Study of effects of downhole conditions on the setting time and compressive strength of MOS settable system by orthogonal experimental design

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Abstract. Lost circulations of drilling fluids during drilling and completion can be an expensive and time-consuming problem. Commonly used lost circulation materials (LCMs) like nutshells or cross-linked polymer gels can’t be easily removed after plugging with a risk of formation damage. Magnesium oxysulfate cement (MOS cement) provides a reliable solution for solving the severe loss that occurs in production and non-production zones. In this paper, the effects of harsh downhole conditions including density, bentonite content, salt content, and temperature on the setting time and compressive strength of the MOS settable system are studied using the orthogonal experimental analysis method. At the same time, laboratory plugging experiments and field application are used to evaluate the ability of the MOS settable system to seal the severe loss. The results show that the downhole conditions have a significant effect on the setting time and compressive strength of the system. By adding calcium carbonate particles, the permeability of the MOS settable system can be reduced and the pressure-bearing capacity of the MOS solidified plug can be improved. Field application shows that this settable system can solve severe loss that conventional LCMs cannot solve and has a good application prospect.

1. Introduction

1.1. Lost circulation and control methods

Lost circulation refers to the phenomenon that various working fluids leak into the underground formation uncontrollably during drilling or workover operations. The lost circulation is different from the filtration loss of the drilling fluid filtrating into the permeable formation (like sandstones) under the positive pressure differential between the drilling fluid column pressure and formation pressure. The latter has a lower fluid velocity and therefore the loss volume is small, and the filtration process will cause the formation of mud cake on the well wall, which is beneficial to the wellbore stability. The former has a higher fluid velocity and a larger loss volume, and it will cause a drop in the drilling fluid level in the wellbore, which is likely to cause safety problems such as wellbore instability even a blowout.

There are many types of lost circulation, which can be roughly summarized into four types, namely, seepage losses (loss rate less than 10 bbl/h, ie, below 1.6 m³/h), partial losses (10~100 bbl/h, ie, 1.6 m³~16 m³/h), severe losses (more than 100bbl/h, ie, above 16 m³/h) and total losses (no turns to surface).
[1]. Seepage losses and partial losses usually occur in high permeability sandstone formations or formations pre-existing micro-fractures or including induced fractures. Severe losses and total losses usually occur in formations including large-fractures or karst caves.

In oil fields, there are usually two treatment methods for lost circulation. One is the method of adding LCMs into the drilling fluids while drilling, the other is the method of plugging after stopping the drilling [2]. The former is applied when the drilling engineer anticipates the seepage loss of the drilling fluids could occur in the formation to be drilled according to adjacent well experiences and add LCMs like calcium carbonates, elastic graphites or fibers with an appropriate particle size distribution (PSD) to the drilling fluids according to the 1/3 bridging theory [3] or other bridging theory [4, 5]. The larger pores or micro-fractures in the formation will be sealed with the mud cake and the added LCMs through forming a seal, thereby avoiding the loss of a large amount of drilling fluid, and preventing the hydraulic pressure from transmitting to the induced fracture tips to stop the fracture propagation according to the “stress cage” theory [6]. However, when the loss rate is too high, it is necessary to adopt the second plugging method-plugging after stopping drilling. A prepared plugging slurry consisting of cement or crosslinked polymer is squeezed into the loss layer to form a plugging pill so that the bearing capacity of the formation is improved and no repeated loss occurs when the drilling operation continues.

Although drilling fluid engineers have adopted various sealing techniques to solve the problem of lost circulation, the field application of oilfields shows that these materials still have major drawbacks. For example, the premise of a successful plugging for the high permeable or fractured formation by using particulate materials of a certain PSD is that the particle size of the materials and the mean pore diameter or fracture aperture of the formation meets certain principles of bridging behavior like “1/3 bridging theory” [3]. However, the exact value of the pore diameter or the fracture aperture of formation is unknown when the loss occurs, which leads to the blindness of the plugging operation and the low probability of success [7]. Organic crosslinked polymer gels can enter the loss zone by virtue of their deformability, however, this also causes problems that the drill bit cannot penetrate a large section of the organic chemical gel plug due to adhering to the drill bit. If the seal or the plug could not be removed completely, the formation damage would be more serious reducing the oil or gas production when the loss occurs in the production zones. Cement plugging is generally the last method of plugging adopted by the drilling fluid engineers. It has the huge advantage of plugging without knowing the exact size of loss channels. But it requires a special cement mixer vehicle and takes long duration (usually 2 to 3 days) until the cement is completely consolidated. This leads to a significant increase in the cost of plugging operations and causes formation damage to the production zones due to its low acid solubility.

1.2. MOS cement as LCM for severe loss

Acid-soluble cements (ASCs) have been developed to reduce the blockage of cement stone to the reservoir pores and fractures and to reduce the extent of formation damage. ASCs mainly include Portland cement adding acid soluble materials like calcium carbonate and Sorel cement [8, 9]. Sorel cement mainly includes three types: magnesium oxy-chloride cement (MOC cement), magnesium oxy-sulfate cement (MOS cement) and magnesium phosphate cement (MPC cement). MOS cement is a MgO-MgSO₄·H₂O ternary gelling system composed of a magnesium sulfate solution and a light-burned magnesium oxide powder and it can solidify into a certain shape (figure 1). MgO is easy to form layered Mg(OH)₂ through hydration, but Mg(OH)₂ has weak cementing ability due to its low the solubility product. By adding a blending agent, the formation of Mg(OH)₂ can be delayed because of the common ion effect and a complex salt (xMgO·yMg(OH)₂·zH₂O) can be formed to enhance the cementation strength. Because of its good fire resistance, wear resistance, corrosion resistance, good adhesion, fast setting and hardening and high early strength, MOS cement has been widely used in the manufacture of light-weight thermal insulation walls, refractory and decorative materials in the construction industry [10]. The MOS cement has a lower production temperature compares to the conventional silicate cement, which is approximately 800 ℃ and 1450 ℃, respectively. So it has the advantages of lower energy consumption, lower production equipment and related manufacturing processes.
Therefore, in order to avoid various drawbacks of the conventional LCMs, a new acid-soluble inorganic gel plugging agent based on the MOS cement has been developed to solve the severe to total loss through full sealing, and it has the characteristics of short curing time and high plugging strength. At the same time, the plugging agent can be dissolved by 15% hydrochloric acid, so that the backflow can be realized after sealing the production formation without formation damage. This newly developed MOS plugging system could help oilfield drilling fluid engineers achieve a fast, efficient and low-cost plugging operation.

1.3. Harsh downhole conditions and orthogonal experimental design

However, the use of MOS cements as LCMs for solving severe loss in wells is different from the use as building materials. It is necessary to consider the effects of downhole conditions on cement performance, especially the setting time and compressive strength. The main factors include loss zone the depth and temperature of loss zones, drilling fluid composition and properties.

In overbalanced drilling, the drilling fluids have a certain density range, and this range is within a safety density window to avoid borehole instabilities and to prevent formation fluid influx. The lower limit of the drilling fluid safety density window is determined by the larger of formation collapse pressure and formation pore pressure, and the upper limit is determined by the smaller of formation fracture pressure and formation lost-circulation pressure. The density of the drilling fluid can be adjusted by adding barite or other weighting materials such as calcium carbonate or hematite [11]. When a severe loss occurs, the plugging slurry needs to be adjusted to a suitable density range by adding weighting materials like barite. Therefore, it is necessary to consider the influence of the addition of barite on the properties of the MOS settable system, especially the setting time and compressive strength.

The drilling fluid also needs to have a certain viscosity and a certain yield stress to be able to suspend weighting materials and transport drill cuttings out of the hole. This is usually achieved by adding viscosity control agents; eg, clays such as bentonite, attapulgite, or sepiolite. When curing additives are added to the drilling fluid to convert it into a settable plugging slurry or the plugging slurry is pumped downhole to contact the drilling fluid in the wellbore, a certain amount of bentonite will be present in the slurry.

Another zone where losses are common is subsalt rubble zones [12]. When drilling into a salt zone, saltwater drilling fluid (sodium chloride or potassium chloride) is usually used for keeping the borehole stability. At this time, the drilling fluid has a high salt content. When formation water with high salt content exists in the loss channels, the plugging slurry is also susceptible to salt invasion. Therefore, the setting time and compressive strength of the plugging slurry may be affected by the bentonite and salt too.

Another factor that must be considered is the effect of downhole temperature. Formations at different depths have different temperatures, and usually the formation temperature rises linearly as
the depth of the formation increases. When the plugging slurry is pumped into the formation, it can be solidified under the excitation of the formation temperature.

Orthogonal experimental design refers to an experimental design method that studies multiple influencing factors and multiple numerical levels. According to the orthogonality, some representative points are selected from the comprehensive experiments for testing. These representative points have the characteristics of uniform dispersion and neat comparability. When the test involves three or more influencing factors, and there may be interactions between the factors, the test workload will become very large and even difficult to implement. In view of this problem, the orthogonal experiment design is undoubtedly a better choice. The main tool for the orthogonal experiment design is the orthogonal table. The experimenter can find the corresponding orthogonal table according to the requirements of the number of factors and levels, and whether there is interaction. Some representative points are selected for testing, which can achieve the equivalent results of a large number of comprehensive tests with the minimum number of tests. Therefore, the orthogonal table design test is an efficient, fast and economical multi-factor test design method.

The characteristic of the orthogonal table is that the arranged test method has the characteristics of balanced matching. In each column, different numbers appear equally. The arrangement of numbers in any two columns is complete and balanced. The above two points fully reflect the two advantages of the orthogonal table, that is, "uniform dispersion and neat comparison." Generally speaking, each level of each factor touches each level of another factor once, and this is orthogonality.

1.4. Calcium carbonate as a filler
In the preliminary experiment, it is found that although the MOS settable slurry has a good setting effect, it still had a high permeability, resulting in its plugging capacity less than 2 MPa. Therefore, the commonly used filling material calcium carbonate is used to increase the compactness, and the effect is verified by laboratory plugging experiments. Calcium carbonate with an average particle diameter of 5-10 μm is also called heavy calcium carbonate and it is widely used. Heavy calcium carbonate can play a skeleton role in plastic products and has a great effect on the dimensional stability of plastic products. It can also improve the hardness of products and improve the surface gloss and surface flatness of products. When used together with concrete, it not only reduces production costs but also increases the toughness and strength of the product in the construction industry.

The addition of heavy calcium carbonate to the gel plugging material can increase the structural compactness of the gel plugging material after solidification, enhance its toughness and strength, and greatly enhance its sealing pressure capacity in the loss zone. It also can help overcome the weakness of conventional plugging materials that are not completely sealed and weak in pressure bearing capacity.

Therefore, in this paper, a basic MOS gelling system composed of light-burned magnesia, magnesium sulfate heptahydrate, boric acid (retarder) and distilled water. In order to study the adaptability of this basic MOS gelling system to different harsh downhole environments, we investigated the density, sodium chloride content, bentonite content and temperatures on the final setting time and compressive strength through orthogonal design methods. Finally, we evaluated the plugging capacity of the basic MOS gelling system through laboratory tests and field tests for severe loss. The discussion of the experimental results will help to drill fluid engineers better understand the influence of various downhole factors on the properties of the MOS gelling plugging system and make more appropriate choices when a severe loss occurs.

2. Materials and methods

2.1. Materials
Light-burned magnesia powder (MgO, Industrial reagent, purity ≥87%) is purchased from Hebei Magnesium Chemical Technology Co. Ltd. (China). The impurities of light-burned MgO (PDF card no. 77-2364) include MgCO3 (PDF card no. 86-2344), SiO2 (PDF card no. 79-0563) and Mg3(OH)2Si4O10 (PDF card no. 13-2558) by X-Ray Diffraction (XRD) analysis (figure 1), and it has a mean particle size of 12.095 μm by Particle Size Analysis (PSA) (figure 3). Its activity is 53.7% tested
by the hydration method. Magnesium sulfate heptahydrate (MgSO\textsubscript{4}·7H\textsubscript{2}O, Industrial reagent, purity ≥ 99%) is a colorless fine needle-like crystal purchased from Zhengzhou Jiahong Chemical Products Co., Ltd. (China). Borax (Na\textsubscript{2}B\textsubscript{4}O\textsubscript{7}·10H\textsubscript{2}O, analytical reagent, purity ≥ 99%) is a white crystalline powder purchased from Energy Chemical (China). NaCl (analytical reagent, purity ≥ 99%) is purchased from Energy Chemical Co., Ltd. (China).

Figure 2. The XRD of light-burned MgO.

Figure 3. The PSA of light-burned MgO.

2.2. Slurry preparation and Orthogonal experiment design

The MgO and MgSO\textsubscript{4} solution can form a xMg(OH)\textsubscript{2}·yMgSO\textsubscript{4}·zH\textsubscript{2}O ternary gel system. According to Demediuk and Cole’s research, the chemical reaction of main complex salt (5Mg(OH)\textsubscript{2}·MgSO\textsubscript{4}·3H\textsubscript{2}O) is as equation (1):

\[
5\text{MgO} + \text{MgSO}_4 \cdot 7\text{H}_2\text{O} + \text{H}_2\text{O} \rightarrow 5\text{Mg(OH)}_2 \cdot \text{MgSO}_4 \cdot 3\text{H}_2\text{O}
\]  

(1)

According to the reaction formula, the reaction molar ratio of the main component MgO to MgSO\textsubscript{4}·7H\textsubscript{2}O is 5:1. Considering that the activity of MgO is only about half, it is necessary to increase the molar ratio of MgO to MgSO\textsubscript{4}·7H\textsubscript{2}O to 10:1, then the mass ratio of them is 1.6:1. At the same time, the amount of the main agent (MgO and MgSO\textsubscript{4}·7H\textsubscript{2}O) should be controlled to 30% of the total volume of water for saving cost. Therefore, the basic MOS settable system is: distilled water + 18.5% MgO + 11.5% MgSO\textsubscript{4}·7H\textsubscript{2}O + 5% boric (retarder).

We studied the effects of densities (1.2 g/cm\textsuperscript{3}, 1.4 g/cm\textsuperscript{3}, 1.6 g/cm\textsuperscript{3}, 1.8 g/cm\textsuperscript{3}), sodium chloride contents (5%, 10%, 15%, 30%), bentonite contents (4%, 6%, 8%, 10%) and temperatures (40 ℃, 60 ℃, 70 ℃, 90 ℃) on the final setting time and compressive strength of the basic MOS settable system through orthogonal design methods. Since the orthogonal experiment includes four experimental factors, and each experimental factor includes 4 experimental levels, so the orthogonal table of L\textsubscript{16}(4\textsuperscript{5}) is selected (Table 1) assuming no interaction between factors. “L” represents the orthogonal table, and the number “16” in the lower right corner represents the number of rows in the table, that is, the total number of tests required is 16. The number “5” in the upper right corner in the bracket represents the experimental factors to be investigated, and the number “4” represents each experimental level, which is the number of numerical gradients set by each factor. The specific experimental arrangement and experimental results are shown in Table 1.

**Table 1.** Orthogonal table for studying the MOS settable system stability

| Sample number | NaCl  \( (%) \) | Bentonite \( (%) \) | Density \( (\text{g/cm}^3) \) | Temperature \( (\text{°C}) \) | Setting time \( (\text{min}) \) | Compressive strength \( (\text{MPa}) \) |
|---------------|-----------------|---------------------|------------------|-----------------|-----------------|-----------------|
| 1             | 5               | 4                   | 1.2              | 40              | 90              | 0.24            |
| 2             | 5               | 6                   | 1.4              | 60              | 170             | 0.75            |
| 3             | 5               | 8                   | 1.6              | 70              | 70              | 0.63            |
### 2.3. Setting time test

The setting time (min) of the MOS settable system is measured by penetration test using the SZR-5 penetration tester (Hebei Kebiao Instrument Co., Ltd. (China)). When the penetration is less than 1.5 mm, the MOS settable system is completely cured, and the corresponding time is the setting time.

### 2.4. Compressive strength measurements

The compressive strength (MPa) is measured by taking the crush strength average of three cubes through WDW-100Y Strength Testing Machine (Jingzhou Modern Petroleum Science & Technology Co., Ltd. (China)).

### 2.5. Plugging evaluation experiment

Different proportions (10%, 15%, 20%) of 3000 mesh heavy calcium carbonate (average particle size of 5 μm) are added to the MOS settable system. Check whether the heavy calcium carbonate can improve the sealing pressure capacity after curing. The plugging experiment is carried out by using a High Temperature High Pressure water loss meter (Qingdao Tongchun Oil Instrument Co., Ltd. (China)) and its physical model is shown as figure 4. Two layer steel beads with a diameter of 8 mm are laid on the bottom of the drilling fluid cup as a simulated large-pore loss zone with a diameter of 1.23 mm. The plugging capacity of the slurry is evaluated by the pressure-loss rate test. 50 mL of basic MOS settable slurry is prepared and poured into the drilling fluid cup. The temperature is set to 40 °C, and then cured for 5 hours. Then open the drilling fluid cup to observe the solidification of the slurry and pour 100 mL of water into the drilling fluid cup, pressurize it with nitrogen through the upper stem. The pressure increment is 1 MPa and stable time of 10 min. When the water is completely lost, the test is finished, and the solidified steel bead bed formed by the MOS settable slurry is observed. The loss rate under different pressures is used to evaluate the ability of the MOS settable system with different proportions of calcium carbonate to plug the severe loss zone.

|   |   |   |   |   |   |
|---|---|---|---|---|---|
| 4 | 5  | 10| 1.8| 90 | 40 | 0.10 |
| 5 | 10 | 4 | 1.4| 70 | 145| 1.06 |
| 6 | 10 | 6 | 1.2| 90 | 30 | 0.28 |
| 7 | 10 | 8 | 1.8| 40 | 75 | 0.18 |
| 8 | 10 | 10| 1.6| 60 | 190| 0.39 |
| 9 | 15 | 4 | 1.6| 90 | 35 | 0.29 |
| 10| 15 | 6 | 1.8| 70 | 70 | 0.77 |
| 11| 15 | 8 | 1.2| 60 | 270| 0.24 |
| 12b| 15| 10| 1.4| 40 | 600| 0   |
| 13| 30 | 4 | 1.8| 60 | 215| 0.31 |
| 14b| 30| 6 | 1.6| 40 | 600| 0   |
| 15| 30 | 8 | 1.4| 90 | 35 | 0.65 |
| 16| 30 |10 | 1.2| 70 | 72 | 0.08 |

*The blank factor column is omitted.*  
*Samples 12 and 14 have lost the setting ability. For data analysis and comparison, the setting time is set to 600 min and the compressive strength is set to 0 MPa.*
3. Results and discussions

3.1. The effect of downhole conditions on the setting time and compressive strength of the MOS settable system

Table 2 and Table 3 show the analysis result obtained from the orthogonal experiment (table 1).

$\bar{K}_x$ ($x = 1, 2, 3, 4$) is the average value of setting time or compressive strength of all samples for an experimental factor (including sodium chloride, density, bentonite, and temperature) at a certain experimental level. For example, for the experimental factor of sodium chloride, $\bar{K}_1$ represents the average setting time or compressive strength of samples 1, 2, 3, and 4 when the sodium chloride content is 5%; $\bar{K}_2$ represents the average setting time or compressive strength of samples 5, 6, 7, and 8 when the sodium chloride content is 10%; $\bar{K}_3$ represents the average value of setting time or compressive strength of samples 9, 10, 11, 12 when the sodium chloride content is 15%; $\bar{K}_4$ represents the average value of setting time or compressive strength of samples 13, 14, 15, 16 when the sodium chloride content is 30%. $R$ is the range of $[\bar{K}_1, \bar{K}_2, \bar{K}_3, \bar{K}_4]$ representing the difference between the maximum value and the minimum value. The specific calculation is as follows.

For setting time analysis of sodium chloride content as in equation (2)–(6):

$$\bar{K}_1 = \frac{90 + 170 + 70 + 40}{4} = 92.50$$ (2)

$$\bar{K}_2 = \frac{145 + 30 + 75 + 19}{4} = 110.00$$ (3)

$$\bar{K}_3 = \frac{35 + 70 + 270 + 600}{4} = 243.75$$ (4)

$$\bar{K}_4 = \frac{215 + 600 + 35 + 72}{4} = 230.50$$ (5)

$$R = \text{Max}[\bar{K}_1, \bar{K}_2, \bar{K}_3, \bar{K}_4] - \text{Min}[\bar{K}_1, \bar{K}_2, \bar{K}_3, \bar{K}_4] = 243.75 - 230.50 = 151.25$$ (6)

For compressive strength analysis of sodium chloride content as in equation (7)–(11):

$$\bar{K}_1 = \frac{0.24 + 0.75 + 0.63 + 0.10}{4} = 0.430$$ (7)
\[
K_2 = \frac{1.06 + 0.28 + 0.18 + 0.39}{4} = 0.478
\] (8)
\[
K_3 = \frac{0.29 + 0.77 + 0.24 + 0}{4} = 0.325
\] (9)
\[
K_4 = \frac{0.31 + 0 + 0.65 + 0.08}{4} = 0.260
\] (10)
\[
R = \text{Max}\{K_1, K_2, K_3, K_4\} - \text{Min}\{K_1, K_2, K_3, K_4\} = 0.478 - 0.260 = 0.218
\] (11)

For other experimental factors, the calculation methods of \(K_x\) (\(x = 1, 2, 3, 4\)) and \(R\) are the same, and the calculation results for setting time of all samples are shown in Table 2 and Table 3.

**Table 2.** Orthogonal experiment results for setting time.

| \(K_i\) and \(R\) | Sodium chloride | Bentonite | Density | Temperature |
|---------------------|-----------------|-----------|---------|-------------|
| \(K_1\)             | 92.50           | 121.25    | 115.50  | 341.25      |
| \(K_2\)             | 110.0           | 217.50    | 237.50  | 211.25      |
| \(K_3\)             | 243.75          | 112.50    | 223.75  | 89.25       |
| \(K_4\)             | 230.50          | 225.50    | 100.00  | 35.00       |
| \(R\)               | 151.25          | 113.00    | 137.50  | 306.25      |

**Table 3.** Orthogonal experiment results for compressive strength.

| \(K_i\) and \(R\) | Sodium chloride | Bentonite | Density | Temperature |
|---------------------|-----------------|-----------|---------|-------------|
| \(K_1\)             | 0.430           | 0.475     | 0.210   | 0.105       |
| \(K_2\)             | 0.478           | 0.450     | 0.615   | 0.423       |
| \(K_3\)             | 0.325           | 0.425     | 0.328   | 0.635       |
| \(K_4\)             | 0.260           | 0.142     | 0.340   | 0.330       |
| \(R\)               | 0.218           | 0.333     | 0.405   | 0.530       |

And the effects of experimental factors on the setting time and compressive strength of the samples are shown in figure 5 and figure 6.

As can be seen from Figure 1 and Figure 2, there are obvious differences in the effects of various experimental factors on the setting time and compressive strength of the MOS settable system. For the experiment factor of sodium chloride (figure 5 (a) and figure 6 (a)), when the addition amount is from 5% to 10%, the setting time and compressive strength of the system slightly increase; when the addition amount is 15%, the setting time of the MOS settable system is significantly extended, however, the compressive strength decreased significantly. When the addition amount is 30%, the setting time and compressive strength of the system decreased slightly, but the change is not large. Experiment results indicate that the proper amount of salt (less than 10%) is beneficial to extend the setting time and increase the compressive strength of the MOS settable system. The presence of Cl\(^-\) will transform part of the MOS hydration products into stronger MOC hydration products, thereby improving the performance. Ronald E. Sweatman and W. Chrys Scoggins also find that salt water has a significant retarding effect on the magnesium settable system and it can slightly increase the compressive strength through comparing the properties of the slurries prepared with freshwater and seawater (its salt content is generally 3% - 5%) respectively. However, when the salt content is too high (greater than 10%), the performance will be deteriorated, which is mainly reflected in the substantial extension of the setting time and the significant decrease in compressive strength. This has an important guiding significance for its field application. The MOS settable system can withstand a small amount of salt contamination without affecting the performance, however, when the high-salt drilling fluid or formation water is contained in the wellbore or loss zone, it is necessary to prevent the
system from being contaminated through technical measures such as pumping a spacer fluid in advance.

Figure 5. Effects of experimental factors on the final setting time of the MOS settable system: (a) Sodium chloride; (b) Bentonite; (c) Density; (d) Temperature.

Figure 6. Effects of experimental factors on the compressive strength of the MOS settable system: (a) Sodium chloride; (b) Bentonite; (c) Density; (d) Temperature.
Bentonite has been used as the viscosity and thixotropic control agent for water-based drilling fluids. For the experiment factor of bentonite (figure 5 (b) and figure 6 (b)), the effect of different dosages on the setting time of the MOS settable system does not reflect obvious regularity, and the curve is jagged. However, the compressive strength of the MOS settable system decreases slowly with the increase of the amount of bentonite. When the amount of bentonite is 10%, the compressive strength of the MOS settable system decreases sharply. It has been estimated that the best bentonite contains about 60 to 70% sodium (Na) montmorillonite. The remaining 30% to 40% might be calcium (Ca) montmorillonite or other low-yielding clays, such as kaolinite [14]. The structure of montmorillonite [15] consisting of an alumina octahedral \([\text{Al}_2\text{(OH)}_3]n^{2+}\) sheet layer sandwiched between two silica tetrahedron \((\text{Si}_2\text{O}_5)n^{2-}\) sheet layers is composed of superposed lamellae, which have an edge absorption at a diffuse negative silica sheet with various cations balancing unsaturated oxygen ions. This negative charge arising on this layer as the result of the substitution is balanced by chemisorption of cations such as Mg\(^{2+}\) compensating ions located between the sheets together with molecules of water. So montmorillonite clays are much more affected by cations. Therefore, when the MOS settable system is invaded by bentonite from drilling fluids, the bentonite will occupy part of the system space, and some Mg\(^{2+}\) will be adsorbed to the sheets due to cation substitution, which affects the formation of the complex salt, resulting in compressive strength decline and setting time change. In the field application, it is necessary to avoid the MOS settable system from being polluted by bentonite causing the system performance to deteriorate.

Barite particles are very stable and difficult to dissolve in water, acids, alkalis and salts. Therefore, its effect on the properties of the MOS settable system is mainly due to the filling effect of inert particles. For the experiment factor of density (Figure 5 (c) and Figure 6 (c)), the setting time of the MOS settable system first increases and then decreases with density. This is mainly because at low density (1.2 g/cm\(^3\) and 1.4 g/cm\(^3\)), a small amount of barite particles are filled between magnesium hydroxide and magnesium sulfate in the MOS settable system, which hinders the formation of complex salts, thereby extending the setting time. At the same time, the particles reduce the porosity and increase the compactness of the system improving the compressive strength [16]. However, when the content of barite is too large, the curing ability will be reduced, which will cause the system performance to fail. Therefore, for field applications, the amount of barite should be carefully controlled. When the density of the plugging slurry is high, the amount of magnesium oxide and magnesium sulfate needs to be increased to ensure that the system can be effectively solidified.

For the experiment factor of temperature (figure 5 (d) and figure 6 (d)), different temperatures have a significant effect on the setting time of the MOS settable system. The setting time decreases approximately linearly with the increasing temperature, which indicates that the increase in temperature will speed up the setting process. In other scholars' research [8, 13, 17], this phenomenon has also emerged. This is mainly due to the acceleration of the micro-Brownian motion of particles, which accelerates the formation of the complex salt. When the temperature is 40 ~ 70 °C, the compressive strength increases obviously, but when the temperature is 90 °C, the compressive strength intensely decreases. High temperatures may cause some damage to the complex salt generated, thereby reducing the structural strength. Therefore, for field application, it is necessary to know the temperature at the loss layer in advance to avoid the failure of the MOS settable system.

3.2. Laboratory plugging evaluation

Table 4 shows the loss rate under different pressure and indicates that the larger the amount of calcium carbonate, the better the plugging effect. When the amount of calcium carbonate is 10%, the MOS plug can withstand a pressure of 3 MPa at a loss rate of 0.2 mL/s. When the amount of calcium carbonate is 15%, the MOS plug has a lower loss rate at a pressure of 3 MPa of 0.012 mL/s. When the amount of calcium carbonate is 20%, the MOS plug can withstand higher pressure of 4 MPa at a loss rate of 0.125 mL/s. The state of the MOS plug with adding different proportions of calcium carbonate after the experiment is shown in Figure 7. The MOS plug formed can firmly bond the steel ball together and is still intact with a certain strength after being taken out. This indicates that the addition of calcium carbonate can significantly improve the plugging capacity of the MOS settable slurry.
Figure 7. MOS plug formed after slurry solidification: (a) and (b) is basic MOS settable slurry + 10% CaCO$_3$; (b) and (d) is basic MOS settable slurry + 15% CaCO$_3$; (e) and (f) is basic MOS settable slurry + 20% CaCO$_3$.

Table 4. MOS plugging experiment result of basic MOS settable slurry with different amount of CaCO$_3$.

| Pressure (MPa) | Loss rate (mL/s) |
|---------------|------------------|
|               | + 10% CaCO$_3$  | + 15% CaCO$_3$ | + 20% CaCO$_3$ |
| 1             | 0                | 0              | 0              |
| 2             | 0                | 0              | 0              |
| 3             | 0.200            | 0.012          | 0.040          |
| 4             | loss out         | loss out       | 0.125          |
| 5             | /                | /              | loss out       |

3.3. Field application

Magnesia settable systems have been used to solve severe loss in wells worldwide like in Appalachians [18], Southeast Lake in Maracaibo, Neuquen basin in Argentina [19] and North Sea [17]. However, it has not been widely used as LCMs for solving severe loss problems in China. The field case (Well A) introduced next is a successful application of MOS settable system in China.

Well A which is located in the Junggar Basin, Xinjiang, is a directional well with a designed vertical depth (TV) of 2835 m and its detail parameters are shown as Table 5. The first total loss
occurred when drilling to after adding LCMs (21.05 lb/bbl; ie, 60 kg/m³) to the drilling fluid. The second loss occurred when drilling to 2192 m. The bottom hole pressure (BHP) was 1.8 MPa after adding LCMs (28.07 lb/bbl; ie, 80 kg/m³) to the drilling fluid. The third total loss occurred when drilling to 2555 m. The bottom hole pressure (BHP) was 1.5 MPa after adding LCMs (35.08 lb/bbl; ie, 100 kg/m³) to the drilling fluid. When drilling to 2705 m, the fourth loss occurred when the drilling fluid density increased to 1.34 g/cm³. A decision is made to use the well cement for curing the severe loss. After two injections of well cement, the well section of 2129 m–2705 m is cemented. When drilling continues to 2138 meters, another severe loss of a rate of 80 m³/h happens. The circulation is resumed after pumping of 40 m³ drilling fluid and 80 m³ LCMs (28.07 lb/bbl; ie, 80 kg/m³) slurry.

Judging from the drilling process, the lost interval of this well is from 1734 m to 2705 m. Due to the high permeability of the sandstone formation and the existence of fractures, the conventional LCMs cannot effectively stay in the lost formation and the blocking layer is easy to be diluted and washed away causing the repeated loss. When well cement is used to cement the loss zones, repeat loss also may happen due to the short curing time and insufficient curing strength. In order to plug the well section of 1700 m–2000 m to resume drilling, a decision is made to use the MOS cement for curing the severe loss. The following MOS plugging formula is adopted: the drilling fluid + 3% nut shell (1–3 mm) + 3% nut shell (3–5 mm) + 2% vermiculite (2–5 mm) + 2% comprehensive LCMs + 3% molecular emulsion + 32.5% MOS settable material, with a density of 1.35 g/cm³. The process of plugging operation is as follows: Firstly, put the drill bit at 2018 m and pump 25.5 m³ MOS plugging slurry to the wellbore, and 19 m³ drilling fluid is been displacement with a pump rate of 1.5 m³/min within 40 min. The MOS plugging slurry is expected to return to the depth of 1700 m. Secondly, the drill bit is lifted to 1600 m and wait for curing by 7 hours. Thirdly, the well is shut-in and pump the drilling fluid with a small rate into the well to test the bearing capacity of the wellbore, and the downhole pressure was 2.2 MPa and the stable pressure of 1.5 MPa. In the subsequent drilling, the density of the drilling fluid is increased to 1.37 g/cm³ and no loss occurs again. The total plugging operation time is 10 hours.

**Table 5. The casing program of Well A.**

| Drilling order | Bit size (mm) | Well Section (m) | Casing size (mm) | Casing depth (m) | Altitude of cement loop (m) |
|---------------|---------------|------------------|------------------|------------------|--------------------------|
| First section | 381.00        | 0–381.00         | 273.10           | 300.00           | 0–300.00                 |
| Second section| 243.30        | 0–2140.00        | 193.70           | 0–3078.49        | 1840–3078.49             |
|               | 215.90        | 2140–3078.49     |                  |                  |                          |
| Depth of kickoff point (m) | 1243.00 |            |                      |                  |                          |
| Deviation angle (°) | 35.54 (at the well depth of 2705.00 m) | | | | |

**4. Conclusion**

The following conclusions can be drawn based on this work.

1. The MOS settable system only can withstand a small amount of salt contamination without affecting the performance.

2. It is necessary to avoid the MOS settable system from being polluted by bentonite causing the system performance to deteriorate.

3. The setting ability of the MOS settable system will be weakened when its density is too high, in other words, it contains too much inert weighting materials.

4. The setting time of the MOS settable system will decrease approximately linearly with the increase of temperature.

5. The addition of calcium carbonate as a filler can significantly improve the plugging capacity of the MOS settable system.

6. MOS settable system as LCM has the characteristics of rapid setting and convenient-use and can solve the severe loss problem that cannot be solved by other conventional LCMs.
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