Development and evaluation of a novel high-density weighted fracturing fluid in ultra-deep reservoirs

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Abstract. Ultra-deep reservoirs make the fracturing operations facing some inevitable problems, especially the higher surface treating pressure, which usually exceeds the limitation of existing fracturing equipment. Weighted fracturing fluid has become one of the most effective methods to deal with this challenge. In this study, a novel high-density weighted fracturing fluid was developed with self-synthesized new weighting agent and crosslinker through extensive laboratory experiments. The density of the new weighted fracturing fluid was up to 1.46 g/cm³. The crosslinking time can be controlled within 3.1~10.2 mins, which helps in limiting the tubing friction. Performance evaluation results show that the fluid system has desired high temperature-stability and shearing resistance. Under 150 °C, 170 s⁻¹, the final apparent viscosity still remains above 50 mPa·s after shearing for 60 mins. The maximum friction reduction rate of the based fluid reached 68.4 %, which is speculated lower than the wellsite application. The newly proposed weighted fracturing fluid is a good choice for ultra-deep and high-temperature reservoirs stimulation and hence improving the recovery.

1. Introduction
Advancement of exploration and drilling technologies impel the oil and gas industry to extend depth for hydrocarbons [1-3]. However, each new extension in depth accompanied by great challenges of abnormally high pressure and ultra-high temperature [4]. Existing fracturing equipment is unable to provide such high surface treating pressures and therefore cannot guarantee safe fracturing operations. To date, two options are available to deal with this challenge, one solution is reducing the tube friction and the other is increasing the density of the fracturing fluid [5]. But it is difficult to make breakthroughs when the friction reduction rate has been exceeded 70 %, and the long transportation distance will inevitably increase the frictional pressure difference. Weighted fracturing fluid has become the most popular method to handle this issue.

Years of studies showed that the weighted fracturing fluid systems used worldwide mainly include the following: borate crosslinking system [6], carboxymethyl hydroxypropyl guar gum zirconium crosslinking system [7], viscoelastic surfactant system [8], and guar gum organoboron crosslinking system [9], etc. The main components of the weighting agents are metal salts, such as sodium chloride, potassium chloride, calcium chloride, potassium bromide, sodium bromide, sodium nitrate, potassium...
formate, sodium formate and the mixture of inorganic and organic salts [4]. However, when the fracturing fluid is weighted by divalent metal salts such as Ca$^{2+}$ and Mg$^{2+}$, it will react with formation water to precipitate and block the formation. Monovalent metal ions are less sufficient in weighting fracturing fluids, and nitrate is forbidden in many regions due to its dangerous chemicals properties. Although potassium bromide and sodium bromide perform better in aggravating, they are too expensive to be applied on a large scale. Organic salt solution usually has relatively lower crystallization temperature, which can meet the requirements of wellsite operations in winter. The fracturing fluid composed of formate solution has the characteristics of density adjustability, high temperature stability, and strong inhibition [10]. The biggest disadvantage of the formate weighting agent is the large dosage, and its solubility can reach more than 80 % at room temperature. In addition, most of the existing weighted fracturing fluid systems suffer from short cross-linking time, high friction, poor field application and other problems. Hence, research and development of a novel high-density weighted fracturing fluid for ultra-deep reservoirs is imminent.

This paper describes a novel fluid system for ultra-deep reservoirs stimulation that has a higher density than existing weighted fracturing fluids. The self-synthesized new weighting agent and crosslinker enable the density of the weighted fluid system to reach 1.46 g/cm$^3$, with a delayed crosslink that can be controlled within 3.1~10.2 mins. Performance evaluation experiments show that the new weighted fluid system can withstand up to 150 °C of formation temperature, and the maximum friction reduction rate of the based fluid reached 68.4 %, indicating the proposed high-density fracturing fluid enable meets the requirements of field hydraulic fracturing operation.

2. Materials and Methods

2.1. Experimental materials
Thickener of super guar gum JK101, industrial grade, made by Petrochemical Kunshan Co. LTD. Crosslinker YP-4, self-made; borax, ethylene glycol, glycerol, N, N-Dimethylformamide, ethyl formate, ammonium persulfate, analytical purity, provided by China State Pharmaceutical Group Chemical Reagent Co. LTD. Potassium formate (CHKO$_2$), sodium formate (CHO$_2$Na), potassium bromide (KBr) and sodium hydroxide (NaOH) were purchased from Hongyan Chemical Reagent Factory (Hedong District, Tianjin, China). All the chemicals used were of analytical grade, and no purification was required before experiments.

2.2. Experimental methods

2.2.1. Synthesis of weighting agent. Use an electronic balance to weigh 280 g potassium formate, 240 g potassium bromide and 160 g sodium formate respectively, pulverize the three solids and stir them evenly. The mixture of 40 g N, N-dimethylformamide and 40 g distilled water was added to the above mixture, and the new weighting agent JZ-1 was obtained after stirring thoroughly.

2.2.2. Synthesis of crosslinker. Put 10 g borax, 10 g glycerol and 20 g distilled water in a three-necked flask, 1 mol/L NaOH solution was used to adjust pH to about 7, heat the system up to 60 °C and stir until complete hydration. Then add 5 g glycerin and 5 g ethylene glycol at the same time. After reacting at 60 °C for 4 h, a clear and transparent organic boron crosslinker YP-4 can be obtained.

2.2.3. Preparation of weighted fracturing fluid. The weighted fracturing fluid was prepared as follow: take a certain amount of distilled water into a blender, adjust the rotation speed until the top of the shaft of the agitator blade can be seen in the vortex formed by the liquid. Slowly add the thickener JK101 guar gum, to avoid the formation of “fish eyes”. Turn off the blender after the solution is stirred evenly and leave it for a few minutes to obtain the base fluid. Add weighting agent, pH regulator and crosslinker in sequence according to the formula, and the weighted fracturing fluid system was formed after fully stirring.
2.2.4. Performance Evaluation of weighted fracturing fluid. Temperature and shearing resistance performance was tested via HAAKE MARS III rheometer provided by Thermo Fisher Scientific Co. LTD. The apparent viscosity of the prepared weighted fracturing fluid was measured under 150 °C and sheared at 170 s⁻¹. If the final viscosity remains above 50 mPa·s after shearing for 60 mins, the weighted fracturing fluid system is deemed to have excellent temperature resistance and shear resistance. The friction reduction performance was evaluated by loop friction test system. Test the frictional pressure difference generated when fresh water (ΔP_w) and the weighted fracturing fluid (ΔP_f) flow through same straight pipe respectively, and the friction reduction rate (FR) can be calculated by the following Equation (1).

\[ FR = \frac{(\Delta P_w - \Delta P_f)}{\Delta P_w} \times 100\% \]  

3. Results and Discussion

3.1. Characterization of the weighting agent

The selection principles of weighting agents for fracturing fluid generally include density, solubility, compatibility with other additives, safety and economy. Guar gum fracturing fluid usually cross-linked in a weak alkaline environment, so strong acid and weak alkaline salts (such as calcium chloride, zinc chloride, etc.) are not suitable as weighting materials. Bromate (potassium bromide, sodium bromide) is a good weighting material, the density of its saturated solution can reach more than 1.8 g/cm³, which can significantly increase the adjustable range of fracturing fluid density [4-6]. However, it is difficult to be widely used due to its high cost. Recently years, the output of formate (potassium formate and sodium formate) has been increasing year by year. The formate weighting materials have the characteristics of adjustable density, high temperature stability, and strong inhibition [10]. Moreover, it has good compatibility with reservoir fluids, and the biodegradable properties can greatly reduce damage to humans and the environment.

The new weighting agent JZ-1 combines the advantages of bromate and formate weighting agents, has high solubility, and the solution after dissolution is neutral. It has good compatibility with fracturing fluid and formation fluid, and will not affect the crosslinking of guar gum. The addition of potassium bromide can increase the density of the formate weighting solution at the same concentration, which can save production costs compared to the use of potassium bromide alone as well as significantly reduce the amount of formate. At room temperature, the relationship between the density of weighted fracturing fluid and the concentration of the novel weighting agent JZ-1 is shown in Figure 1. In general, as the mass concentration of JZ-1 weighting agent increases, the density of the fracturing fluid base liquid presents a linear increase. At a mass concentration of 54.3 %, the solution density reaches 1.46 g/cm³. By linear regression of experimental data, the linear equation between the density of weighted fracturing fluid and the mass concentration of JZ-1 can be obtained, where \( R^2 \) is 0.998, indicating that the fitting is good. Comparing the density change curve of potassium formate in the figure, it can be seen that under the same concentration conditions, the density of the new weighting agent solution is significantly higher than that of the potassium formate solution. Therefore, the dosage of potassium formate can be effectively reduced when preparing the same density solution.

According to the above experimental results, a series of base liquids with different densities were prepared to study the effect of the new weighting agent JZ-1 on the apparent viscosity of JK101 guar gum base fluid. The experimental results are shown in Figure 2. It can be seen from the figure that as the concentration of the weighting agent increases, the apparent viscosity of the fracturing fluid base fluid gradually decreases. Guar gum molecules are continuously dispersed and swelled in water to form random coils. At this time, the viscosity of the fracturing fluid base fluid is the highest. With the addition of the weighting agent, a large amount of potassium formate and sodium formate are dissolved in the water. The formate is completely ionized in the water and combined with water molecules, inhibiting the extension of guar gum molecules in the water, thus affecting the apparent viscosity of the base fluid. Comparing the apparent viscosity of potassium formate-weighted fracturing
fluid, it can be seen that under the same mass concentration, the viscosity of the fracturing fluid formed by the new weighting agent JZ-1 is generally higher than that of potassium formate-weighted fracturing fluid, with the difference being more pronounced at higher concentrations. This is because the sodium bromide in the new weighting agent can slightly increase the apparent viscosity of the guar gum base fluid [11].

![Figure 1](image1.png)

**Figure 1.** The relationship between density of fracturing fluid and mass concentration of weighting agent.

![Figure 2](image2.png)

**Figure 2.** Apparent viscosity changes with the mass concentration of weighting agents.

3.2. **Evaluation of organic boron crosslinker YP-4**

The organic boron crosslinker YP-4 is a colorless and transparent liquid, easily soluble in water, and the pH is between 6 and 7. JK101 guar gum was slowly added into the weighted brine and stirred for 15 min to make the guar gum fully swell. The fracturing fluid base liquid with a mass concentration of 0.45 % was prepared, and the pH value of the base fluid was adjusted to 11~12 with 1mol/L NaOH solution. Add a certain amount of YP-4 crosslinker to the base fluid, and stir evenly with a glass rod to form a crosslinking fracturing fluid system. The crosslinking effect of the fracturing fluid can be verified through the “tongue out” and “picking up” experiments, as shown in Figure 3. The experimental results show that the novel organic boron crosslinker has an excellent crosslinking effect.
“Tongue out” (a) and “picking up” (b) performances of the cross-linked fracturing fluid.

In order to ensure the crosslinking effect of the crosslinker and save economic cost as well, it is necessary to carry out optimization experiments on the dosage of the crosslinker. Add unequal dosages of organic boron crosslinker YP-4 to the base fluid with pH=12, observe and record the crosslinking and picking effects of the system, as shown in Table 1. The experimental results show that when the dosage of YP-4 reaches 0.55 %, the formed fracturing fluid system has relatively superior crosslinking and picking effects. Continue increasing the dosage of YP-4, the crosslinking and picking performances are still very good, but under the premise of fully considering the cost, the preferred dosage of YP-4 is 0.55 wt%.

Table 1. Crosslinking and picking effects of the formed fracturing fluid system.

| No. | The dosage of YP-4, wt% | Crosslinking effect | Picking effect  |
|-----|------------------------|---------------------|-----------------|
| 1   | 0.40                   | Weakly              | Unable          |
| 2   | 0.45                   | Weakly              | Slightly        |
| 3   | 0.50                   | Weakly              | Slightly        |
| 4   | 0.55                   | Strongly            | Completely      |
| 5   | 0.60                   | Strongly            | Completely      |
| 6   | 0.65                   | Strongly            | Completely      |

The crosslinking time of the organic boron crosslinker is affected by the pH value of solution. Appropriately prolonging the crosslinking time is beneficial to lower the friction pressure difference of wellbore flow, thereby reducing the pumping pressure of the wellhead equipment [7]. On the premise that the optimal dosage of guar gum is 0.45 % and the dosage of crosslinker is 0.55 %, this study further explored the influence of the pH value of the base fluid on the crosslinking time. The delayed crosslinking time is defined as the time at which the vortex of fracturing fluid appears to close. As shown in Figure 4, the crosslinking time of fracturing fluid increases with the increase of pH value of base fluid. An increase in the pH value leads to a slower hydrolysis of the complex compounds and a reduction of borate ions in the base solution, so the crosslinking time is prolonged. Relevant literatures show that the temperature resistance of cross-linked fracturing fluids increases as the pH increases, but excessively high pH will cause "over-crosslinking" phenomenon and seriously affect the crosslinking strength. Hence, the pH value of YP-4 crosslinker is preferably in the range of 9~12, and the crosslinking time can be controlled within 3.1~10.2 mins.
3.3. Optimization the weighted fracturing fluid formula

Through the above extensive optimization experiments about thickener, weighting agents and crosslinker of the weighted fracturing fluid, the final formula with a density of 1.46 g/cm$^3$ was determined as follows: 54.3 wt% JZ-1 + 0.45 wt% JK101 super guar gum + 0.3 wt% NaOH (1mol/L) + 0.55 wt% YP-4.

3.4. Performance of temperature and shear resistance

The weighted fracturing fluid system was prepared according to the above formula. HAAKE rheometer was employed to measure the apparent viscosity of the fracturing fluid after shearing for 60 mins under 150 °C and 170 s$^{-1}$ condition. The rheological curve of the apparent viscosity changes with temperature is shown in Figure 5. As can be seen from the figure, the final viscosity of the weighted fracturing fluid remains above 50 mPa·s after shearing, indicating the proposed fracturing fluid has desired high temperature-stability and shearing resistance performance, which can meet the requirements for most wellsites operations.

![Figure 4. Curve of crosslinking time versus pH value.](image)

![Figure 5. Viscosity-temperature curve of JK101 with 0.45 wt% concentration.](image)
3.5. Friction reduction performance

The friction reduction performance of fracturing fluid is mainly characterized by the friction reduction rate, and the loop friction test system always employed to measure this parameter. During the experiment, the displacement of the screw pump gradually increased from 250 kg/h to 2500 kg/h with an increment of 250 kg/h. However, 8 mm diameter pipeline was used in laboratory tests while in field operations 3.5 inches tube (diameter 75.9 mm) was utilized [12]. There is a fact that the flow velocity of the same displacement fluid in different pipe diameters is different, and the linear velocity can be used as a transition variable to combine the experiment results with the field practice. Moreover, it is worth noting that the friction reduction rate test only needs to use the weighted base fluid instead of the cross-linked fracturing fluid, which reflects the delayed crosslinking characteristics of the fracturing fluid during the actual wellbore flow. The friction reduction rate test result is shown in Figure 6. The friction reduction rate increases with the increase of the linear velocity, and the maximum friction reduction rate of 0.45 wt% JK101 based fluid reached 68.4 %. Since the maximum displacement in laboratory (2500 kg/h) corresponds to the field displacement of 3.70 m³/min, which is much smaller than most wellsite operation displacements. It is speculated that the actual field friction reduction rate will be greater than 68.3%.

![Figure 6. Friction reduction rate of JK101 with 0.45 wt% concentration varied with flow velocity.](image)

4. Field Application

This novel weighted fracturing fluid was used in the fracturing operation of a deep exploration well in Yubei block, Tuha oilfield, Xinjiang, China. The reservoir has a vertical depth of 5984 ~ 6017 m, and the temperature is 148 °C. According to the existing data of field operation, the breakdown pressure was about 108 MPa when using the conventional density (1.0 g/cm³) fracturing fluid system, which has a huge security risks. However, when firstly employing the novel weighted fracturing fluid with a density of 1.46 g/cm³, the construction pressure was drop to 84 MPa, the maximum injection rate was 4.8 m³/min. During the subsequent construction process, the injection pressure ranged from 78 to 86 MPa, indicating the novel fluid system can effectively decrease the surface treating pressure. After fracturing, the flowback rate was 95%, and the flowback effect was improved. Moreover, daily oil production was up to 12 t/d, which was higher than adjacent wells. The successful field application of this novel weighted fracturing fluid provides strong technical support for ultra-deep reservoir reconstruction in Tuha oilfield.

5. Conclusion

In this research, a novel high-density weighted fracturing fluid was developed with self-synthesized new weighting agent JZ-1 and crosslinker YP-4 through extensive laboratory experiments. The density of the novel weighted system was 1.46 g/cm³, which can effectively reduce wellhead equipment pressure, ensuring safe fracturing operations in ultra-deep reservoirs. Compared with traditional
formate weighting agents, the new weighting agent enable increases the viscosity of the fracturing fluid base fluid. The synthesized organic boron crosslinker YP-4 has an excellent delayed crosslinking characteristics, and the crosslinking time can be controlled within 3.1~10.2 mins. Performance evaluation experiments show this weighted fracturing fluid has desired high temperature-stability and shearing resistance. The final apparent viscosity still remains above 50 mPa·s after shearing for 60 mins under 150 °C, 170 s⁻¹. In addition, the friction reduction rate results show that the weighted system itself has good friction reduction performance, and the maximum friction reduction rate of 0.45 wt% JK101 based fluid reached 68.4 %, which is speculated lower than the wellsite application. The novel fluid system has a good field application in Tuha oilfield, and the stimulation effect is remarkable after fracturing. This study provides a good choice for ultra-deep and high-temperature reservoirs when other weighted systems are not feasible.

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