Effect of surface wettability of ceramic proppant on oil flow performance in hydraulic fractures

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Abstract
Ceramic proppants are critical products for holding induced fractures open and enhancing hydrogen productivity in low-permeability reservoirs. Though the effects of proppant size and proppant type on the well productivity have been well studied in the past, the role of the proppant surface wettability in the oil flow performance has not been thoroughly investigated. To help the readers and the industries understand the wetting behavior of ceramic proppants, this research was designed to analyze the effect of the proppant wettability on the flow efficiencies of oil and water under different conditions (for example, various proppant size, water saturation, and waterflooding). Also, it was required to determine the optimal wettability of proppants in the available candidates for the enhancement of oil recovery in the hydraulic fracturing process. Results of the work indicate that the proppant wettability plays an essential role in the productivities of water and oil in tight sandstones. Compared with the same-size oil-wet proppants, the mixed-wet proppants contribute to increasing the oil flow efficiency and reducing the produced volume of water. However, an increased value of proppant size can significantly enhance the oil recovery. Besides, using the oil-wet proppants, the waterflooding phenomenon can lead to a lower oil recovery than that of the non-water-flooded specimen. Based on the findings of the microscopic structural characteristics of the proppant surface, a fluid channel mechanism was proposed to help understand the proppant wettability effect on the flow behaviors of oil and water. This research can provide guidance on the determination of proppants in the designs of hydraulic fracturing operations. These findings can help the reservoir engineers dealing with hydraulic fracturing better design the surface wettability of the proppants, especially for those reservoirs with a multi-phase flow.

Keywords
ceramic proppants, fluid channel mechanism, hydraulic fracturing, oil flow, surface wettability, waterflooding
1 | INTRODUCTION

To successfully stimulate the productivity of the low-permeability unconventional reservoirs (e.g., tight oil sandstones and gas shale reservoirs), hydraulic fracturing has been widely used to create tiny fractures and connect natural fractures by injecting highly pressurized fracturing fluid.\textsuperscript{4} In this process, transferred with the fracturing fluid, those solid proppants play an essential role in holding the fracture open and providing a high fracture conductivity, thus leading to an economic hydrocarbon extraction from the fractured unconventional pay zones.\textsuperscript{5-7} The proppant should have a high resistance to the fracture closure stress after decreasing the fracturing pressure, in case that they would be crushed under a tremendous value of loading.\textsuperscript{5,8,9} Compared with sand-based proppants, ceramic proppants have been widely used in the hydraulic fracturing industries due to its high strength and uniform size.\textsuperscript{10,11} To maximize the fracture conductivity, the proppants are also required to be distributed uniformly inside a fracture, especially inside a fracture with a low concentration of proppants.\textsuperscript{12-15} Meanwhile, the proppant embedment into the fracture face can occur due to an increased fracture closure pressure in a depleted reservoir or a weakened rock matrix in the water-sensitive formations.\textsuperscript{5} Such a proppant embedment can significantly reduce the fracture conductivity and increase the probability of clogging tight fracture networks in the shale formations due to a softened fracture face after exposure to water.\textsuperscript{16,17} In addition, many kinds of literature have focused on the effects of types, shapes, size, and compositions of proppants on oil and gas flow efficiency.\textsuperscript{18-24} Among all the available selections, those proppants exhibiting lightweight, uniform-size, high-strength, chemically inertia, and economical cost are said to be preferable for unconventional hydrocarbon reservoirs.\textsuperscript{5,14,16-25} Moreover, the intermediate strength ceramic proppants should be more durable than clay-based ceramic proppants and sand proppants under cyclic stresses.\textsuperscript{11}

However, until now, the task of understanding the effect of proppant wettability on oil transfer performance is still tricky. The available studies of proppant wettability are mainly relevant to the clean-up of fracturing fluid residues inside the fracture. This is to increase the fracture conductivity, which can be exhausting work in traditional fracturing operations due to the water-wet surfaces of traditional proppants.\textsuperscript{26} To increase the flow back recovery, Bestaoui-Spurr et al.\textsuperscript{27-29} developed a neutrally wet proppant by bonding the hydrophobic and oleophobic molecules to the proppant surface and demonstrated it could significantly fasten the fluid movement and the clean-up of treatment fluids by eliminating the capillary pressure. This kind of neutrally wet proppant can significantly reduce the pressure losses of the multi-phase flow through the proppant pack, and increase fluid transfer conductivity of water, oil, and gas.\textsuperscript{30} For sand proppants widely used in frac operations, by altering the surface wettability of proppants from hydrophilic to hydrophobic, it can significantly achieve a higher fracture conductivity of water by maximizing clean-up of the frac fluid residues.\textsuperscript{31,32} However, in another research involved in a multi-phase transfer process, this alteration of surface wettability to hydrophobic could achieve a high fracture conductivity of oil by blocking the water movement inside the proppant matrix.\textsuperscript{33,34} The effect of proppant wettability on fluid transfer tends to diminish with increased permeability of the proppant matrix.\textsuperscript{33}

In the rock matrix, the wettability affects the relative permeability of oil and water vs water saturation by controlling the flow behaviors and spatial distribution of multi-phase fluids.\textsuperscript{35-37} One primary focus of this study is to figure out the effect of proppant surface wettability on oil and water flow performance. To help the readers understand the discrepancy in various experimental results, a fluid channel mechanism was developed to describe the different fluid flow behaviors through the proppant matrix. Meanwhile, a scanning electron microscope (SEM) was used to characterize the surface topographical characteristics of the proppants. By combining the microlevel observation with the fluid channel mechanism, we can have a better understanding of the significance in altering the surface wettability of ceramic proppants. According to the experimental conditions, the wetting behavior of the proppants on the oil/water flow was quantified and evaluated. Finally, the optimal proppants with specific surface wettability were proposed for enhancement of oil recovery in the hydraulic fracturing stimulation.

2 | EXPERIMENTAL METHODS

2.1 | Test apparatus and specimen preparation

In this research, one primary component of the equipment is the high-pressure high-temperature (HPHT) core holder shown in Figure 1. It can hold a maximum pressure of 15 000 psi and a maximum temperature up to 250°F. The core diameter is suggested to be 2 inches, and the maximum length of tested cores can reach 24 inches. After the core is put into the chamber, a confining pressure can be applied to the confining sleeve through the back pressure regulator (BPR) connected to a gas tank. Meanwhile, there are multiple pressure sensors (PT) and temperature sensors (TT) along the core holder, which can be sued to collect pressure loss and temperature changes along with the fluid transfer process. However, in this experiment, we did not do any measurements of the changes in temperature or pressure. Besides, Parker sandstone cores were used for a simulation of the low-permeability reservoir. It had a relatively low permeability of about 15-30 mD.\textsuperscript{38}

Using the same kind of tight sandstone, the cores were prepared as 22-inch-length cylinders which had a diameter of 2 inches. Meanwhile, the core also consisted of a planar diametrical aperture at one end, which had a slot width of 0.10
inch and a slot length of 6 inches to mimic a fracture. Then, three kinds of proppants were used to fill the fracture, including 20/40 mixed-wet proppant (MP), 20/40 oil-wet proppant (OP-1), and 12/18 oil-wet proppant (OP-2). The qualitative measurement on the proppant surface wettability can be seen in Figure 2. As shown in the picture below, MP can absorb both the water and the oil droplets while the oleophilic proppants of OP-1 and OP-2 exhibited a hydrophobic property. Moreover, the two-phase injection system, as shown in Figure 1, used a 49°-API-Gravity oil and the local tap water.

### 2.2 | Experimental procedure

To mimic the initial production performance of low-permeability reservoirs after being hydraulic fractured, we can follow the following instructions: Firstly, determine the rock porosity (Ø) using vacuum-saturation method and quantify the permeability (K) from displacement tests. Then, inject oil into the core at a flow rate of 10 mL/min until no more oil can be injected, which can lead to an initial water saturation (S_wi). Prepare the tight sandstone core with a 6 × 0.10 (length × diameter, in) diametrical fracture at one end and fill it up with specific proppants. In the next, simultaneously inject the multi-phase fluid at a particular injection water cut (ω) using the same fluid flow rate of 10 mL/min. Finally, collect the production-related data from the outlet near the fracture, such as the oil production rate and the water cut history, until the produced water cut reaches a stabilized cumulative water cut. Over the test procedure, the confining pressure was set at 3000 psi, and all the tests were conducted under room temperature and pressure (72°F, 14.7 psi).

To mimic the reservoirs after being water-flooded, all the procedures are the same as those demonstrated above, except the re-injection of water. After we obtain the maximum injection volume of oil, we re-inject water into the core until the intermediate produced water cut reach the injection water cut. The workflow chart of the experimental procedure is summarized in Figure 3.
2.3 | Experimental design

To investigate the effect of proppant wettability on multiphase flow performance, the experimental program was designed in Table 1.

3 | RESULTS

To describe the production characteristics, the water cut of the accumulative fluid production was used to quantify the fracture conductivity of water and oil. A high water cut means a high volume of produced water and a low amount of produced oil in the total liquids production. Also, a new dimensionless parameter was defined as the fluid injection volume (FIV) below:

\[
FIV = \frac{\sum \text{Fluid injection amount}}{\sum \text{Total pore volume}}
\]

Cumulative water cut = \[
\frac{\sum \text{Total water production}}{\sum \text{Total fluid production}}
\]
Thus, we can plot the cumulative water cut vs FIV under various conditions from the time when the fluid production occurred.

### 3.1 Proppant wettability effect on flow performance before waterflooding

As shown in Figure 4, all the samples (i.e., Specimen 1, Specimen 2, Specimen 3) exhibited a stable value of produced water cut similar with the injection water cut along with an increasing production history. To specify the changes in the cumulative water cut with an increased fluid injection volume, Figure 4A shows the differences between OP-1 proppants and the same-size MP proppants, Figure 4B shows the readers the effect of increasing the oil-wet proppant size on the oil/water flow efficiency, and Figure 4C compares the oil flow performances through MP proppants and the large-size OP-2 proppants. With the same proppant size, mixed-wet proppants displayed a slightly lower produced water cut than that of oil-wet proppants at the beginning of production, as shown in Figure 4A. This means they can increase the oil productivity and inhibit the water transfer through the proppants. Compared to OP-1 oil-wet proppants, the time required for a stable produced water cut (i.e., 40%) was significantly longer in the mixed-wet proppants. However, an increased value of the proppant size substantially contributes to improving the oil flow efficiency by blocking the water phase inside the proppants. As displayed in Figure 4B,C, the OP-2 oil-wet proppants exhibited its advantage in reducing the produced water cut which was lower than that of the OP-1 oil-wet proppants and that of the mixed-wet proppants.

### 3.2 Proppant wettability effect on flow performance after waterflooding

As defined in Figure 3, a waterflooding refers to the produced water cut immediately reached the injection water cut. After the waterflooding, the changes of cumulative water cut with an increasing fluid injection volume is shown in Figure 5A for the multi-phase flow performance within OP-1 proppants and the same-size MP proppants. Figure 5B shows the readers the effect of large-size proppant size on the oil/water flow efficiency. Besides, the oil flow performances through MP proppants and the large-size OP-2 proppants were summarized in Figure 5C. After being water-flooded, the wettability effects of proppants on the fluid flow performances were illustrated in Figure 5. The water-flooded specimens exhibited similar experimental results with the specimens from Specimen 1 to Specimen 3. Filled with the mixed-wet proppants, Specimen 5 showed a higher affinity for transferring oil because the produced water cut was lower than that of Specimen 4 containing OP-1 oil-wet proppants. Also, by altering the proppant size from 20/40 mesh to 12/18 mesh, the water production can be significantly reduced in Specimen 6, as shown in Figure 5B,C. Moreover, all these specimens displayed a similar produced water cut with the injection water cut after FIV exceeded 3.

### 3.3 Comparison of proppant wettability effects on flow performance

To study the waterflooding influence on the proppant wettability effect on fluid flow efficiency, we conducted another statistical analysis of the production data. Figure 6A shows the readers the waterflooding influence on the effect of OP-1 proppants on fluid flow efficiency while Figure 6B shows the same of OP-2 proppants. As for the effects of MP proppants on oil/water flow efficiency, the cumulative water cut was plotted in Figure 6C. As shown in Figure 6A, by comparing to a water-flooded specimen, OP-1 oil-wet proppants showed a slightly better enhancement on oil recovery in a non-water-flooded specimen. However, in Figure 6B, the large-size OP-2 oil-wet proppants tended to have a higher potential in increasing the oil flow efficiency in the water-flooded specimen. When it came to the mixed-wet proppants, MP, it did not make a big difference whether the specimen was water-flooded or not.

### 4 DISCUSSION

To enhance the completion effectiveness and the production economics, the hydraulic fracturing operation should achieve
Due to the highly water-wet surface of natural proppants, the traditional proppants can easily retain the water phase within the proppant pack.\textsuperscript{26-29} In the hydraulic fracturing process, the water entrapment can lead to a high pressure loss of the fracturing fluid within the proppant pack and a decreased fracture length.\textsuperscript{26} As a result, it usually results in a high water saturation inside the proppants pack, a decreased fracture conductivity, and lower production rate than expected.\textsuperscript{26} To reduce the amount of the water entrapment within the proppants pack, the neutrally wet proppants are encouraged by many scholars and the industries.\textsuperscript{26-29} Designed for eliminating the capillary pressure, the neutrally wet surface has no preferential affinity for both the aqueous phase and hydrogen, simultaneously being hydrophobic and lipophobic. Based on the data from indoor experiments and field applications, they have been proved to significantly increase the fracture permeability to all the phase of water, oil, and gas.\textsuperscript{26,30}

Compared with mixed-wet proppants, in this research, the oil-wet proppants resulted in higher water productivity. This can be explained by the increased hydrophobicity of the proppant surface.\textsuperscript{31} Along with an increasing hydrophobicity, the sweep efficiency of water can get increased due to the broadening of invasion fingers, which can explain the increased water recovery from OP-1 water-flooded specimens.\textsuperscript{31,39} By increasing the particle size, the dominant parameter controlling the flow performance should be related to the increased porosity and permeability. Compared with pore geometry, the effect of surface wettability of ceramic proppants on the fluid flow performance can be diminished, especially in a porous medium with a high permeability and a large amount of micro fluid channels.\textsuperscript{33,39,40} Nevertheless, those findings failed to explain the oil-increasing flow within the mixed-wet proppants pack. Since the mixed-wet proppants simultaneously had preferential affinities for oil and water, they should lead to low water recovery and low oil recovery. Thus, new mechanisms should be developed to explain the wetting behaviors of the mixed-wet proppants.

Due to the difference in the proppant wettability, oil and water can show up in various phases when transferring through the pack of proppants. As shown in Figure 7, when

**FIGURE 4** Proppant wettability effects on flow performance before waterflooding
Case A scenario is applied, a water coating can be formed on the surface of the water-wet proppant particles, thus creating an interconnective water flow channel and reducing the water flow resistance. However, the oil phase can be divided into droplets and be left discontinuously in the pore throats. As a result, the water-wet proppants can inhibit oil movement and increase the water mobility. Similarly, when oil-wet proppants are used in Case B, there can be an interconnective fluid channel of the oil phase near the proppants’ surface. As a result, it brings difficulties for the water to transfer through the oil-wet proppants pack but contributes the oil flow.

According to the fluid channel mechanism, we can explain the experimental results as shown in Figure 8. Due to its dual affinity to water and oil, the mixed-wet proppant matrix can have a fluid channel simultaneously comprised of oil and water. Thus, with an injection water cut, the fluid channels of the same-size mixed-wet proppant should be more interconnective than those of the oil-wet proppants. Meanwhile, there can be a high-oil-fraction in the two-phase fluid channel due to a low value of surface tension of oil molecules. As a result, using the same proppant size, the mixed-wet proppants should be better in transferring the oil phase than the oil-wet proppants. A high porosity can explain the proppant size effect on the oil stimulation performance. Also, a bigger oil-wet proppant size contributes to a relative smoothness of oil flow channel. As for the waterflooding effect on the proppant wettability performance, it can still be understood from the fluid channels. Due to the dual affinity of oil and water, the waterflooding should not significantly affect the conductivity of the fluid channels of the mixed-wet proppants. However, as for the oil-wet proppants, the waterflood can result in difficulties in the interconnection of the fluid channels, thus leading to decreased oil productivity in Specimen 4 with OP-1 proppants.

In addition, SEM and energy-dispersive system were used to observe the characteristics of the proppant surface. As shown in Figure 9, the resin-coated oil-wet proppant surface is much smoother than that of the mixed-wet proppant. According to the oxide analysis result, there exists a layer of oleophilic materials which can account for the oil affinity of the oil-wet proppants. Also, the complex structures of

**FIGURE 5** Proppant wettability effects on flow performance after waterflooding
FIGURE 6  Proppant wettability effects on flow performance under different conditions

FIGURE 7  Fluid transfer inside the hydraulic fractures filled with (A) water-wet proppants and (B) oil-wet proppants (modified from Raza et al's work)
mixed-wet proppant can help to explain the dual affinity of oil and water due to the capillary cohesion.

5 | CONCLUSIONS

In order to advance the understanding on the effect of surface wettability of ceramic proppants on hydrogen transfer, this research innovatively proposes an experimental study of the wetting behavior of ceramic proppants on the flow performance of oil and water through hydraulic fractures packed with proppants. In this research, tight sandstones were used as the simulation of low-permeability reservoirs while an artificial slot was prepared at one end to mimic a hydraulic fracture. Then, we filled the fracture with ceramic proppants with various surface wettability and proppant sizes. Based on the production history of water cut using the traditional displacement tests, the key findings can be summarized below:

1. For a proppant pack with a small proppant size, the surface wettability plays an essential role in determining the flow efficiency of oil and water. Using an increased proppant size, the effect of surface

![Surface topography](image)

**FIGURE 8** Multi-phase fluid channels around proppants with different wettability

![Spectrum identification](image)

**FIGURE 9** Scanning electron microscope observations to proppant surface of proppants OP-1 and MP
wettability on hydrogen transfer can be diminished due to high permeability.

2. Compared with the same-size oil-wet proppants, the mixed-wet proppants exhibited a better performance in increasing the oil flow efficiency and blocking the water inside the proppant pack.

3. The waterflooding can reduce the oil flow efficiency through large-size oil-wet proppants but increase the same through small-size oil-wet proppants. Meanwhile, it does not significantly affect the oil flow efficiency inside the mixed-wet proppants.

4. Combining with the proppant wettability and the microscopic structures of the proppant surface, a fluid channel mechanism was proposed to help the readers see the formation of interconnective fluid channels because of the preferential affinity of the proppant surface.

Though the oil-wet proppants showed similar wetting behaviors on the oil/water transfer with others’, the mixed-wet proppants were studied for the first time and need to be further developed in the future. Also, water-wet proppants and neutrally wet proppants need to be included in the study of surface wettability of ceramic proppants on the oil flow performance. Meanwhile, some quantification methods should be used to describe the degree of the hydrophilicity and the lipophilicity.

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CONFLICT OF INTEREST

The authors declare no competing financial interest.

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