Characterizing the Compressive Piezoresistive Behavior of Smart Cement with Varying Water-to Cement Ratios Using Vipulanandan Model

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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 fibers (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. 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. The compressive strength of the smart cement with 0.1% CF and w/c ratio of 0.38, 0.44 and 0.54 were increased by over 10 % after 28 days of curing compared to the unmodified cement. Linear correlations were observed between resistivity index (RI_{24hr}) and compressive strength of smart cement for different curing times.

Introduction

Cement is used in multiple applications to build the infrastructures such as 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. Hence there is need to develop highly sensing cement so that it' perfomce and changing properties over time can be monitored.

Recent studies have suggested that replacing the DC measurement with the AC measurement can eliminate the polarization effect (Zhang et al. 2010, Vipulanandan et al. 2013-2021).

Past studied have investigated the changes in electrical resistivity with applied stress referred to as piezoresistive behavior of modified cement-based and polymer composites (Vipulanandan et al. 2008). 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 (Zuo et al. 2014; Vipulanandan et al. 2002-2021).

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

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 less than 0.1% of carbon fibers (CF) by the weight of cement was mixed with all the samples. 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 A/L 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:

(i) Conductivity Probe

Commercially available conductivity probe was used to measure the conductivity (inverse of resistivity) of the slurries. In the case of cement, this meter was used during the initial curing of the cement. The conductivity measuring range was from 0.1μS/cm to 1000 mS/cm, representing a resistivity of 0.1Ω.m to 10,000 Ω.m.

(ii) Digital Resistivity Meter

Digital resistivity meter (used in the oil industry) was used measure the resistivity of fluids, slurries and semi-solids directly. The resistivity range for this device was 0.01 Ω-m to 400 Ω-m.

The conductivity probe and the digital electrical resistivity device were calibrated using standard solution of sodium chloride (NaCl).

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 (Vipulanandan et al. 2013; Vipulanandan et al. 2014a). 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 factor (Eqn.1) for a time period of initial five hours of curing. This K factor was used to determine the resistivity of the cement with the curing time.

Compressive Strength Test

The cylindrical specimen (50mm dia.*100 mm height) was capped and tested at a predetermined controlled displacement rate. Compression tests were performed on cement samples after 1, 7 and 28 days of curing using a hydraulic compression machine. At least three specimens were tested under each testing condition and average results are reported.

Piezoresistivity Test

Piezoresistivity 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 (Vipulanandan et al. 2013).

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) in curve fitting as defined in Eqns. (3) and (4) were quantified.

$$RMSE = \sqrt{\frac{\sum_{i=1}^{n}(y_i - x_i)^2}{N}} \quad (3)$$

$$R^2 = \left(\frac{\sum_i (x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum_i (x_i - \bar{x})^2} * \sqrt{\sum_i (y_i - \bar{y})^2}}\right)^2 \qquad (4)$$

where y_i = actual value; x_i =calculated value from the model; $\bar{y}$ =mean of actual values; $\bar{x}$= mean of calculated values and N is the number of data points.

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 resistivities at the end of 24 hours (RI_{24hr}) and 7 days $\left(RI_{7days}\right)$ were defined in Eqn. (7) and Eqn. (8) as follows:

$$RI_{24hr} = \frac{\rho_{24hr-}\rho_{min}}{\rho_{min}} * 100 \qquad (7)$$

$$RI_{7\ days} = \frac{\rho_{7days-}\rho_{min}}{\rho_{min}} * 100 \qquad (8)$$

(a)w/c =0.38

Unit weight of the smart cement with w/c of 0.38 was 16.48 ppg. 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 2. 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 2. 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 16.12 ppg. 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 2. 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 15.78 ppg. 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 2) and the electrical resistivity reduced to reach the ρ_{min} of 0.78 Ω-m after 128 minutes (t_{min}) as summarized in Table 2. 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 2. 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%.

Summary: The initial electrical resistivity (ρ_o) of the smart cement decreased by 3% and 13% when the w/c ratio increased from 0.38 to 0.44 and 0.54 respectively as summarized in Table

1. The minimum electrical resistivity (ρ_{min}) of the smart cement also decreased by 10% and 21% when the w/c ratio was increased from 0.38 to 0.44 and 0.54 respectively as summarized in Table 1. The time to reach the minimum electrical resistivity (t_{min}) increased by 15% and 21% when the w/c ratio increased from 0.38 to 0.44 and 0.54 respectively as summarized in Table 1.

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

Piezoresistivity and strength of smart cement

Additional of about 0.1% CF substantially improved piezoresistive behavior of the cement. 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:

$$\frac{\sigma}{\sigma_f} = \left[\frac{\frac{x}{x_f}}{q_2+(1-p_2-q_2)\frac{x}{x_f}+ p_2\left(\frac{x}{x_f}\right)^{\left(\frac{p_2}{p_2-q_2}\right)}}\right] \tag{11}$$

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, a 14% and 53% reduction when the w/c ratio increased from 0.38 to 0.44 and 0.54 respectively as summarized in Table 3. 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% CF increased the strength by 3%, 17% and 15% for cement with w/c ratio of 0.38, 0.44 and 0.54 respectively as summarized in Table 2.

The change in electrical resistivity at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ for the unmodified oil well cement with different w/c ratios of 0.38, 0.44 and 0.54 were 0.70%, 0.60% and 0.48% respectively as summarized in Table 3. 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 as summarized in Table 2.

Using the p-q Piezoresistive model (Eqn. 11)), 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. (11)) predicted the measured stress- change in resistivity relationship very well (Fig. 4a and Fig. 5a). 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

The compressive strength (σ_f) of the cement with w/c ratio of 0.38, 0.44 and 0.54 after 7 days of curing increased by 61%, 56% and 115% respectively compared with the compressive strength (σ_f) of the cement after one day of curing as summarized in Table 3. 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 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 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 0.62%, 0.55% and 0.41% respectively as shown in Fig. 4b. 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 5b). Additional of 0.1% CF increased the change in electrical resistivity of oil well 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 as summarized in Table 2.

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 p-q piezoresistive model (Eqn. (11)). The piezoresistive model (Eqn. (11)) predicted the measured stress- change in resistivity relationship very well (Fig. 4b and Fig. 5b). The piezoresistive model parameters q_2 and p_2 are summarized in Table 3. 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.

(iii) 28 days of curing

The compressive strength (σ_f) of the cement with w/c ratio of 0.38, 0.44 and 0.54 for 28 day of curing increased by 12%, 16% and 14% respectively compared with the 7 day compressive strengths. 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 as shown in Fig. 4c and summarized in Table 3. 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 5c). Additional of 0.1% CF increased the change in electrical resistivity of oil well 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 as summarized in Table 2.

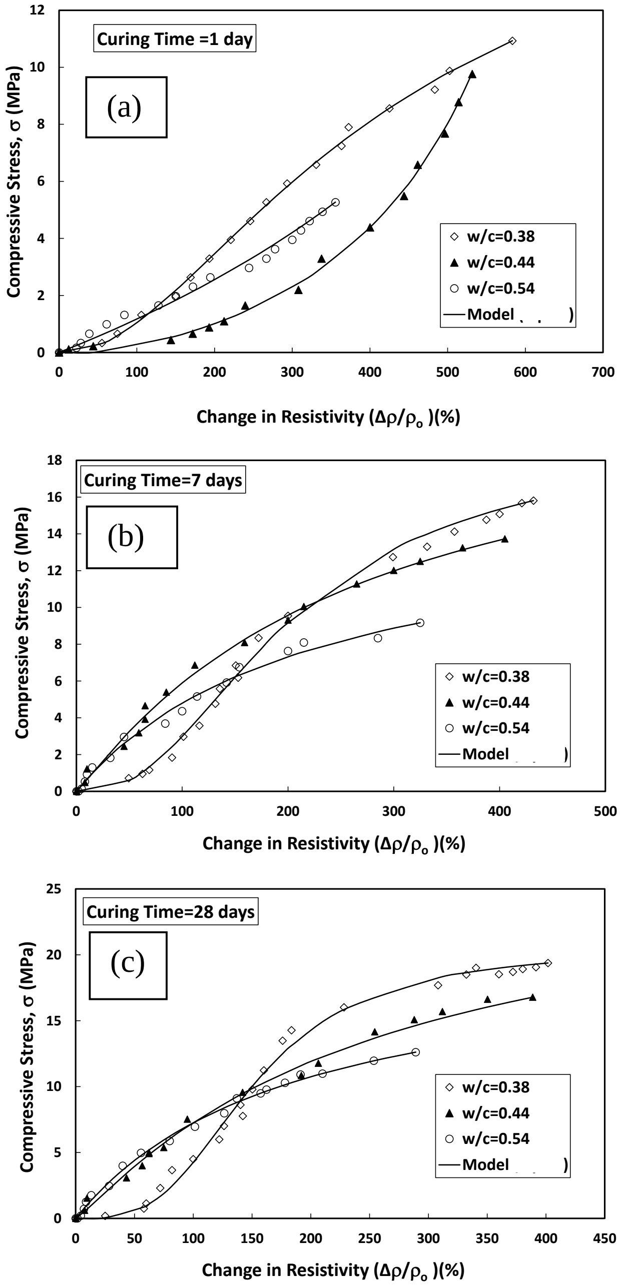

Figure 1: Measured and predicted piezoresistive behavior of smart cement with curing time (a) one day (b) 7 days and (c) 28 days

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. (11)). The piezoresistive model (Eqn. (11))

predicted the measured stress- change in resistivity relationship very well (Figure 1(c)). 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.

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

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 summarized in Table 1.

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

$$\sigma_{1day} = 0.03 \times RI_{24hr} + 3.3 \qquad R^2=0.81 \qquad (12)$$

$$\sigma_{7days} = 0.031 \times RI_{24hr} + 6.5 \qquad R^2=0.89 \qquad (13)$$

$$\sigma_{28days} = 0.03 \times RI_{24hr} + 9.7 \qquad R^2=0.94 \qquad (14)$$

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

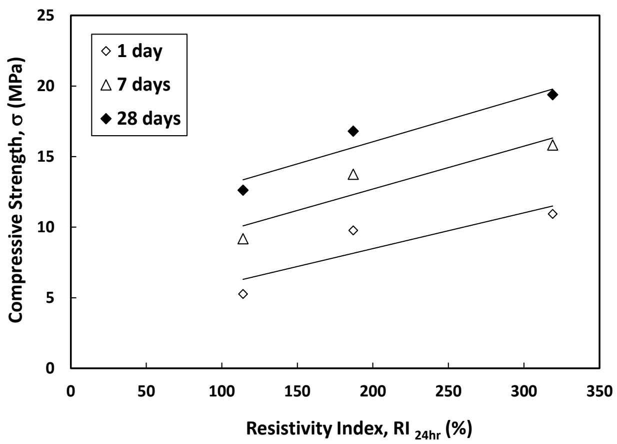

Figure 2: Relationship between resistivity index ($RI_{24\,hr}$) and compressive strength of smart cement for water-to-cement ratio of 0.38 to 0.54

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:

1. 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% reduce with increasing the water-to-cement ratio from 0.38 to 0.44 and 0.54 respectively. 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% conductive filler (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. Additional of 0.1% CF also increased the 28 day compressive strength by over 10%.

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.

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