IOP Conference Series: Materials Science and Engineering

PAPER • **OPEN ACCESS**

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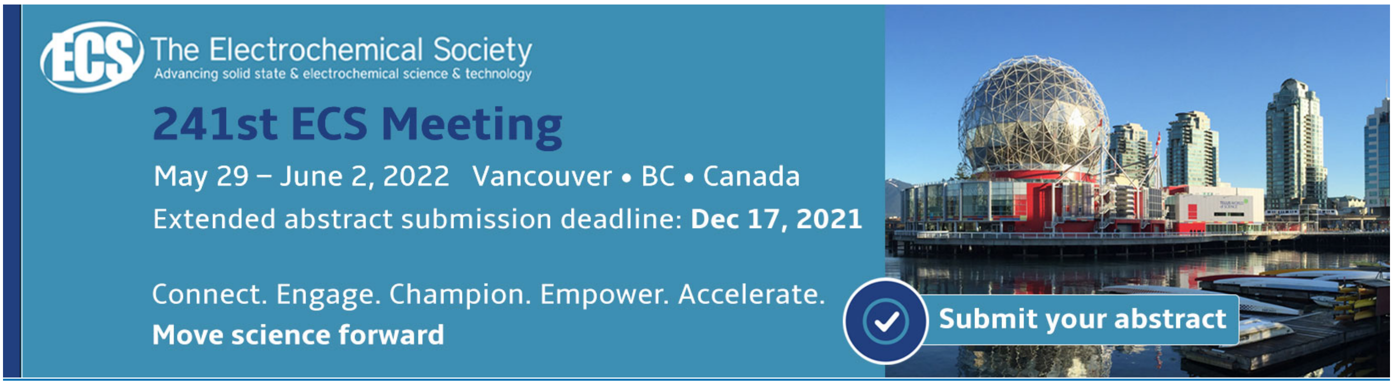
To cite this article: J Nori *et al* 2021 *IOP Conf. Ser.: Mater. Sci. Eng.* **1201** 012054



Effect of SiO_2 and SiO_2/TiO_2 hybrid nanoparticles on cementitious materials

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Abstract. In this paper, we report the effect of SiO_2 nanoparticle solution on the properties of the neat industry and environmental cements. Moreover, the hybrid SiO_2/TiO_2 nanoparticles solution impact on the Portland G-class cement. Both destructive and non-destructive tests were used to characterize the properties of the slurries and the cement plugs. Results indicate that the optimum concentration of the nanoparticles improved the elastic, energy absorption, rheological, heat development, and the mechanical load carrying capacity of the cements. The selected optimal nanoparticles concentrations results showed that

- the addition of 0.56 % SiO_2 by weight of cement (bwoc) increased the uniaxial compressive strength (UCS) of the neat industry cement by 16.7%.
- the 0.13% SiO_2 bwoc increased the UCS of the neat environmental cement by 50.2%.
- the blending of 0.264 %SiO_2 / 0.044% TiO_2 bwoc increased the UCS of neat G-class cement by 8.5%. However, by changing the curing temperature and pressure, different results can be achieved.

1. Introduction

Cement is an important well barrier element in oil and gas wells. During well construction process, cementing job is categorized as primary and remedial cementing. Primary cementing is the process of cement placement around casing. The main functions of cement among others are to provide zonal isolation, to prevent migration of fluids in the annulus, to provide structural integrity for the casing, and protection of the casing string from corrosive fluids [1]. The remedial cementing operations are performed to repair when primary cementing fails. A petroleum well will be permanently plugged with cement and abandoned when the well is found no longer economically profitable [2]. Cement quality and good cementing job are the main factors to ensure a long-term integrity of the well. However, due to pressure and temperature loading, the permeability of cement will be increased by cracking, and debonding [1]. This as a result allows reservoir fluid leakage through cement and outside of casing, between cement and inside of casing, cement itself, in cement fractures, and between cement and rock [3].

For long-term structural integrity, NORSOK D-010 defined the well integrity as the "a*pplication of technical, operational, and organizational solutions to reduce risk level of undesired formation fluids leaks throughout the life cycle of a well*" [4]. Additionally, the NORSOK D-010 demands criteria for cement properties to be impermeable, have long-term integrity, non-shrinking, ductile (non-brittle) to be able to withstand mechanical loads/impact and have resistance to different chemicals / substances (H_2S, CO_2 and hydrocarbons) as well has wetting to ensure bonding to steel.

Nevertheless, a well integrity survey study conducted by Vignes and Aadnøy [5] indicated that several wells have shown integrity issues in the North Sea. As displayed in Figure 1, survey results from the considered 31 production and 40 injections showed that cement related failure recorded about 11% rate.

Theresa and Bachu [6] have evaluated the CO_2 leakage potential of several abandoned wells in Alberta, Canada. Assessment results have shown that most of the leakages were above the top of the cement. Additionally, the cement bond logs indicated channeling through which formation fluids leaked and resulted in casing corrosion. From the integrity survey, it is evident that the cement does not satisfy the NORSOK D-010 requirements, and this suggests the need to improve the properties of the conventional cement.

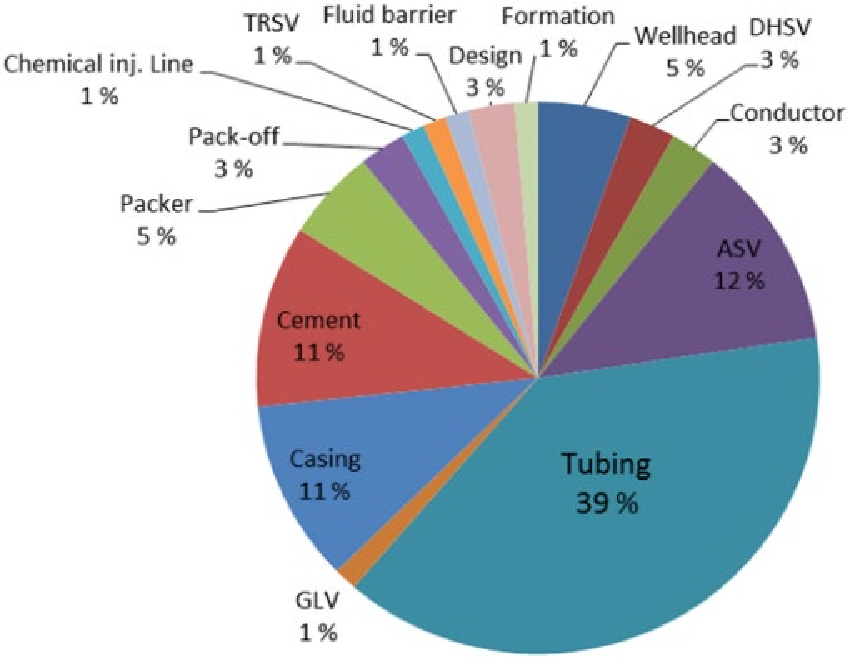


Figure 1. Barrier element failure [5].

In the recent years, the application of nanoparticles has shown remarkable effect on cement properties. The application of nanotechnology (1-100 nanometers) has shown proven solution in several industries such as biomedical and electronics. The surface area of nanoparticle is higher than the micro sized particles. Through chemical and physical interactions, nanoparticles create a new material having properties such as light weight and relatively greater strength [7]. The nanoparticle research results documented in literature have shown impressive impact on drilling fluid, cement, and enhanced oil recovery. However, its application is not fully investigated. For instance, some of the application of nanoparticle on cement has shown an improvement on the mechanical, rheological and petrophysical parameters. These nanoparticles include SiO_2 and Fe_2O_3 [8], Nano silica ([9 - 11], Nanotube [12] Nano-engineered API G-class cement [13], Nano clay [14], MWCNT [15], MgO [16] Graphite [17], Nano zeolite [18], Titanium Dioxide [19, 20].

In this paper, the impact of SiO_2 nanoparticle on the properties of industry (C-class) and environmental cements will be investigated. From the literature study, it is noted that there are positive impacts of the separate application of SiO_2 and TiO_2 on the G-class Portland oil well cement. However, in this paper, the effect of hybrid (SiO_2+TiO_2) nanoparticles on G-class will also be investigated.

2. Experimental works

This section presents the materials, methods of characterization, and cement slurry preparations.

2.1 Materials and methods

2.1.1 Description of Cements: Three different cements were used for the study, namely industry cement (C-class), Portland G-class cement and environmental cement.

*Industry **c**ement* **-** The Industry cement was provided by NORCEM [21]. The industry cement (also called C-class cement) is most commonly used on the top section of the petroleum wellbore because of its strength.

Environmental cement - The environmental cement was provided by CEMEX [22]. The cement consists of a lot of slags and has a great strength. The cement can thus be of good candidate for use on the top section of petroleum and geothermal wells.

*Portland G-class **c**ement* - The Portland G-class cement was obtained from NORCEM Co., Ltd [21]. The Portland G-class cement is the most commonly used oil well cement. In accordance with API SPEC 10A/NS-EN ISO 10426-1, the G-class cement is tested and found to have a higher sulfate resistance.

2.1.2 Description of Nanoparticle: To investigate the effect of nanoparticles on the cements, two types of nanoparticles in water solution were used, namely, colloidal silica nanoparticle solution and titanium oxide nanoparticle solution.

*Colloidal **s**ilica **n**anoparticle **s**olution* - The colloidal silica solution has a concentration of 50 wt.% suspension in H2O[23]. The solution has a density of 1.4 g/mL at 25°C with a pH ranging from 9.0 – 10.5. The nanoparticle was purchased in solution form from Merck Life Science AS/Sigma Aldrich Norway AS.

Titanium oxide nanoparticle solution **–** The rutile-titanium oxide solution has a concentration of 15 wt.% suspension in H2O [24]. The size of the nanoparticle ranges from 5-15nm. The nano-solution was purchased from the US Research Nanomaterials, Inc.

2.1.3 Characterization methods - The characterization of the cement plug specimens is through destructive and non-destructive methods. The first phase of testing is non-destructive tests, where the samples are characterized through the measurement of their ultrasonic, rheology, and heat development. The second phase is mechanical destructive test with uniaxial compressive tests.

*Compressive **s**trength* - Uniaxial compressive strength (UCS) is the material's strength to carry the compressive loading until it fractures. The UCS test procedures is according to NS-EN 196-1 standard (Norway, 2005) (ASTM, 2013). The UCS of the material is estimated from the peak load and the cross-sectional area of the specimens [25].

$$\mathrm{UCS} = \frac{\mathrm{F_{max}}}{\mathrm{A}} \tag{1}$$

where UCS is the Uniaxial compressive strength (MPa), F_{max} is the maximum force/load at the time of failure (N) and A is the cross-sectional area of the specimen (mm^2).

Sonic travel time **-** Ultrasonic inspection is a non-destructive method of investigating the materials ability to transmit sonic wave through its body. If structure contains cracks, pores, trapped air, and not very well cemented, the travel time will be higher than the very well compacted and strong materials. CNS Farnell Pundit 7 device was used for the non-destructive test. The travel time of the ultrasonic pulse that has been emitted from source and propagated through the cement specimen was recorded at the receiver. The compressional wave velocity through the plug specimen is estimated as:

$$\mathrm{V_p} = \frac{l}{t} \tag{2}$$

Where V_p is the compressional wave's velocity (m/s), l is the length of cement plug (m), t is the compressional wave's travel time through the plug (μs)

Viscosity of **cement slurry** - Fann viscometer was used to measure the viscosity of cement slurries. The measurements are at 300, 200, 100, 6, and 3 revolution per minute dial reading. The rheological parameters of the cement slurries were analyzed with the Casson rheological model. The Casson model describes viscoelastic fluids at high and low shear rate. Mathematically, the model is expressed as [1].

$$\tau^{0.5} = \tau_c^{0.5} + \mu_c^{0.5}\gamma^{0.5} \tag{3}$$

where τ is shear stress (Pa), τ_c is yield stress (Pa), μ_c is viscosity (Pa.s), γ is shear rate (sec^{-1})

Heat development: An exothermic reaction occurs when cement is in contact with water. The release of heat will increase the temperature of cement during hydration process. Based on the uniaxial compressive strength test results, the optimal nanoparticle concentration effect on the viscosity of the slurry will be compared with the neat cement slurries. The cement slurries were placed in an insulated compartments and connected with temperature sensors. During the hydration process, the sensors were measuring the temperature of the cements every 5 minutes for three days.

2.2 Experimental test matrix design

Test design 1- Investigation of silica on C-class cement

The first test design is aimed at investigating the effect of nano-silica solution on the C-class cement. The water/cement ratio (WCR) of slurry is 100/178.57 ≈ 0.56. A total of 4 nanoparticle-based cement plugs and one nanoparticle free plug were synthesized. For statistical purpose, four samples were made for plugs #1-4. To evaluate the impact of the higher concentration of nanoparticles, plug #5 with 0.84wt% concentration was synthesized having only one sample. Table 1 shows the amount of water, cement, and silica nanoparticles used for test design 1. The nano-free plugs are referred as a reference (Ref), or control and the nanoparticle blended cement plugs will be compared with. To maintain the concentration of fluids (i.e., 100 g), as the concentration of nano-solution increases, the same amount of water was reduced. The weight percent (wt.%) of nanoparticles are calculated by weight of cement (bwoc).

Table 1. Test design-1

Plug (#)	Freshwater (g)	Cement (g)	SiO_2 (aq) (g)	SiO_2 (aq) (% bwoc)
1	100	178.57	0.0	0.0
2	99.50	178.57	0.50	0.28
3	99.25	178.57	0.75	0.42
4	99.00	178.57	1.00	0.56
5	98.50	178.57	1.50	0.84

Test design 2- Investigation of silica on environmental cement

The design idea here is to evaluate the possibility of using environmental cement for the oil and gas well provided that it qualifies the industry requirement. The slurry was synthesized with a water/cement ratio of 100/192.3 ≈ 0.52. Table 2 provides the composition of test design-2.

Table 2. Test design-2

Plug (#)	Freshwater (g)	Cement (g)	SiO_2 (aq) (g) (aq)	SiO_2 (aq) (% bwoc)
1	100	192.3	0.0	0.0
2	99.75	192.3	0.25	0.13
3	99.50	192.3	0.50	0.26
4	99.25	192.3	0.75	0.39

Test design 3- Investigation of silica and titanium oxide hybrid on G-class cement

As reviewed, several investigators have analyzed the separate impact of SiO_2 and TiO_2 on G-class cement. In this paper, Test design 3 is aimed at investigating if there is synergy between 0.264 % bowc SiO_2 with different concentration of TiO_2 nanoparticle. The water cement ratio of the G-class cement was according to API given as: 100g /227.27g ≈ 0.44. Table 3 shows the composition of test design-3.

Table 3. Test design-3

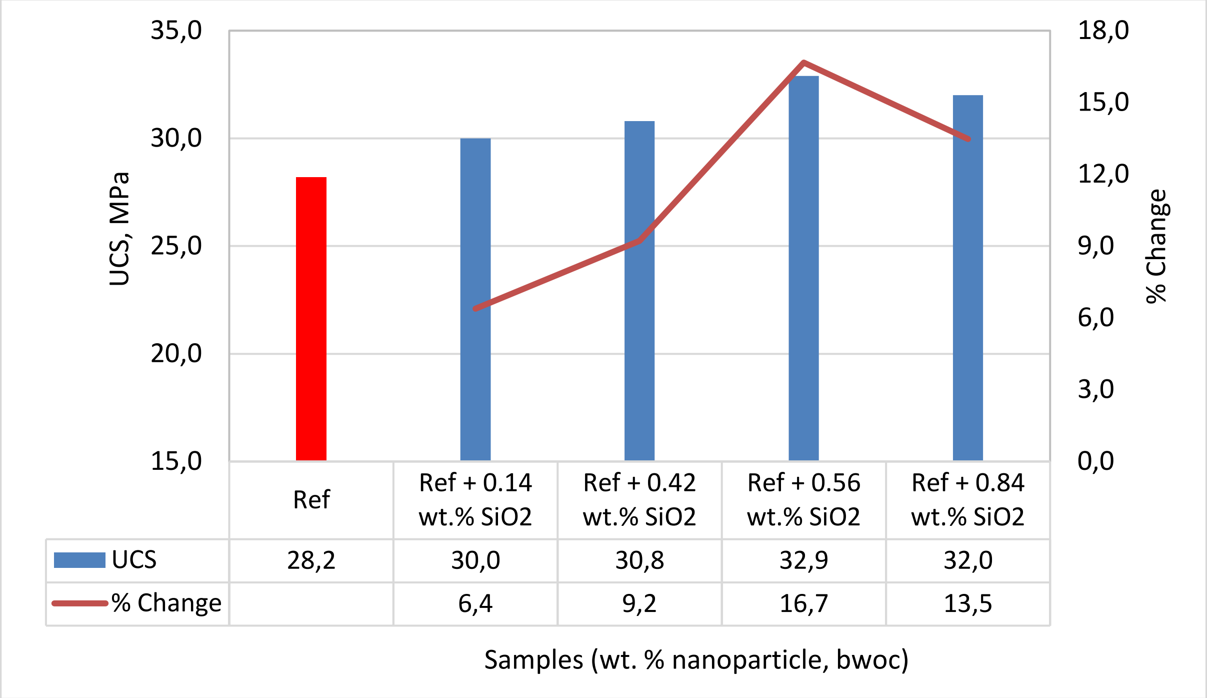
Plug (#)	Freshwater (g)	Cement (g)	SiO_2(aq) (g) (% bwoc)	TiO_2 (aq) (g) (% bwoc)
1	100	227.27	(0.0 g) (0.0)	(0.0) (0.0)
2	99.3	227.27	(0.6 g) (0.264)	(0.1) (0.044)
3	99.2	227.27	(0.6g) (0.264)	(0.2) (0.088)
4	99.1	227.27	(0.6 g) (0.264)	(0.3) (0.132)

3 Results

This section presents the experimental test results obtained from the three test designs. Both the destructive and non-destructive results of the nanoparticle blended slurries are compared with the nano-free neat cement. The results reported here are the average values of tested samples that have been cured in air at room temperature and pressure for 28 days. The standard deviation of the measured dataset has been calculated and, in most cases, varies from samples to sample. For instance, for the C-class cement, it varies in the range of 0.09 -2.8 MPa. The higher deviation was due to the defects on the specimen and when molding the slurry, it might contain trapped air in the system. Another observation was that some of the samples had imperfect flat surface, where plugs experiences non-uniform loading. As a result, a point load causes early breakdown. However, most of the test samples showed relatively closer measured datasets. The test results trend documented in this paper is quite similar to the one reported in literatures.

3.1 Effect of SiO_2 on uniaxial compressive strength of C-class cement

Portland G-class cement is the commonly used oil well cement for well construction and plug and abandonment. C-class cement is used on the top section of the wellbore with the objective of providing strong structural integrity. Figure 2 shows the effect of SiO_2 on the uniaxial compressive strength of the C-class cement. Results shows that among the considered SiO_2 nanoparticles concentrations, the 0.56 %bwoc increased the UCS of the neat cement by about 17%.

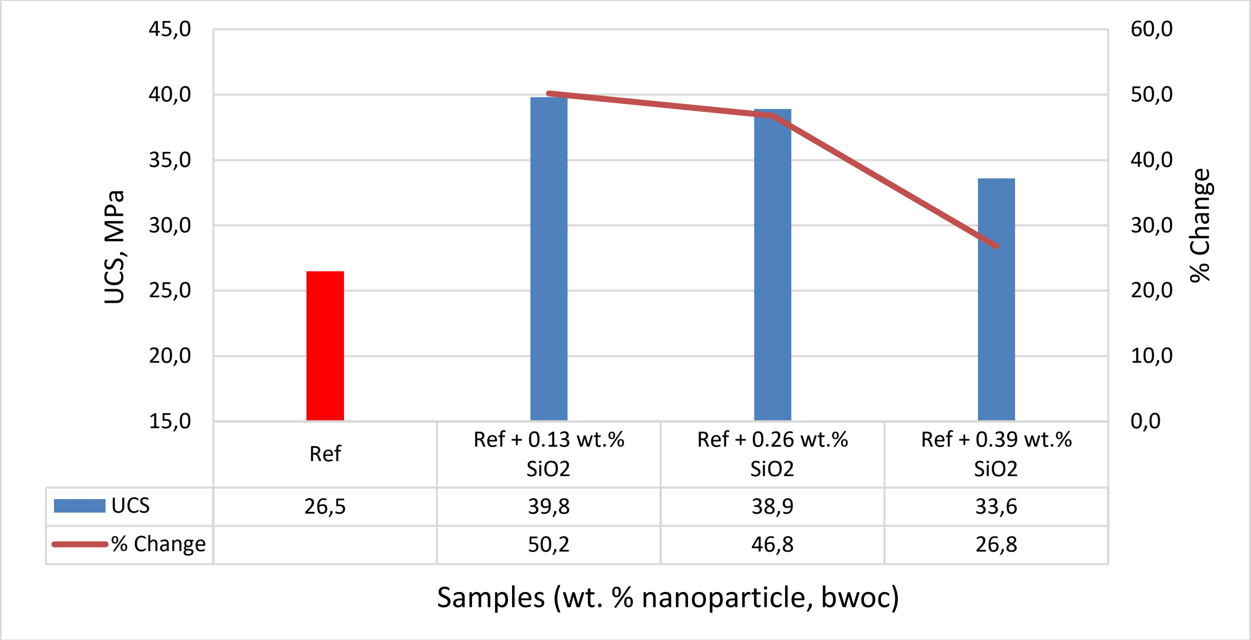


	Ref	Ref + 0.14 wt.% SiO2	Ref + 0.42 wt.% SiO2	Ref + 0.56 wt.% SiO2	Ref + 0.84 wt.% SiO2
UCS	28,2	30,0	30,8	32,9	32,0
% Change		6,4	9,2	16,7	13,5

Figure 2. Effect of SiO_2 on the uniaxial compressive strength of C-class cement

3.2 Effect of SiO_2 on Uniaxial Compressive Strength of Environmental Cement

Figure 3 shows the effect of SiO_2 on the UCS of the environmental neat cement. Results show that as the nanoparticle increases from 0.13wt%, 0.26wt%, and 0.39wt% SiO_2 bwoc, the UCS increase by 50%, 47%, and 27% respectively relative to the reference. Both the neat C-class cement and the environmental cement exhibited nearly equivalent UCS for the considered water cement ratio. Nevertheless, the impact of SiO_2 is very significant in the environmental cement than in the C-class cement. The different effects are due to the different chemical compositions of the cements and their interactions with the SiO_2 nanoparticle.



	Ref	Ref + 0.13 wt.% SiO2	Ref + 0.26 wt.% SiO2	Ref + 0.39 wt.% SiO2
UCS	26,5	39,8	38,9	33,6
% Change		50,2	46,8	26,8

Figure 3. Effect of SiO_2 on the uniaxial compressive strength of environmental cement

3.3 Effect of SiO_2 - TiO_2 on uniaxial compressive strength of Portland cement

The nano blending was formulated by fixing 0.264% SiO_2 bwoc concentration and varying the concentration of TiO_2 as 0.044%, 0.088, and 0.132% bwoc. As displayed in figure 4, results relative to the reference neat cement show that as the titanium oxide increases from 0.044wt%, 0.088wt%, and 0.132wt% TiO_2 bwoc, the UCS increase by 9%, 8%, and reduced by 0.1%, respectively.

Please note that the observations are at 28 cuing days. In the three test designs, one clear observation is that the cements blended with a higher nanoparticle concentration might have degraded the cement-cement bonding or slower down the hydration process. By extending the curing days, changing temperature and pressure, one may achieve different results. The reason for the performances could be explained through element and internal structure analysis. This is considered as a future work.

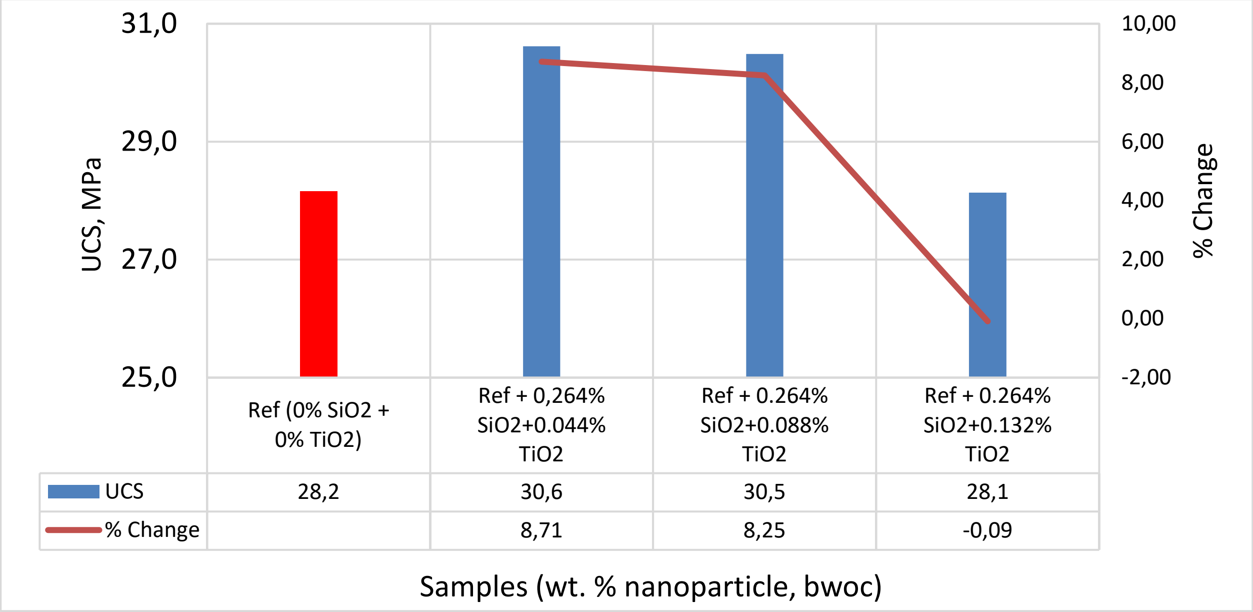


Figure 4. Effect of SiO_2 + TiO_2 hybrid on the uniaxial compressive strength of G-class cement

3.4 Effect of nanoparticles on viscosity of cement slurries

The pumpability of cement slurries is one of the parameters to be in consideration for the cement placement in an oil well. The flow of cement in the well is controlled by the viscosities of the cement slurry. The effect of nanoparticles on the rheological properties of three cement types are compared with the nanoparticle untreated neat cement slurries. Except for the C-class cement, the nanoparticle concentrations in the other two cement slurries were selected based on the highest uniaxial compressive strength. On the other hand, the viscosity of the C-class cement blended with the optimal nanoparticle concentration for the highest UCS (i.e., 0.56 % SiO_2 bwoc) has shown that the reading was outside the measurement scale and found out to be very viscous. For this reason, we used the lowest concentration (i.e., 0.14 % bwoc) for the viscosity evaluation purpose. Table 4 provides the calculated yield stress and plastic viscosity for the neat and nano treated cements. For all the cement types, nanoparticles reduced the viscosities and hence reduces the flow resistances for the cement placement job.

Table 4. Casson yield stresses and Casson plastic viscosities

Temperature (^{0}C)	Neat C-class	Neat C-class+ 0.14% SiO_2	Neat Environmental	Neat Environmental +0.13% SiO_2	Neat G-class	Neat G-class + 26% SiO_2 + 0.044% TiO_2
Yield stress (Pa)	4.0	7.7	10.1	8.0	5.7	5.5
Plastic viscosity (cP)	138.2	131.6	97.7	75.8	163.4	148.1

3.5 Effect of Nanoparticles on the heat development of cement slurries

Heat is liberated (i.e., exothermic reaction) when cement mixes with water. Based on the best UCS results obtained from the nanoparticles, the heat development phenomenon in the three cement types is measured comparing with the neat nano-free cements. It can be observed from Figure 5 that the peak temperature for the neat industry cement is measured to be 70.5ºC and the nano treated cement recorded 65 ºC. As shown in Figure 6, the nanoparticles increased the peak temperature of the environmental neat cement by 4ºC. On the other hand, as displayed in Figure 7**,** the considered SiO_2 /TiO_2 hybrid nanoparticles did not show any impact on the peak temperature of the neat G-class cement. However, one can observe the temperature lag between the nano-free and the nano-blending cement systems, where the temperature development in the neat system exhibited about 4ºC faster during the early peak temperature development phases. It is important to note that the results presented here are valid only for the considered nanoparticle concentrations.

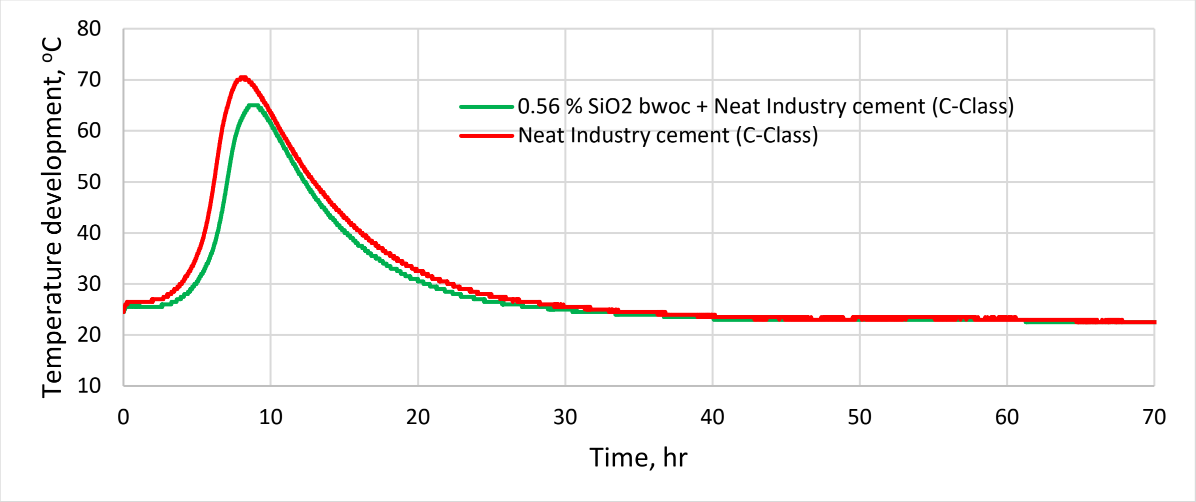


Figure 5. Temperature development in the neat-and 0.56% SiO_2 blended C-class cement

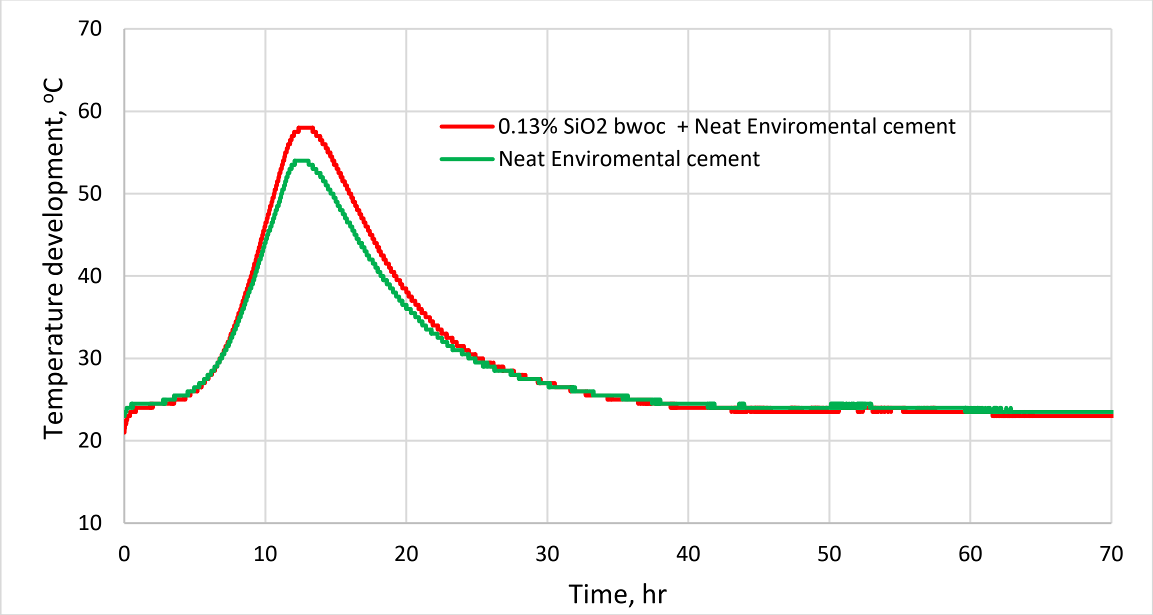


Figure 6. Temperature development in the neat and 0.13% SiO_2 mixed environmental cement

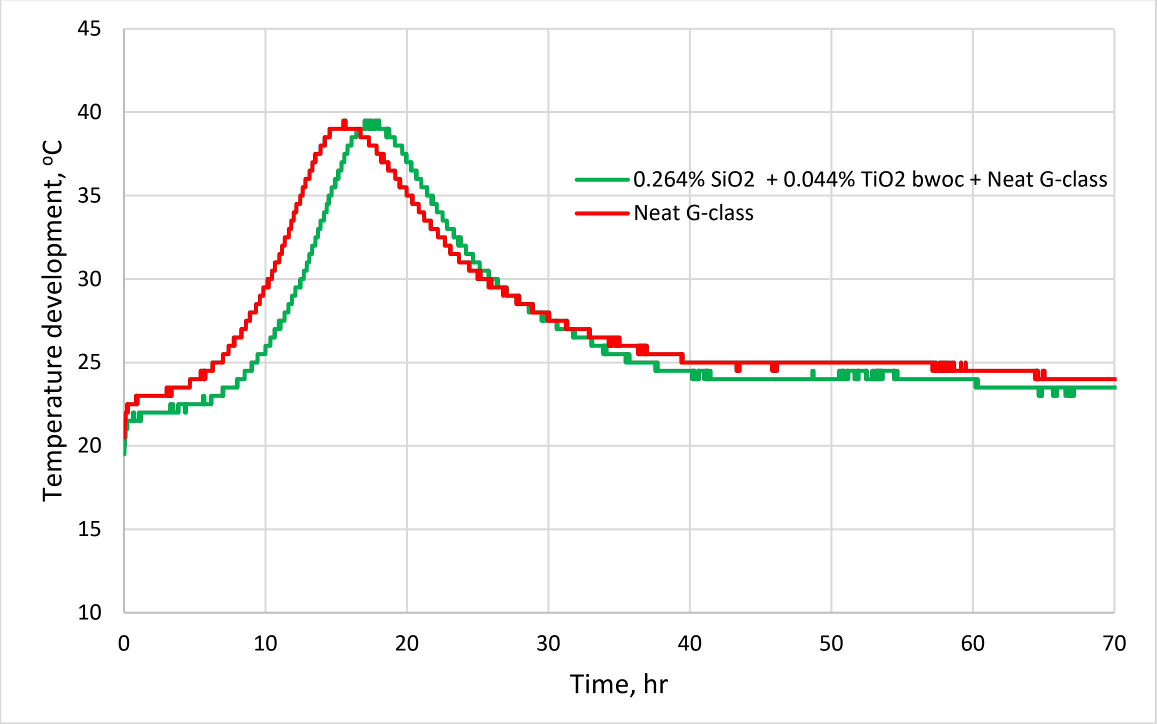


Figure 7. Temperature development in the neat and 0.264% SiO_2 + 0.044% TiO_2 blended G-class cement

4 Modelling and testing

An empirical model was derived by coupling the destructive (UCS) with the non-destructive (compressional wave velocity) data. Similar, empirical models are available in literature, which have been developed based on cementitious and rock-based dataset.

4.1 Modelling

During the process of empirical model development, the average values of the UCS and the compressional wave velocities of the plugs were used. The velocity data was measured the day when the cement plugs were tested with mechanical destructive test. The 7-and 28 days C-class and environmental cement-based plugs data were considered for the modelling. Figure 8 shows the power law model with R^2 =0.8334. The model reads:

$$UCS = 0.1255V_p^{4.4748} \tag{4}$$

where UCS (MPa) and V_p is the compressional wave velocity (km/s)

The model prediction of the newly developed model is compared with Horsrud’s (2001) [26] empirical model, which was derived from North Sea shale. The Horsrud´s UCS –V_p model reads:

$$UCS = 0.77V_p^{2.93} \tag{5}$$

where UCS (MPa) and Vp (km/s)

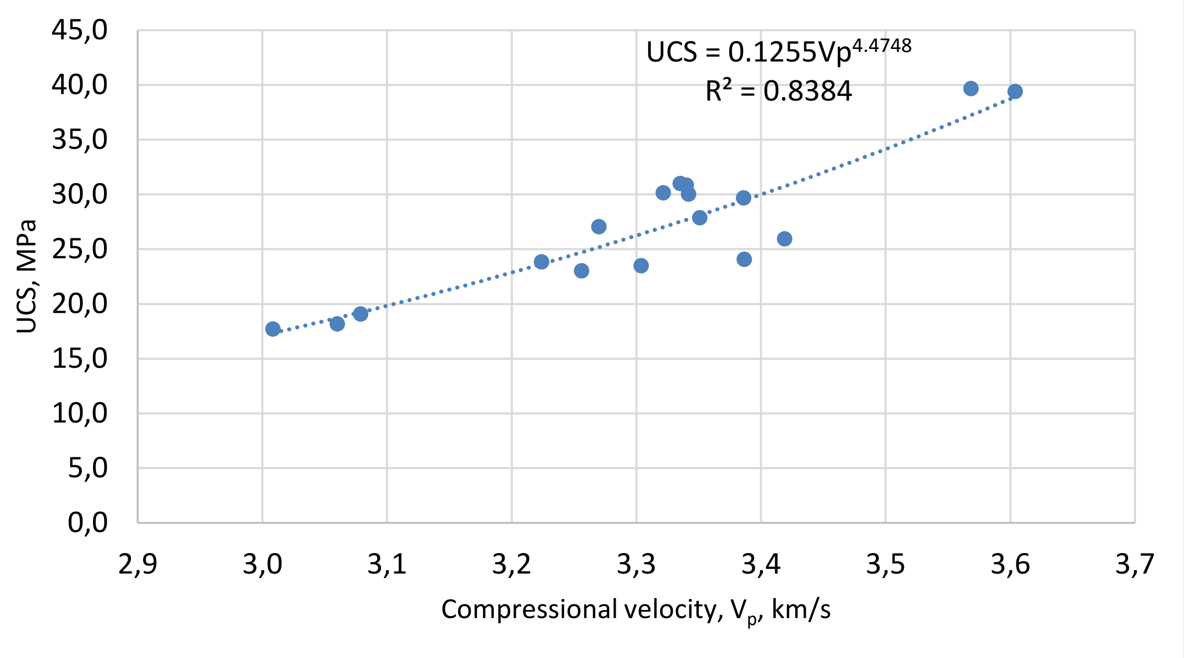
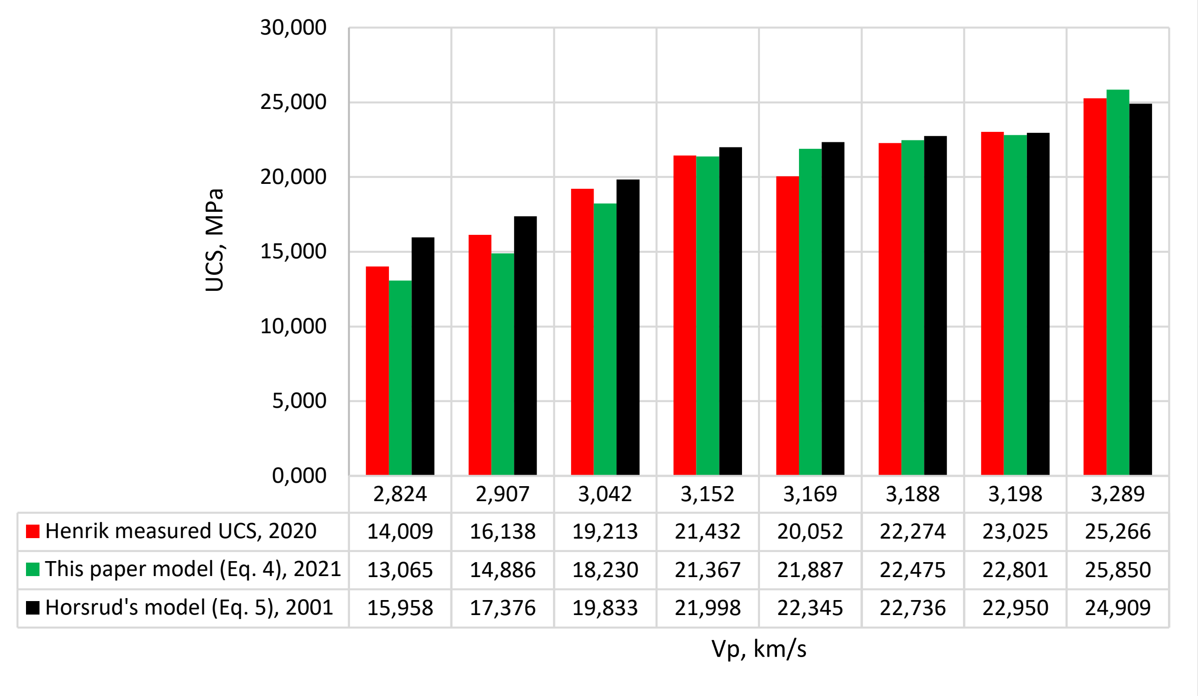


Figure 8. UCS – Vp empirical model

4.2 Testing

This paperwork model (equation (4)) and the Horsrud's model (equation (5)) prediction were tested against the experimental dataset measured by Henrik Nerhus [27]. The datasets are different nanoparticles based G-class cement plug specimens. As displayed in Figure 9, the percentile deviation of the measurement from this paper's model shows in range of 0.3-9.2% and the Horsrud´s model deviation is in the range of 0.3-13.9%.



	2,824	2,907	3,042	3,152	3,169	3,188	3,198	3,289
🟥 Henrik measured UCS, 2020	14,009	16,138	19,213	21,432	20,052	22,274	23,025	25,266
🟩 This paper model (Eq. 4), 2021	13,065	14,886	18,230	21,367	21,887	22,475	22,801	25,850
⬛ Horsrud's model (Eq. 5), 2001	15,958	17,376	19,833	21,998	22,345	22,736	22,950	24,909

Figure 9. This paperwork and Horsrud's models prediction of Henrik´s dataset

5. Summary

The desire of the NORSOK D-010 standard for the cement is to have a long term well barrier performance. However, well integrity survey results indicated that the conventional cement has shown integrity issue. With the objective of improving the C-class, environmental, and G-class cement properties, this paper experimentally investigated the impact of SiO_2 and the hybrid SiO_2 /TiO_2 nanoparticles on the neat cements.

Results from the study are summarized as:

- The impact of nanoparticles on the neat cements (C-class, environmental, and G-class) is not a linear function of the nanoparticle concentration. As the concentration nanoparticles reaches to the optimal value, a desired cement property can be obtained.
- Based on the mechanical and elastic properties, the 0.56 % and 0.13 % SiO_2 bwoc were found to be the optimal concentration for the C-Class and for the environmental cements, respectively.
- It is observed that SiO_2 nanoparticle has shown a significant impact on the UCS of the environmental cement.
- The optimum SiO_2 nanoparticle concentrations reduced the peak temperature developments of the neat C-class and the environmental cement. The hybrid SiO_2/TiO_2 did not show any impact on the neat G-class cement. However, by changing the concentration of the nanoparticles and water cement ratio one may achieve different results.

Please note that the results presented in this paper are valid for the considered curing temperature and pressure. In general, the results reported in this paper and documented in literature indicate the huge potential of nanoparticles to improve the conventional cement properties and enhance structural integrity of construction works. However, up to this level of research, the mechanisms for the performance of the nanoparticles in the cements are not investigated yet. As the future work, element analysis and scan electron microscopic analysis of the cement plugs might reveal the internal structure of the cements as well as other phenomenon that promote the hydration processes.

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