Piezoresistive behavior of Smart Cement Grouts with Sodium Silicate Additive Predicted Using Vipulanandan Curing and Piezoresistive Stress-Strain Models

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Abstract

In this study highly sensing smart cement grouts with and without sodium silicate additive were developed and tested and also the curing and piezoresistive behavior were modelled. Sensing cement grout was prepared using water-to-cement ratio of 0.8. Commercially available table top mixer was used at a speed of 1000 rpm for five minutes and 0.075% carbon fibers (based on the cement weight) were added during the mixing process. Curing process of the cementitious materials was monitored with changes in electrical properties which can also be easily monitored in the field. Electrical resistivity (sensing property) of the grouts were measured using a digital resistivity meter. Also the electrical resistance changes with the curing time were measured using the LCR (inductance (L), capacitance (C) resistance (R)) meter with the two-probe method. The test specimens were capped and then tested at a controlled displacement rate to fail the specimen in about 30 minutes. Tests were performed up to twenty-eight days of curing. The one day compressive strength of the smart cement grout was 3 MPa and it increased to over 16 MPa after 28 days of curing. Addition of up to 3% sodium silicate reduced the compressive strength of the grout. Change in sensing parameter, the resistivity of the smart cement grout material under stress was investigated. During the compression test, electrical resistance was measured along the stress axis. Also the change in sensing property was related to the applied stress to develop the piezoresistive relationship. The piezoresistive strain after one day curing was 155% and it increased to 179% after 28 days of curing. The failure strain of cement grout is about 0.2%, so the smart cement grout was over 775 times (77500%) more sensitive than the regular grout. Vipulanandan curing model, correlation model and piezoresistive p-q model were used to characterize the smart grout behaviors.

Introduction

Cement grouts are used in number of applications in infrastructure construction, maintenance and repairing of bridges, highways, airport runways, buildings, tunnels, pipelines, piles and wells (oil, gas and water) (Ahossin Guezo et al. 2014; Vipulanandan 2021 and 2024). In year 2013 there was a building failure in Bangladesh with over thousand deaths. In year 2018 there was bridge failure in Italy with 37 deaths and many injuries. Also cement grouts are used in soil stabilization, repairing rocks, stabilizing slopes and tunnels and solidifying contaminated soils (Bowen, 1981; Vipulanandan et al. 1992; Anagnostopoulos 2014; Vipulanandan 2021 and 2024; Vipulanandan et al. 2022). With the wide spectrum of applications, there is a need to make the cement grouts sensing so that it can be monitored from the time of mixing to the entire service life and also make the repaired sections highly sensing for real-time monitoring.

In recent years highly sensing smart cements have been developed (Vipulanandan et al. 2015, 2018, 2021, 2022). The smart cement materials are bulk sensors where the entire material becomes a 3 dimensional (3D) sensing tool.

Objectives

The overall objective of this study was to investigate the potential of making the cements grouts highly sensing. The specific objectives of this study are follows:

(a) To develop and characterize the highly sensing smart cement grouts with and without sodium silicate (SS) additive.

(b) Model the curing and piezoresistive behavior of the smart cement grouts using Vipulanandan Models.

Materials and Methods

(a). Materials

Smart cement grout: Cement was mixed with 0.075% carbon fibers with diameter in the micrometer range to make it a chemo-thermo-piezoresistive material (U.S. Patent 2019).

(b). Methods

Cement Grout Mixing

Sensing cement grout was prepared using water-to-cement ratio of 0.8 with and without sodium silicate (SS) addition in this study. Commercially available table top mixer was used at a speed of 1000 rpm for five minutes and 0.075% carbon fillers (based on the cement weight) were added during the mixing process.

Cement Specimen

The test specimens were prepared in plastic cylindrical molds. All specimens were capped to minimize moisture loss and were cured under room condition (23^{o}C and relative humidity of 50%) up to the day of testing. At least three specimens were tested under each condition and average results are discussed.

Electrical Resistivity

Curing process of the cementitious materials can be monitored with changes in electrical properties (Vipulanandan et al. 2015, 2018). Electrical resistivity (sensing property) of the grouts was measured using a digital resistivity meter. Also the electrical resistance changes with the curing time were measured using the LCR (inductance (L), capacitance (C) resistance (R)) meter using the two-probe method (Vipulanandan and Prashanth, 2013). Each specimen was first calibrated to obtain the effective parameter K (K_e) using the resistivity (ρ) and the measured electrical resistance (R) based on the Eqn. (1) as follows:

$$\rho = RA/L = R/(L/A) = R/K \quad (1)$$

To determine the nominal parameter K (K_n), especially for conductors, L is the spacing between the conductors used to measure the electrical resistance, A is the area through current will flow. Compared to conductive metals, cement slurry used in this study is an insulator and hence effective parameter K_e was determined by measuring the resistivity and resistance using Eqn. (1). Based on the monitoring it was determined that after 180 min. of curing of the cement, the parameter K_e became stable and was 54 m^{-1} and was used to determine resistivity of the hardened cement specimen. The changes in resistivity (Δρ) can be related to the change in resistance (ΔR) under applied stress and represented in Eqn. (2) as follows:

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

Compression Test

The test specimens were capped and then tested at a controlled displacement rate to fail the specimen in about 30 minutes (CIGMAT GR02-02). Tests were performed up to twenty 28 days.

Changes in resistivity with applied stress

Change in sensing parameter of the grout material under stress was investigated. The changes in resistivity with applied stress of the sensing cement grouts for repairing the 28 days old damaged sensing cement specimens were investigated under compressive loading. During the compression test (CIGMAT GR 2-02, 2002), electrical resistance was measured along the stress axis. Also the change in sensing property was related to the applied stress to develop the piezoresistive relationship (Vipulanandan 2021).

Results and Discussions

The unit weight of the smart cement grout with 0.8 w/c ratio was 15.79 kN/m^3. Also the Vipulanandan Impedance characterization model identified the electrical resistivity as material property to monitor (Vipulanandan et al. 2013).

Curing

The change of electrical resistivity with curing time for the sensing cement grout up to 28 days of curing was monitored. The initial resistivity (ρ_o), minimum resistivity (ρ_{min}), time to reach the minimum resistivity (t_{min}) are summarized in Table 1. Also the percentage of maximum change in resistivity at the end of 24 hours (RI_{24hr}), 7 days (RI_{7days}), and 28 days ($RI_{28\ days}$) are defined in Eqn. (3), Eqn. (4) and Eqn. (5) as follows:

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

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

$$RI_{28\ days} = \frac{\rho_{28days} - \rho_{min}}{\rho_{min}} * 100 \quad (5)$$

Table 1. Summary of bulk resistivity parameters for the smart cement grout with and without SS cured at room temperature up to 28 days

Mix Type (w/c = 0.8)	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)	$\rho_{28\ days}$ (Ω.m)	$RI_{24\ hr}$ (%)	$RI_{7\ days}$ (%)	$RI_{28\ days}$ (%)
Grout (SS = 0%)	15.79	1.08	1.04	180	2.16	6.16	9.37	108	492	801
Grout (SS = 1%)	15.89	0.69	0.54	300	1.01	2.20	4.85	87	307	798
Grout (SS = 3%)	15.93	0.52	0.41	300	0.56	1.28	3.29	37	212	702

The initial sensing resistivity property (ρ_o) of the smart cement grout with w/c ratio of 0.8 was 1.08 Ohm.m and the sensing property reduced to reach the ρ_{min} of 1.04 Ohm.m after 180 minutes (t_{min}) as summarized in Table 1. With 1% and 3% SS, the initial resistivity and the minimum resistivity decreased and the time to reach the minimum (t_{min}) increased to 300 minutes. With 1% SS, the initial resistivity (ρ_o) decreased by 36% and ρ_{min} decreased by 44% and with 3% SS, the ρ_o decreased by 51% and ρ_{min} decreased by 60%. The 24 hours resistivity (ρ_{24hr}) of the smart cement grout only was 2.16 Ω.m. Hence the maximum change in resistivity after 24 hours (RI_{24hr}) was 108% as summarized in Table 1. The seven and twenty eight days resistivity (ρ_{7days} and ρ_{28days}) of the smart cement grout was 6.16 Ω.m and 9.37 Ω.m, hence the maximum change in resistivity after seven days and twenty eight days (RI_{7days} and $RI_{28\ days}$) were 492% and 801% respectively. The addition of SS decreased the resistivity compared to that of the smart cement grout only. Addition of 1% SS decreased the 24 hours, seven days and twenty eight days resistivity of the smart cement grout by about 52%, 60% and 48% respectively and hence the resistivity indices RI_{24hr}, RI_{7days} and $RI_{28\ days}$ also decreased compared to that of smart cement grout only. Addition of 3% SS decreased the 24 hours, seven days and twenty eight days resistivity of the smart cement grout by about 74%, 79% and 65% respectively and hence the resistivity indices RI_{24hr}, RI_{7days} and $RI_{28\ days}$ also decreased.

Vipulanandan Curing Model

Since the changes in the electrical resistivity was monitored during the curing of the cement grout the Vipulanandan Curing Model (Vipulanandan 2021) is as follows:

$$\frac{1}{\rho} = \left(\frac{1}{\rho_{min}}\right)\left[\frac{\left(\frac{t+t_o}{t_{min}+t_o}\right)}{q_1+(1-p_1-q_1)*\left(\frac{t+t_o}{t_{min}+t_o}\right)+p_1*\left(\frac{t+t_o}{t_{min}+t_o}\right)^{\frac{q_1+p_1}{p1}}}\right] \tag{6}$$

where ρ is the electrical resistivity (Ω-m); ρ_{min} is the minimum electrical resistivity (Ω-m); t_{min} is the time corresponding to minimum electrical resistivity (ρ_{min}); Parameters p_1, t_o and q_1 are model parameters and t is the curing time (min). The parameter q_1 represents the initial rate of change in the resistivity and parameter p_1 influences the prediction of the changes in resistivity with time. Also the parameter ratio q_1/p_1 influences the long-term prediction of the resistivity and also the type of additives used in the cement. The model will also predict the initial resistivity (ρ_0) when the time t = 0. The curing model parameter p_1 for the smart cement grout was 0.710 after twenty eight days summarized in Table 2. The curing model parameter q_1 for smart cement grout was 0.295 after twenty eight days as summarized in Table 2. The Vipulanandan curing model predicted the measured curing resistivity very well as shown in Figure 1. The root mean square of error (RMSE) varied from 0.09 Ω.m to 0.45 Ω.m for twenty eight days of curing.

Table 2. Vipulanandan Curing Model parameters for the the smart cement grout with and without SS cured at room temperature up to 28 days

Mix Type (w/c = 0.8)	Curing Time	ρ_{min} (Ω.m)	t_{min} (min)	p_1	q_1	t_o (min)	RMSE (Ω.m)	R^2
Grout (SS = 0%)	28 days	1.04	180	0.710	0.295	110	0.45	0.97
Grout (SS = 1%)		0.54	300	0.236	0.161	70	0.09	0.99
Grout (SS = 3%)		0.41	300	0.113	0.092	51	0.03	0.99

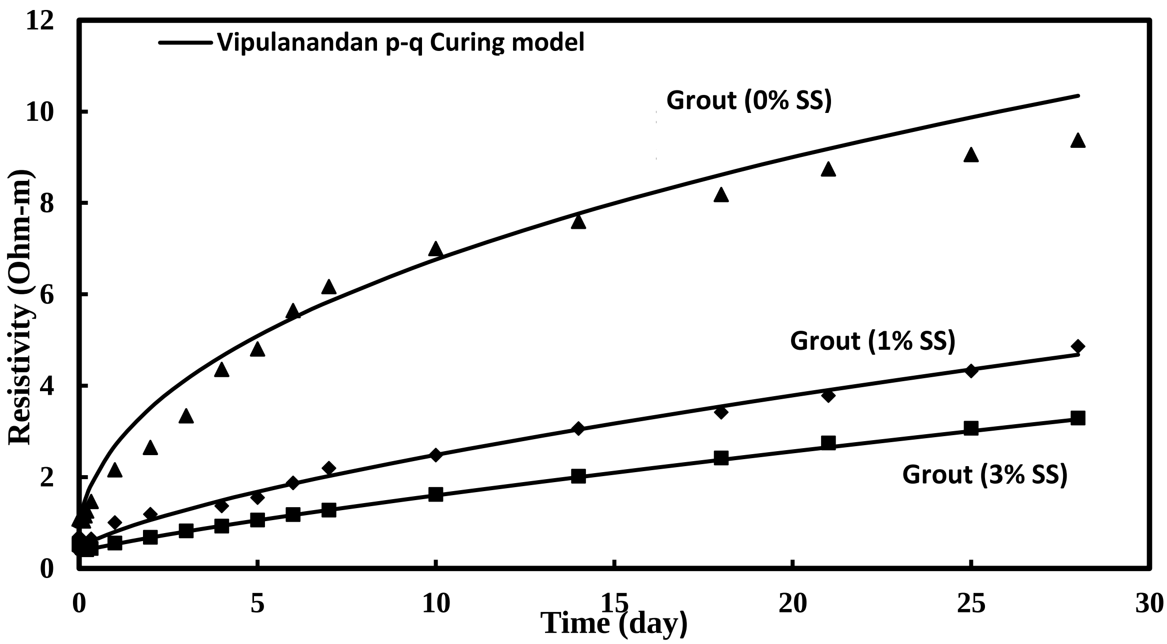

Figure 1 Variation of the curing resistivity with time for smart cement grouts up to twenty eight days of curing and modeled using the Vipulanandan p-q curing model.

Compressive Behavior

(a) Piezoresistivity

The piezoresistive axial strain at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ for the sensing cement grout only after one, seven and twenty eight days of curing were 155%, 156% and 179% which decreased to 117%, 116% and 125% respectively for sensing cement grout with 1% SS. Thus the changes in resistivity with applied stress of the grouts were reduced by 24%, 25% and 30% after one, seven and twenty eight days of curing respectively with addition of 1% SS. By the addition of 3% SS, the changes in resistivity with applied stress after 1day, 7 days and 28 days of curing were 106%, 94% and 103%, which reduced by 31%, 40% and 42% respectively.

Vipulanandan p-q Piezoresistive Model

Based on experimental results, The Vipulanandan p-q piezoresistive model was used to predict the behavior and the equation is as follows:

$$\frac{\sigma_c}{\sigma_{cf}} = \left[\frac{\frac{x}{x_f}}{q_2+(1-p_2-q_2)\frac{x}{x_f}+p_2\left(\frac{x}{x_f}\right)^{\left(\frac{p_2+q_2}{p_2}\right)}}\right] \tag{7}$$

where the stress is σ_c (MPa); stress at failure is σ_{cf} (MPa): $x = \left(\frac{\Delta\rho}{\rho}\right) * 100$ = Percentage of change in piezoresistive axial strain due to the stress; $x_f = \left(\frac{\Delta\rho}{\rho}\right)_f * 100$ = Percentage of piezoresistive axial strain at failure; $\Delta\rho$ is the change in the resistivity; ρ is the initial resistivity (at σ=0 MPa) and p_2 and q_2 are model parameters.

Using the p-q piezoresistive model (Eqn. (7)), the relationships between compressive stress and the piezoresistive axial strain $\left(\frac{\Delta\rho}{\rho}\right)$ of the smart cement grout with and without SS for one, seven and twenty eight days of curing were modeled. The piezoresistive model (Eqn. (7)) predicted the measured stress-change in resistivity relationship very well in Figure 2 for the smart cement grout, Figure 3 for the smart cement grout with 1% SS and Figure 4 for the smart cement with 3% SS. The model parameters q_2 and p_2 are summarized in Table 3. The R^2 were 0.95 to 0.99. The root mean square of error (RMSE) varied between 0.04 MPa and 0.64 MPa as summarized in Table 3.

Table 3. Strength, piezoresistive axial strain at failure, model parameters for the smart cement grouts cured after 1 day, 7 days and 28 days.

Mix Type (w/c = 0.8)	Curing Time (day)	Strength σ_{cf} (MPa)	Piezoresistivity at peak stress, $(\Delta\rho/\rho)_f$ (%)	p_2	q_2	R^2	RMSE (MPa)
Smart Grout SS =0%	1 day	2.96	155	0.031	0.607	0.99	0.08
Smart Grout SS = 1%		2.23	117	0.037	0.706	0.95	0.17
Smart Grout SS = 3%		1.82	106	0.183	1.193	0.99	0.04
Smart Grout SS = 0%	7 days	9.94	156	0.035	0.642	0.99	0.18
Smart Grout SS = 1%		6.98	116	0.052	0.596	0.99	0.15
Smart Grout SS = 3%		5.45	94	0.07	1.582	0.99	0.16
Smart Grout SS =0%	28 days	16.47	179	0.012	0.613	0.99	0.10
Smart Grout SS = 1%		13.42	125	0.01	0.561	0.97	0.64
Smart Grout SS = 3%		11.75	103	0.03	0.492	0.99	0.20

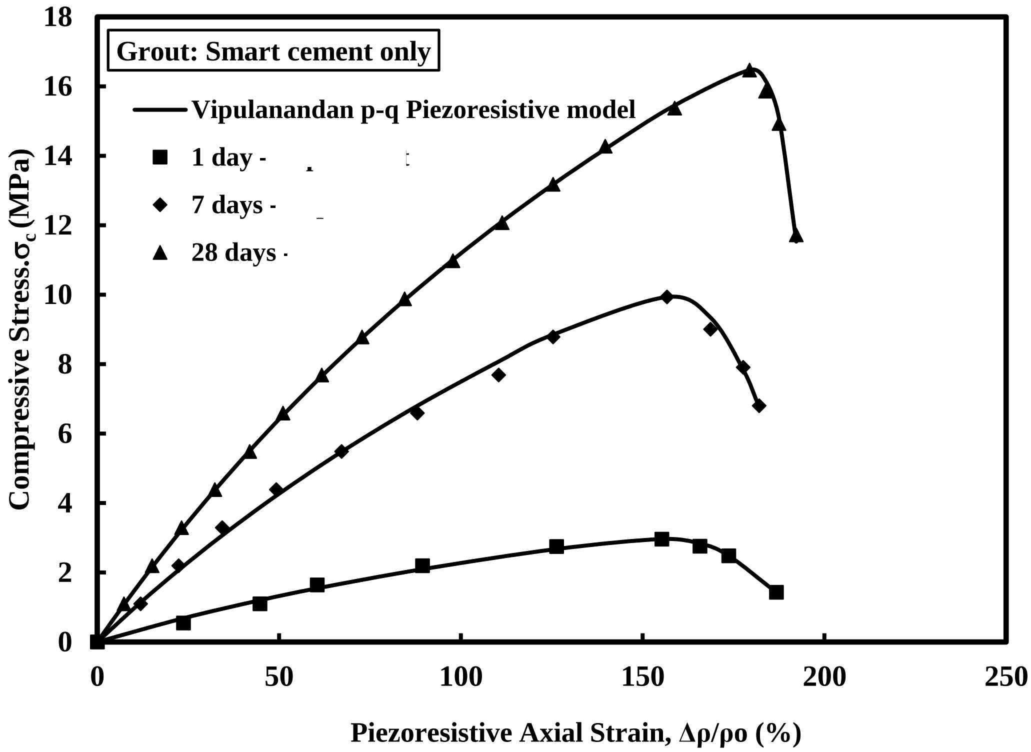

Figure 2. Piezoresistive response of the smart cement grout after one day, seven days and twenty eight days of curing and modeled using Vipulanandan p-q piezoresistivity model.

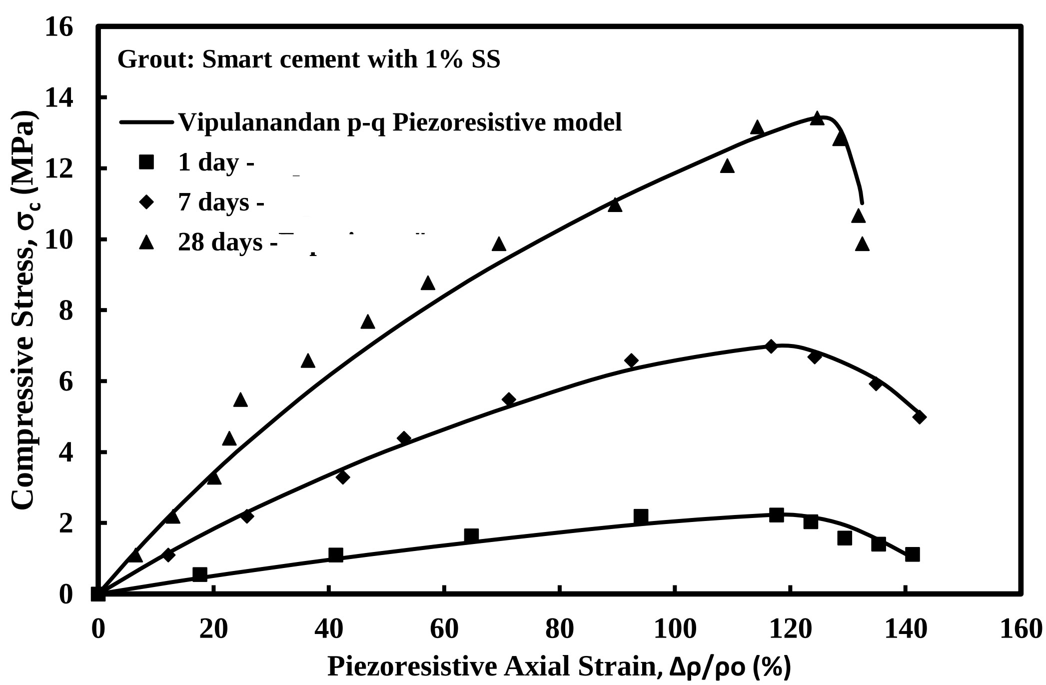

Figure 3. Piezoresistive response of the smart cement with 1% sodium silicate after one day, seven and twenty eight days of curing and modeled with p-q piezoresistive model.

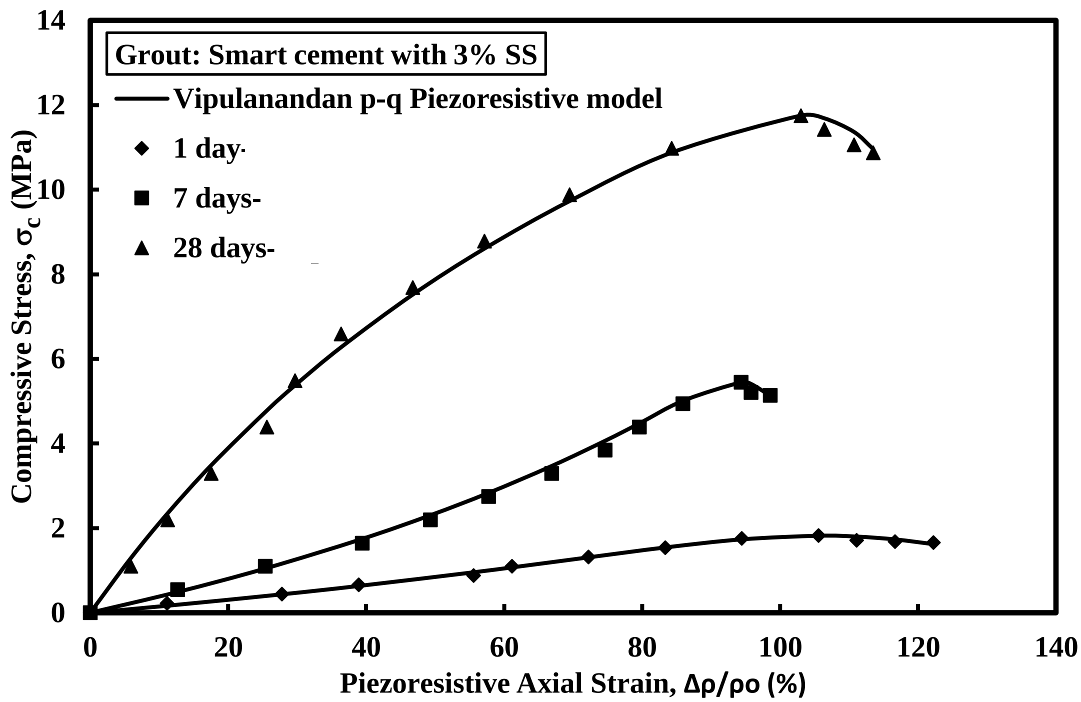

Figure 4. Piezo-sensing response of the smart cement with 3% sodium silicate after one day, seven days and twenty eight days of curing modeled with Vipulanandan p-q piezoresistive model.

(b) Strength

The σ_{cf} of the smart cement grout after one, seven and twenty eight days of curing were 2.96 MPa, 9.94 MPa and 16.47 MPa. With 1% SS the strength decreased to 2.23 MPa, 6.98 MPa and 13.42 MPa respectively for the smart cement grout as summarized in Table 10.3. Thus the compressive strength of the grouts were reduced by 24%, 29% and 19% after one, seven and twenty eight days of curing with addition of 1% SS. With the addition of 3% SS, the compressive strengths after 1day, 7 days and 28 days of curing were 1.82 MPa, 5.45 MPa and 11.75 MPa, reduction of 38%, 45% and 28% respectively.

Strength with curing time

The strength of smart cement grouts increased with time. The relationship between the compressive strength and time was modeled using the Vipulanandan Correlation Model (Vipulanandan et al. 1993, 2017) as follows:

$$\sigma_c = t/(E + Ft) \tag{8}$$

Where,

σ_c = Compressive strength of the grout (MPa)

t = time of curing (day)

In the relationship E and F are the material parameters. The parameter E represents the initial rate of change in the strength and parameter F determines the ultimate strength. For the cement grouts, experimental results matched very well (Fig. 5) with the proposed model with R^2 of 0.95 to 0.99. For sensing cement grout only, parameters E and F were 0.347 MPa^{-1} and 0.048 MPa^{-1}. For sensing cement grout with 1% SS, parameters E and F were 0.586 MPa^{-1} and 0.054 MPa^{-1}. For sensing cement grout with 3% SS, parameters E and F were 0.841 MPa^{-1} and 0.055 MPa^{-1}. The parameter E and F were linearly related to SS concentration as follows:

$$E = 0.16 *(\%SS) + 0.34, \qquad R^2 = 0.97 \tag{9}$$

$$F = 0.002*(\%SS) + 0.05, \qquad R^2 = 0.95 \tag{10}$$

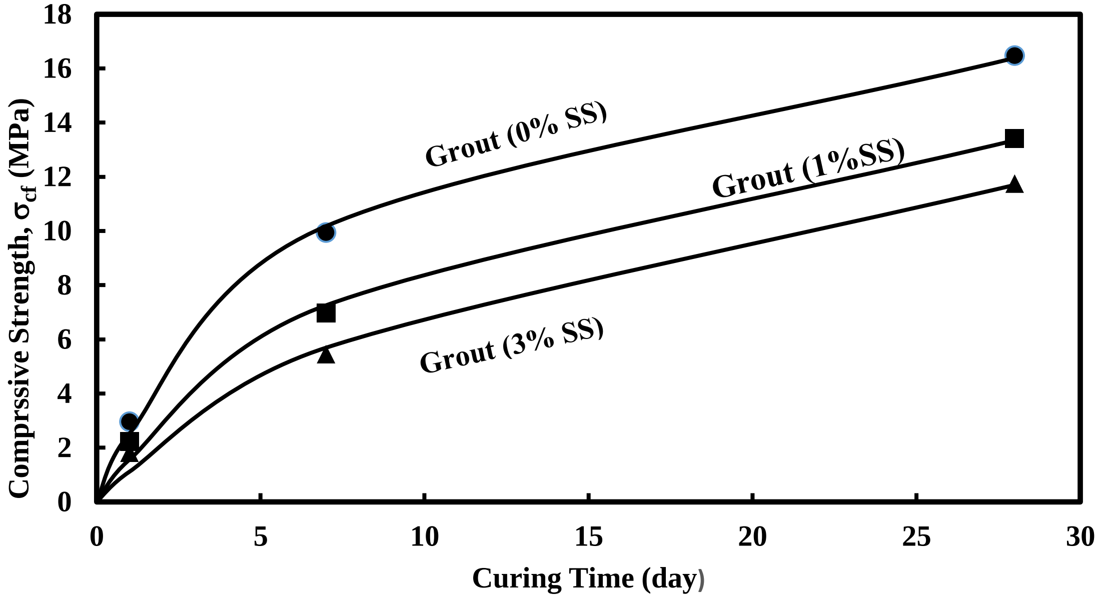

Figure 5 Relationship between compressive strength and the curing time for the grout modeled using the Vipulanandan correlation model.

Conclusions

Based on the testing and modeling of the sensing smart cement grout cured under room condition (23°C, relative humidity 50%), the following conclusions are advanced:

1. The initial sensing resistivity property (ρ_o) of the smart cement grout was 1.08 Ohm-m and it reduced with the addition of sodium silicate. The minimum sensing property (ρ_{min}) of the smart cement grout decreased to 1.04 Ohm-m and it reduced with the addition of sodium silicate.. The maximum change in the sensing electrical resistivity after one day, RI_{24hrs} was 108%. Hence the electrical sensing property resistivity can be used not only for quality control but also monitoring the smart cement grout curing.
2. The curing of the grouts were modeled using the Vipulanandan curing model and the model predicted the experimental results very well based on the coefficient of determination and the root mean square error (RMSE).
3. The smart cement grouts with water-to-cement ratio of 0.8 showed increase in resistivity with the applied compressive stress verifying the piezoresistive cement concept. The Vipulanandan p-q piezoresistive model predicated the compressive stress–change in sensing property relationship of the smart cement grout very well. Also the changes in the secant piezoresistive modulus and strengths with the curing time were modelled using the Vipulanandan Correlation Model.

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