An Experimental Study on optimizing the particle size distribution of bridging agents in drilling fluids

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Abstract. Managing lost circulation has always been a serious challenge in the oil industry. This situation becomes further complicated when shale formation is involved. Particulate lost circulation materials (LCMs) have been used to prevent the drilling fluid from entering fractured, cavernous, or high-permeability formations for many years. However, existing LCMs are inefficient in terms of size and application methods in solving severe-to-total losses in shales due to the uncertainty of particle size distribution (PSD) in drilling fluids. This study focuses on an experimental method optimizing the PSD of solid particles in the drilling fluid at high temperature and pressure. An orthogonal experiment of L9 (3)4, including four factors and three levels, was designed to investigate the relationship between the PSD of solid particles and filtration loss. Results indicate that the orthogonal design method contributes to the optimization of the PSD of solid particles in the drilling fluid and reduce the test time and experimental workload during the drilling fluid process in the design stage. In addition, a polynomial relationship between the cumulative filtration loss and D90 or D50 is verified experimentally in this study. This study will remarkably benefit the drilling technologists in resolving the issue of lost circulation.

1. Introduction
Lost circulation is still a serious issue in the drilling industry. Lost circulation can result in more than 12% of nonproduction time in the Gulf of Mexico [1]. The leak-off test has been used to estimate the pressure of lost circulation [2]. In general, the design of the particle size distribution (PSD) can effectively control the lost circulation. Abrams [3] found that the plugging of the pore throat caused by the drilling fluid can occur at a concentration level of at least 5% by volume of the mud solids with depths of at least 1 in. Meanwhile, the diameters of bridging particles were greater than or equal to one-third of the formation median pore sizes. Smith [4] suggested that the d1/2 rule should be considered, except for the 1/3 rule. Hands [5] recommended that the D90 (90% of the particles should be smaller than size x) of the PSD of bridging particles should be equal to the pore size of the rock. Cargnel [6] offered that an effective mud cake could be established immediately when the PSD of bridging agents...
was 1/7 D pore throat < D particle < 1/3 D pore throat. Subsequently, Chellappah [7] further investigated the relationship of cumulative volume percent $\propto dx$ (where $d =$ particle diameter). He found that choosing $x \sim 1$ could be more suitable than using the proposed $x = 0.5$ in ideal packing theory. However, Vickers [8] suggested that five parameters, namely, D90, D75, D50, D25, and D10, should be utilized to evaluate the pore plugging ability of bridging materials, especially when insufficient pore data are known. A new bridging theory was proposed to effectively bridge and seal the fracture of a certain width. The theory stated that D50 and D90 should be greater than or equal to 3/10 and 6/5 of the fracture width, respectively [9]. In addition, the drilling industry can also benefit from the developments in nanotechnology. The use of nanoparticles in drilling fluids will enable the drilling technologists to modify the drilling fluid rheology and PSD swiftly [10–13]. Some researchers have also experimentally revealed the appropriate proportion in the mixture of micro- and nanoparticles to optimize the drilling mud PSD and minimize filtrate reduction. Salehi et al. [14] stated that the plug capacity would be largely dependent on the PSD in the mixed solid particles. These theories and methods have been successfully applied in the laboratory and field. However, certain scholars have argued that the application of the classic bridging theory is impractical for low-permeability formations due to low pore sizes [15]. Notably, the majority of these theories are based on knowing the size of rock matrix pores, but precisely predicting the real pore size of the rock matrix or the width of the fracture is difficult in an actual case. Therefore, the challenge is the efficient and rapid selection of bridging agents in an actual case.

The main objective of this study is to recommend a comprehensive test method or procedure for quantitatively selecting sealing agents in drilling fluids. Moreover, the method proposed can remarkably reduce the experimental workload and cost of the drilling fluid design to cater to the current trend of oil development.

2. Experimental methods

2.1. Experimental Setup

To test the loss of drilling fluid, a permeability plugging tester (PPT) with a 400 mD disk was used in this study. The test procedure recommended in API RP 13B-1 must be strictly followed. In addition, a Rasterizer 3000 from Malvern Instruments was used to measure the PSD of solid particles in drilling fluids, and the test procedure was based on the instrument manual.

2.2. Experimental Procedure

An orthogonal design of L9 (3)4, including four factors and three levels, was designed to investigate the effect of solid particles in the drilling fluid on the cumulative filtration loss. The experimental formulas are listed in Table 1. We first prepared the drilling fluid according to these formulas and aged it for 16 h at 150 °C. Then, the filtration test was performed according to the previous instruction.

| Factor | Level | A (%) | B (%) | C (%) | D (%) |
|--------|-------|-------|-------|-------|-------|
|        | 1     | Starch | Asphalt | NPs | Fiber |
|        | 2     | 2     | 2     | 1.5  | 2     |
|        | 3     | 3     | 1     | 3    | 0     |

3. Results and discussion

According to the array of orthogonal experiment, nine experiments were conducted. The test results are listed in Table 2. Notably, each test was repeated thrice under the same conditions to reduce the experimental error.
Table 2. Experimental plan and results

| Number | A (Starch) | B (Asphalt) | C (NPs) | D (Fiber) | Results | Filtration (mL) |
|--------|------------|-------------|---------|-----------|---------|----------------|
|        | I          | II          | III     | Mean      |         |                |
| 1      | 1          | 3           | 0       | 4         | 13.2    | 14.2           | 12.2           | 13.2           |
| 2      | 1          | 2           | 1.5     | 2         | 16.5    | 17.4           | 15.6           | 16.5           |
| 3      | 1          | 1           | 3       | 0         | 18.5    | 20.8           | 16.7           | 18.67          |
| 4      | 2          | 3           | 1.5     | 0         | 19      | 19.3           | 19              | 19.10          |
| 5      | 2          | 2           | 3       | 4         | 8.8     | 10             | 7.8            | 8.87           |
| 6      | 2          | 1           | 0       | 2         | 15.5    | 13.5           | 17.6           | 15.53          |
| 7      | 3          | 3           | 3       | 2         | 16      | 14.5           | 17.8           | 16.10          |
| 8      | 3          | 2           | 0       | 0         | 18.1    | 17.1           | 19.1           | 18.10          |
| 9      | 3          | 1           | 1.5     | 4         | 9       | 8.1            | 10.2           | 9.10           |

3.1. Influence of Different Types of Sealing Agents for Filtration Loss.

According to the mechanism of orthogonal experimental design, \( \overline{K}_j \) (\( I = A, B, C, D ; j = 1, 2, 3, 4 \)) is defined as the mean value of all levels of each factor. This process is designed for selecting the optimal level and the best combination of factors. The finest level of each factor can be obtained at the largest value of \( \overline{K}_j \). In addition, \( R_i \) is defined as the difference between the maximum and the minimum value of \( \overline{K}_j \). \( R_i \) is utilized to assess the conspicuousness of each factor. The high value of \( R_i \) increases the impact on the test results. Then, the computational process of all levels of the A factor is listed in the following.

\[
\overline{K}_{A1} = \frac{(13.2 + 16.5 + 18.67)}{3} = 16.12
\]
\[
\overline{K}_{A2} = \frac{(19.10 + 8.87 + 15.53)}{3} = 14.50
\]
\[
\overline{K}_{A3} = \frac{(16.10 + 18.10 + 9.10)}{3} = 14.43
\]
\[
R_i = \max(\overline{K}_{A1}) - \min(\overline{K}_{A3}) = 16.12 - 14.43 = 1.69
\]

The laboratory data presented in Table 3 show that the sequence of the effect of the four factors on the cumulative filtration loss is \( R_D > R_B > R_A > R_C \). This order shows that a small change in D can result in a significant change in the cumulative filtration loss. However, the cumulative filtration loss only slight changes with the fluctuations of C. Further analysis of the relationship between each factor and the cumulative filtration loss is shown in Fig. 1. Notably, the horizontal coordinate shown in Fig. 1 is the mean values of the tests conducted thrice at each level of each factor under the same conditions. On the basis of the change in the mean value of each factor (Figs. 1a–1d), the different shape curves can be observed. For D additive, the cumulative filtration loss sharply decreased from 18.62 ml to 10.39 ml when the concentration of D increases from 0% to 4%. For A additive, the increase in A from 1%wt to 3%wt (Fig. 1b) potentially results in a slight decline in the cumulative filtration loss, which is a similar trend to that observed in B (Fig. 1b). For C additives, the cumulative filtration loss decreases steadily from 15.61 ml to 14.54 ml (Fig. 1c). C seems to have a minimal effect on the cumulative filtration loss. Thus, we can conclude through the above-mentioned work that the best combination of lost circulation materials (LCMs) in the study is A2B2C1D3. The results are also cost-optimisation. The combination of plugging agents with the best drilling fluid system should consist of 2%wt starch, 2%wt asphalt, 0%wt NPs, and 4%wt fiber.
Table 3. Analysis Results of the Orthogonal Experiment

| Source | Degrees of freedom | $K_{41}$ | $K_{42}$ | $K_{43}$ | $R_i$ | Sum of squares | Mean squares | $F_i$ | P (%) |
|--------|--------------------|---------|---------|---------|------|---------------|-------------|------|-------|
| A      | 2                  | 16.1    | 14.5    | 14.4    | 1.6  | 16.47         | 8.23        | 4.54 | 4.60  |
| B      | 2                  | 16.1    | 14.4    | 14.4    | 1.7  | 16.79         | 8.40        | 4.63 | 4.69  |
| C      | 2                  | 15.6    | 14.9    | 14.5    | 1.0  | 5.31          | 2.65        | 1.46 | 1.48  |
| D      | 2                  | 10.3    | 16.0    | 18.6    | 8.2  | 319.25        | 159.63      | 88.03| 89.2  |
| Error  | 18                 |         |         |         |      | 32.64         | 1.81        |      |       |
| Total  | 26                 |         |         |         |      |               |             |      |       |

Fig. 1 On the basis of the change in the mean value of each factor

Analysis of variance (ANOVA): The laboratory data fluctuation of each factor caused by trial errors or trial conditions may seriously influence the test result to a certain extent. Thus, an ANOVA examination was utilized to investigate the experimental error. In ANOVA, several parameters were involved, including the sum of all test indexes (T), the sum of squared deviation (SSi), degree of freedom (dfi), and mean squares (Si); the sum of squared deviation (SSe), the error degree of freedom (dfe), and the error mean squares (Se) for the experimental error (Se) factors. Si and Se were used to calculate F. The value of F is an indicator of the effect of each factor on the experimental results. These calculation formulas are listed as follows.

Table 3 summarizes the computational results. The results show that A is the prominent factor affecting the cumulative filtration loss given that $F_D > F_{0.01(2,18)}$, whereas C insignificantly affected the filtration loss because $F_C < F_{0.01(2,18)}$ and $F_C < F_{0.05(2,18)}$. Furthermore, the percentage contribution indicates that the most prominent factor contributing to the reduction of the cumulative filtration loss is factor D (89.23%), followed by factors A (4.60%), B (4.69%), and C (1.48%). Therefore, fibrous materials are crucial in the control of lost circulation.
\[ T = \sum_{i=1}^{n} \sum_{r=1}^{l} x_{ir} \]  

(5)

\[ SS_i = \frac{1}{r \cdot s} \sum_{j=1}^{m} K_{ij}^2 - \frac{T^2}{ns} \]  

(6)

\[ df_i = m - 1 \]  

(7)

\[ SS_a = \frac{1}{3 \times 3} (K_{i1}^2 + K_{i2}^2 + K_{i3}^2) - \frac{T^2}{9 \times 3} = 16.47 \]  

(8)

\[ SS_e = \sum_{i=1}^{9} \sum_{r=1}^{3} x_{ir}^2 - \frac{1}{3} \sum_{i=1}^{9} (\sum_{r=1}^{3} x_{ir})^2 = 32.64 \]  

(9)

\[ df_a = df_b = df_c = df_e, 3 - 1 = 2, df_e = 9 \times (3 - 1) = 18 \]  

(10)

\[ S_a = \frac{SS_a}{df_a} = \frac{8.23}{1.81} = 4.54 \]  

(11)

\[ F_A = \frac{S_A}{S_e} = \frac{8.23}{1.81} = 4.54 \]  

(12)

where SSi = the sum of square deviation, dfi = the degree of freedom, Si = the variance, F = ratio, SSe = the error sum of squares deviation, dfc = the error degree of freedom, Si = the error variance, and T = the sum of all test data.

3.2. Analysis of the PSD.

PSD analysis of the nine experiments is presented in Table 4. All test points, which were fitted in several mathematical models, are depicted in Figs. 2–3 to determine the relationship between the PSD and the cumulative filtration loss. The cumulative filtration loss generally reduces as D10, D50, or D90 increases. From the correlation coefficient, the key factors contributing to the reduction of the cumulative filtration loss are D90 and D50. In addition, the cumulative filtration loss has a polynomial relationship with the D90 and D50 rather than a linear relationship because the correlation coefficient is the largest among all the fitting curves. Moreover, the value of D10 is greater than the pore size of the disk at the minimum cumulative filtration loss volume. This condition indicates that only less than 10% of the particles can invade the disk pore to form an internal mud cake. This conclusion is clearly different from aforementioned bridging theories in literature. Thus, this study experimentally confirms that the application of the classic bridging theory is impractical for smaller pore throat.

| Table 4. Particle Size Distribution of Different drilling fluids |
|---|---|---|---|---|---|---|---|---|
|   | 1   | 2   | 3   | 4   | 5   | 6   | 7   | 8   | 9   |
| D10 | 2.84 | 1.5 | 1.58 | 1.69 | 2.55 | 2.58 | 2.12 | 1.76 | 2.65 |
| D50 | 29.1 | 15.9 | 16.4 | 15.8 | 29.4 | 24.6 | 24   | 15.8 | 28.1 |
| D90 | 140  | 112 | 72.2 | 62.6 | 163  | 125  | 121  | 62.1 | 146 |
3.3. Validating the optimal combination.

With the intention of verifying the validity of the conclusions above, a freshwater drilling fluid was designed. The composition of the best drilling fluid was based on mud + 2% starch + 2% asphalt + 4% fiber. Compared with the nine experiments aforementioned in the previous parts, the filtration rate of drilling fluids designed by the above conclusion is the minimum (Figs. 4 and 5), and the value of the D10 is equal to 2.39 \( \mu \)m, which is greater than the pore size of the disk. These rheological parameters of drilling fluids can also meet the operational requirements in the field (Table 5). Therefore, the theory and method obtained in this study can be applied to the efficient and economical design of drilling fluids.

| Heat Time (h) | Density (g/cm\(^3\)) | AV (cP) | PV (cP) | YP (Pa) |
|---------------|----------------------|---------|---------|---------|
| 0             | 1.25                 | 40.5    | 31      | 8.5     |
| 16            | 1.25                 | 41.5    | 32      | 9       |
4. Conclusions
This study presents an orthogonal experimental design for investigating the relationship between the PSD of bridging particles and HPHT filtration. The specific results and conclusions can be summarized as follows:

1. The sealing capacity of the drilling fluid is highly dependent on the optimized PSD of particles in the drilling fluid. The optimal combination of bridging particles can contribute to the minimization of filtrate losses of drilling fluids.

2. An orthogonal experimental design can help in the selection of the optimal concentration of plugging agents. Thus, the method proposed in the paper can remarkably reduce the experimental workload and cost.

3. This study shows that the combination of sealing agents determined by the orthogonal experiment exhibits a broad particle distribution that can match the voids of the rock matrix in the case of unknown porosity-related data. In addition, the best sealing capacity of drilling fluids is obtained when a polynomial relationship exists between the cumulative filtration loss and the PSD.

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