Novel acid soluble consolidating material to overcome lost circulation problems in reservoir intervals

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Abstract. Based on lost circulation problem, a new kind of acid soluble consolidating material was developed, and the sealing characteristics were evaluated. This acid soluble consolidating material had the characteristics of rapid water loss, high filter-cake consistency and high acid dissolution. The results indicated that the obtained curing slug had good stability at high temperature, and the sealing strength of curing slug was as high as 10 MPa. High temperature and high pressure (HTHP) sealing analog experiment indicated that the maximum pressure-bearing capacity of plugged zone was up to 18.5 MPa. Using this consolidating material to seal the shale is a very powerful and economical approach to address lost circulation problem in troublesome thief zones.

1. Introduction

During the drilling operation of abnormal high pressure formation, the fracturing induced leakage is often encountered [1-3]. Due to great differences among leakage points, it is very difficult to locate the leakage zone of these induced leakages, so it will bring great challenge to plugging technology [4-7].

Due to the particular characteristics of multiple leakage points, it is very difficult to determine the accurate leakage zone [8-10]. The conventional plugging technology, including cement plugging technology, bridge plugging technology, gel polymer plugging technology, and so on, has the characteristics of easily repeated leakage, long plugging operation time and poor plugging effect, so the drilling cycle and drilling cost should be increased greatly [11-13]. In recent years, the expansion pipe plugging technology has provided an effective means for the complex lost circulation, but there are disadvantages such as high cost and complicated application technology.

A new type of acid soluble consolidating material (ASCM) is developed by one-pot method, and the properties of ASCM are evaluated by HTHP water loss at high temperature, consolidating ability and pressure bearing capacity. The results indicate that ASCM has the characteristics of high water loss, high acid dissolution and high bearing pressure. In the process of plugging operation, due to low viscosity and small particle size, ASCM could enter the thief zones and exhibit rapid water losses, so the leakage channel could be plugged by obtained filter cake. Using ASCM to control lost circulation is a very powerful and economical approach to address lost circulation problems in multiple leakage zones. In the future, ASCM might hold great promise to resolve instability problems in reservoir intervals.
2. Preparation of acid soluble consolidating material
Given the nature of plugging material, the responsive consolidating material is composed by main material, suspending agent, curing agent and filter aid. All things considered, ASCM should have the characteristics of rapid water loss, good stability, high filter-cake consistency and high acid dissolution.

2.1. Host materials
The primary feature of ASCM is high water loss. The host materials (HM) should have good porous structure and compressibility, and they could play an important role in filling and sealing the leakage channel. As shown in table 1, four kinds of porous structural materials are evaluated.

| Number | Porous materials | Filtration time/s | Filter loss/mL | Cake thickness/mm | Acid solubility/\% | Consistency |
|--------|------------------|-------------------|----------------|-------------------|-------------------|-------------|
| 1      | HM-1             | 38                | 145            | 9                 | 75                | hard        |
| 2      | HM-2             | 35                | 150            | 9                 | 65                | hard        |
| 3      | HM-3             | 34                | 130            | 10                | 69                | hard        |
| 4      | HM-4             | 23                | 120            | 8                 | 99                | medium      |

The experimental results indicate that HM-2 has big water loss, low acid dissolution, and high filter-cake consistency. HM-4 has big water loss, excellent acid dissolution, and low filter-cake consistency. So both HM-2 and HM-4 are chosen as composite host materials. Based on the requirement for acid solubility, the host materials could be adjusted by the proportion of HM-2 and HM-4.

2.2. Suspending agent
The suspending agent is used to suspend the host materials and other materials, and it could improve the suspension stability of plugging slurry. The suspending agent (SA) could play a role in bridging in the lost circulation channel, and improve the filter-cake consistency and toughness. The properties of suspending agents are evaluated, as shown in table 2.

| Number | Suspending agent | Stability/\% | Filter loss/mL | Cake thickness/mm |
|--------|------------------|--------------|----------------|------------------|
| 1      | SA-1             | 85           | 160            | 8                |
| 2      | SA-2             | 95           | 170            | 9                |
| 3      | SA-3             | 90           | 175            | 10               |

The experimental results demonstrate that SA-2 has good suspension effect. SA-2 has good water dispersibility, so it could improve the suspension stability of plugging slurry greatly. In addition, SA-2 has good compatibility and bonding strength, so it could play a role in toughening modification of solidified filter cake.

2.3. Curing agent
The plugging material could form filter cake easily in the leakage channel, but the pressure-bearing capacity of filter cake is low. To improve the strength of filter cake, the curing agent (CA) should be used, so the filter cake could be solidified. The properties of curing agents are tested in table 3.

The results indicate that three kinds of curing agents have little influence on the strength of filter cake. The filter cake formed by CS-3 has the highest strength, so CS-3 has the best curing effect. The curing effect of CS-3 is being improved with the increase of dosage of CS-3. But when the dosage of CS-3 is more than 10%, the gelation of plugging slurry could be increased greatly. The results indicate that CS-3 has enough curing effect when the dosage of CS-3 is 5~10%.
Table 3. The evaluation of properties of curing agents.

| Number | Curing agent | Curing strength /KN | Cake thickness /mm | Filtration time /s | Filter loss /mL |
|--------|--------------|---------------------|-------------------|-------------------|----------------|
| 1      | CS-1         | 16                  | 11                | 25                | 190            |
| 2      | CS-2         | 12                  | 9                 | 23                | 185            |
| 3      | CS-3         | 20                  | 10                | 23                | 180            |

2.4. Filter aid
The filter aid is used to further improve the filtration performance of plugging agent, and accelerate the filtration rate of plugging slurry. The effect of the dosage of filter aid (FA) on the properties of plugging slurry is shown in table 4.

Table 4. The influence of the dosage of FA on the properties of plugging slurry.

| Number | Dosage /% | Filtration time /s | Filter loss /mL | Cake thickness /mm | Stability /% | Curing strength /KN |
|--------|-----------|--------------------|-----------------|-------------------|--------------|---------------------|
| 1      | 4         | 30                 | 180             | 10                | 95           | 19                  |
| 2      | 6         | 25                 | 180             | 9                 | 95           | 20                  |
| 3      | 8         | 18                 | 185             | 10                | 90           | 20                  |
| 4      | 10        | 16                 | 190             | 10                | 85           | 20                  |
| 5      | 12        | 16                 | 190             | 10                | 85           | 21                  |
| 6      | 15        | 15                 | 190             | 11                | 80           | 21                  |

The experimental results indicate that this filter aid could accelerate the filtration rate, increase the filter loss, and improve the curing strength of filter cake. The filtration rate of plugging agent could be improved with increase of the dosage of filter aid. But when the dosage of filter aid is more than 12%, the stability of plugging agent could be weakened. All things considered, the filter aid has enough filtration rate when the dosage is 8~12%.

2.5. Acid soluble consolidating material
Taking into consideration of various components, the proportion of main material, suspending agent, curing agent and filter aid is optimized, and a new kind of acid soluble consolidating material is developed.

Formula of ASCM: 40%HM-2+30%HM-4+12%SA-2+8%CS-3+10%FA

After ACSM and water are mixed and stirred fully, a type of plugging slurry with good suspension and strong fluidity is prepared. The filter cake formed at high pressure could be used to seal the formations with porous leakage or fractured leakage.

3. Results and discussion

3.1. The evaluation of HTHP water loss at different temperatures
For a consolidating material, rapid water loss and big water loss at high temperature and high pressure (HTHP) conditions is very important. The high-temperature and high-pressure filter press is used to test the filtration properties of ASCM. The water loss and filtration time of plugging slurry at high temperature are shown in table 5.

The experimental results indicate that this plugging slurry containing ASCM has high water loss in 60-75 second. In addition, the water loss could be reduced with the increase of temperature. The water loss remains unchanged when the temperature is above 150°C. So ASCM has good filtration properties at high temperature.
Table 5. The test of HTHP water loss of plugging slurry containing ASCM.

| Temperature | 80℃ | 100℃ | 120℃ | 150℃ | 180℃ |
|-------------|-----|------|-------|------|------|
| Water loss/mL | 325 | 320  | 310   | 310  | 305  |
| Filtration time/s | 75  | 68   | 67    | 60   | 61   |

^a Formula for plugging slurry: fresh water + 20% ASCM.

3.2. The consolidating ability of ASCM

The high-temperature stability of solidified filter cake is of great importance for the completion operation. The maintenance intensity of obtained curing slug at 180℃ is shown in figure 1. The curing strength could be enforced with the increase of curing time, but the curing strength is reduced when the curing time is more than 15 d. Therefore, the curing slug has good stability at high temperature when the curing time is less than 15 d. In addition, the sealing strength could be up to 10 MPa, so it could meet the requirement of completion operation.

![Figure 1](image1.png)

**Figure 1.** The relation curve between the curing intensity of curing slug and curing time.

3.3. The test of acid dissolution of ASCM

For the leakage zone in reservoir intervals, the plugging material should have good solubility in acid. Different quantity of hydrochloric acid with the concentration of 15% is added to the same amount of ASCM, and ASCM samples are dissolved rapidly in hydrochloric acid. After soaking for 24 h, filtration and desiccation, the sample weight before and after acidification is weighed and measured, and the acid dissolution is calculated as bellow,

\[ \eta = \left( \frac{M_1 - M_2}{M_1} \right) \times 100\% \]  \hspace{1cm} (1)

Where \( \eta \) refers to acid dissolution, %; \( M_1 \) refers to ample weight before acidification, g; \( M_2 \) refers to sample weight after acidification, g.

![Figure 2](image2.png)

**Figure 2.** The curve of acid dissolution of ASCM.
The curve of acid dissolution of ASCM samples is shown in figure 2. The experimental results indicate that the acid dissolution of samples is raised with the increase of the dosage of hydrochloric acid. The maximum acid dissolution of ASCM is 91%. So ASCM has good acid dissolution.

The curing slug is obtained by solidified filter cake from ASCM. The acid dissolution of curing slug is evaluated by immersion in hydrochloric acid. The curve of acid dissolution of curing slug is shown in figure 3. The end-face immersion rolling test indicates that the acid dissolution of curing slug is above 85% when the immersion time is above 16 h.

![Figure 3. The curve of acid dissolution of curing slug from ASCM.](image)

3.4. The evaluation of plugging performance of ASCM

The plugging slurry containing ASCM is prepared, and the plugging performance of ASCM is evaluated by HTHP sealing analog device. Using 10 mm slotted templet to simulate the leakage zone, the sealing strength of plugged zone produced from plugging slurry is shown in figure 4.

![Figure 4. The squeeze pressure curve of plugging slurry containing ASCM.](image)

The results indicate that the plugging slurry could reside and pile up in the simulated leakage zone. When the squeezing amount is more than 250 mL, the complete filtration of plugging slurry is encountered. The squeeze pressure rises gradually with the increase of squeezing amount. The plugging slurry containing ASCM is compacted gradually in the leakage zone, and the highest pressure-bearing capacity is as high as 18.5 MPa.

4. Conclusions

- A new type of acid soluble consolidating material is prepared. ASCM has the characteristics
of rapid water loss, big water loss and high filter-cake consistency.

- The curing slug has good stability even at 180°C, the sealing strength of curing slug is as high as 10 MPa in 15 d, so ASCM could meet the requirement of completion operation.
- The acid dissolution of ASCM could reach up to 91%. The highest pressure-bearing capacity of plugged zone is up to 18.5 MPa. ASCM could seal the reservoir with porous leakage or fractured leakage effectively, and thus improve wellbore strength greatly.

Acknowledgments
This study is supported by the National Science and Technology Major Project (No. 2016ZX05020-004, 2016ZX05051, 2017ZX05030-004) and CNPC Project (No. 2018E-18, 2018D-5009-05, 2017D5008-03, 2016E-0109, 2016E-0608).

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