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Characterizing Smart Cement with Sodium Metasilicate for Real Time Monitoring of Ultra-Deepwater Oil Well Cementing Applications

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Abstract

During cementing operation, it is critical to determine the flowing of cement slurry between the casing and formation, depth of the circulation losses and fluid loss, setting of cement in place and performance of the cement after hardening. Recent case studies on cementing failures have clearly identified some of these issues that resulted in various types of delays in the cementing operations. At present there is no technology available to monitor cementing operations in real time from the time of placement through the borehole service life. Also, there is no reliable method to determine the length of the competent cement supporting the casing.

In this study oil well cement with better sensing properties, henceforth called smart cement, was used to verify that its behavior can be monitored at various stages of construction and during its service life. A series of experiments evaluated the smart well cement behavior with and without up to 0.3% sodium metasilicate (SMS) to determine the sensitivity of the electrical resistivity of cement from curing to hardened state was investigated. The test results showed that SMS reduced the electrical resistivity of the water and cement slurries based on the amount. The SMS up to 0.3% also affected the rheological properties, setting characteristics, and the piezoresistive properties of the smart cement. In a 24-hour period the maximum change in the electrical resistivity (RI_{24}) for the cement without SMS (0.4 water-to-cement ratio) was 175%. The RI_{24} for the cement with SMS varied with the amount of SMS. Addition of 0.2% SMS had a minimal effect on the compressive strength. The smart cement was piezoresistive with the addition of SMS but the sensitivity was decreased.

Introduction

As deepwater exploration and production of oil and gas expands around the world, there are unique challenges in well construction beginning at the seafloor. Recent case studies on cementing failures have clearly identified several issues that resulted in various types of delays in the cementing operations. Also preventing the loss of fluids to the formations and proper well cementing have become critical issues in well construction to ensure wellbore integrity because of varying down hole conditions (Labibzadeh et al., 2010; Eoff et al., 2009; Ravi et al,. 2007; Gill et al., 2005; Fuller et al., 2002). Moreover, the environmental friendliness of the cements is a critical issue that is becoming increasingly important (Dom et al., 2007; Thaemlitz et al., 1999; Durand et al., 1995). Lack of cement returns may compromise the casing support, and excess cement returns can cause problems with flow and control lines (Ravi et al. 2007; Gill et al. 2005; Fuller et al. 2002). Hence there is a need for monitoring the cementing operation in real time. At present there is no technology available to monitor the cementing operation real time from the time of placement through the entire service life of the borehole. Also, there is no reliable method to determine the length of the competent cement supporting the casing.

In characterize the behavior of cementitious materials several methods such as X-ray diffraction, calorimetric analysis, scanning electron microscopy, and ultrasonic methods have been in used. Electrical resistivity measurement has been applied by many researches on concrete and other cementing applications (Taylor and Arulanandan, 1974; McCarter et al., 1998and 2006; Vipulanandan et al. 2006), but there are no reports in the literature of electrical resistivity measurements for characterizing oilwell cement. Electrical response characteristics measurement has appropriate sensitivity in monitoring the characteristics of cementitious materials (McCarter et al., 1998). The advantages in using this technique include its accuracy, ease of testing and procedures, and nondestructive characteristics (Vipulanandan et al., 2004-2013). Additionally, this method can be used for monitoring the long term behavior of cement in practice.

Electrical resistivity of cement is affected by a number of factors, such as pore structure (continuity and tortuosity), pore solution composition, cementitious content, water-cement (w/c) ratio, moisture content, and temperature (Polder et al., 2001). Moreover, electrical resistivity of cement is dramatically affected by admixtures, due to the resistivity contrast between cement and the admixture substances. Vipulanandan et al. (2004, 2006-2013) have studied the change in electrical resistivity with applied stress, referred to as piezoresistive behavior of modified cementitious and polymer composites. 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.

Sodium metasilicate (SMS)

From the initial use in the late 1800's sodium silicate based compounds have been used in a number of applications including cementing, grouting, emulsifying, and in cleaning agents. Of the various forms of sodium silicate based compounds, sodium meta silicates (anhydrous) have been used in oil and gas industry related applications. Because of its emulsification and interfacial tension reduction characteristics, SMS has been used in alkaline flooding, a chemical recovery method to recover oil from various types of geological formations and sand (Larrondo et al. 1985).

Based on the chemical composition of sodium metasilicate (Na_2SiO_3), it can react with the cement hydration products, forming various types of calcium silicate hydration gels. Fasesan et al. (2005) investigated the use of 0.5% of sodium metasilicate (by weight of cement) in 50:50 class H cement slurry to replace the 2% bentonite. The study showed that the sodium metasilicate was effective in controlling free water and fluid loss. In many parts of the world, severe lost circulation and weak formations with low fracture characteristics are common. These situations require the use of low-density cement systems that reduce the hydrostatic pressure of the fluid column during the cement placement process. Hence lightweight additives (also known as extenders) are used to reduce the weight of the slurry. Malyshev et al. (2013) used SMS as the extender for a lightweight cement system of density 1500 kg/m^3 (12.6 ppg). Hence SMS has multifunctional ability to modify the oil well cement.

Objectives

The overall objective of the study was to determine the effect of sodium metasilicate on the behavior of smart oil well cement. The specific objectives are as follows:

(i) Effect of SMS on the rheological, curing, and electrical properties of smart oil well cement.

(ii) Effect of SMS on the piezoresistive behavior of hardened smart oil well cement.

Materials and Methods

Materials
Smart Cement

Commercially available oil well cement (Class H cement) was modified with additives to make it a piezoresistive material.

Sodium Metasilicate

Sodium metasilicate, also known as disodium metasilicate, used in this study is a white, odorless, granular anhydrous powder. Based on the data sheet provided by the chemical manufacturer, it has a molecular weight of 122 g/mol, pH of12.5 at 10 g/l at 20 °C (68 °F), melting point of1,090 °C (1,994 °F), density of 2.61 g/cm3 at 20 °C (68 °F), and water solubility of 350 g/L at 20 °C (68 °F).

Methods

Sodium metasilicate (SMS) solution

The samples were prepared by mixing selected amount of SMS powder (by weight of water) in water at room temperature. The mixture was blended using a table top blender and the pH was monitored at the end of mixing. Up to 5% SMS solution was prepared to characterize the electrical resistivity properties. Water with and without SMS was used to prepare the cement specimens.

Cement Mixture

The samples were prepared according to the API standards. Smart cement with a cement-to-water ratio of 0.4 was used in this study. Two series of cement slurries were prepared with and without selected amount of SMS (0.2%).

Cement Specimen Preparation

After mixing, specimens were prepared using cylindrical molds with a diameter of 2 inches and a height of 4 inches. Two conductive wires were placed in all of the molds which were 5 cm apart. All specimens were capped to minimize moisture loss and were cured up to the day of testing for the piezoresisitivity under compressive loading.

Rheological Tests

Rheological properties determine the ability of cement to be pumped. The rheology tests were performed by utilizing a rotational viscometer at room pressure and temperature at rpms ranging from 3 to 600, and related shear stresses were recorded. The viscometers were calibrated using several standard solutions.

Electrical Resistivity
SMS Solution

A commercially available conductivity probe was used to measure the conductivity (inverse of resistivity) of the fluids. The resistivity measuring range was from 0.1 Ω.m to10,000Ω.m.
Cement

Based on the results of past studies, electrical resistivity was selected as a monitoring parameter to quantify the performance of modified cement during the curing and hardening process (Vipulanandan et al.2004-2013). Electrical resistivity of the slurries was measured using an API standard resistivity meter. Further, electrical resistance was measured using an inductance – capacitance - resistance (LCR) meter during the curing time. To minimize the contact resistances, the resistance was measured at 300 kHz using the two-wire

method (Vipulanandan et al., 2013). The principle of measuring the resistance is shown in Figure1. Each specimen was calibrated to obtain the electrical resistivity (ρ) from the measured electrical resistance (R) based on the Eqn. (1).

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

where L is the distance between the wires, A is the cross-sectional area through which the current is flowing, and L/A is called the geometry factor K. Where parameter K=L/A, the ratio of the cross-sectional area and the length of the material is defined for a particular set-up. If we know the resistivity and the resistance of the material then from the relationship in Eqn. (1) we can determine the parameter K. The resistivity of each cement slurry was determined using the API resistivity meter. Figure 2 shows K values determine for four different cement slurries such as cement slurry with and without Sodium Meta-silicate (SMS). After about 5 hours (300 min), the K value stabilized and it was used to determine the resistivity of the hardened cement specimens.

Compressive Strength Tests.

Compressive strength of cement determines the ability of cement to stabilize casing in the wellbore. The cylindrical specimen was capped and tested at a predetermined controlled displacement rate. Compression tests were performed on cement samples after 2 and 7 days of curing using a hydraulic compression machine.

Piezoresistivity Tests.

Piezoresisitivity describes the change in electrical resistivity of a material under pressure. Since oil well cement serves as a pressure-bearing part of wells in real applications, the piezoresistivity of smart cement with and without SMS was investigated under compressive loading. During each compression test, electrical resistance was measured in the stress axis. To eliminate the polarization effect, alternating current (AC) resistance measurements were made using a LCR meter at a frequency of 300 KHz. Furthermore, changes in resistivity were related to the applied stress.

Results and Discussions

Results

The average unit weight of the cement slurry was 121. 5 pcf.

SMS Solution
pH

Addition of SMS to the water increased the pH as shown in Figure 3. With the addition of 0.1% SMS, the water pH increased from 7.7 to 11.8, a 50% change in the pH. With the addition of 0.3% SMS the pH was 12.4.

Resistivity

The resistivity of sodium metasilicate solution was determined with the conductivity probe. SMS solution was very sensitive to electrical resistivity. The resistivity of water

decreased from 21Ω.m to 4.15 Ω.m with an addition of only 0.1% SMS (Figure 4), 80% reduction in resistivity. The addition of more SMS further reduced the resistivity of the solution. The following relationship is proposed based on the experimental results:

$$\rho = \rho_o - S/(E + DS) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(2)$$

where:
ρ = resistivity of the sodium metasilicate solution
ρ_o = resistivity of tap water without sodium metasilicate (21 Ω.m)
S   = Concentration of sodium metasilicate (% by weight)

Parameters E and D are model parameters: parameter E represent the initial rate of change and parameter D determines the ultimate resistivity. Experimental results matched very well with the proposed model with a coefficient of determination (R^2) of 0.98, and parameters E and D were found as 0.0016/Ω.m and 0.047/Ω.m, respectively.

Rheological properties
Gel Strength

For the smart cement, after 10 seconds gel strength was 12 lb/100 ft^2, and after 10 minutes gel strength was 14 lb/100 ft^2.With the addition of 0.2% SMS to the cement the 10-second gel strength was 15 lb/100 ft^2 and the 10-minute gel strength was 17 lb/100 ft^2. Hence, addition of 0.2 SMS increased the 10-second gel strength by 25% and the 10-minute gel strength by 21%.

Effect of SMS

It was evident from the rheological tests on cement slurries with and without 0.2% SMS that the addition of SMS negatively affects the shear-thinning behavior of cement. Addition of SMS increased the viscous behavior of the cement. For instance, the viscosity of cement without SMS at a shear strain rate of 100 (1/sec) was 146 cP. At the same shear strain rate the 0.2 percent SMS sample had viscosity of 225 cP, a 54% increase in viscosity.

Modeling

Due to the shear-thinning behavior of the cement slurries the Bingham plastic model was not an accurate model to estimate the shear strain rate – shear stress relationship. To predict the shear strain rate - shear stress relationship, the Herschel-Bulkley model (Eqn.3) and hyperbolic model (Eqn. 4) were used to predict the experimental data.

The Herschel–Bulkley (H-B) model follows:

$$\tau = \tau_o + k \, \gamma^n \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

where:
τ = shear stress
γ = shear rate
τ_o = yield stress
k and n are model parameters.
From the smart cement, the k and n were found to be

1.09 and 0.78, respectively (Figure 5). The coefficient of determination (R^2) was 0.97. The yield stress was found as 1.87 lb/100 ft^2.

For the cement slurry with 0.2% sodium meta-silicate the k and n (for Herschel–Bulkley model)were found to be 0.61 and 0.88, respectively (Figure 6). The coefficient of correlation was 0.99. The yield stress was 13.8 lb/100 ft^2, a notable increase in the yield stress with the addition of 0.2% SMS.

The hyperbolic model is as follows:

$$\tau = \tau_o + \gamma/(A+B\gamma) \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots(4)$$

where.

τ= shear stress

τ_o= yield stress

A and B are model parameters.

Here for the smart cement parameters A and B were found as 2.83(Pa.sec)$^{-1}$and 0.001Pa^{-1},respectively, and the coefficient of determination (R^2) was 0.97. The yield stress was 5.01 lb/100 ft^2. The apparent viscosity at 600 RPM was 106 cP.

For the smart cement with 0.2 SMS, the hyperbolic model parameters A and B were found as3.21(Pa.sec)$^{-1}$ and 0.0004Pa^{-1},respectively, and the coefficient of determination (R^2) was found to be 0.99. The yield stress was17.4lb/100 ft^2.With the addition of 0.2% SMS the apparent viscosity at 600 RPM was 136 cP, a 28% increase.

Fluid Loss

For the smart cement the total fluid loss was 137 cc. Smart cement (w/c ratio of 0.4) with the addition of 0.2% SMS had a fluid loss of 145 cc (Figure 7), hence a 6% increase in the fluid loss.

Electrical Resistivity
Initial Resistivity

The electrical resistivity of the cement slurry with and without SMS was determined. The initial resistivity of the smart cement slurry was 0.97 Ω.m, and it decreased with the addition of sodium metasilicate, as shown in Figure8. With the addition of 0.1% SMS the resistivity decreased to 0.92 Ω.m, a 5% reduction. With the addition of 0.2% and 0.3%SMS the resistivity were 0.9 Ω.m and 0.88 Ω.m. Hence the resistivity was sensitive to the concentration of SMS in the cement.

Resistivity during Curing Process

Electrical resistivity could be used as a fingerprint of the curing process. Figure 9 illustrates the change in electrical resistivity (ρ) during curing time for smart cement with and without SMS. It was observed that the curves of the different samples, with and without SMS, follow a similar trend with time. The electrical resistivity dropped to a minimum value, and then gradually increased with time. After initially mixing cement with water, resistivity decreased to a minimum value (ρ_{min}), and the corresponding time to reach the minimum resistivity was t_{min}. The decrease in resistivity immediately after mixing was due to dissolution of soluble ions from the cement particles after cement was mixed with water, and the dissolving process of the ions caused the resistivity decrease during early periods. The term t_{min} can be used as an index of speed of chemical reactions and cement set times. With the formation of resistive solid hydration products that block the conduction path, resistivity increased sharply with curing time. The following increase in electrical resistivity was caused by the formation of a large amount of hydration products in the cement matrix. Finally, a relatively stable increasing trend was reached by the ion diffusion control of hydration process. Resistivity increased steadily up to 24 hours and reached a value of ρ_{24}. Change in electrical resistivity with respect to minimum resistivity quantifies the formation of solid hydration products, which leads to the strength development in the curing cement. Therefore, by tracking the change in resistivity of oil well cement, a clear understanding of the hydration process and strength development can be obtained. Hence the Resistivity Index at 24 hours (RI_{24}) is defined as the maximum change in resistivity in 24 hours to reflect the changes in resistivity.

Variations in electrical resistivity with time for samples with different amounts of SMS are summarized in Table 1. Increasing SMS content decreased the minimum resistivity of cement (ρ_{min}). This is another indicator of the increased chemical reaction between the cement and SMS. With 0.2 percent SMS the minimum resistivity of cement decreased from 0.81 Ω.m to 0.75 Ω.m, a 7.5% percent decrease. Also, addition of SMS increased both the t_{min}, and the rate of change in resistivity. $RI_{(24)}$ for the smart cement was 175 percent (Table 1). With the addition of SMS, $RI_{(24)}$ varied from 178 to 196 (%). In general, higher change in electrical resistivity ($\rho_{24} - \rho_{min}$) indicates that increased hydration products are developed in the hydrating cement system.

Compressive Strength
Effect of SMS

The compressive strength of smart cement without any SMS was 1.0 ksi and 2.5 ksi after 2 days and 7 days of curing, respectively. As shown in Figs. 10 and 11, the addition of 0.2% SMS did not affect the compressive strength of cement.

Piezoresistivity

The piezoresistive behavior (compressive stress versus change in resistivity) of cement with and without SMS is shown in Figs. 10 and 11 after 2 days and 7 days curing, respectively. Electrical resistivity was sensitive to compressive stress. Figures 10 and 11 show that electrical resistivity increased during compressive loading. Addition of SMS reduced the piezoresistive response of smart cement. After two days of curing the smart cement failed at a resistivity change of 550 percent, while the 0.2 percent SMS added cement failed at a resistivity change of 150 percent. After seven days of curing the smart cement failed at a resistivity change of 800 percent, while the 0.2 percent SMS added cement failed at a resistivity change of 200 percent. The failure strain is 0.25 percent, and hence the change in the resistivity of cement with 0.2 percent of SMS was 800 times higher than the strain.

Conclusions

Based on this experimental and analytical study on smart cement with the addition of sodium metasilicate (SMS), the following conclusions are advanced:

1) Addition of 0.1% SMS increased the pH and reduced the resistivity of water by 50% and 80%, respectively. The change in resistivity of water with the addition of SMS has been modeled.

2) Addition of 0.2% SMS increased both the 10-second and 10-minute gel strength by over 20%.

3) Addition of 0.2% SMS increased the yield strength, viscosity, and fluid loss. Shear stress and shear strain rate relationships have been modeled.

4) Electrical resistivity developments with hydration time of the cement with different amounts of SMS follow a similar pattern: they first drop to a minimum point and then gradually increase with time. The minimum resistivity decreased, and the time to achieve minimum resistivity (rate of change) increased with the addition of increasing SMS. The resistivity index ($RI_{(24)}$) of the cement with and without SMS were comparable.

5) The smart cement showed piezoresistive behavior. Addition of SMS reduced the piezoresistive behavior of the smart cement. Addition of 0.2% SMS did not affect the compressive strength of the smart cement.

Acknowledgements

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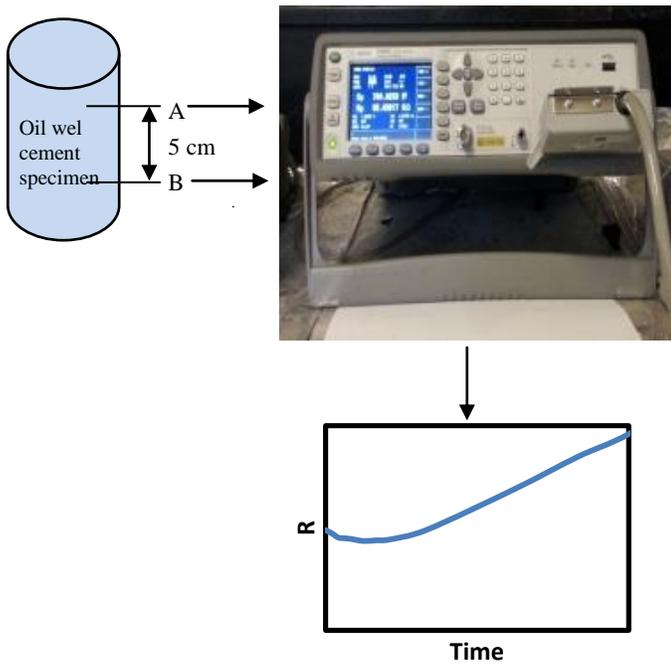
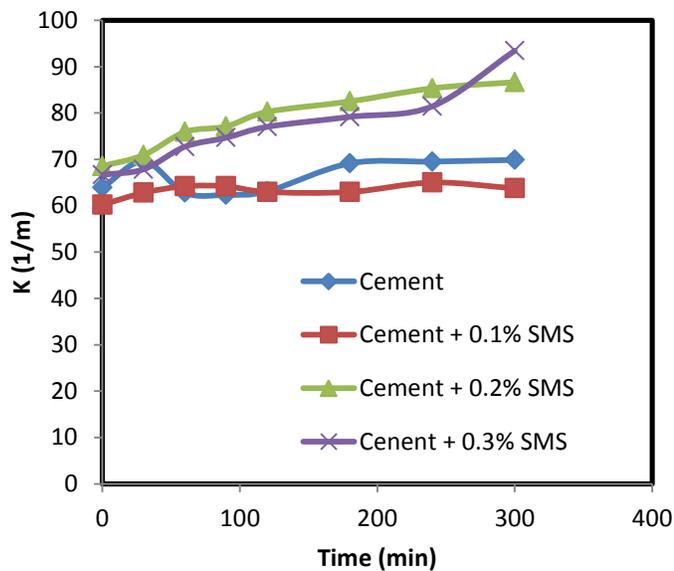**Figure 1.** Typical components in the resistance measurements in the cement specimen.

Figure 2. Variation of the K value (1/m) determined from the resistivity and the set up shown in Figure 1.
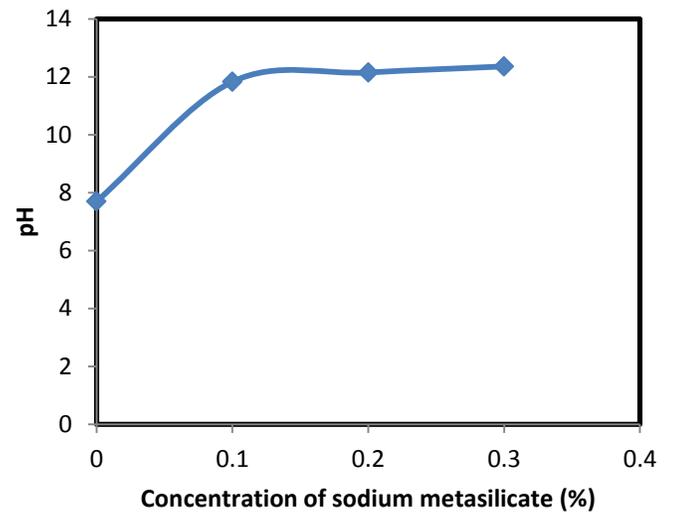

Figure 3. Variation of pH of water with sodium Metasilicate concentration (% by weight of water)
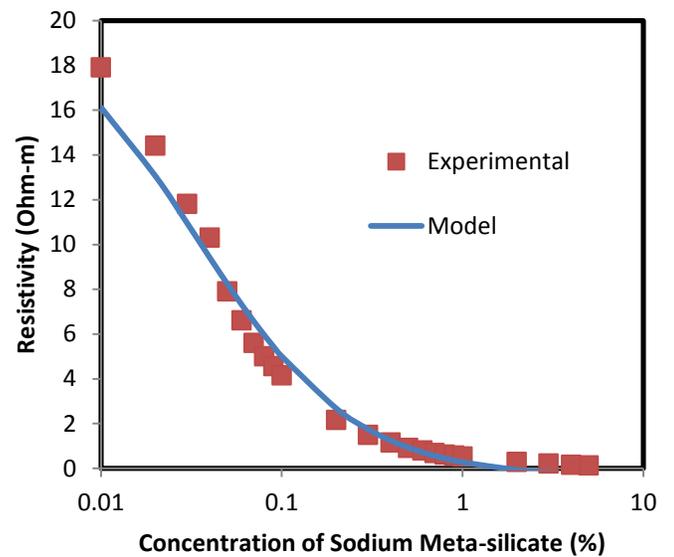

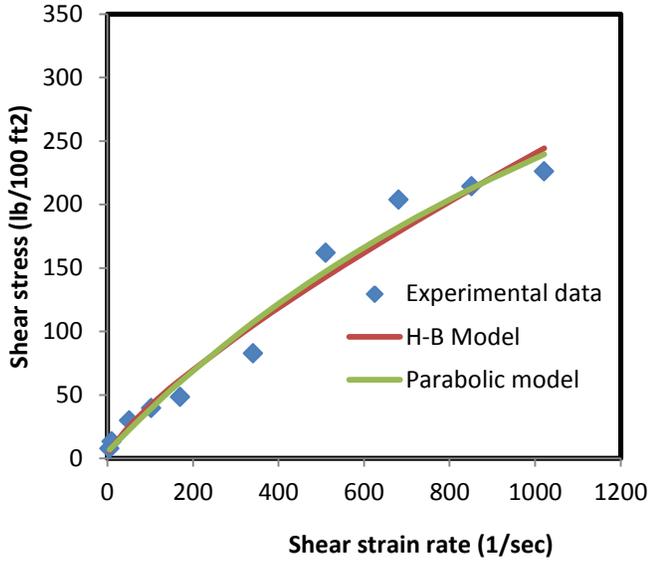**Figure 4.** Variation of resistivity of water with sodium metasilicate concentration (% by weight of water)

Figure 5. Shear stress – shear strain rate relationship for a smart cement slurry without sodium metasilicate
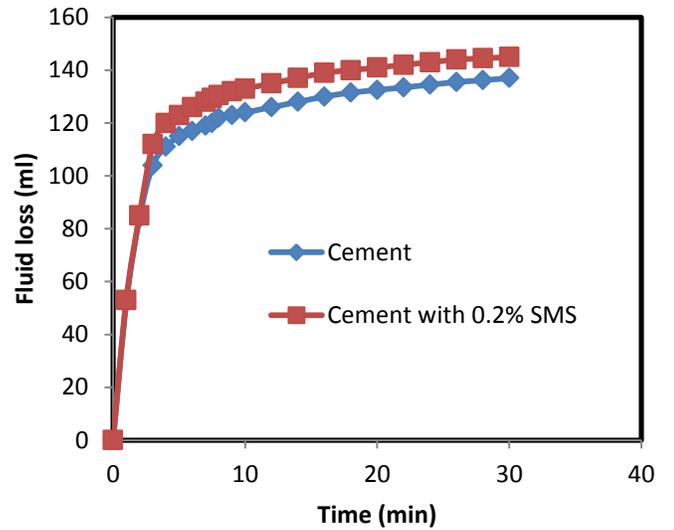
Figure 7. Fluid loss with time for smart cement slurry with and without sodium metasilicate
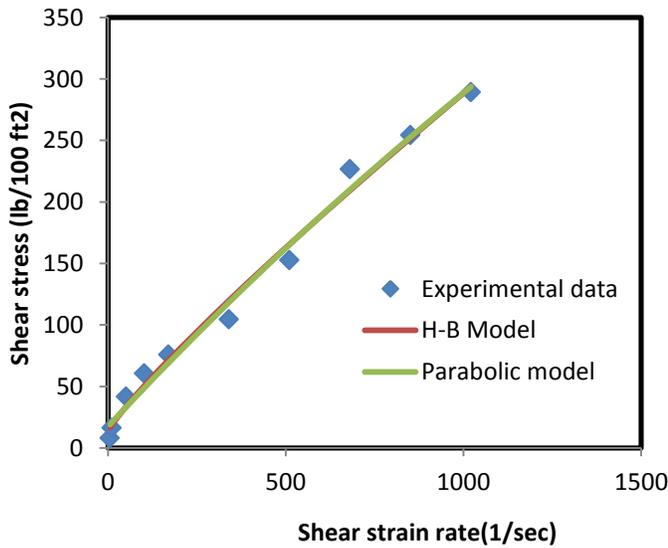

Figure 6. Shear stress – shear strain rate relationship for a smart cement slurry with 0.2% sodium metasilicate
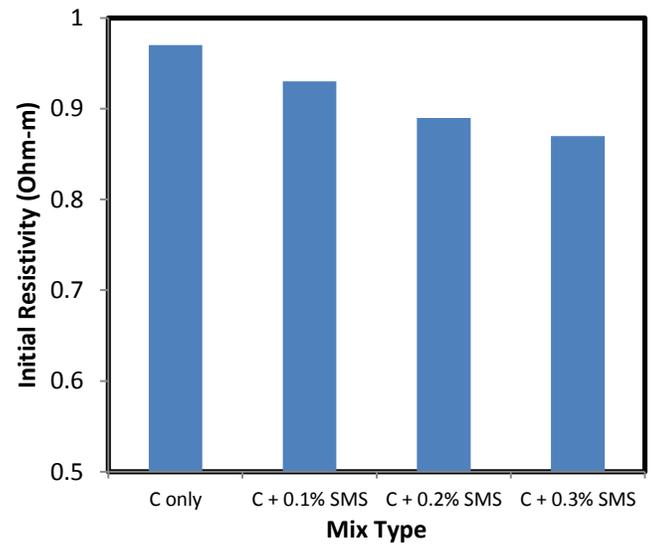

Figure 8. Initial resistivity of the cement slurry with different concentration of sodium meta-silicate (C = smart cement)
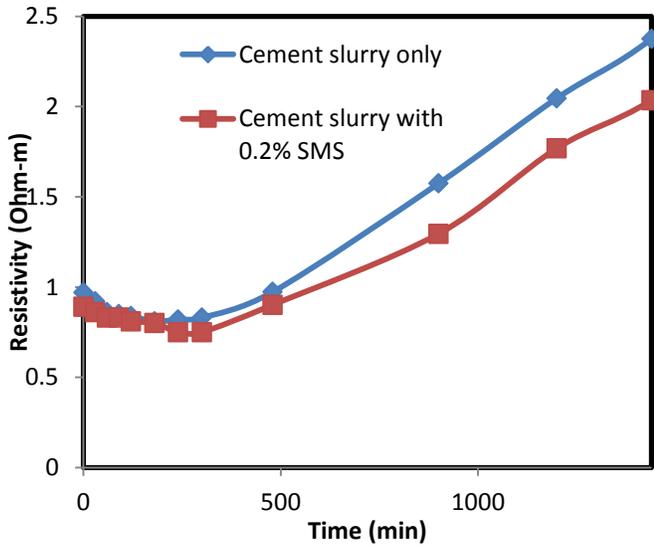
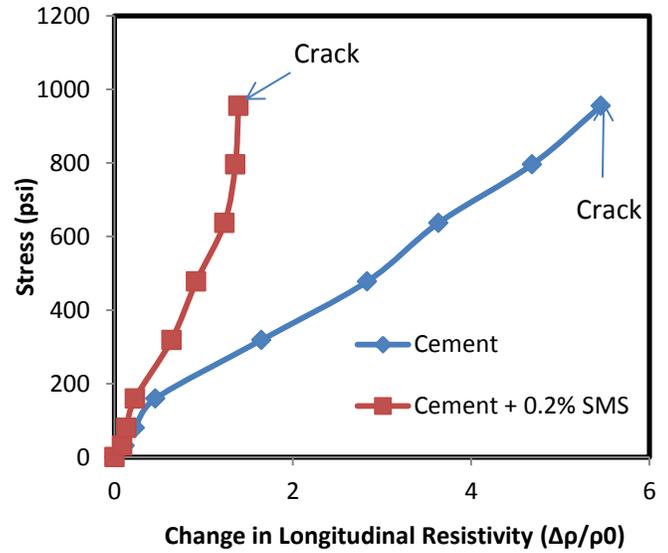**Figure 9.** Variation of resistivity of curing smart cement slurry with time

Figure 10. Piezoresistive response of the smart cement with and without sodium metasilicate after 2 days of curing.

Table 1. Resistivity change with curing time for smart Oil Well Cement with different percentage of Sodium Metasilicate (SMS)

Mix Proportions		ρ_o $(\Omega\text{-m})$	ρ_{min} $(\Omega\text{-m})$	t_{min} (min)	ρ_1 $(\Omega\text{-m})$	ρ_{24} $(\Omega\text{-m})$	$RI_{(24)}$ (%)
w/c= 0.4	Cement (C)	0.97	0.81	180	0.86	2.23	175
	C+0.1% SMS	0.93	0.80	240	0.81	2.24	180
	C+0.2% SMS	0.89	0.75	240	0.83	2.22	196
	C+0.3% SMS	0.87	0.61	300	0.77	1.7	178

ρ_o = Initial resistivity
ρ_{min} = Minimum resistivity
t_{min} = Time to reach minimum resistivity
ρ_1 = Resistivity at 1 hour
ρ_{24} = Resistivity at 24 hour
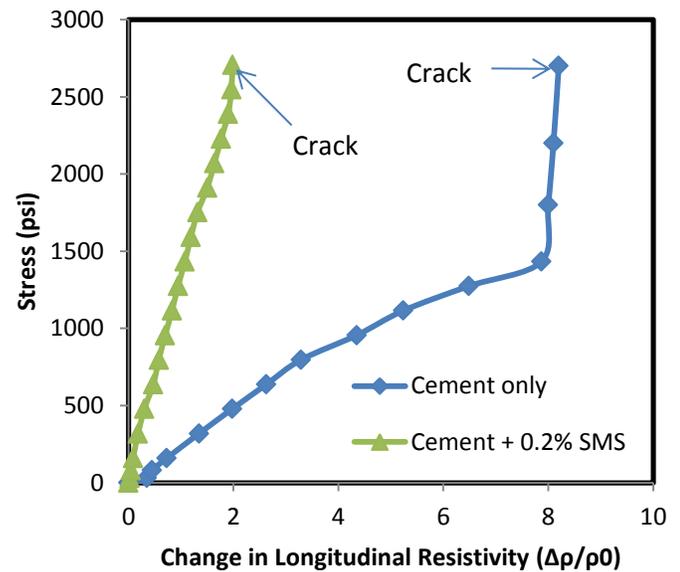$RI_{(24)}$ = Resistivity Index = $[(\rho_{24}-\rho_{min})/\rho_{min}]$ (%)

Figure 11. Piezoresistive response of the smart cement with and without sodium metasilicate after 7 days of curing.