Experimental Investigation on Low-damage Nanofracture Fluid System in a Tight Reservoir Based on Nanoemulsion

He Shumei1, Zhang Jin1, Wei Pu2, Li Hui3* Yu Qin4, Yang Jing4, Lv Qi Chao3
1PetroChina Research Institute of Exploration & Development Dagang Oilfield Company,
2CNPC Xin Jiang Oilfield Xiyou Shixi Operation District, Karamay, China
3China University of Petroleum, Beijing,
4Northeast Petroleum University

*Corresponding author: lihui@cup.edu.cn; ORCID: 0000-0002-7948-4241

Abstract. A tight reservoir has low permeability, low porosity, poor pore-throat connectivity, and strong heterogeneity. Hydraulic fracture is an effective technology to develop such reservoirs. However, in the process of fracturing, fracture fluid will cause damage to the formation. This formation damage can be reduced by nanomaterials due to their nano-level structure. In this paper, experimental studies on the use of nanoemulsion to reduce formation damage caused by fracture fluid are performed through pressure conduction experiments. A low-damage nanofracture fluid system that is suitable for tight reservoirs is optimized, and the injection time is optimized. Finally, a high-efficiency and low-damage nanofracture fluid system that meets the development needs of tight reservoirs is formed. This system provides support for the application of nanomaterials in tight reservoir fracture fluid. Experimental results show that the nanoemulsion fracture fluid can reduce the permeability damage caused by conventional slick water fracture fluid with a removal rate of 16.36% and a recovery rate of 80.00%. The damage rate of nanoemulsion fracture fluid to the fracture surface is 20.33%. The damage rate of the fracture surface under the synergistic effects of nanoemulsion fracture fluid and conventional slick water fracture fluid is between 20.92% and 31.89%. The optimal injection time of nanoemulsion fracture fluid is 2 h.

1. Introduction
In tight oil and gas reservoirs, nanoscale pore-throat systems (<1000 nm in diameter) are widely developed, leading to small pore throats, low porosity, and low permeability. Horizontal well and volume fracturing are effective techniques for the development of such reservoirs (Shi et al., 2014). At present, the most commonly used fracture fluid is water-based fracture fluid (Tian, 2017). During the application of water-based fracture fluid, clay will swell, permeability will decrease, and fluid return will be slow, leading to formation damage (Nurudeen et al., 2018; Li et al., 2018). A system composed of nanoparticles has many special properties that are different from those of the common macroscopic mass material system because of the small size (1–100 nm) of nanomaterials. Adding nanomaterials to a fracture fluid system can reduce the damage of fracture fluid to formation (Wang et al., 2016). Huang et al. (2007) introduced a nanotechnology application for maintaining viscosity at high temperatures and controlling the fluid loss of VES fluid without generating formation damage. Lafitte et al. (2012) investigated the synthesis of 15 nm boronic acid-functionalized polymeric nanomaterials (also called nanolatexes) by using microemulsion polymerization. The synthesis of a series of boronic acid-functionalized nanoparticles and their application to guar crosslinking results in cost reduction, improved logistics, and less damage to the fracture conductivity. A high dosage of acrylamide based on
polymers may cause formation damage due to factors such as incomplete degradation. Liang et al. (2015) demonstrated the advantage of using selected nanomaterials to enhance the thermal stability of the crosslinked synthetic fluids and to reduce the polymer loading. Lv et al. (2017) investigated the effects of silica nanoparticles on the foam filtration performance in porous media. The negative effects of pressure drop and permeability increase to foam filtration were weakened by adding SiO2 nanoparticles. Their results indicated that silica nanoparticles can be used as a high-performance filtrate reducer for a foam fluid in porous media. Reza (2015) combined polyelectrolyte complex and silica nanoparticles successfully to reduce fluid loss when mixed with low-concentration HPG solutions for low-permeability and tight cores. However, high-permeability cores showed no sensitivity to the addition of nanoparticles.

In this paper, we use the pressure transmission method to evaluate fracture fluid with nanoemulsion additive and to optimize the injection time, thereby providing a new technical method for the application of nanomaterials in tight reservoir development.

2. Experimental theory

The pressure stability time of the permeability test is longer than that of a conventional reservoir because of the low permeability of a tight reservoir. The experimental error caused by temperature change is large, and the accuracy of experimental results is affected. The core stress sensitivity needs more time for measurement (Zhang et al., 2019; Qiu et al., 2014; Kang et al., 2013; Brace et al., 1968). Therefore, traditional steady-state pressure measurement is not suitable for tight core samples. In this paper, a pressure transmission apparatus was used to establish a new method for formation damage evaluation of tight reservoirs (Xue et al., 2016; Zhang et al., 2019; Qiu et al., 2014; Zhang et al., 2018). Fig. 1 shows a schematic of the model for measuring the permeability of a tight core sample by using the pressure transmission method. In the initial stage, the pressure in the vessel under the core sample remains unchanged at P0, and the displacement above the sample is carried out at constant pressure Pm. This physical model can be simplified to a one-dimensional flow problem to establish a mathematical model to solve permeability (Zhang et al., 2019; Zhang et al., 2018).

\[
\frac{\partial^2 P}{\partial x^2} = \frac{1}{\omega} \frac{\partial P}{\partial t}
\]

(1)

\[
\omega = \frac{K}{\phi \mu C}
\]

(2)

where \(\omega\) is the conductivity of rock, cm\(^3\)·s\(^{-1}\);
K is the rock permeability, mD;
\(\phi\) is the rock porosity, \%;
\(\mu\) is the viscosity of the fluid, mPa·s;
C is the compressibility coefficient of the fluid, mPa\(^{-1}\).

The initial condition and the boundary condition are as follows:
Initial condition: \[ P(x,0) = P_0 \] (3)

Boundary conditions: \[ P(0,t)=P_{hao} \] (4)

Then, we obtain

\[ \frac{\partial P}{\partial (L,t)} = \frac{q\mu}{KA} \] (5)

The boundary condition is modified using compressibility coefficient C, as shown below

\[ C = -\frac{1}{V} \frac{\partial V}{\partial P} \] (6)

\[ \partial V = -CV \partial P \] (7)

\[ q = \frac{\partial V}{\partial t} \] (8)

\[ q = -CV \frac{\partial P}{\partial t} \] (9)

It is concluded that

\[ \frac{\partial P}{\partial x(L,t)} = -\frac{CV \mu \partial P}{KA} \frac{\partial t}{\partial t} \] (10)

The solution of Eq. 10 is obtained as follows (Carslaw et al., 1968):

\[ P(x,t) - P_0 \]

\[ \frac{P_m - P_0}{P_m - P_0} = 1 - 2\sum_{n=1}^{\infty} \exp \left( -\Phi_n \frac{\omega t}{L^2} \right) \sin \left( \frac{x \Phi_n}{L} \right) \] (11)

where \( \Phi \) is the rock porosity, \%;

\( V \) is the downstream reservoir volume, \( \text{cm}^3 \);

\( L \) is the length of the rock sample, \( \text{cm} \);

\( A \) is the cross-sectional area of the rock sample, \( \text{cm}^2 \);

\( \Phi_n \) strongly depends on the ratio of the pore volume of the sample to the volume of the downstream reservoir. When this ratio is very small, Eq. 11 can be simplified to Eq. 13 at \( x = L \) (Tranter et al., 1959):

\[ P(L,t) - P_0 \]

\[ \frac{P_m - P_0}{P_m - P_0} = 1 - \exp \left( -\Phi_n \frac{\omega t}{L^2} \right) \cos \Phi_n \sin \Phi_n + \Phi_n \] (13)

Taking the natural logarithm of Eq. 13, we can obtain

\[ \ln \left[ \frac{P_m - P(L,t)}{P_m - P_0} \right] = \lambda t \] (14)

where

\[ \lambda = -\frac{AK}{\mu CVL} \] (15)

It is concluded that

\[ K = \frac{\mu BV L}{A} \times \frac{1}{\Delta t} \times \Delta \ln \left( \frac{P_m - P_0}{P_m - P(L,t)} \right) \] (16)

where \( A \) is the cross-sectional area of the rock sample, \( \text{cm}^2 \);

\( C \) is the compression coefficient of the working liquid, \( \text{MPa}^{-1} \);

\( V \) is the volume of the downstream container, \( \text{ml} \);

\( L \) is the length of the rock sample, \( \text{cm} \).
3. Experimental materials and methods

3.1 Core and nanomaterials description

The core samples used are tight cores that are mainly composed of feldspar sandstone and lithic feldspar sandstone with poor overall physical properties. The porosity is mainly distributed in the range of 6%–10%, the permeability is between 0.01–0.1 mD, and the reservoir throat radius is between 0.3–0.5 μm. The pore-throat separation is relatively good and most of them are microthroats with fine or medium pores (Xi et al., 2015; Lin et al., 2006), which belong to typical tight sandstone reservoirs. The experimental cores are shown in Fig. 2. The basic parameters of core samples are shown in Table 1.

![Fig. 2 Core samples used in the experiment](image)

Table 1: Basic data table of core samples

| Core No. | D (cm) | L (cm) | Porosity (%) |
|----------|--------|--------|--------------|
| C7V-1    | 2.497  | 4.951  | 5.21         |
| C7V-2    | 2.506  | 5.039  | 5.74         |
| C7V-3    | 2.488  | 5.020  | 10.05        |
| C7V-4    | 2.500  | 5.020  | 8.27         |
| C7V-5    | 2.479  | 4.981  | 5.82         |
| C7V-6    | 2.470  | 4.970  | 9.81         |

The nanoadditive used in this study is the nanoemulsion, which has a small particle size and ultra-low concentration. In terms of weight percentage, its homogeneous microemulsion is mixed with the following raw materials: 8%–40% surfactant, 0.5%–10% polymer, 10%–30% alcohol, 3%–30% oil, and water. The nanoemulsion is a homogeneous transparent liquid–liquid dispersion system in which the emulsion particle size is 5–30 nm (Figure 3).

![Fig. 3 (a) Pure nanoemulsion; (b) nanoemulsion with 0.2% concentration](image)

3.2 Experimental methods
3.2.1 Experimental apparatus

The experimental apparatus used in this study includes a pressure transmitter (Fig. 4a), surface interface tension meter (Fig. 4b), wetting angle tester (Fig. 4c), and PDP200 gas permeability tester (Fig. 4d).

![Experimental apparatus images](a) (b) (c) (d)

Fig. 4 (a) Pressure conduction device; (b) surface tension meter (BZY-2) (c) wetting angle meter; (d) PDP200 pulse attenuation gas permeability meter

3.2.2 Experimental procedures

1. Preparation of core: Epoxy resin adhesive and hardener were fully mixed at a 1:1 ratio to cement the core. After solidification, the core was cut into 0.25-inch-thick core slices. The cemented core samples and the sample slice are shown in Fig. 5.

![Cemented samples](a) (b)

Fig. 5 (a) Cemented samples; (b) Sample slice

2. The pressure transmitter was connected, and the core slice was placed in it. The upper and lower reaches were vacuumed at the same time for 1 h.

3. The treated formation water was injected into the downstream at a certain pressure (e.g., 0.1 MPa), and the downstream pressure $P_0$ was recorded after the pressure stabilized.

4. Formation water was injected into the upstream at a certain flow pressure $P_m$ (0.5 MPa) and back pressure (0.51 MPa). The change in the downstream pressure $P(L, t)$ was...
continuously monitored and recorded with time. The curve of \( \ln[(P_m-P(L, t))/(P_m-P_0)] \) and time \( t \) was plotted, and the slope of the curve was obtained. According to Eq. (16), the initial permeability \( K_0 \) was calculated.

(5) Upstream pressure was kept constant (4.5 MPa), and the conventional fracture fluid was injected in the core surface after 5 h. Steps (2)–(3) were repeated, and damage permeability \( K_1 \) was calculated.

(6) The conventional fracture fluid was replaced with nanoemulsion. The nanoemulsion was injected continuously into the upstream for 2 h. Steps (2)–(3) were repeated, and recovery permeability \( K_2 \) was calculated.

(7) According to the initial permeability, damage permeability, and recovery permeability, the damage rate, removal rate, and recovery rate can be calculated according to Eqs. 17 to 19.

\[
\text{Damage rate } \gamma : \quad \gamma = \frac{K_0 - K_1}{K_0} \times 100\% \quad (17)
\]

\[
\text{Removing rate } \nu : \quad \nu = \frac{K_2 - K_1}{K_0} \times 100\% \quad (18)
\]

\[
\text{Recovery rate } \eta : \quad \eta = \frac{K_2}{K_0} \times 100\% \quad (19)
\]

where \( K_0 \) is the initial permeability before the core was damaged;
\( K_1 \) is the damage permeability after the core was damaged by conventional fracture fluid;
\( K_2 \) is the recovery permeability after nanoemulsion was injected in the core slice;
\( K_0 \) is the initial permeability before the core was damaged.

4. Optimization and evaluation of nano fracture fluids

4.1 Reducing permeability damage by nanoemulsion

In this study, the permeability recovery rate was measured after the nanoemulsion was applied on the core, which was damaged by slick water fracture fluid. The slick water fracture fluid consists of 0.07% resistance reducer, 1% iron stabilizer, and 0.5% drainage aid.

A core sample named c7v-1 was used to investigate the nanoemulsion effect on reducing the fracture fluid damage. First, the damage to the fracture surface caused by conventional slick water fracture fluid was tested; the initial permeability \( (K_0) \) of the core slice before the injection of the conventional slick water fracture fluid is \( 2.20 \times 10^{-4} \text{mD} \), the damage permeability \( (K_1) \) after the conventional slick water fracture fluid damage is \( 1.40 \times 10^{-4} \text{mD} \), and the damage rate is 36.36%, according to Eq. 17. After the slick water fracture fluid was injected for 5 h, the nanoemulsion was continuously injected into the core for 2 h. Experimental results showed that the recovery permeability \( (K_2) \) is \( 1.76 \times 10^{-4} \text{mD} \) (Table 2). According to Eqs. 17 and 18, the removal rate of nanoemulsion on the permeability damage caused by slick water fracture fluid is 16.36%, and the corresponding recovery rate is 80.00% (Table 3).

| \( K_0 \) (\( \times 10^{-4} \text{mD} \)) | \( K_1 \) (\( \times 10^{-4} \text{mD} \)) | \( K_2 \) (\( \times 10^{-4} \text{mD} \)) |
|---|---|---|
| 2.20 | 1.40 | 1.76 |
Table 3: Damage rate, removal rate, and recovery rate of cv7-1

| Damage rate (%) | Removal rate (%) | Recovery rate (%) |
|-----------------|------------------|-------------------|
| 36.36           | 16.36            | 80.00             |

4.2 Comparison of conventional fracture fluid and nanofracture fluid

A total of 0.2% nanoemulsion was added to slick water fracture fluid to form a nanoemulsion fracture fluid system. Nanoemulsion fracture fluid and conventional slick water fracture fluid were injected into the core slices for 5 h to investigate the different effects of nanofracture fluid system and slick water fracture fluid on formation permeability damage, respectively. The experimental results are shown in Table 4. The results indicate that the damage rate of slick water fracture fluid to formation permeability is 36.36%, and the damage rate of nanoemulsion fracture fluid to formation permeability is lower, which is 20.33%.

Table 4: Experimental results before and after injury of different fracture fluids

| Cores | Fracture fluid types                  | K₀ (10⁻⁴ mD) | K₁ (10⁻⁴ mD) | Damage rate (%) |
|-------|---------------------------------------|--------------|--------------|-----------------|
| c7v-1 | slick-water fracture fluid             | 2.20         | 1.40         | 36.36           |
| c7v-2 | Nano-emulsion fracture Fluid          | 1.82         | 1.45         | 20.33           |

4.3 Optimization of injection time of nanofracture fluid

In this work, the optimal injection time of nanoemulsion fracture fluid on the fracture surface was studied. Six different injection times (0, 1, 2, 3, 4, and 5 h) of nanoemulsion fracture fluid were compared and optimized; see Table 5. The experimental results are as follows:

(1) Conventional slick water fracture fluid was injected for 5 h, and nanoemulsion fracture fluid was injected for 0 h.

The initial permeability of the core slice without fracture fluid damage is K₀ = 2.2 × 10⁻⁴ mD, the permeability of the core slice with fracture fluid damage is K₁ = 1.40 × 10⁻⁴ mD, and the damage rate is 36.36% after being invaded by conventional fracture fluid for 5 h.

(2) Conventional slick water fracture fluid was injected for 4 h and nanoemulsion fracture fluid was injected for 1 h.

The initial permeability of the core slice without fracture fluid damage is K₀ = 1.91 × 10⁻⁴ mD, the permeability of the core slice with fracture fluid damage is K₁ = 1.32 × 10⁻⁴ mD, and the damage rate is 31.89% after being invaded by conventional fracture fluid for 4 h and by nanoemulsion fracture fluid for 1 h.

(3) Conventional slick water fracture fluid was injected for 3 h, and nanoemulsion fracture fluid was injected for 2 h.

The initial permeability of the core slice without fracture fluid damage is K₀ = 1.80 × 10⁻⁴ mD. The permeability after 3 h invasion by conventional fracture fluid and 2 h invasion by nanoemulsion fracture fluid is K₁ = 1.41 × 10⁻⁴ mD, and the damage rate is 21.67%.

(4) Conventional slick water fracture fluid was injected for 2 h, and nanoemulsion fracture fluid was injected for 3 h.

The initial permeability of the core slice without being damaged by fracture fluid is K₀ = 1.99 × 10⁻⁴ mD. The permeability is K₁ = 1.57 × 10⁻⁴ mD after being penetrated by conventional
slick water fracture fluid for 2 h and continued to be invaded by nanoemulsion slick water fracture fluid for 3 h, and the damage rate is 21.11%.

(5) Conventional slick water fracture fluid was injected for 1 h, and nanoemulsion fracture fluid was injected for 4 h.

The initial permeability of the core slice without being damaged by fracture fluid is \( K_0 = 1.96 \times 10^{-4} \text{ mD} \). The permeability after being invaded by conventional slick water fracture fluid for 1 h and then by nanoemulsion slick water fracture fluid for 4 h is \( K_1 = 1.55 \times 10^{-4} \text{ mD} \), and the damage rate is 20.92%.

(6) Conventional slick water fracture fluid was injected for 0 h, and nanoemulsion fracture fluid was injected for 5 h.

The initial permeability of the core slice without fracture fluid damage is \( K_0 = 1.82 \times 10^{-4} \text{ mD} \). The permeability of the core slice with fracture fluid damage is \( K_1 = 1.45 \times 10^{-4} \text{ mD} \), and the damage rate is 20.33% after being invaded by nanoemulsion fracture fluid for 5 h.

As shown in Table 5, the damage rate of core slices under the synergistic effects of nanoemulsion slick water fracture fluid and conventional slick water fracture fluid is between 20.92% and 31.89%, which is lower than that of conventional slick water fracture fluid (36.36%). However, the damage rate is higher than that caused by only nanoemulsion slick water fracture fluid invasion (damage rate: 20.33%).

Table 5: Comparison of experimental results before and after different injection times of nanoemulsion slick water fracture fluid

| Cores | Injection time of slick water fracture fluid (hr) | Injection time of nano-emulsion fracture fluid (hr) | Initial Permeability \(10^{-4}\text{mD}\) | Permeability after damage \(10^{-4}\text{mD}\) | Injury rate (%) |
|-------|-----------------------------------------------|-----------------------------------------------|----------------------------------------|----------------------------------------|----------------|
| c7v-1 | 5                                             | 0                                             | 2.20                                   | 1.40                                   | 36.36          |
| c7v-3 | 4                                             | 1                                             | 1.91                                   | 1.32                                   | 31.89          |
| c7v-4 | 3                                             | 2                                             | 1.80                                   | 1.41                                   | 21.67          |
| c7v-5 | 2                                             | 3                                             | 1.99                                   | 1.57                                   | 21.11          |
| c7v-6 | 1                                             | 4                                             | 1.96                                   | 1.55                                   | 20.92          |
| c7v-2 | 0                                             | 5                                             | 1.82                                   | 1.45                                   | 20.33          |

According to Fig. 6, the damage rate of formation permeability decreased with the increase in the nanoemulsion fracture fluid injection time. The damage rate decreased rapidly when the nanoemulsion fracture fluid injection time is less than 2 h. When the injection time of nanoemulsion fracture fluid is more than 2 h, the effect of fracture surface damage is not obvious. Therefore, we recommend that the injection time of nanoemulsion fracture fluid should be 2 h.
5. Conclusion

(1) The nanoemulsion fracture fluid can reduce the permeability damage caused by conventional slick water fracture fluid with a removal rate of 16.36% and a recovery rate of 80.00%.
(2) The damage rate of nanoemulsion fracture fluid to the fracture surface is 20.33%, which is lower than that of conventional slick water fracture fluid (36.36%).
(3) The damage rate of fracture surface under the synergistic effects of nanoemulsion fracture fluid and conventional slick water fracture fluid is between 20.92% and 31.89%, which is lower than that of slick water fracture fluid. The optimal injection time of nanoemulsion fracture fluid is 2 h.

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