Nano silica and Biosurfactant Modified Smart Oil Well Cement Grout

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Abstract

Cement and polymer grouts are being used in various construction and rehabilitation applications related to oil wells, piles, structures, leaking pipelines and ground improvement. In this study oil well cement (Class H) was used as representative cement grout. The smart cement grout was developed using conductive fillers to make them piezoresistive materials. The highly sensing properties of the smart grout will help with the real-time monitoring of the performance of the grout from the time of installation to the entire service life. A series of experiments were conducted to evaluate the smart grout behavior with and without 1% nano silica (NS) and 0.5% biosurfactant (BS) to determine the sensitivity in terms of piezoresistivity. The Marsh Funnel flow time and the viscosity increased by addition of NS and BS for both types of grouts. Addition of 1% NS improved the compressive strength and piezoresistivity of both smart cement grouts. For the smart oil well cement grout the piezoresistivity at compressive peak stress varied from 155 to 179% and it increased to 209-237% with 1% nano-silica. With 1% nano-silica and 0.5% biosurfactant the piezoresistivity reduced to 64 to70%. For the Portland cement grout with with 1% NS and 0.5% BS the piezoresistivity varied from 124% to 178%. Vipulanandan p-q piezoresistive model predicated the compressive stress–piezoresistivity strain relationship of the smart cement grouts with and without NS and BS very well.

1. Introduction

Grouting materials are being used for not only construction but also for repairs and maintenance of onshore and offshore infrastructure facilities. Grouts have been commonly used for leak control in below grade wet wells or holding tanks; manholes; sewer and storm lines, cracked retaining walls and other underground structures [Vipulanandan, 1996a, Vipulanandan, 2000; Ozurel et al. 2004], repairing of cracks in massive concrete structures and masonaries [Anagnostopoulos 2014], to coat pre-stressed cables, to stabilize ground near tunnels [Stille and Gustafson, 2010], rehabilitating old defective masonaries in historical buildings [Yeon and Han, 1997, Baltazar et al. 2012], and to repair cracked oil well cement sheath [Chun et al. 2008]. The primary goal of grouting these facilities is to return the structure to its original working conditions by the oldest trenchless technology method, in-situ grouting [Krizek et al. 1985 and Vipulanandan et al 1996-2009]. Several types of grouting materials have been used in controlling leaks in concrete wastewater systems and storm systems [Vipulanandan, 1996b]. The grouting or grout application are now a days very much common [Lim et al. 2013] and cement-based grouts have been widely used since the 1800s and even earlier [Bowen 1981]. Grouting is a process of injecting the cementitious fluid that enters and set into fissures, cracks or voids [Nonveiller, 1989] of the damaged structure or rock and provide a sealing to the damaged part.

Recently, smart cement has been developed with class H cement and 0.075% of conductive filler (CF) which has a sensing ability in terms of electrical resistivity [Vipulanandan et al., 2014-2015]. If the water cement ratio of the slurry is changed and/or any kind of additives are added to the slurry or the cement slurry is contaminated, the resistivity can sense that change both in the cement slurry and hardened cement. The most important characteristics is the piezoresistivity of the hardened cement (i.e., the change in resistivity due to applied stress) where the change in resistivity due to applied stress was found form 300-400% form the initial resistivity which is very high compared to the change in the axial strain (which is about 0.2% only at failure). The rheological properties were not affected by the addition of 0.075% conductive filler with class H cement. This smart cement can be used to monitor the structural health of the borehole from cementing time to its entire service life by developing a wired electrical monitoring system. If for any reason this smart cement is damaged and required to use cement grout then the question arises whether that grout can provide the sealing, strength and the piezoresistivity at the same time.

2. Objectives

The overall objective of the study was to determine the curing and piezoresistive behavior of smart cement and smart polymer grouts with and without nano-silica (NS) and biosurfactant (BS). The specific objectives are as follows:

(i) Characterize the piezoresistive behavior of smart oil well cement grout with and without NS and BS.

(ii) Characterize the piezoresistive behavior of smart Portland cement grout with and without NS and BS

(iii) Model the piezoresistive responses of the smart cement grouts using Vipulanandan p-q Piezoresistive Model.

3. Materials and Methods

Materials

Cement: Commercially available oil well cement (API class H cement) was modified with 0.075% conductive filler (CF) to make it piezoresistive material.

Smart cement grout: Smart cement grout was prepared using smart cement mixed with water- to-cement ratio of 0.8.

Nanosilica: Nano silica (NS) used in this study is a white nano particles of silica (SiO_2) in powder form having average particle size 12 nm, surface area 175 - 225 m2/g, pH 3.7 - 4.7, water content < 1.50 %, melting point/freezing point > 1,600 °C (> 2,912 °F), initial boiling point - 2,230 °C (4,046 °F), relative density 2.600 g/cm3.

Biosurfactant: The surfactant used in this study is the UH-Biosurfactant which was produced from used vegetable oil in continuously stirred batch reactors and Serratia.sp. bacteria.

Methods

Mixing

The samples were prepared by mixing the water with the cement or polymer solution using standard mixers. Smart cement grout with water-to-cement ratio of 0.8 was used in this study.

Specimen Preparation

After mixing, specimens were prepared using cylindrical molds with a diameter of 50.8 mm and a height of 101.6 mm. Two conductive wires were placed in all of the molds which were 50.8 mm apart. All specimens were capped to minimize moisture loss and were cured up to the day of testing for the piezoresisitivity under compressive loading.

Flow test

Marsh cone test was done to characterize the cement grouts with and without additives.

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 oil well 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:

$$\rho = R * K \tag{1}$$

where R is electrical resistance. The calibration parameter K was determined based on the resistance measured at 300 kHz of frequency and the resistivity (digital resistivity meter and conductivity meter) measured at the corresponding time. Normalized change in resistivity with the changing conditions is represented as

$$\frac{\Delta\rho}{\rho_0} = \frac{\Delta R}{R_0} \tag{2}$$

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

Initial resistivity

Two Different methods were used for electrical resistivity measurements of oil well 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:

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.

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 grouts

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 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). 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 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 1, 7 and 28 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 grout with and without nano silica and biosurfactant additives was investigated under compressive loading. During 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 (Vipulanandan 2021).

4. Results and Discussions

Marsh cone viscosity of the grouts

The flow time for the cement grouts were determined using the Marsh Funnel viscometer. After determining the flow time, the viscosity is calculated from the relationship, $\mu = d(t-25)$, where μ = viscosity in centipoise (cP), d = density of the mud in gm/cc, t = flow time of the grout in seconds. The determined flowtime and calculated viscosities are summarized in Table 1. The smart oil well cement grout flow time was 55 sec which increased to 83 sec with the addition of 1% NS. Addition of 0.5% BS decreased the flow time to and 64 sec with 1% NS.

Table 1. Marsh Funnel flow time and viscosity of cement grouts

Mix Type	Density, d (gm/cc)	Flow Time, t (sec)	Marsh Funnel Viscosity, μ (cP)
Grout (H, w/c=0.8 only)	1.61	55	48
Grout (H, w/c=0.8, NS = 1%)	1.72	83	100
Grout (H, w/c=0.8, NS=1%, BS=0.5%)	1.62	64	63

Initial Electrical Resistivity

(a) **Smart Oil Well Cement Grout**: The initial resistivity (ρ_o) of the smart cement grout increased from 1.08 Ω-m to 1.13 Ω-m with 1% NS, a 5% increase; but with 1% NS and 0.5% BS, ρ_o increased by 6% to 1.14 Ω-m. The minimum resistivity (ρ_{min}) of the smart cement grout and the smart cement grout with 1% NS was same at 1.04 Ω-m, but increased to 1.09 Ω-m, a 5% increase. The changes in the electrical resistivity were higher than the changes in the unit weight of the cement grout. Hence the electrical resistivity can also be used for quality control of smart cement grout curing.

Table 2. Density and Initial Resistivity of Smart Grouts

Grout Mix	Density (g/cc)(ppg)	Initial resistivity, ρ_o (Ω.m)
Grout (class H, w/c=0.8 only)	1.62 (13.44)	1.08
Grout (H, w/c=0.8, NS=1%)	1.73 (14.39)	1.13
Grout (H, w/c=0.8, NS=1%, BS=0.5%)	1.63 (13.54)	1.14

Piezoresistivity and strength of smart cement grouts

Addition of 0.075% CF substantially improved piezoresistive behavior of the cement grout. Based on experimental results, p-q model developed by Vipulanandan and Paul, 1990 was modified and used to predict the change in electrical resistivity of cement with applied stress for 1, 7 and 28 days of curing. The new piezoresistive model was defined as follows:

$$\frac{\sigma}{\sigma_f} = \left[\frac{\frac{x}{x_f}}{q_2 + (1-p_2-q_2)\frac{x}{x_f} + p_2 \left(\frac{x}{xf}\right)^{\left(\frac{p_2}{p_2-q_2}\right)}} \right] \tag{3}$$

where σ: stress (MPa); σ_f: stress at failure (MPa); $x = \left(\frac{\Delta\rho}{\rho_o}\right) * 100$ = 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; $\Delta\rho$: change in electrical resistivity; ρ_o : Initial electrical resistivity (at σ=0 MPa) and p_2 and q_2 are piezoresistive model parameters

Smart oil well cement grout

The compressive strength ($\square_f$) of the smart cement grout after 1day, 7 days and 28 days of curing were 2.96 MPa, 9.94 MPa and 16.47 MPa which were increased to 3.49 MPa, 10.94 MPa and 18.55 MPa respectively for smart cement grout with 1% NS which were summarized in Table 3. Thus the compressive strength of the grouts were increased by 18%, 10% and 13% after 1day, 7 days and 28 days of curing with addition of 1% NS. By addition of 1% NS and 0.5%BS, the strengths after 1day and 7 days of curing were 2.72 MPa and 9.05 MPa which are 8% and 9% reduction respectively.

The change in electrical resistivity at failure $\left(\frac{\Delta\rho}{\rho_o}\right)_f$ for the smart cement grout only after 1day, 7 days and 28 days of curing were 155%, 156% and 179% which were increased to 237%, 227% and 209% respectively for smart cement grout with 1% NS (Table 3). Thus the piezoresistivity of the grouts were increased by 53%, 45% and 18% after 1day, 7 days and 28 days of curing with addition of 1% NS. By addition of 1% NS and 0.5%BS, the piezoresistivity after 1day and 7 days of curing were 70% and 64%, which are 55% and 66% reduction respectively.

Using the p-q Piezoresistive model (Eqn. 3)), the relationships between compressive stress and the change in electrical resistivity $\left(\frac{\Delta\rho}{\rho_o}\right)$ of the smart cement grout with and without NS and BS for 1 day, 7 days and 28 days of curing were modeled. The piezoresistive model (Eqn. (3)) predicted the measured stress- change in resistivity relationship very well (Fig. 1, Fig. 2, Fig. 3). The model parameters q_2 and p_2 are summarized in Table 5. The coefficients of determination (R^2) were 0.98 to 0.99. The root mean square of error (RMSE) varied between 0.08 MPa and 0.23 MPa as summarized in Table 3.

Table3. Compressive Strength and Piezoresistivity Model Parameters for Smart Oil Well Cement grout.

Mix Type	Curing Time (day)	Strength σ_f (MPa)	Piezoresistivity at peak stress, $(\Delta\rho/\rho_o)_f$ (%)	p_2	q_2	R^2	RMSE (Mpa)
Grout (H, w/c=0.8 only)	1 day	2.96	155	0.031	0.607	0.99	0.08
Grout (H, w/c=0.8, NS = 1%)		3.49	237	0.022	0.616	0.98	0.13
Grout (H, w/c=0.8, NS=1%, BS=0.5%)		2.72	70	0.135	0.840	0.99	0.05
Grout (H, w/c=0.8 only)	7 days	9.94	156	0.035	0.642	0.99	0.18
Grout (H, w/c=0.8, NS = 1%)		10.94	227	0.02	0.72	0.99	0.23
Grout (H, w/c=0.8, NS=1%, BS=0.5%)		9.05	64	0.08	0.72	0.99	0.17
Grout (H, w/c=0.8 only)	28 days	16.47	179	0.012	0.613	0.99	0.10
Grout (H, w/c=0.8, NS = 1%)		18.55	209	0.020	0.77	0.99	0.08

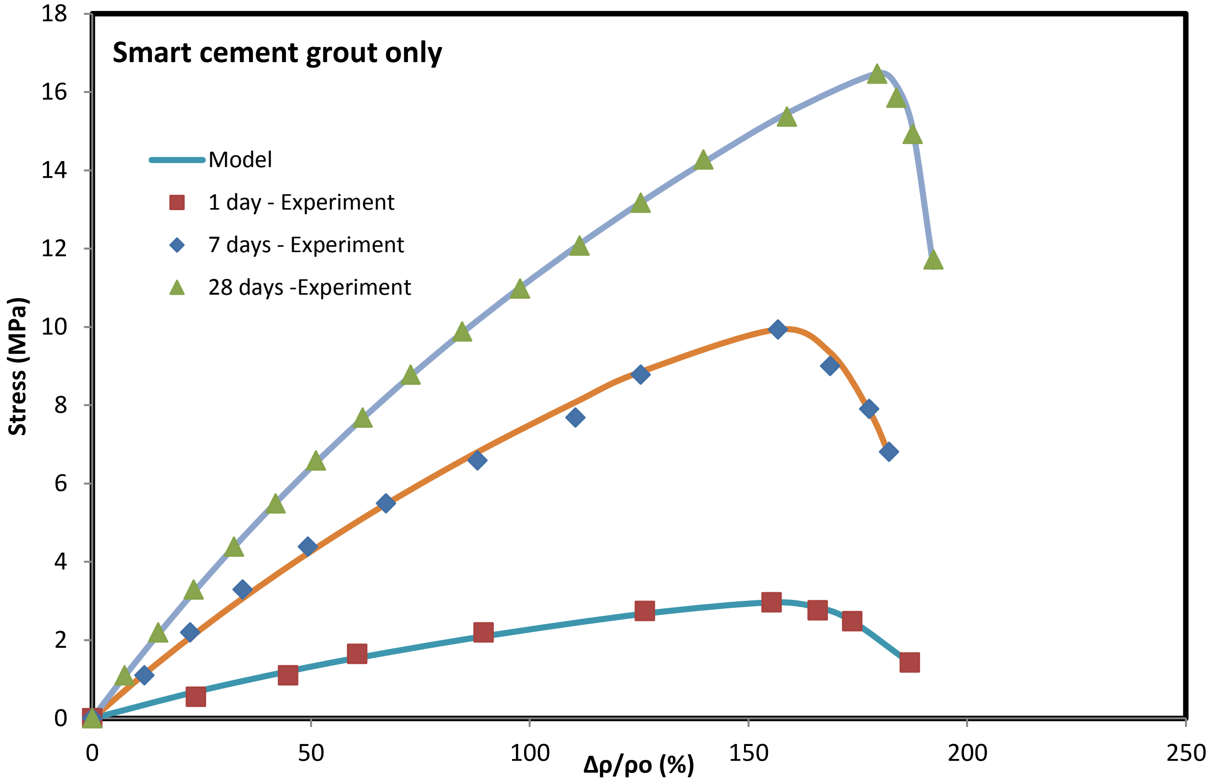

Figure 1. Piezoresistive response of the smart oil well cement grout only after 1 day, 7 days and 28 days of curing and modeled using the p-q model.

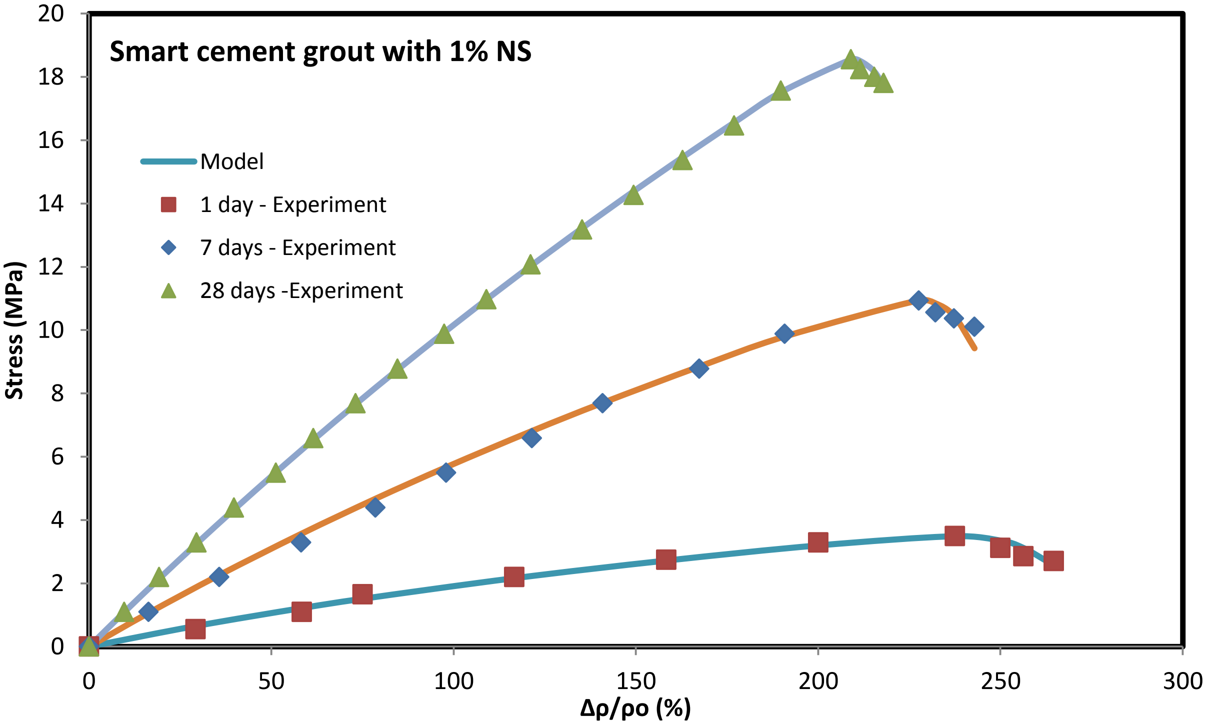

Figure 2. Piezoresistive response of the smart cement grout with 1% NS after 1 day, 7 days and 28 days of curing and modeled using the p-q model.

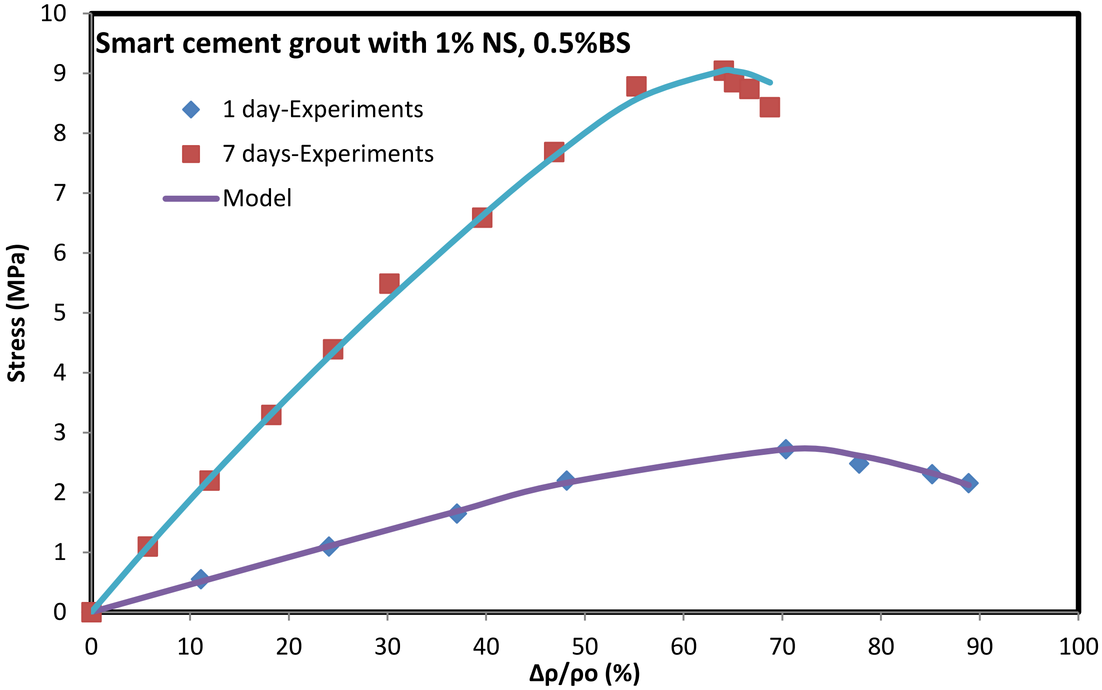

Figure 3. Piezoresistive response of the smart cement grout with 1% NS, 0.5%BS after 1 day and 7 days of curing and modeled using the p-q model.

5. Conclusions

Based on experimental and analytical study on smart cement grout with and without NS and BS cured at room temperature, the following conclusions are advanced:

1. The Marsh cone flow time and the viscosity increased by addition of NS and BS for the smart oil well cement grouts.
2. The initial resistivity (ρ_o) of the smart cement grout increased from 1.08 Ω-m to 1.13 Ω-m with 1% NS, a 5% increase; but with 1% NS and 0.5% BS, ρ_o increased by 6% to 1.14 Ω-m. The minimum resistivity (ρ_{min}) of the smart cement grout and the smart cement grout with 1% NS was same at 1.04 Ω-m, but increased to 1.09 Ω-m, a 5% increase. The changes in the electrical resistivity were higher than the changes in the unit weight of the cement grout. Hence the electrical resistivity can also be used for quality control of smart cement grout curing.
3. The smart cement grout showed piezoresistive behavior under compressive stress. Without any additive, piezoresistivity at peak stress was varying from 155-179% which was increased to 209-237% with 1% NS. But with 1% NS and 0.5% BS the piezoresistivity reduced to 64-70%.
4. Vipulanandan p-q piezoresistive model predicated the compressive stress–change in resistivity relationship of the smart cement grout with and without NS and BS very well.

6. Acknowledgement

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