Characterizing the Compressive Piezoresistive Behavior of Smart Cement Using Vipulanandan Model

C. Vipulanandan, Ph.D., P.E.
Center for Innovative Grouting Materials and technology (CIGMAT)
Department of Civil and Environmental Engineering
University of Houston
Houston, Texas 77204-4003
Email: CVipulanandan@uh.edu

Abstract - **The effect of water-to-cement ratio (w/c) on the piezoresistive behavior of smart cement was investigated. The sensing property of the smart cement was modified with 0.1% carbon fiber (CF) and the behavior was investigated up to 28 days of curing. Electrical resistivity was identified as the sensing and monitoring property for the smart cement. The initial resistivity (ρ_o) of the smart cement decreased from 1.03 Ω-m to 1 Ω-m and 0.9 Ω-m, a 3% and 12% reduction when the w/c ratio was increased from 0.38 to 0.44 and 0.54 respectively, higher than the changes in the initial unit weights of the cement slurry. For the smart cement the electrical resistivity increased with the applied compressive stress (New Technology). The piezoresistive axial strain of the smart cement at failure with water-cement ratio of 0.38 and curing of 28 days was over 300% compared to the failure strain cement of 0.2%, 1500 times (150,000%) higher make it a highly sensing material. The Vipulanandan p-q piezoresistive model predicated the piezoresistive compressive stress – change in resistivity relationship of the smart cement very well. Linear correlations were observed between resistivity index (RI_{24hr}) and compressive strength of smart cement for different curing times.**

Keywords: Water-to-cement ratio, Electrical resistivity, Piezoresistivity, Modeling.

I. INTRODUCTION

Cement is used in multiple applications to build the infrastructures such as all sizes and types of buildings, bridges, highways, underground storage facilities, pipelines and wells (oil, gas and water) for centuries. Cement is produced around the world and its unique binding properties, strength, durability and cost makes it a unique material compared all other human made materials. Over the past few decades there have been failures of cement based infrastructures resulting in losses and human deaths [1, 2]. Hence there is need to develop highly sensing cement so that its performance and changing properties over time can be monitored [3-9].

Past studies have investigated the changes in electrical resistivity with applied stress referred to as piezoresistive behavior of modified cement-based and polymer composites [2]. The studies showed that the changes in resistivity with the applied stress were 30 to 50 times higher than the strain in the materials. Hence the change in resistivity has the potential to be used to determine the integrity of the materials and modeling the nonlinear behavior of the smart cement is important to better understand the effects of various parameters investigated in the study [3, 5, 7, 8, 9].

Recent studies have suggested that replacing the DC measurement with the AC measurement can eliminate the polarization effect [4, 5, 7, 8, 9].

II. OBJECTIVES

The overall objective was to quantify the effect of different w/c ratio on the electrical resistivity and piezoresistive behavior of smart cement. The specific objectives are as follows

(i) Experimentally verify the piezoresistive behavior of smart cement with different water-to-cement ratios up to 28 days of curing.
(ii) Model the piezoresistive behavior of smart cement with different water-to-cement ratios

ISBN: 978-624-6150-05-1

up to 28 days of curing using the Vipulanandan p-q Piezoresistive Model.

III. MATERIALS AND METHODS

In this study, cement with water-to-cement of 0.38, 0.44 and 0.54 was used. To improve the sensing properties and piezoresistive behavior of the cement modified with less than 0.1% of carbon fibers (CF) by the weight of cement was mixed very well for all the samples (no change in cement sresistivity). After mixing, specimens were prepared using 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 prepared for each mix.

Density

The density of smart cement with and without CF was measured immediately after mixing using the standard mud balance cup.

Electrical Resistivity

It was very critical to identify the sensing properties for the cement that can be used to monitor the performance. After numerous studies and based on the current study on cements, electrical resistivity (ρ) was selected as the sensing property for cement-based materials. Hence two parameters (resistivity and change in resistivity) were used to quantify the sensing properties of cement. Electrical resistivity is given by:

$$\rho = R/K_e \quad (1)$$

where R is electrical resistance, and Ke is the effective correlation parameter. In the literature the nominal correlation parameter (developed for conductors) Kn which is equal to the ratio L/A where L is the linear distance between the electrical resistance measuring points, A is the effective cross sectional area. Current study has shown that the Ke was in the range of 50 to 55 while the Kn was in the rage of 25 to 30. Normalized change in resistivity with the changing conditions is represented as

$$\frac{\Delta\rho}{\rho_0} = \frac{\Delta R}{R_0} \quad (2)$$

where R_o, ρ_o: Initial resistance and resistivity respectively and ΔR, Δρ: change in resistance and change in resistivity respectively.

Initial Resistivity of Smart Cement Slurry

Two Different methods were used for electrical resistivity measurements of the cement slurries. To assure the repeatability of the measurements, the initial resistivity was measured at least three times for each cement slurry and the average resistivity was reported. The electrical resistivity of the cement slurries were measured using conductive probe and digital resistivity meter used in the oil industry.

Resistivity of smart cement

In this study high frequency AC measurement was adopted to overcome the interfacial problems and minimize the contact resistances. Electrical resistance (R) was measured using LCR meter (measures the inductance (L), capacitance (C) and resistance (R)) during the curing time. This device has a least count of 1 μΩ for electrical resistance and measures the impendence (resistance, capacitance and inductance) in the frequency range of 20 Hz to 300 kHz. Based on the impedance (z) – frequency (f) response it was determined that the smart cement was a resistive material [5, 7, 8, 9]. Hence the resistance measured at 300 kHz using the two probe method was correlated to the resistivity (measured using the digital resistivity device) to determine the K_e factor (Eqn.1) for a time period of initial five hours of curing. This K_e factor was used to determine the resistivity of the cement with the curing time.

Piezoresistivity Test

*Piezores*istivity describes the change in electrical resistivity of a material under stress. Since oil well cement serves as pressure-bearing part of the oil and gas wells in real applications, the piezoresistivity of smart cement (stress – resistivity relationship) with different w/c ratios were investigated under compressive loading at different curing times. During the compression test, electrical resistance was measured in the direction of the applied stress. To eliminate the polarization effect, AC resistance measurements were made using a LCR meter at frequency of 300 kHz [9].

Statistical Parameters

In order to determine the accuracy of the model predictions, both coefficient of determination (R^2) and the root mean square error (RMSE) were used.

IV. RESULTS AND DISCUSSION

Density and Resistivity

Several characteristic resistivity parameters can be used in monitoring the curing (hardening process) of the cement. The parameters are initial resistivity (ρ_o), minimum electrical resistivity (ρ_{min}), time to reach the minimum resistivity (t_{min}) and percentage of maximum change in resistivity at the end of 24 hours (RI_{24hr}) and 7 days (RI_{7days}) were defined in Eqn. (3) and Eqn. (4) as follows:

$$RI_{24hr} = \left(\frac{\rho_{24hr} - \rho_{min}}{\rho_{min}}\right) 100 \quad (3)$$

$$RI_{7\ days} = \left(\frac{\rho_{7days} - \rho_{min}}{\rho_{min}}\right) 100 \quad (4)$$

(a)w/c =0.38

Unit weight of the smart cement with w/c of 0.38 was 19.38 kN/m^3. The initial electrical resistivity (ρ_o) of the smart cement with w/c ratio of 0.38 modified with about 0.1% CF was 1.03 Ω-m. and the electrical resistivity reduced to reach the ρ_{min} of 0.99 Ω-m after 99 minutes (t_{min}) as summarized in Table 1. The 24 hours electrical resistivity (ρ_{24hr}) of the cement was 4.15 Ω.m. Hence the maximum change in electrical resistivity after 24 hours (RI_{24hr}) was 319% as summarized in Table 1. The 7 days electrical resistivity (ρ_{7days}) of the cement grout was 7.75 Ω.m, hence the maximum change in electrical resistivity after 7 days (RI_{7days}) was 683%.

(b) w/c =0.44

Unit weight of the smart cement with w/c of 0.44 was 18.96 kN/m^3. The initial electrical resistivity (ρ_o) of the smart cement with w/c ratio of 0.44 and modified with 0.1% CF was 1 Ω-m. The electrical resistivity reduced to reach the ρ_{min} of 0.89 Ω-m after 114 minutes (t_{min}) as summarized in Table 1. The 24 hours electrical resistivity (ρ_{24hr}) of the sample was 2.55 Ω.m. Hence the maximum change in electrical resistivity after 24 hours (RI_{24hr}) was 187%. The 7 days electrical resistivity (ρ_{7days}) of the sample was 5 Ω.m, hence the maximum change in electrical resistivity after 7 days (RI_{7days}) was 462%.

(c) w/c=0.54

Unit weight of the smart cement with w/c of 0.38 was 18.56 kN/m^3. The initial electrical resistivity (ρ_o) of the smart cement with w/c ratio of 0.54 modified with 0.1% CF was 0.9 Ω-m (Table 1) and the electrical resistivity reduced to reach the ρ_{min} of 0.78 Ω-m after 128 minutes (t_{min}) as summarized in Table 1. The 24 hours electrical resistivity (ρ_{24hr}) of the sample was 1.67 Ω.m. Hence the maximum change in electrical resistivity after 24 hours (RI_{24hr}) was 114% as summarized in Table 1. The 7 days electrical resistivity (ρ_{7days}) of the sample was 4.6 Ω.m, hence the maximum change in electrical resistivity after 7 days (RI_{7days}) was 490%.

Table 1. Curing Electrical Resistivity Parameters for the Smart Cement

w/c	Density (kN/m^3)	Initial resistivity, ρ_o (Ω.m)	ρ_{min} (Ω.m)	t_{min} (min)	ρ_{24hr} (Ω.m)	$\rho_{7\ days}$ (Ω.m)	$RI_{24\ hr}$ (%)	$RI_{7\ days}$ (%)
0.38	19.38	1.03	0.99	99	4.15	7.75	319	683
0.44	18.96	1.0	0.89	114	2.55	5.0	187	462
0.54	18.56	0.9	0.78	128	1.67	4.6	114	490

ISBN: 978-624-6150-05-1

Piezoresistivity and strength of smart cement

*Additi*onal of about 0.1% carbon fibers substantially improved piezoresistive behavior of the cement and the electrical resistivity increased with the application of compressive loading, all new compared to the information in the literature [5, 6, 7, 8]. Vipulanandan p-q piezoresistive model was used to predict the change in electrical resistivity of cement during with applied stress for 1, 7 and 28 days of curing. The Vipulanandan p-q piezoresistive model was defined as follows [5, 7, 8]:

$$\frac{\sigma}{\sigma_f} = \left[\frac{\frac{x}{x_f}}{q_2+(1-p_2-q_2)\frac{x}{x_f}+p_2\left(\frac{x}{x_f}\right)^{\left(\frac{p_2}{p_2-q_2}\right)}}\right] \qquad (5)$$

where σ: stress (psi); σ_f: stress at failure (psi); $x = \left(\frac{\Delta\rho}{\rho_o}\right) * 100$ = Percentage of change in electrical resistivity due to the stress; $x_f = \left(\frac{\Delta\rho}{\rho_o}\right)_f * 100$ = Percentage of change in electrical resistivity at failure; Δρ: change in electrical resistivity; ρ_o : Initial electrical resistivity (σ=0 MPa) and p_2 and q_2 are piezoresistive model parameters.

(i) 1 day of curing

The compressive strength (σ_f) of the cement with w/c ratio of 0.38, 0.44 and 0.54 for one day of curing were 10.6 MPa, 8.4 MPa and 4.6 MPa respectively. Addition of 0.1% CF to the cement (smart cement) with w/c ratio of 0.38, 0.44 and 0.54 increased the compressive strength to 10.9 MPa, 9.8 MPa and 5.3 MPa respectively. Hence the addition of 0.1% carbon fiber (CF) increased the strength by 3%, 17% and 15% for cement with w/c ratio of 0.38, 0.44 and 0.54 respectively. The change in electrical resistivity at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ for the unmodified cement with different w/c ratios of 0.38, 0.44 and 0.54 were 0.70%, 0.60% and 0.48% respectively. With 0.1% CF addition to the smart cement the electrical resistivity at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ for the smart cement with w/c of 0.38, 0.44 and 0.54 were 583%, 531% and 355% respectively. Additional of 0.1% CF to the cement substantially enhanced the change in electrical resistivity of oil well cement at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ with w/c ratios of 0.38, 0.44 and 0.54 by a factor of 832, 697 and 729 respectively compared to the unmodified cement.

Using the Vipulanandan p-q Piezoresistive model (Eqn. (5)), the relationships between compressive stress and the change in electrical resistivity $\left(\frac{\Delta\rho}{\rho_o}\right)$ of the cement with different w/c ratios of 0.38, 0.44 and 0.54 for one day of curing were modeled. The piezoresistive model (Eqn. (5)) predicted the measured stress- change in resistivity relationship very well (Fig. 1a). The model parameters q_2 and p_2 are summarized in Table 2. The coefficients of determination (R^2) were 0.98 and 0.99. The root mean square of error (RMSE) varied between 0.02 MPa and 0.04 MPa as summarized in Table 2.

(ii) 7 days of curing

Addition of 0.1% carbon fibers (CF) to the cement (smart cement) with w/c ratio of 0.38, 0.44 and 0.54 increased the compressive strength to 17.2 MPa, 13.7 MPa and 9.2 MPa respectively. Hence the addition of 0.1% CF to the cement increased the compressive strength by 9%, 5% and 4% for cement with w/c ratio of 0.38, 0.44 and 0.54 respectively.

The change in electrical resistivity of unmodified cement at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ with different w/c ratio of 0.38, 0.44 and 0.54 were 0.62%, 0.55% and 0.41% respectively. With 0.1% CF addition to the smart cement the electrical resistivity at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ for the smart cement with w/c of 0.38, 0.44 and 0.54 were 432%, 405% and 325% respectively (Fig. 1b). Additional of 0.1% CF increased the change in electrical resistivity of cement at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ with w/c ratio of 0.38, 0.44 and 0.54 by a factor of 697, 736 and 792 respectively compared to the unmodified cement.

The relationships between compressive stress and the change in electrical resistivity $\left(\frac{\Delta\rho}{\rho_o}\right)$ of the cement with different w/c ratio of 0.38, 0.44 and 0.54 for 7 days of curing were modeled using the Vipulanandan p-q piezoresistive model (Eqn. (5)). The piezoresistive model (Eqn. (5)) predicted the measured stress- change in resistivity relationship very well (Fig. 1b). The piezoresistive model parameters q_2 and p_2 are summarized in Table 2. The coefficients of determination (R^2) were 0.99. The root mean square of error (RMSE) was varied between 0.02 MPa and 0.04 MPa as summarized in Table 3.

(iii) 28 days of curing

Addition of 0.1% CF to the cement (smart cement) with w/c ratio of 0.38, 0.44 and 0.54 increased the compressive strength to 19.4 MPa, 16.8 MPa and 12.6 MPa respectively. Hence the addition of 0.1% CF to the cement increased the compressive strength by 12%, 11% and 12% for cement with w/c ratio of 0.38, 0.44 and 0.54 respectively.

The change in electrical resistivity of oil well cement at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ with different w/c ratio of 0.38, 0.44 and 0.54 were 0.55%, 0.41% and 0.33% respectively. With 0.1% CF addition to the cement (smart cement) the electrical resistivity at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ for the smart cement with w/c of 0.38, 0.44 and 0.54 were 401%, 389% and 289% respectively (Fig. 1c). Additional of 0.1% CF increased the change in electrical resistivity of cement at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ with different w/c ratios of 0.38, 0.44 and 0.54 after by 729, 948 and 875 respectively compared to the unmodified cement.

The relationships between compressive stress and the change in electrical resistivity $\left(\frac{\Delta\rho}{\rho_o}\right)$ of the cement with different w/c ratio of 0.38, 0.44 and 0.54 after 28 day of curing were modeled using the p-q Piezoresistive model (Eqn. (5)). The piezoresistive model (Eqn. (5)) predicted the measured stress- change in resistivity relationship very well (Fig. 2c). The piezoresistive model parameters q_2 and p_2 are summarized in Table 2. The coefficients of determination (R^2) were 0.99. The root mean square of error (RMSE) was varied between 0.02 MPa and 0.04 MPa as summarized in Table 2.

Compressive Strength – Resistivity Relationship

During the entire cement hydration process both the electrical resistivity and compressive strength of the cement increased gradually with the curing time. For cement pastes with various w/c ratios, the change in resistivity was varied during the hardening. The cement paste with a lower w/c ratio had a lowest electrical resistivity change (RI_{24hr}) than cement with higher w/c ratio as shown in Table 1.

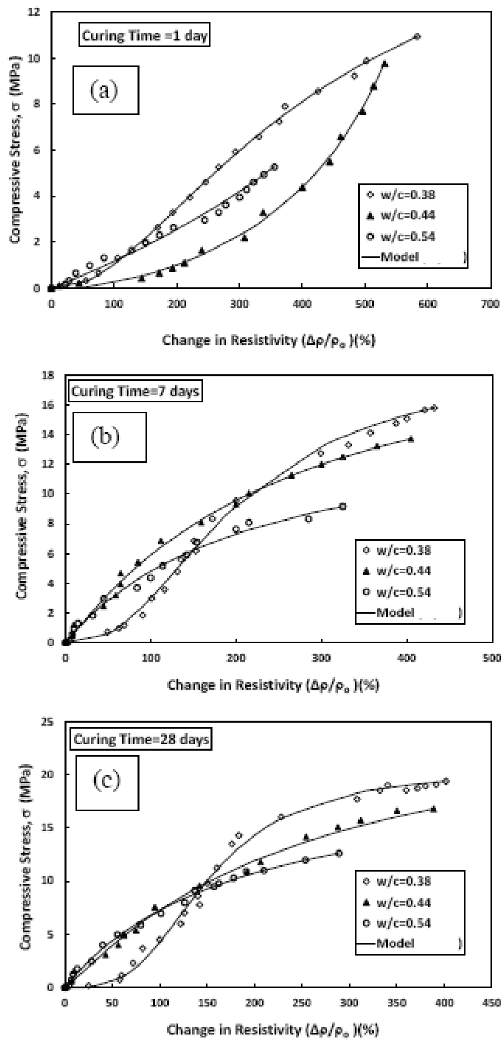

Fig. 1. Measured and predicted piezoresistive behaviour of smart cement with curing time (a) 1 day (b) 7 days and (c) 28 days

The relationship between (RI_{24hr}) and the one day, 7days and 28 days compressive strength (MPa) (Fig. 2) were:

$$\sigma_{1day} = 0.03 \times RI_{24hr} + 3.3 \quad R^2=0.81 \qquad (6)$$

$$\sigma_{7days} = 0.031 \times RI_{24hr} + 6.5 \quad R^2=0.89 \qquad (7)$$

$$\sigma_{28days} = 0.03 \times RI_{24hr} + 9.7 \quad R^2=0.94 \qquad (8)$$

Hence the compressive strength of the smart cement after various curing times was linearly related to the electrical resistivity index, RI_{24hr}.

ISBN: 978-624-6150-05-1

Since RI_{24hr} can be determined in one day, it can be used to predict the compressive strength of smart cement up to 28 days.

V. CONCLUSIONS

Based on the experimental study and analytical modeling of the curing and piezoresistivity behavior of smart cement with w/c ratio of 0.38, 0.44 and 0.54, following conclusions are advanced:

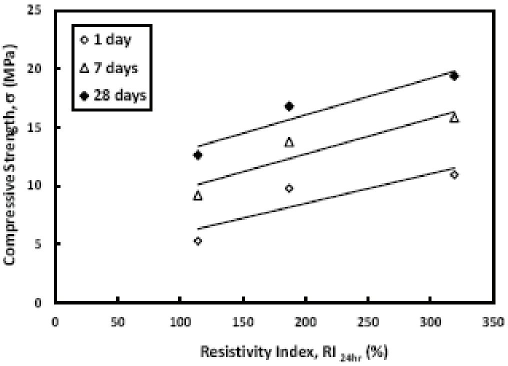

Fig. 2. Relationship between resistivity index ($RI_{24\ hr}$) and compressive strength of smart cement for water-to-cement ratio of 0.38 to 0.54

1. The changes in the electrical resistivity were higher than the changes in the unit weight of the cement. Hence the electrical resistivity can also be used for quality control.
2. The smart cement showed enhanced piezoresistive behavior compared to unmodified cement. With 0.1% carbon fiber (CF) modification the piezoresistivity strain at peak stress was over 300%. The piezoresistivity enhancement was depended on the water-to-cement ratio and curing time. The Vipulanandan p-q piezoresistive model predicted the compressive stress- changes in resistivity relationship very well.
3. Linear relationship was observed between resistivity index (RI_{24hr}) and compressive strength of smart cement for different curing times. Since RI_{24hr} can be determined in one day, it can be used to predict the compressive strength of smart cement up to 28 days.

VI. ACKNOWLEDGMENTS

This study was supported by the Center for Innovative Grouting Materials and Technology (CIGMAT) at the University of Houston, Houston, Texas with funding from various industries.

Table 2. Piezoresistive Model Parameters for the Smart Cement

Material	w/c	Curing Time (day)	$(\Delta\rho/\rho o)_f$ (%)	σ_f (MPa)	q_2	p_2	RMSE (MPa)	R^2
Smart cement	0.38	1	583	10.9	0.30	0.16	0.01	0.99
		7	432	17.2	0.14	0.09	0.03	0.99
		28	401	19.4	0.05	0.03	0.03	0.99
	0.44	1	531	9.8	1.59	0.85	0.02	0.99
		7	405	13.7	0.33	0.07	0.02	0.99
		28	389	16.8	0.41	0.06	0.02	0.99
	0.54	1	355	5.3	1.37	0.0	0.04	0.99
		7	325	9.2	0.41	0.0	0.03	0.99
		28	289	12.6	0.39	0.0	0.02	0.99

REFERENCES

[1] D. Izon and M. Mayes (2007). “Absence of fatalities in blowouts encouraging in MMS study of OCS incidents 1992-2006.” Well Control, 86-90.

[2] C. Vipulanandan and V. Garas (2008). “Electrical resistivity, pulse velocity and compressive properties of carbon fiber

reinforced cement mortar." Journal of Civil Engineering Materials, 20, 93-101.
[3] Y. Zuo, J. Zi and X. Wei (2014). "Hydration of cement with retarder characterized via electrical resistivity measurements and computer simulation." Construction and Building Materials, 53, 411–418.
[4] J. Zhang, E. A. Weissinger, S. Peethamparan, and G. W.Scherer, (2010). "Early hydration and setting of oil well cement." Cement and Concrete Research, 40, 1023-1033.
[5] C. Vipulanandan (2021). Smart Cement: Development, Testing, Modeling and Real-Time Monitoring, Taylor and Francis CRC Book, 440 pp.
[6] D. D. L. Chung (2001). "Functional properties of cement-matrix composites." Material Science, 36, 1315-1324.
[7] C. Vipulanandan and K. Ali (2018) "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.
[8] C. Vipulanandan and N. Amani (2018) "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.
[9] C. Vipulanandan and P. Prashanth, (2013). "Impedance spectroscopy characterization of a piezoresistive structural polymer composite bulk sensor." Journal of Testing and Evaluation, 41, 898-904.

 ISBN: 978-624-6150-05-1