(\frac{(\Delta\rho/\rho)}{(\Delta\rho/\rho)_0} \right)}{q_2 + (1-p_2-q_2) \times \left(\frac{(\Delta\rho/\rho)}{(\Delta\rho/\rho)_0} \right) + p_2 \times \left(\frac{(\Delta\rho/\rho)}{(\Delta\rho/\rho)_0} \right)^{\left(\frac{p_2+q_2}{p_2} \right)}} \quad (4)$$

Where σ_{max} is the maximum stress, $(\Delta\rho/\rho)_0$ is the piezoresistivity of the hardened cement under the maximum stress and p_2 and q_2 are model parameters influenced by the material properties.

Material Characterization

It is important to first characterize the materials based on the electrical properties, which can be easily adopted in the field.

Vipulanandan Impedance Model

Vipulanandan et al. (2018, 2021) studied different possible equivalent circuits for composite materials with two probes measurement and found appropriate equivalent circuits to represent materials.

Case 1: General Bulk Material – Capacitance and Resistance

In the equivalent circuit for Case1, the contacts were connected in series, and both the contacts and the bulk material were represented using a capacitor and a resistor connected in parallel. In the equivalent circuit for Case 1, R_b and C_b are resistance and capacitance of the bulk material, respectively; and R_c and C_c are resistance and capacitance of the contacts, respectively. Both contacts are represented with the same resistance (R_c) and capacitance (C_c), as they are identical. Total impedance of the equivalent circuit for Case 1 (Z_1) can be represented as:

$$Z_1(\sigma) = \frac{R_b(\sigma)}{1 + \omega^2 R_b^2 C_b^2} + \frac{2R_c(\sigma)}{1 + \omega^2 R_c^2 C_c^2} - j \left\{ \frac{2\omega R_c^2 C_c(\sigma)}{1 + \omega^2 R_c^2 C_c^2} + \frac{\omega R_b^2 C_b(\sigma)}{1 + \omega^2 R_b^2 C_b^2} \right\}, \quad (5)$$

where ω is the angular frequency of the applied signal. When the frequency of the applied signal is very low, $\omega \rightarrow 0$, $Z_1 = R_b + 2R_c$, and when it is very high, $\omega \rightarrow \infty$, $Z_1 = 0$.

Case 2: Special Bulk Material - Resistance Only

Case 2 is a special case of Case 1 in which the capacitance of the bulk material (C_b) is assumed to be negligible. The total impedance of the equivalent circuit for Case 2 (Z_2) is

$$Z_2(\sigma) = R_b(\sigma) + \frac{2R_c(\sigma)}{1 + \omega^2 R_c^2 C_c^2} - j \frac{2\omega R_c^2 C_c(\sigma)}{1 + \omega^2 R_c^2 C_c^2} \tag{6}$$

When the frequency of the applied signal is very low, $\omega \rightarrow 0$, $Z_2 = R_b + 2R_c$, and when it is very high, $\omega \rightarrow \infty$, $Z_2 = R_b$ (Fig. 1).

The shape of the curves shown in Figure 1 is very much influenced by material response and the two probes used for monitoring. Testing of smart cement and concrete indicated that Case 2 represented their behaviors and hence the bulk material properties can be represented by resistivity and characterized at a frequency of 300 kHz using the two probes.

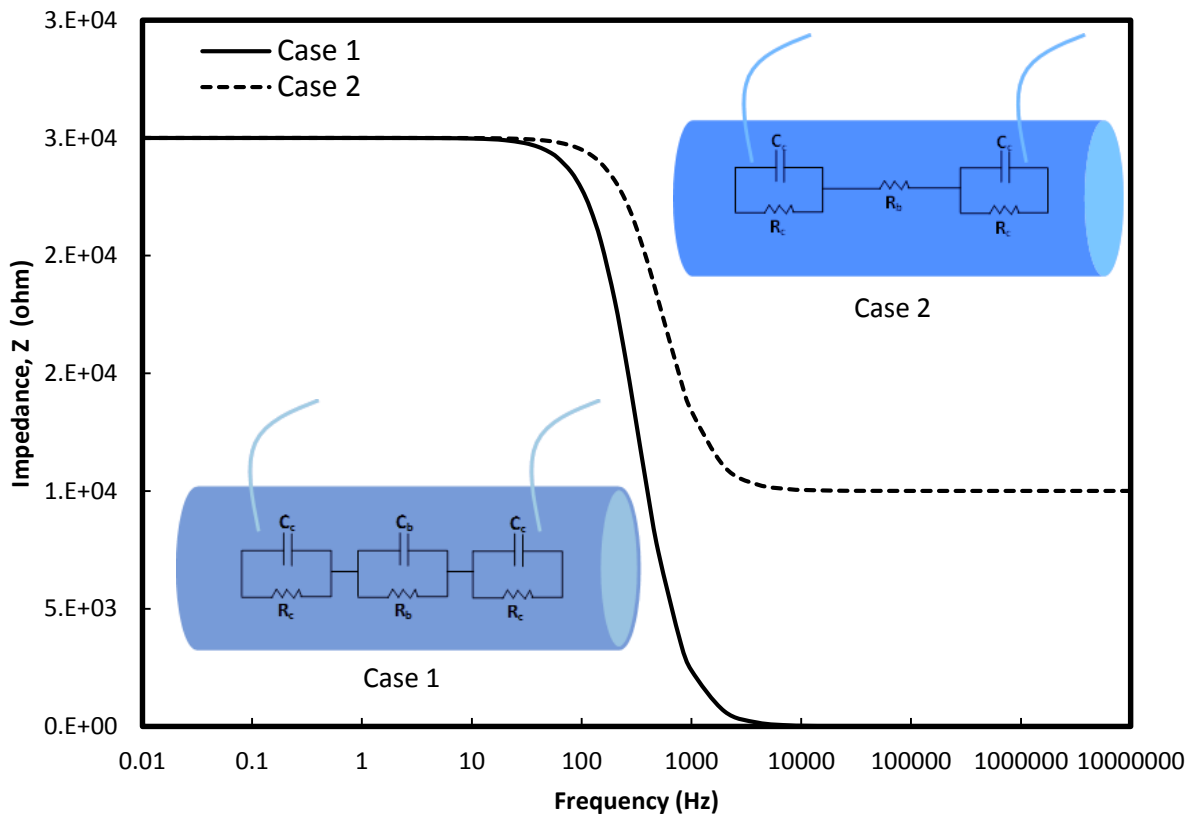


Figure 1. Vipulanandan impedance-frequency models for composite materials

4. Results and Analyses

Material Characterization

Impedance Vs Frequency Relations

Investigation of the impedance versus frequency relationship tested immediately after mixing and also after 28 days of curing for the smart cement grout and smart cement concrete is

shown in Figures 2 and 3. The observed shape of the curve represents the Case 2, indicating that the bulk material can be represented by resistance. This has been verified for over 5 years.

Initial resistivity

Initial electrical resistivity increased with the addition of aggregates.

(a) Smart Cement:

The average initial electrical resistivity of the smart cement was 1.02 Ω.m.

(b) Smart Cement Concrete:

75% Gravel: The average initial electrical resistivity of the smart cement concrete with 75% gravel increased by 267% to 3.74 Ω.m. This increment was due to gravel content in the concrete.

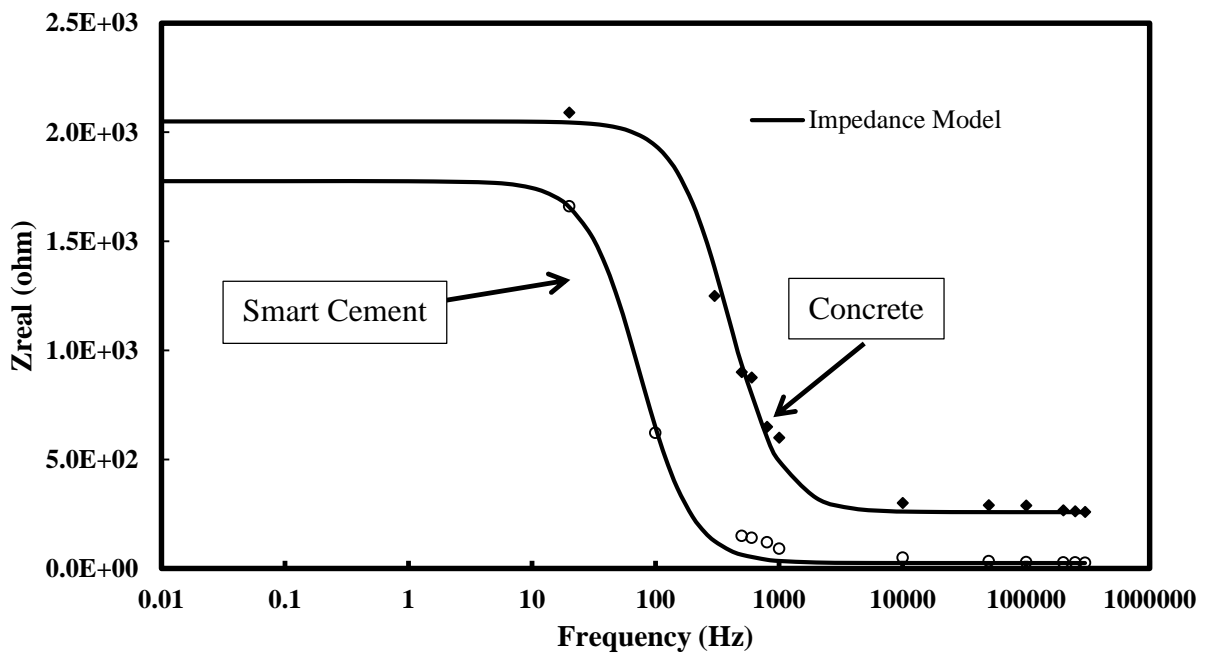


Figure 2. Impedance Characterization of the Smart Cement and Concrete Immediately after Mixing

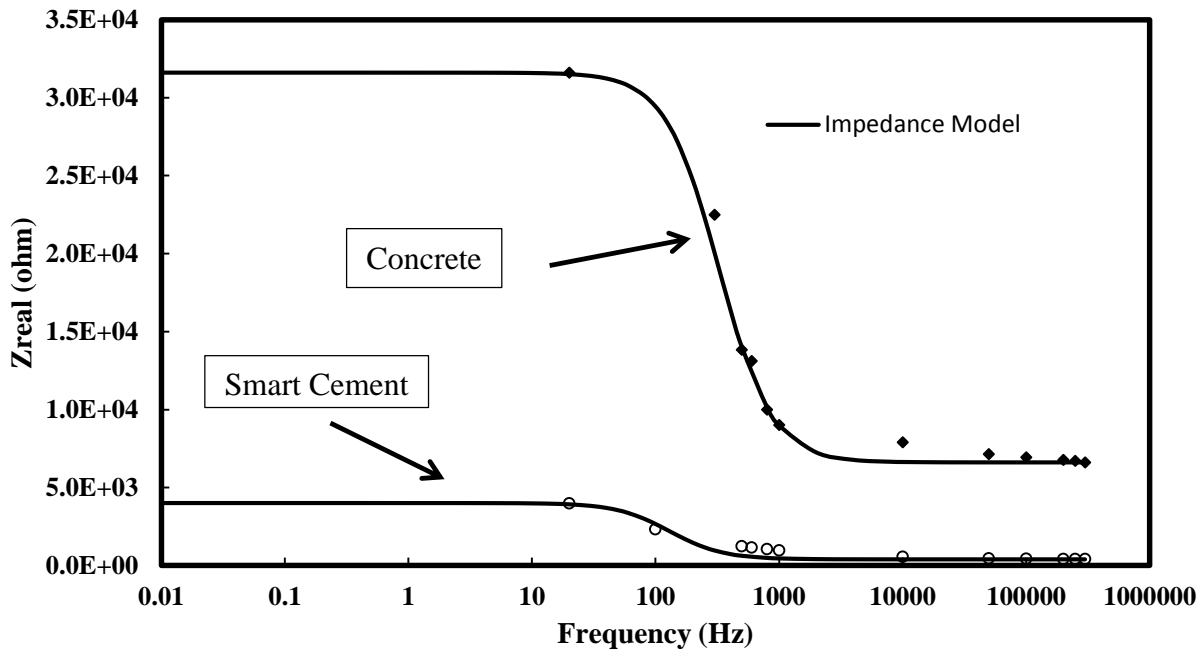


Figure 3. Impedance Characterization of the Smart Cement and Concrete after 28 Days of Curing

Resistivity during curing

Electrical resistivity of a concrete is determined mainly by the porosity and conductive ion concentration in the pore solution. From the standpoint of conductivity, concrete can be regarded as a two-component composite material, pore solution and solid phase (aggregate + hydration products + unhydrated binders) (Xiao and Li, 2008). During the setting of the cement, the capillary porosity is constant and changes in the pore solution resistivity leads to determine the evolution of the slurry resistivity (Zhiyong Liu et al., 2014). As shown in Figure 10, the pore solution resistivity decreased initially and reached a minimum resistivity of ρ_{min} at specific time of t_{min} which is due to increment of ionic concentration in pore solution. By preceding the hydration, production of C-S-H network caused later increment in bulk paste resistivity (Jie Zhang et al., 2009).

(a) Smart Cement:

The minimum electrical resistivity of the smart cement after 90 minutes of mixing was 0.79 $\Omega.m$ (Table 1, Figure 4).

(b) Smart Cement Concrete:

75% Gravel: The minimum electrical resistivity of the 75% gravel smart cement concrete increased by 339% to 3.46 $\Omega.m$. The time corresponds to the minimum resistivity of 75% gravel smart cement concrete reduced by 30 minutes to 60 minutes compare to the smart cement.

Table 1. Electrical resistivity parameters of the smart cement composites slurries

Smart Cement Concrete (by volume)	ρ_0 ($\Omega.m$)	ρ_{min} ($\Omega.m$)	t_{min} (minute)	ρ_{24} ($\Omega.m$)	$\frac{\rho_{24} - \rho_{min}}{\rho_{min}}$ %
No Gravel	1.02	0.79	90	5.14	550%
75% Gravel	3.74	3.46	60	20.01	478%

28 Days

(a) Smart Cement:

After 28 days of curing, the electrical resistivity of smart cement was 14.14 $\Omega.m$. (Fig. 5).

(b) Smart Cement Concrete:

75% Gravel: After 28 days of curing the electrical resistivity of 75% gravel smart cement composite increased by 333% to 61.24 $\Omega.m$.

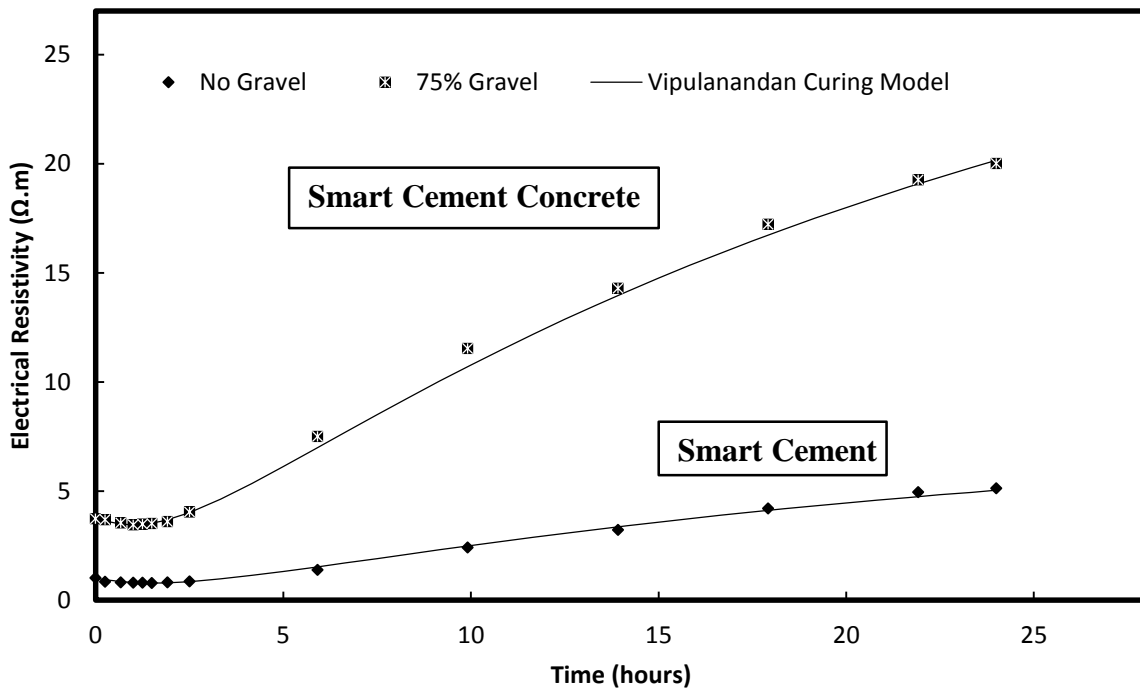


Figure 4. Development of electrical resistivity of smart cement composites during the initial 24 hours of curing

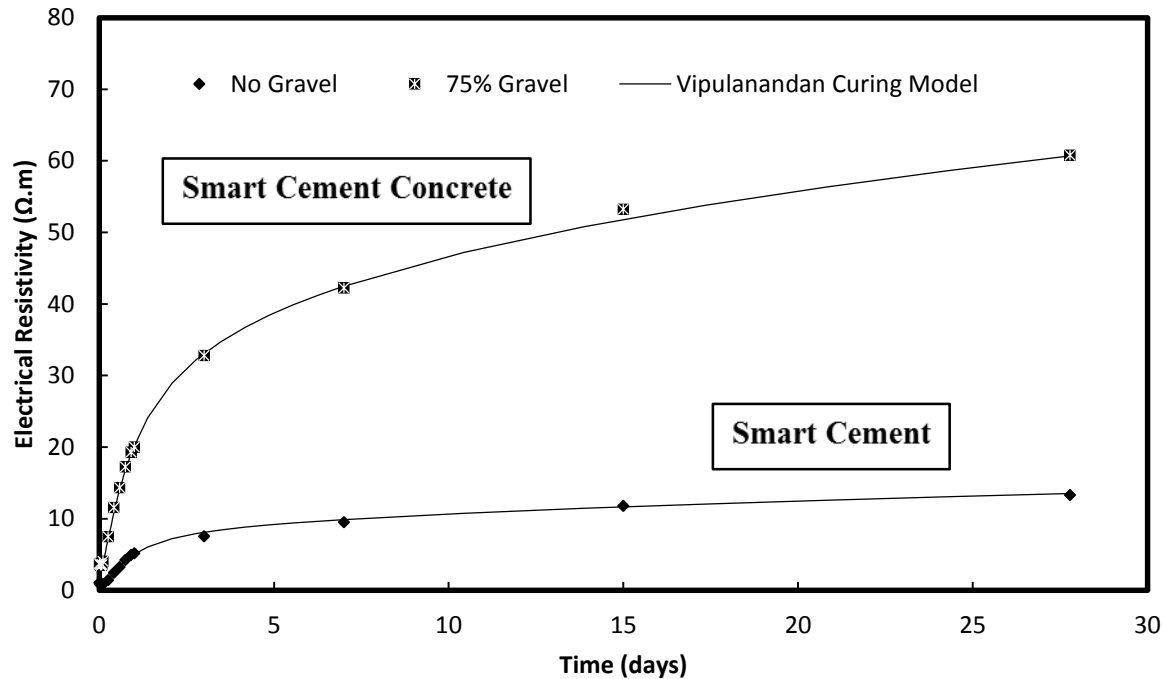


Figure 5. Development of electrical resistivity of smart cement composites during 28 days of curing

Compressive Behavior

Compressive Strength

Compressive strength of smart cement and smart concrete were tested after 1 and 28 days of curing.

1 day curing

(a) Smart Cement:

After 1 day of curing, the compressive strength of the smart cement was 8.6 MPa.

(b) Smart Cement Concrete:

75% Gravel: The compressive strength of the 75% gravel smart composite decreased by 29% to 6.1 MPa compare to the smart cement with no gravel.

28 days curing

(a) Smart Cement:

After 28 days of curing, the compressive strength of the smart cement was 21.7 MPa.

(b) Smart Cement Concrete:

75% Gravel: The compressive strength of the 75% gravel concrete decreased by 43% to 12.4 MPa compare to the smart cement with no gravel.

Changes in compressive strength of the concrete can be justified with the percentage of cement in

the concrete.

Piezoresistivity

Piezoresistive behavior of smart cement and smart cement concrete was evaluated after 1 day and 28 days of curing as shown in Figure 6.

1 day curing

(a) Smart Cement:

After 1 day of curing, the piezoresistivity of the smart cement at the peak compressive stress was 375% (Fig. 6. Table 2). Parameters p_2 and q_2 for the model were 0.61 and 0.57 respectively.

(b) Smart Cement Concrete:

75% Gravel: The piezoresistivity of the 75% gravel smart composite reduced by 57% to 163% compare to the smart cement. Parameters p_2 and q_2 for the model were 0.40 and 0.80 respectively.

28 days curing

(a) Smart Cement:

After 28 days of curing, the piezoresistivity of the smart cement was 204%. Parameters p_2 and q_2 for the model were 0.83 and 0.42 respectively.

(b) Smart Cement Concrete:

75% Gravel: The piezoresistivity of the 75% gravel smart composite reduced by 51% to 101% compare to the smart cement. Parameters p_2 and q_2 for the model were 0.81 and 0.40 respectively.

Table 3. Model parameters of p-q model for evaluating the piezoresistivity behavior of the concrete

Smart Cement Concrete	p_2	q_2	R^2	Compressive Strength (MPa)	Ultimate Piezoresistivity (%)	RMSE (MPa)
1 Day Curing						
No Gravel	0.61	0.57	0.99	8.6	375	0.3
75% Gravel	0.40	0.80	0.99	6.1	163	0.3
28 Days Curing						
No Gravel	0.83	0.42	0.98	21.7	204	1.0
75% Gravel	0.81	0.40	0.99	12.4	101	0.4

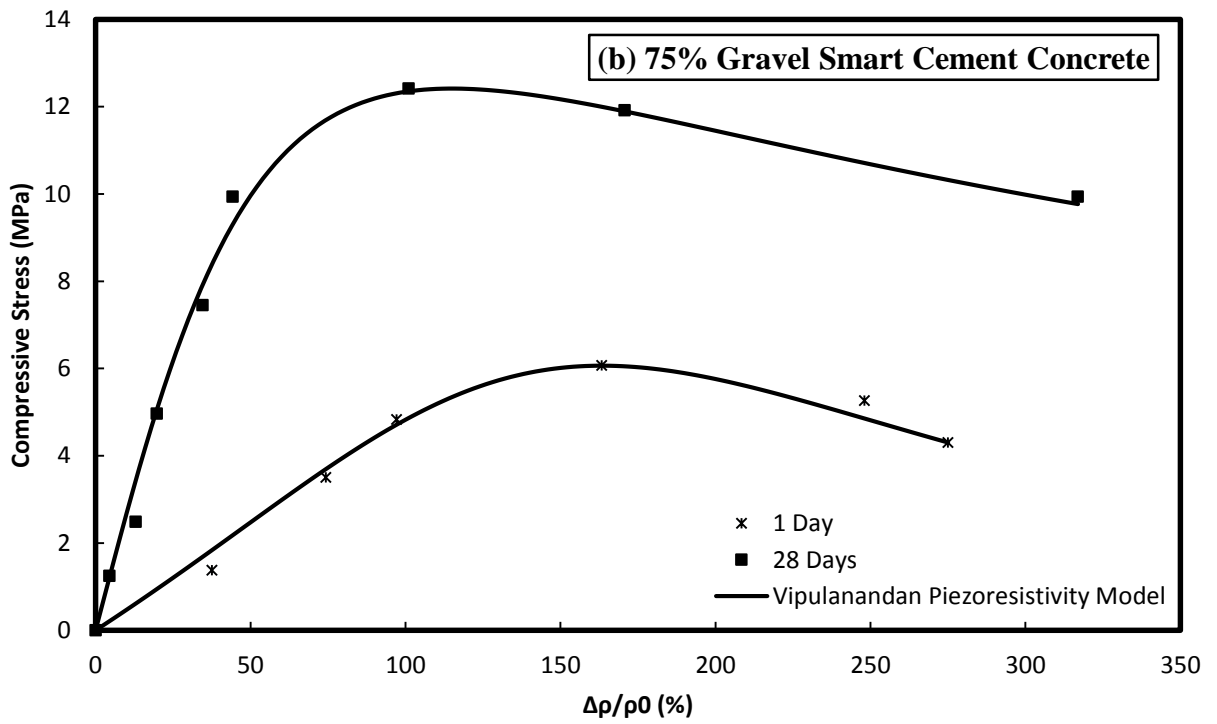
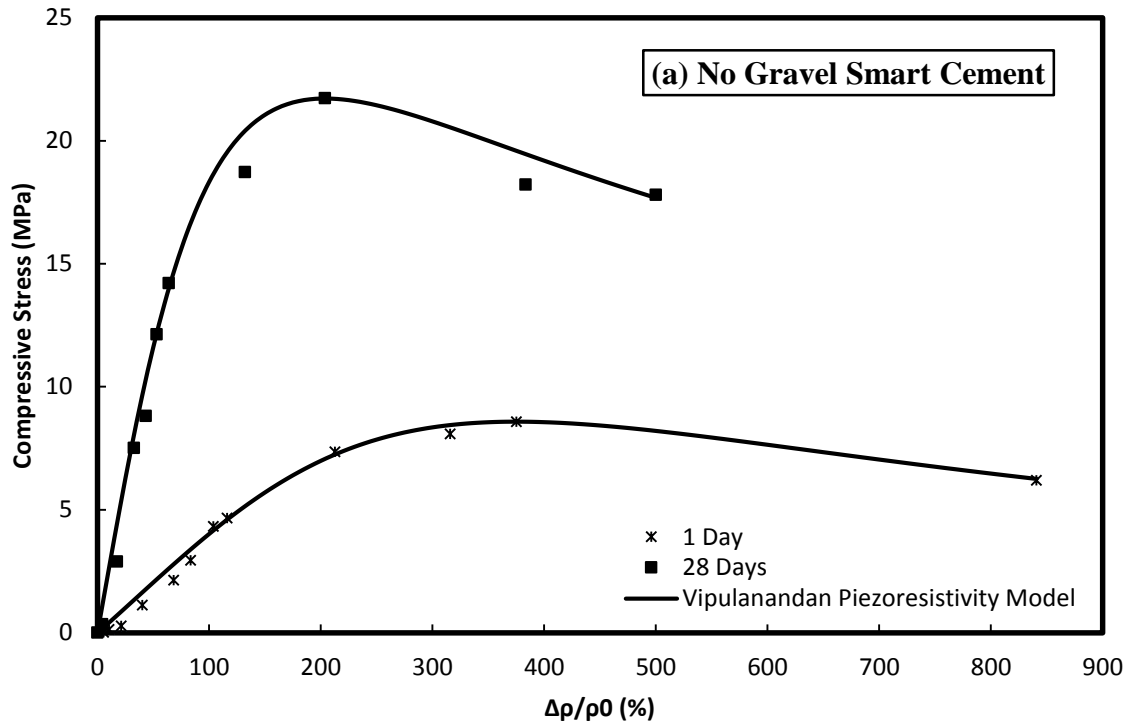


Figure 6. Piezoresistivity of smart cement composites after 1 and 28 days of curing: (a) No gravel and (b) 75% Gravel

5. Conclusions

The smart cement was used as the binder in the concrete to make it a highly bulk sensing concrete. It is also important to develop real-time monitoring systems that can be easily adopted in the field. Based on experimental and analytical study on the behavior of smart concrete (with smart cement binder) with the real-time monitoring in the field following conclusions are advanced:

1. Addition of coarse aggregate increased the initial electrical resistivity of the smart cement composite as well as long term electrical resistivity during curing. The initial electrical resistivity of smart cement was $1.02 \Omega \cdot m$ which increased to $3.74 \Omega \cdot m$ with 75% gravel respectively. After 28 days of curing, the electrical resistivity of smart cement was $14.14 \Omega \cdot m$ which increased to $61.24 \Omega \cdot m$ with 75% gravel respectively. Also Vipulanandan Curing Model predicted the electrical resistivity development in the concrete very well.
2. The piezoresistivity of the smart cement with 0% and 75% gravel content after 28 days of curing were 204% and 101% at a peak compressive stress respectively. Vipulanandan Piezoresistivity Model can be used to predict the piezoresistivity behavior of the smart cement concrete very well.
3. The failure strain of concrete is 0.3%, hence piezoresistive concrete has magnified the monitoring resistivity parameter by 336 times (33,600%) or more higher based on the aggregate content and making the concrete a bulk sensor.
4. Vipulanandan Curing Model and Vipulanandan p-q Stress-Piezoresistive Strain Model Predicted the concrete with smart cement binder behavior very well.

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References

1. Vipulanandan, C., and Ali, K., (2018a) "Smart Cement Grouts for Repairing Damaged Piezoresistive Cement and the Performances Predicted Using Vipulanandan Models" *Journal of Civil Engineering Materials*, American Society of Civil Engineers (ASCE), Vol. 30, No. 10, Article number 04018253.
2. Vipulanandan, C., and Amani, N., (2018b) "Characterizing the Pulse Velocity and Electrical resistivity Changes In Concrete with Piezoresistive Smart Cement Binder Using Vipulanandan Models" *Construction and Building Materials*, Vol. 175, pp. 519-530.
3. Vipulanandan, C.(2021) *Smart Cement Development. Testing, Modeling and Real-Time Monitoring*, Taylor and Francis, CRC Press, 440 pages.