Effect of various testing parameters on the experimental evaluation of oil well cement cured under simulated downhole conditions

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Abstract. It is well known that regular oil well cement systems experiences strength retrogression at curing temperatures above 110°C and silica sand is typically added to mitigate strength reduction. However, due to the complex experimental procedures associated with high temperature and high pressure curing of oil well cement specimens in the laboratory, historical test data tend to suffer significant uncertainties. A comprehensive study of the various influencing factors on the physical and mechanical property test results of oil well cement system cured under 200°C is conducted here. The specific influencing factors investigated include curing time (from 14d to 69d), depressurization rate, curing pressure (50MPa vs 150MPa), specimen size (diameter of 25mm vs 50mm) and curing autoclave design. The property of the set cement evaluated include strength and modulus, permeability, indentation modulus and hardness, etc. Test results suggest bigger specimen size and faster depressurization rate may lead to significant specimen damage, causing experimental artifact. Different autoclave design may also lead to significant differences in the mechanical property of the set cement even under the same curing temperature and pressure.

1. Introduction

Oil well cement is a special type of Portland cement. The most commonly used oil well cement are high-sulfate-resistant grade Class G and Class H cement, whose main differences from ordinary Portland cement (OPC) are their low content of tricalcium aluminate (C₃A) [1]. Similar to OPC, the hydration products of oil well cements at low temperatures (<100°C) are mainly C-S-H gel and Ca (OH)₂. These hydration products have a compact structure and good mechanical properties. At temperatures above 110°C, C-S-H gel is gradually converted to crystalline phases such as α-C₂SH, causing the mechanical properties of the set cement to degrade and this phenomenon is known as strength retrogression. Silica flour is mixed with oil well cement to decrease the calcium oxide to silicon dioxide (Ca/Si) ratio of the mixture, which is desirable for the formation of stable non-retrogressing phases at high temperatures, such as 11 Å tobermorite and xonotlite [2-4].

Due to the need to simulate the high pressure and high temperature (HPHT) condition in the downhole environment of oil wells, the preparation of set oil well cement specimens for property
evaluation is a highly complex process [1, 5]. It typically consists of the following main steps: 1) casting the cement slurry in curing molds; 2) put the slurry-filled molds in a HPHT autoclave; 3) ramp the temperature and pressure of the autoclave to target values in a pre-specified period of time; 4) hold the temperature and pressure constant for the duration of the test; 5) ramp the temperature and pressure of the autoclave to the ambient condition; 6) take out the specimens and remove the molds. Sometimes, in order to simplify the experimental procedures or to simulate field condition of steam injection wells, the cement may be allowed to set outside of the autoclave at lower temperatures and demolded before being subjected to HPHT curing. These complicated specimen preparation procedures can bring significant uncertainties in the experimental results, which may subsequently lead to experimental artifact. For example, it is very well known that the depressurization rate employed at the end of the HPHT curing has a very important impact on the integrity of the specimens [6]. To further complicate the situation, the severity of such effect may be dependent on cement mixture composition and specimen size [7]. In extreme cases, the specimens could be completely disintegrated upon taking out of the molds [6]. Iverson [8, 9] et al. performed a comprehensive study regarding the influences of silica dosage (40% to 80%), curing temperatures (177°C to 343°C) and curing time (14 d to 180 d) on the physical and mechanical properties of oil well cement formulations. However, test data were found to be highly scattered and no obvious trend of strength change with silica dosage, curing temperature, or curing time could been found. Therefore, it is important to study the influences of various testing parameters on the experimental evaluation results of oil well cement.

Although oil well cement specimens are generally cured under simulated downhole environment, the evaluation of their physical and mechanical properties are typically performed at the ambient condition. In order to eliminate depressurization damage to the cement specimens as well as to obtain their “in-situ” properties under HPHT environment, significant efforts have been made to develop customized testing equipment that may cure and test oil well cement mixtures within the same device [10, 11]. However, until now, the only commercialized in-situ test device of such type is based on measurement of ultrasonic transit time (i.e. the Ultrasonic Cement Analyzer, or UCA) [12, 13]. The advantage of UCA test is its ability to evaluate the physical property of cement continuously under HPHT environment, while its disadvantage is the measured acoustic property can only be used to calculate compressive based on empirical equations, which can be highly inaccurate. Reddy [12] et al. conducted UCA tests on oil well cement cured under HPHT conditions and found that the heating rate during curing had a great influence on the sonic strength development of set cement. As the employment of different cement curing chambers may lead to differences in heating rate, their influences on the properties of set cement should also be studied.

Unlike curing temperature, curing pressure is generally believed to have only a minor impact on the mechanical property development of oil well cement systems. It is believed that curing pressure will not change the chemical composition of cement hydration products and hence its effect on the mechanical properties is primarily from compaction. Early research [14] has showed that increasing curing pressure up to 13.8MPa would improve the shorter term compressive strength of cement, while further increases in curing pressure has little effects. More recently, it has been found that curing pressure has qualitatively similar, but quantitatively much smaller effects than curing temperature in terms of accelerating the hydration reaction rate of oil well cement [15-19]. The influences of curing pressure in a much higher pressure range (50MPa-150MPa) will be studied here to improve our understanding in this area.

In summary, due to the lack of sufficient experimental data as well as the complex influencing factors on experimental results, the long-term performance of silica-enriched oil well cement systems is not fully understood. The goal of this study is to investigate the influences of various testing parameters on the physical and mechanical performance of oil well cement systems. The testing parameters studied include depressurization rate, temperature rising rate, specimen size, curing pressure, and curing time. Several previously optimized cement formulation with 70%-90% by weight of cement (bwoc) total silica addition were cured for different durations varying from 14 d to 69 d. The specimens were directly cured in HPHT chambers with molds to simulate the cementing of ultra-
deep wells and geothermal wells. The properties evaluated include compressive strength, elastic modulus, Poisson’s ratio and permeability.

2. Experimental procedure

2.1. Raw materials

Aksu Class G oil well cement (3.127 g/cm³, D50 19.83 μm), silica fume (2.332 g/cm³, D50 0.37 μm), fine silica flour (2.626 g/cm³, D50 15.52 μm) and coarse silica flour (2.666 g/cm³, D50 53.06 μm) were provided by Tarim Oilfield Company. The specific gravities of the powder materials were tested by a pycnometer (model UltraPYC 1200e) from Quantachrome Instrument, USA. The particle size of powder materials was analyzed by a laser particle analyzer (Malvern Mastersizer 2000). The D50 (i.e. median particle size) of cement and silica flour are of the same order of magnitude, while that of silica fume is 1-2 orders of magnitude lower. Silica fume is known to accelerate the process of cement hydration and enhance the compactness of set cement [20]. X-ray fluorescence (XRF) spectrum analysis results of dry cement and silica fume was shown in Table 1. Retarder (BCR-300L), fluid loss reducing agent (BXF-200L), dispersant (BCD-210L), defoaming agent (G603), high temperature suspension agent (BDJ-300S) and other oil well cement admixtures were obtained from Tianjin PetroChina Boxing Technology Co., Ltd.

| Oxide | Al₂O₃ | CaO | Fe₂O₃ | K₂O | MgO | Na₂O | SO₃ | SiO₂ | Free lime |
|-------|-------|-----|-------|-----|-----|------|-----|------|-----------|
| Silica fume | 0.297 | 0.597 | 0.162 | 0.776 | 0.322 | 0.268 | 0.578 | 96.697 | N/A |
| Cement | 2.985 | 65.134 | 6.675 | 0.805 | 1.962 | 0.179 | 3.15 | 18.452 | 1.65 |

2.2. Mixture proportion and preparation of samples

Four different cement formulations were designed as shown in Table 2. The dosages of different components are expressed in % by weight of cement (bwoc). The preparation of cement sample consists of four steps: mold preparation, slurry mixing, HPHT curing in water-saturated condition, specimen cutting and grinding. All final test samples were 25×50mm cylinders except for the test series that studies the influences of specimen size on experimental results.

| Formula | Cement | fine silica | coarse silica | Silica fume | BCJ-300L | BCD-210L | BCR-300L | Water |
|---------|--------|------------|---------------|-----------|---------|---------|---------|-------|
| Z1      | 100    | 15         | 50            | 5         | 4       | 5.5     | 5       | 49.6  |
| Z2      | 100    | 30         | 40            | 0         | 4       | 5.5     | 4.5     | 50.6  |
| Z3      | 100    | 40         | 40            | 0         | 3.75    | 6       | 4       | 54.14 |
| Z4      | 100    | 50         | 40            | 0         | 3.5     | 6.5     | 4       | 57.01 |

*The dosages of BXF-200L and G603 in each formulation are 6% bwoc and 0.5% bwoc, respectively.

2.3. Test methods

(1) Mechanical property test

UTM5105X microcomputer-controlled electronic universal testing machine from the Shenzhen Sansi Zongheng Company was used for compressive strength test. The testing machine was equipped with a video extensometer (MercuryRT series) from Sobriety S.R.O., Czech Republic, which can simultaneously measure the horizontal and vertical deformation of the sample for calculating Young’s modulus and Poisson’s ratio.

(2) Permeability test

For water permeability test, samples taken from the curing autoclave were directly used. The applied confining pressure was 5MPa while the applied pressure differential along the sample was about 1.5-2 MPa. The water permeability was calculated according to Formula 1. For gas permeability
test, the specimens were dried by vacuum in a freezer dryer at room temperature for one week before testing. Gas permeability of each specimen was measured at four different upstream pressures (0.5-2 MPa) with a constant confining pressure of 4 MPa and calculated using Formula 2. Klinkenberg gas permeability was obtained as the intercept of the plot of gas permeability as a function of the inverse of average applied pressure, i.e. \( \frac{1}{P_1+P_0} \).

\[
K_g = \frac{Q_0 \mu_g L}{A \Delta P} \times 1000
\]

(1)

\[
K_w = \frac{Q_0 \mu_w L}{A \Delta P} \times 1000
\]

(2)

where \( K_g \) is the gas permeability, mD; \( P_1 \) is the inlet pressure for gas permeability measurement, atm; \( P_0 \) is atmospheric pressure, which is 1 atm; \( \mu_g \) is the nitrogen gas viscosity, 0.0178 mPa\( \cdot \)s; \( Q_0 \) is the volume flow of gas at atmospheric pressure, cm\( ^3 \)/s; \( L \) is the sample length, cm; \( A \) is the sample cross-sectional area, cm\(^2\); \( K_w \) is the water permeability, mD; \( Q \) is the water flow rate, cm\(^3\)/s, \( \Delta P \) is the pressure difference for water permeability test, atm; \( \mu_w \) is the water viscosity, 1 mPa\( \cdot \)s.

3) Nanoindentation test

A Bruker TI Premier nano-indentation instrument with a pressure range of 1-10000\( \mu \)N and an indentation depth of 0-3\( \mu \)m was used for mico-mechanics evaluation of selected specimens. A small piece of sample was impregnated with epoxy resin and subsequently grinded and polished using an automatic grinder-polisher. All tests were programmed in load control mode such that the indenter came into contact with the sample surface with a maximum load of 3500 \( \mu \)N. A total of 400 indents (a matrix of 20 \( \times \) 20) were conducted per specimen. The spacing between the indents was 10 \( \mu \)m in both directions, resulting in a total indentation area of 200 \( \times \) 200 \( \mu \)m.

3. Results and discussion

3.1. Influence of curing time on experimental results

In the first test series, two HPHT autoclaves from Chandler Engineering (Model 1910) were used to cure the four slurries listed in Table 2 for durations of 14 d and 69 d, respectively. The temperature and pressure profiles during the initial ramp-up are shown in Figure 1 (a). It took approximately 4 hours for the autoclaves to reach the target temperature of 200\( ^\circ \)C and pressure of 150MPa. The temperature profiles of the two tests were almost overlapped, but the pressure profiles deviated between 150min to 250min due to pressure fluctuations of the 14-day-cure test. At the end of the curing period, the autoclaves were switched off to allow depressurization during the process of natural cooling, which took about 1-2 days.

![Figure 1. Influence of curing time on experimental results (200°C and 150MPa) (a) Temperature and pressure profile during ramping up (b) Mechanical properties.](image)

The compressive strength and elastic modulus test results of the four formulations cured for 14-day and 69-day are shown in Figure 1(b). It can be seen that with the increase of curing time, the compressive strengths of slurry Z1 and Z2 were little changed, while those of slurry Z3 and Z4
declined significantly (about 41%). The Young’s modulus of slurry Z1, Z3 and Z4 apparently decreased over time (about 19-28%) while that of Slurry Z2 slightly increased (~10%). The results also indicate that increasing the dosage of fine silica from 30% to 50% significantly increased the compressive strength and Young’s modulus of the set cement during the relatively early age of 14 d, but this also resulted in more severe strength retrogression in the long-term. Slurry Z2 seems to be the optimal formulation for long-term strength stability.

Table 3. Water / Gas permeability of Z1 to Z4.

|        | Z1      | Z2      | Z3      | Z4      |
|--------|---------|---------|---------|---------|
| 14 d Water permeability (mD) | 0.00091 | 0.00130 | 0.00091 | 0.00090 |
| 14 d Gas permeability (mD)   | 0.01900 | 0.01200 | 0.02000 | 0.02500 |
| 69 d Water permeability (mD) | 0.00260 | 0.00150 | 0.00170 | 0.00170 |
| 69 d Gas permeability (mD)   | 0.03300 | 0.01900 | 0.01800 | 0.01400 |

Permeability test results of the four formulations are shown in Table 3. Each test result is the average of two specimens. Overall, the repeatability/consistency of both gas and water permeability test results are very good. At 14 d, the water permeability test results showed very little dependence on slurry composition (approximately 0.001 mD for all formulations). At 69 d, all formulations showed increased water permeability, with slurry Z1 being the most dramatic and slurry Z2 being the most stable. The gas permeability of all formulations is an order of magnitude higher than the water permeability. This is due to the coarsening effect of the cement microstructure during drying [21].

3.2. Influence of depressurization rate on experimental results
In the second test series, the curing autoclave used was the same as those in the first test series (i.e. Chandler Engineering, Model 1910). The temperature and pressure profiles during the ramp-up period were also similar to the previous tests with a curing period of 14 d. In order to study the effect of the depressurization speed on the experimental test results of the set cement, the temperature and pressure were slowly reduced over a period of 200 hours (or 8 days) by manual control, as shown in Figure 2(a). Figure 2(b) compares the test results of slurries Z1 and Z2 cured for the same duration of 14 days at 200°C and 150MPa, but with different depressurization schemes. Quick depressurization is achieved by natural cooling as described earlier. Comparing the test results, it can be seen that the specimens’ water permeability values are dramatically reduced with slow depressurization (65% reduction for slurry Z1 and 46% reduction for slurry Z2), possibly due to reduced specimen damage. At the same time, the compressive strengths are increased with slow depressurization. The amount of increase in compressive strength is 11% for slurry Z1 and 29% for slurry Z2. The increase in Young’s modulus and Poisson’s ratio of slurry Z2 appear to be consistent with compressive strength. However, no statistically meaningful change is observed for the Young’s modulus and Poisson’s ratio of slurry Z1. As discussed earlier, quick depressurization may lead to destruction of the sample, which will affect the test results. Test results obtained here seem to suggest that the severity of property deterioration associated with depressurization may also depend on the cement composition.

3.3. Influence of curing pressure on experimental results
In the third test series, the test procedure was similar to the second test series with the exception that the maximum pressure was reduced to 50MPa to study the influence of curing pressure on experimental results. The manual depressurization took about 80 hours, while the temperature reduction scheme were kept the same as the second test series (Figure 3(a)). It can be seen from Figure 3(b) that, with the increase of the curing pressure, the mechanical property of slurry Z1 is apparently increased possibly due to the compaction effect with higher pressure while the mechanical property of slurry Z2 is little changed. Similarly, the water permeability of slurry Z1 is significantly reduced with increasing curing pressure, while that of slurry Z2 is almost constant. These results indicate that the
curing pressure has a greater influence on the formulation with silica fume, but had little effect on the formulation without silica fume.

Figure 2. Influence of depressurization rate on experimental results (200°C/150MPa, 14d cure) (a) Temperature and pressure profiles during ramping down (b) Physical and mechanical properties.

Figure 3. Influence of curing pressure on experimental results (200°C, 14d cure) (a) Temperature and pressure profiles during ramping down (b) Physical and mechanical properties.

3.4. Influence of specimen size on experimental results
Cement specimen damage during depressurization is primarily caused by fast release of pore pressure. The transport path of pore water is strongly dependent on specimen size. Theoretically, for the same depressurization rate, larger specimens would be more susceptible to damage due to longer pore water transport path, which lead to longer equilibrium time and hence larger pressure differential. In the previous two test series, specimens of two different sizes were produced (diameter × height): 25×50mm and 50×100mm. Test results of the smaller specimens were reported previously in section 3.3. The influences of specimen size on compressive strength are shown in Figure 4. Under the curing pressure of 50 MPa, the compressive strength of the big samples was much less than that of the small samples (reduced 48%-64%). However, under the curing pressure of 150MPa, the compressive strength of the two size specimens are relatively close, with a difference of about 15%.

In order to further investigate the causes of strength differences between big and small samples, nano indentation method was used to analyze the micromechanic properties. The test data are presented in Figure 5. For the big sample, the average elastic modulus is 17.35 GPa and the average hardness is 0.70 GPa; for the small sample, the average elastic modulus is 13.19 GPa and the average hardness is 0.45 GPa. Since the elastic modulus and hardness of the big sample on the microscopic scale is higher than those of the small sample (which may be attributed to inhomogeneity of the material), its mechanical property degradation on the macroscopic scale is unlikely to be caused by changes of hydration products. It is suspected that the cracking during depressurization caused the
macroscopic property degradation of the big samples. Therefore, using small specimens can potentially help reduce the risk of cracking during the pressure release period after HPHT curing.

![Figure 4. Comparison of 14-day test results of different size samples.](image)

3.5. Influence of curing autoclave on experimental results
In the final test series, an additional HPHT autoclave from Jiangsu Hongbo Manufacturing Company was employed to study the influence of different curing chambers on experimental results. The main difference between the curing chambers is the location of the heating elements: they are located inside the curing chamber directly exposed to curing water in Chandler Engineering autoclaves, and outside of the curing chamber in direct contact with the chamber external wall for Jiangsu Hongbo autoclaves. The different design methodology lead to different temperature and pressure profiles during the ramp-up and ramp-down period (See Figure 6), where temperature was measured inside the curing chamber in both cases. Additionally, the pressure of Jiangsu Hongbo autoclaves was controlled by fully automatic syringe pumps, which allows much more precise control during both curing and ramping periods. Slurry Z1 and Z2 was cured in the two different types of autoclave under the condition of 200°C and 50 MPa for a period of 14 d. Test results are shown in Figure 7. There was almost no difference in the water permeability test results for specimens produced by different autoclaves. However, the compressive strength and Young’s modulus of specimens produced by the Jiangsu Hongbo autoclave are significantly higher than those produced by the Chandler autoclave. These results suggest that the curing chamber design has a strong influence on the experimental results, possibly due to the different temperature and pressure profiles. As discussed earlier, Reddy et al. [12] had previously reported that heating rate during initial temperature ramp had a strong influence on the sonic strength development of oil well cement under HPHT conditions.

![Figure 5. Nanoindentation test results of slurry Z2 (a) elastic of modulus (b) hardness.](image)
Figure 6. Temperature and pressure profiles during ramp-up and ramp-down of different autoclaves

Figure 7. Influence of curing autoclave on experimental results (200°C and 50MPa, 14d)

4. Conclusions

Five influence factors on experimental results of oil well cement cured under high temperature and high pressure condition were studied in this article. The main conclusions are as follows:

(1) The long-term strength retrogression of oil well cement is dependent on the formulation design. Slurry Z2 of this study is much more stable than the other formulations during the curing period from 14d to 69d.

(2) Quick depressurization after high pressure curing will lead to specimen damage, which can significantly increase its water permeability; decreases in mechanical property is also observed in certain formulation. Slow depressurization method is preferred to improve the quality of the samples.

(3) Increasing curing pressure (from 50MPa to 150MPa) had little effect on the permeability and mechanical property test results of the formulation without silica fume, but improved the properties of the formulation with silica fume.

(4) For the same depressurization rate, bigger-size specimens are much more susceptible to damage than smaller-size specimens. The specimen damage can lead to significant deterioration in macroscopic mechanical properties, but no influence on microscopic mechanical properties.

(5) The curing chamber design can have a significant influence on the experimental results possibly because of the different temperature and pressure profiles. Therefore, it is critical to use the same type of curing chamber for the same test series.

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