Research on ecologically safe flokulants in clay fluid fluids

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Abstract. Currently, a large number of flocculants differing in properties and composition and working differently in various conditions is manufactured. Drilling fluids are complex systems differing in component composition, thus, it is not possible to recommend a universal flocculant. The present study carries out the analysis with the use of two methods with the objective to assess reagents’ flocculating ability. Having analyzed the results obtained, the following conclusions can be drawn: the reagents BEN-EX, BENTOPLUS and DRB-9 can be used to increase the clay solution viscosity provided that the additives BENTOPLUS and DRB-9 do not exceed the concentration of 0.2 kg/m³; in a concentration of up to 0.1 kg/m³ flocculant SPECFLOC A 7950-20 can be used to increase a clay solution viscosity, at a concentration of 0.2 and 0.3 kg/m³ the additive affects a drilling fluid mainly as a flocculant, thus, reducing the static shear stress of the solution and increasing filtrate flow. If dynamic shear stress is the only determining parameter in drilling with drilling fluid with SPECFLOC N6919 additive, then it can be recommended for being used. In all other cases additional research is required.

1. Introduction
Organic synthetic high molecular weight polymers called flocculants are used to accelerate and improve the separation of solid and liquid phases. When introduced into dispersed systems, these polymers are adsorbed or chemically bonded to the surface of the dispersed phase particles, combine the particles into agglomerates (flocculas) and facilitate their rapid deposition. A wide range of flocculants is presented due to the variety of drilling fluids. The flocculants have different composition and technical characteristics. The optimal type is selected depending on the properties of a suspension, which is to be cleaned, the cleaning method and the desired result. The number of flocculants entering the market is increasing every year. Various tests are carried out with the objective to select a flocculant suitable for the given conditions. In this case, it is advisable to conduct laboratory tests in order to limit the number of grades for further testing. The choice of a test method depends on the problem statement with regards to production.

Flocculants were tested on the basis of IRNITU, the research and development laboratory of drilling fluids and well fixing, for being used in clay wash fluids. The list of the applied chemicals is presented in table 1.
### Table 1. Chemical reagents for comparative analysis

| №  | Chemical                  | Manufacturer         | Chemical role                                                                 | Recommended concentration, kg/m³ |
|----|---------------------------|----------------------|--------------------------------------------------------------------------------|----------------------------------|
| 1  | Bentonite clay powder     | Kurgan               | gelling additive                                                              | 50.0                             |
| 3  | DRB-9                     | -                    | Selective flocculant and bentonite additive                                    | 0.5 – 1.5                        |
| 4  | BENTOPLUS                 | Lamberti             | Synthetic polymer, bentonite additive                                          | 0.1 – 1.0                        |
| 5  | BEN-EX                    | M-I Drilling Fluids  | Bentonite clay modifier                                                       | from 0.14                        |
| 6  | SPECFLOC N6919            | Corporate group “Himalyans” | Polycrylamide with a basic substance content of at least 90%, nonionic series | -                                |
| 6  | SPECFLOC A 7950-20        | -                    | Polycrylamide with a basic substance content of at least 90%, anionic series  | -                                |

Laboratory studies were carried out in a room with a temperature of 20 ± 2° C and a relative humidity of at least 50%.

2. Research Methodology

Analysis with the use of two methods was carried out with the objective to observe the effect of chemicals on the properties of the drilling fluid.

It was decided to add SPECFLOC N6919 and SPECFLOC A 7950-20 in the same amount as the rest reagents due to the fact that the accompanying documents for these chemicals do not contain recommendations for their introduction into the drilling fluid (see Table 1).

3. Methodology

**Bentonite suspension preparation**

1. Weigh 100 g of bentonite clay powder.
2. Pour two liters of distilled water into a glass and place it in a mixer.
3. Introduce the clay powder with a stirrer on. Continue mixing until the clay powder has completely dissolved.
4. The resulting suspension is divided into 4 equal parts.
5. Treat the samples of solution 2, 3, 4 with the test chemical while constant stirring on a laboratory stirrer in compliance with a concentration presented in table 2.
6. Measure the rheological parameters of the obtained samples of the solution (1 – 4), fill table 2 with the results. To ensure the results reliability, the drilling fluid was measured twice (due to the limited duration of the test) on each prepared sample of the solution. Table 2 presents the arithmetic mean values of the indicators.
7. Build a diagram of the dependence of the solution sample dynamic shear stress on the chemical’s concentration.
Table 2 Research results

| №  | Solution composition, kg/m³ | Chemical concentration, kg/m³ | Rheological parameters | Dynamic shear stress, daPA |
|----|-----------------------------|-------------------------------|-----------------------|---------------------------|
|    |                             |                               | at 600 RPM, DR        | at 300 RPM, DR          | PV, cPs                  |
| 1  | Bentonite suspension        | 50.0                          | 31.1                  | 23.5                     | 7.6                      | 76.3                     |
|    | DRB-9                       | 0.1                           | 30.7                  | 24.1                     | 6.6                      | 84.0                     |
|    |                             | 0.2                           | 37.0                  | 30.4                     | 6.6                      | 114.2                    |
|    |                             | 0.3*                          | 47.4                  | 37.2                     | 10.2                     | 129.6                    |
| 2  | Bentonite suspension        | 50.0                          | 30.1                  | 22.8                     | 7.3                      | 74.4                     |
|    | BENTOPLUS                   | 0.1                           | 34.1                  | 26.5                     | 7.6                      | 90.7                     |
|    |                             | 0.2                           | 38.6                  | 30.3                     | 8.3                      | 105.6                    |
|    |                             | 0.3*                          | 42.2                  | 29.1                     | 13.1                     | 76.8                     |
| 3  | Bentonite suspension        | 50.0                          | 31.8                  | 24.0                     | 7.8                      | 77.8                     |
|    | BEN-EX                      | 0.1                           | 35.2                  | 27.2                     | 8.0                      | 92.2                     |
|    |                             | 0.2                           | 39.1                  | 30.9                     | 8.2                      | 109.0                    |
|    |                             | 0.3                           | 43.9                  | 34.1                     | 9.8                      | 116.6                    |
| 4  | Bentonite suspension        | 50.0                          | 31.6                  | 23.6                     | 8.0                      | 74.9                     |
|    | SPECFLOC A 7950-20          | 0.1                           | 42.3                  | 35.7                     | 6.6                      | 139.7                    |
|    |                             | 0.2*                          | 67.7                  | 58.9                     | 8.8                      | 240.5                    |
|    |                             | 0.3*                          | 26.8                  | 19.0                     | 7.8                      | 53.8                     |
| 5  | Bentonite suspension        | 50.0                          | 29.8                  | 22.6                     | 7.2                      | 73.9                     |
|    | SPECFLOC N6919              | 0.1                           | 27.8                  | 25.5                     | 2.3                      | 111.4                    |
|    |                             | 0.2                           | 32.7                  | 28.9                     | 3.8                      | 120.5                    |
|    |                             | 0.3                           | 49.3                  | 41.6                     | 7.7                      | 162.7                    |

* Note: Solutions in selected concentrations were characterized by a separation of the suspension into two phases, flocculation.

The diagrams of the dependence of the sample solution dynamic shear stress on the concentration of the chemical were constructed with the objective to determine the optimal concentration of the additive to bentonite, at which the maximum value of the sample solution dynamic shear stress is achieved.

The results presented in Figure 1 and in Table 2 show that the value of a dynamic shear stress parameter is maximum for the SPECFLOC A 7950-20 reagent at a concentration of 0.2 kg/m³. However, at this saturation, the solution was divided into phases, which can affect other parameters of the washing liquid. Thus, the maximum dynamic shear stress value has a solution with SPECFLOC N6919 additive at a concentration of 0.3 kg/m³.
In accordance with the Provisional regulations concerning the preparation and processing of drilling mud during the construction of production wells in Eastern Siberia, mud was mixed according to the formulation for drilling for direction and conductor (Table 3).

**Table 3. Composition of initial solution**

| №  | solution composition      | Amount of chemical, kg/m³ |
|----|---------------------------|---------------------------|
| 1  | Sodium carbonate Na₂CO₃  | 1.5                       |
| 2  | Bentonite clay powder     | 50.0                      |
Bentonite suspension preparation
1. Prepare all chemicals included in the drilling fluid.
2. Make a sample of each chemical in accordance with table 1.
3. Pour 800 cm$^3$ of distilled water into a measuring cup and place it under a mixer.
4. Turn on a stirrer at a stirring speed of 600 rpm.
5. Insert all components of the solution into a beaker while the stirrer is on. Stir it at constant speed.
6. Introduce sodium carbonate Na$_2$CO$_3$ to remove Ca$^{2+}$ ions.
7. Introduce clay powder while vigorous stirring.
8. Continue mixing until a homogeneous suspension is obtained.
9. Divide the obtained solution into 4 equal parts.
10. Treat the samples of solution 2, 3, 4 with the test chemical while constant stirring on a laboratory stirrer in a concentration presented in table 4.
11. Maintain solution samples with stirring on a laboratory stirrer at low speeds for 2 hours.
12. Measure the parameters of the obtained samples of the solution (1–4); enter the results in table 4.
13. For the reliability of the results obtained, the drilling fluid was measured twice (due to the limited duration of the test) on each prepared sample of the solution. Table 5 presents the arithmetic mean values of indicators.

Fig. 2 Diagram of changes in conditional viscosity depending on the concentration of the chemical
| №  | Solution composition | Chemical reagent concentration, kg/m³ | ρ, g/cm³ | Assumed viscosity 7000 sec, cP | PV, cPs | Dynamic shear stress, daPA | Gel static shear stress, 10⁻⁴Pa, aPA | F₃₀ on API, sm³ | Cake, mm | pH  |
|----|---------------------|--------------------------------------|--------|-----------------------------|--------|--------------------------|--------------------------------------|--------|--------|-----|
| 1  | base solution       | 1.02 6                               | 0.1    | 26.4                        | 6.8    | 87.0                     | 75.0/127.5                           | 20.0   | 2.0    | 10.6 |
|    | DRB-9               | 1.02 6                               | 0.2    | 29.9                        | 5.6    | 116.0                    | 103.0/149.0                          | 19.0   | 2.0    | 10.5 |
|    | base solution       | 1.02 6                               | 0.3    | 39.3                        | 10.6   | 140.0                    | 54.5/52.0                            | 25.5   | 2.0    | 10.6 |
| 2  | BENTOPLUS          | 1.02 6                               | 0.1    | 31.6                        | 7.4    | 92.5                     | 94.0/156.0                           | 18.6   | 2.0    | 10.6 |
|    | base solution       | 1.02 6                               | 0.2    | 36.8                        | 8.4    | 121.0                    | 75.0/78.0                            | 19.8   | 2.0    | 10.5 |
|    | base solution       | 1.02 6                               | 0.3    | 24.6                        | 12.6   | 80.0                     | 63.0/89.5                            | 73.0   | 2.0    | 10.6 |
| 3  | BEN-EX              | 1.02 6                               | 0.1    | 29.2                        | 5.3    | 108.5                    | 105.0/134.0                          | 18.0   | 2.0    | 10.5 |
|    | base solution       | 1.02 6                               | 0.2    | 37.8                        | 7.1    | 129.0                    | 101.0/104.0                          | 17.0   | 2.0    | 10.7 |
|    | base solution       | 1.02 6                               | 0.3    | 52.3                        | 7.3    | 161.0                    | 104.0/112.0                          | 16.5   | 2.0    | 10.6 |
| 4  | SPECFLOC A 7950-20  | 1.02 6                               | 0.1    | 30.1                        | 7.5    | 116.5                    | 93.0/101.0                           | 17.5   | 2.0    | 10.6 |
|    | base solution       | 1.02 6                               | 0.2    | 36.8                        | 10.8   | 103.0                    | 40.0/41.0                            | 62.0   | 2.0    | 10.5 |
|    | base solution       | 1.02 6                               | 0.3    | 26.8                        | 6.7    | 24.5                     | 18.0/36.0                            | 46.0   | 2.0    | 10.6 |
| 5  | SPECFLOC N6919     | 1.02 6                               | 0.1    | 29.2                        | 4.9    | 119.0                    | 101.0/155.0                          | 18.0   | 2.0    | 10.6 |
|    | base solution       | 1.02 6                               | 0.2    | 37.3                        | 6.7    | 145.0                    | 100.0/89.0                           | 16.5   | 2.0    | 10.6 |
|    | base solution       | 1.02 6                               | 0.3    | 57.1                        | 7.9    | 184.0                    | 79.0/87.0                            | 18.5   | 2.0    | 10.6 |

Diagrams of the dependence of the parameters of the washing liquid on the concentration of the concentration of additives in the bentonite suspension were constructed on the basis of the results obtained (Fig. 2-7).
**Fig. 3** Diagram of changes in plastic viscosity depending on the chemical concentration

**Fig. 4** Diagram of changes in static shear stress during 10 seconds depending on chemical concentration
Fig. 5 Diagram of changes in static shear stress for 10 min depending on chemical concentration

Fig. 6 Diagram of changes in dynamic shear stress depending on chemical concentration
Laboratory tests enable to make the following conclusions:

1. All the chemicals provided do not affect the density and pH of the drilling fluid.
2. The main function of reagents BEN-EX and BENTOPLUS is to improve the technological properties of a bentonite suspension.

BEN-EX conforms to the properties declared in the accompanying documents. When it is added in various concentrations to a bentonite suspension, the rheological properties and conditional viscosity of the latter increase and the mud filtration slightly decreases (Fig. 7).

If taken at 0.1 and 0.2 kg/m\(^3\) concentration, BENTOPLUS improves the properties of bentonite. If it was added in the amount of 0.3 kg/m\(^3\), the solution began to separate into layers (photo 1), its rheological characteristics decreased, and the filtration increased sharply (see Fig. 7). Thus, it can be assumed that if the reagent is used to improve the technological properties of a washing liquid, it is not recommended to exceed a concentration of 0.2 kg/m\(^3\).

3. Reagents SPECFLOC A 7950-20, SPECFLOC N6919 are flocculants with different functional groups, while DRB-9 is a selective flocculant. According to literature, a selective flocculant should flocculate cuttings, but not increase the viscosity of a bentonite solution. The test results show that when DRB-9 is added to the wash liquid, an increase in the viscosity at all concentrations is observed due to an increase in the size of the flocculas. With the addition of a larger amount, specifically, up to 0.3 kg/m\(^3\), individual molecules form flocculas with an even larger size, which leads to an increase in the filtrate yield of the solution due to an increase in the amount of free water.

The flocculants SPECFLOC A 7950-20 and SPECFLOC N6919 at a concentration of 0.1 kg/m\(^3\) have the same effect, that is, they increase the rheological and lower filtration properties of the washing liquid. With an increase in the concentration of the SPECFLOC A 7950-20 additive to 0.2 and 0.3 kg/m\(^3\), the solution was stratified and a clarified liquid appeared on the surface (photo 2). At a concentration of 0.3 kg/m\(^3\), the filtration increased sharply and the rheological properties decreased.

When SPECFLOC N6919 was added at a concentration of 0.2 and 0.3 kg/m\(^3\), the conditional viscosity and dynamic shear stress increased. However, a decrease in the static shear stress of the washing liquid was observed. This may indicate that the reagent began to manifest itself as a flocculant. In addition, low values of static shear stress can lead to the fact that the solution will not cope with its most important functions of keeping the cuttings in suspension [9].

Important indicators of the drilling fluid are gel strength and bearing capacity, which are responsible for keeping the drilled particles in suspension and cleaning the borehole from the cuttings, respectively.
Conclusion

Thus, basing on the foregoing, it can be stated that the following are distinguishing characteristics of this type of bentonite clay powder:

1. Reagents BEN-EX, BENTOPLUS and DRB-9 can be used to increase viscosity of a clay solution, provided that the additives BENTOPLUS and DRB-9 do not exceed the concentration of 0.2 kg/m$^3$.
2. Flocculant SPECFLOC A 7950-20 can be used to increase the viscosity of a clay solution in a concentration of up to 0.1 kg/m$^3$, but only after additional studies aimed at obtaining a drilling fluid with a given set of technological parameters by varying the initial concentrations of the base reagents. At a concentration of 0.2 and 0.3 kg/m$^3$, the additive affects the drilling fluid mainly as a flocculant, reducing the static shear stress of the solution and increasing the filtration yield.
3. If dynamic shear stress is the only determining parameter in the process of drilling a drilling fluid with SPECFLOC N6919 additive, then it can be recommended for being used. In all other cases, additional research is needed.

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