

Evaluating the Performance of Surfactants in Enhancing Flowback and Permeability after Hydraulic Fracturing through a Microfluidic Model

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Summary

Hydraulic fracturing can create a large fracture network that makes hydrocarbon production from low-permeability reservoirs economical. However, water can invade the rock matrix adjacent to the created fractures and generate water blockage that impairs production. Using surfactants as fracturing-fluid additives is a promising method to enhance the fluid flowback, and thus mitigate the water blockage caused by invasion. It is imperative to understand how surfactants work during the fracturing and production stages, so as to maximize their effectiveness in production enhancement. In this study, an experimental investigation is conducted using a “chipflood” sequence that simulates fluid invasion, flowback, and hydrocarbon production from hydraulically fractured reservoirs. All experiments are conducted in a 2.5D glass micromodel that provides direct observation of in-situ phase changes when different Winsor types of microemulsions formed in the porous medium. The results provide direct evidence of the impact of the matrix–fracture interaction as well as water removal when surfactants are used. They further elucidate why surfactants under different Winsor-type conditions perform differently in mitigating the water blockage. This helps to clarify the screening criteria for optimizing flowback surfactant in hydraulic fracturing.

Introduction

Hydraulic fracturing increases the contact area with the formation, thus making hydrocarbon production from low-permeability reservoirs economical. During the hydraulic-fracturing job, a massive amount of water is pumped into the formation to create the fracture network; however, only 5–50% of water can flow back before and during the production stage (King 2012; Wasylshen and Fulton 2012; Cao et al. 2017; Xu et al. 2017c). Although a fraction of water is retained within the wellbore, in the bottom of vertical fractures (Agrawal and Sharma 2013), and in the closed “induced unpropped fractures” (Sharma and Manchanda 2015), well productivity is mainly affected by the water invading the formation adjacent to the created fractures. It has been experimentally observed that water imbibition can be pronounced even in tight and ultratight rocks with permeability ranging from hundreds of nanodarcies to tens of microdarcies (Pagels et al. 2013; Bostrom et al. 2014; Dutta et al. 2014; Engelder et al. 2014; Lan et al. 2014). Since the estimated ultimate oil-recovery rate in low-permeability reservoirs is likely less than 10% (EIA 2015; Wachtmeister et al. 2017; Hu et al. 2018) and the production is mainly attributed to the formation adjacent to the propped fractures (Patzek et al. 2014; Yu and Sepehrnoori 2014; Male et al. 2016; Zuo et al. 2016), the invading water, even if not deep into the formation, can hinder hydrocarbon flow from the formation to the fractures. This eventually impairs the well productivity because of the multiphase-flow effect and, in particular, the matrix–fracture interaction (Longoria et al. 2017; Liang et al. 2017b, 2018).

In the field, there are two methods applied to mitigate the blockage caused by the invading water: shutting in the well and using surfactants as fracturing-fluid additives (Liang et al. 2017b). Shutting in the well allows the invading water to dissipate into the deeper formation through spontaneous imbibition (Almulhim et al. 2014; Bertoncello et al. 2014; Dutta et al. 2014). However, this works only within the water-wet region of the formation, and its efficiency decreases with the square root of rock permeability (Liang et al. 2018). Therefore, for low-permeability reservoirs, shutting in the well requires a long delay of hydrocarbon production. In addition, the contact of the reservoir rock with the fracturing fluid can trigger clay swelling in sandstones and shales (Madsen and Müller-Vonmoos 1989; Caenn et al. 2011; Gupta et al. 2013) and matrix softening that aggravates proppant embedment under the closure stress (Alramahi and Sundberg 2012; Das et al. 2014). Both of the above would potentially impair the well productivity after the shut-in period.

Using surfactants as fracturing-fluid additives can either promote spontaneous imbibition to dissipate the invading water into the deeper formation (Wang et al. 2012; Kim et al. 2016; Neog and Schechter 2016; Alvarez et al. 2017; Liang et al. 2017c) or promote flowback to recover the invading water (Liang et al. 2015; Kim et al. 2016; Shuler et al. 2016; Liang et al. 2017a, 2017b). Although both approaches can mitigate the water blockage and enhance the relative permeability to hydrocarbon, the former requires a larger use of surfactants to sustain the imbibition; considering the loss of surfactant onto the rock surface by adsorption, this approach might not be economical for low-permeability rocks that have small pores and, thus, a large specific surface area (Loucks et al. 2009; Nelson 2009). The second approach is to achieve an ultralow oil–water interfacial tension (IFT) to reduce the invasion depth of water and also the capillary pressure that retains the water within the pore space, especially near the fracture face (Liang et al. 2017a, 2017b). Ultralow-IFT surfactants can be screened according to reservoir conditions (including temperature, pressure, and salinity), just as those screened for chemical enhanced oil recovery (CEOR) (Hirasaki et al. 2011; Lake et al. 2014). However, in contrast to the CEOR approach, mitigating water blockage does not require the surfactants to migrate deep into the formation, and it can be applied in both water-wet and oil-wet reservoirs (Liang et al. 2017c). Therefore, using ultralow-IFT surfactants to promote flowback is a promising method to enhance the production after hydraulic fracturing, and it is necessary to understand how this type of surfactant works during the fracturing and production stages, so as to maximize surfactant effectiveness in production enhancement.

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Previous studies using surfactants and computed-tomography (CT) -monitored corefloods have also shown that ultralow-IFT surfactants can perform differently in mitigating water blockage and enhancing rock relative permeability to oil at different salinities when different Winsor types of microemulsions (Winsor 1948) form in situ: permeability-regaining performance is found to be the best under Winsor-I conditions, followed by Winsor-III conditions; it is at the worst under Winsor-II conditions (Liang et al. 2017a). Lacking a direct observation on phase changes during the mixing process in the porous medium, it remains unknown which properties control the performance of ultralow-IFT surfactants in enhancing the flowback and rock relative permeability to oil.

Microfluidic models (i.e., micromodels) can provide a means for direct observation of multiphase flow within porous media. Starting with a single layer of closely packed glass beads between two parallel glass plates in the 1950s (Chatenever and Calhoun 1952), micromodels have evolved into fabricated chips with complex pore networks and/or flow channels/pores in the micro-meter to submicro-meter scale (Karadimitriou and Hassanizadeh 2012; Xu 2018). Among various materials, glass/silicon and polydimethylsiloxane (PDMS) are two commonly used materials to fabricate the transparent micromodels for direct observation (Abgrall and Gué 2007; Ren et al. 2013). Complex-pore-network models with submicro-meter pore-throat sizes are easier to manufacture with PDMS than glass/silicon. However, PDMS models are oil-wet and they are difficult to alter to water-wet state. Second, PDMS models are hard to fabricate with 3D or pseudo-3D (i.e., 2.5D) features that can better approximate a real rock (Karadimitriou et al. 2013; Ren et al. 2013; Xu 2018). Third, PDMS itself is permeable, thus it can swell and deform after coming in contact with oil. For these reasons, PDMS models are more suitable for observing the displacement between gas/foam and water within porous media (Ma et al. 2012; Conn et al. 2014).

Xu et al. (2017a) developed an isotropic etching technique that can fabricate a 2.5D glass micromodel with 3D features at the pore throats. This new model enables capturing flow behaviors such as “capillary trapping” (Lake et al. 2014) and “capillary snap off” (Roof 1970) within pores, and thus provides a good approximation of real rock. The 2.5D glass micromodels have been successfully applied to observe the effect of a nanoparticle/surfactant-stabilized emulsion system on enhanced oil recovery (EOR) (Xu et al. 2017b) and the formation of a Winsor III microemulsion phase in the porous medium during the coinjection of an oil and surfactant solution (Tagavifar et al. 2017).

In this study, an equivalent three-step “chipflood” sequence [like the three-step coreflood sequence introduced in studies by Liang et al. (2017a, 2017b)] was conducted in a 2.5D glass micromodel. This micromodel enabled the direct observation of in-situ phase changes during the mimicked fracturing-fluid invasion and the flowback when different Winsor microemulsions formed in the porous medium. The observation could supplement the previous analyses from CT-monitored corefloods (which provide only changes of water saturation) and phase behavior observed in pipette tests, and further reveal the key properties controlling the performance of surfactants in enhancing flowback. The results of this study provide direct evidence of the impact of the matrix–fracture interaction (i.e., the capillary end effect at the fracture face) and its removal when the surfactant is used, elucidate why the Winsor-I condition leads to the best performance of the flowback surfactant, and clarify the screening criteria of flowback surfactants for mitigating water blockage and enhancing oil production after fracturing.

Materials and Methods

Micromodel. The 2.5D glass micromodel was fabricated using isotropic hydrofluoric acid etching (Xu et al. 2017a). A schematic and the microscopic characterization of the model are shown in **Fig. 1**. In this micromodel, the dimensions of pore throats were significantly smaller than those of pore bodies, both horizontally and vertically. This pore-throat geometry enables capturing different key multiphase-flow behavior caused by capillarity, thus making this model a powerful tool for observing the formation and mitigation of water blockage in the presence or absence of surfactants. **Table 1** lists the detailed geometric dimensions of the micromodel used in this study.

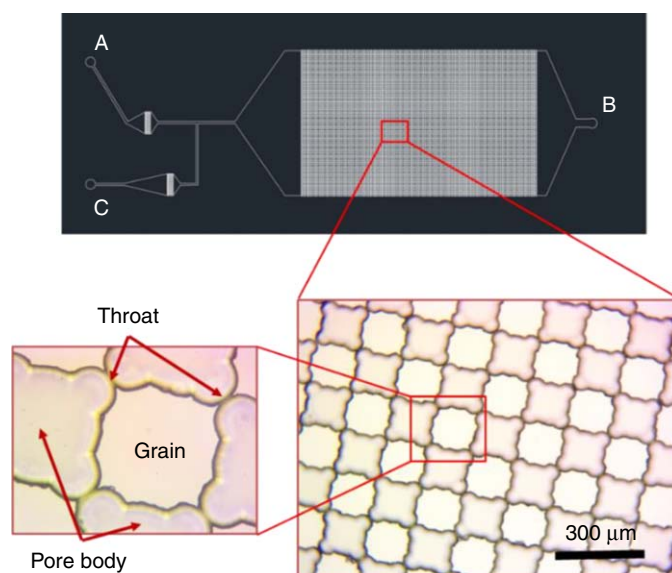


Fig. 1—Schematic (top) and microscopic characterization (bottom) of a 2.5D glass micromodel (Xu et al. 2017a).

Pore diameter	200 μm	Matrix length	3.8 cm
Pore depth	20 μm	Matrix width	2.4 cm
Throat width	23 μm	Pore volume	7 μL
Throat depth	3 μm	Porosity	38.5%
Matrix wettability	Water-wet		

Table 1—Geometric dimensions of 2.5D glass micromodel.

Choice of Hydrocarbon and Surfactant-Aided Fracturing Fluid. In this study, *n*-pentane and *n*-hexane were chosen to mimic the light oil typically produced from tight oil reservoirs.

Water with and without surfactant additives was chosen to mimic the water-based fracturing fluid. Although components such as friction reducer or gel can cause significant formation damage, the focus of this study was the formation and mitigation of water blockage caused by the water phase. For the surfactant additive, the identical formulation as introduced in studies by Liang et al. (2017a, 2017b) was used to form microemulsions between water and either of the above alkanes. It was a mixture of an internal olefin sulfonate, an alcohol propoxylated sulfate, and isobutyl alcohol, with a total concentration of 1 wt% in deionized (DI) water (i.e., 1 wt% surfactant). Sodium chloride (NaCl) was used to tune the salinity of this surfactant solution, so that different Winsor microemulsions could be formed. Through pipette phase-behavior tests, it was found that 1 wt% surfactant with a salinity of 50,000 ppm generated a Winsor-I microemulsion with *n*-hexane and with 60,000 ppm salinity generated a Winsor-III microemulsion with *n*-hexane; with 57,500 ppm salinity, it generated a Winsor-II microemulsion with *n*-pentane. Because the salinity required for this surfactant formulation to generate a Winsor-II microemulsion with *n*-hexane was beyond the salinity limitation of forming a stable solution without precipitation, *n*-pentane was used as the hydrocarbon to test Winsor-II conditions. Fig. 2 shows the results of phase-behavior tests in the pipettes at three different salinities when different Winsor microemulsions formed. From the solubilization ratio read from each pipette, reduction in oil–water IFT could be calculated using the Huh equation (Huh 1979; Levitt et al. 2009). IFT was 1.4×10^{-3} mN/m when Winsor-I microemulsion forms, 3.3×10^{-4} mN/m when Winsor-III microemulsion forms, and 2.9×10^{-3} mN/m when Winsor-II microemulsion forms.

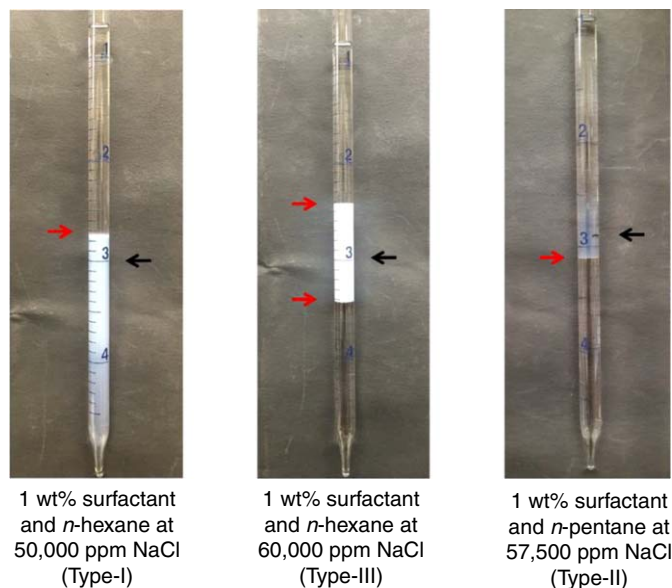


Fig. 2—Results of phase-behavior tests between *n*-pentane or *n*-hexane and 1 wt% surfactant at different salinities at 25°C. Hydrocarbon (2 mL) and surfactant solution (2 mL) were added to each pipette, which were then gently shaken several times and allowed to equilibrate. Black arrows point to the interfaces before mixing, and red arrows point to the interfaces after mixing and being equilibrated. All surfactant solutions passed the aqueous stability test without any precipitation at their assigned salinities.

“Chipflood” Schemes. A three-step chipflood sequence, equivalent to the coreflood sequence introduced in the study by Liang et al. (2017a), was conducted in the glass microfluidic chip. The experimental temperature was kept at 25°C during the coreflood and phase-behavior tests of the chosen surfactants. All chipflood experiments were conducted under atmospheric pressure; however, it has been reported that the pressure has a weak influence on the phase changes at different salinities within the tested range (Nelson 1983; Roshanfekr et al. 2012; Jang et al. 2014). The three-step process consists of saturating with hydrocarbon, invading with the fracturing fluid, and then flowing back with hydrocarbon. The entire experimental process, including the formation and removal of microemulsions when different surfactants were used, was directly recorded with a microscope that was equipped with a digital camera.

To study the influence of surfactants on mitigating the water blockage, it is crucial to choose an appropriate hydrocarbon flow rate in Step-3 of this chipflood sequence. The chosen flow rate (or superficial velocity) can create a similar flow condition as the ones in the previous corefloods with tight rocks and allow two major forms of capillarity-dominated water blockage to show in the micromodel: capillary trapping and matrix–fracture interaction. In previous corefloods, both forms of water blockage were observed at a superficial velocity of hydrocarbon up to 2.58 ft/D in 8-md Texas Cream Limestone, or up to 1.30 ft/D in 3.2-md Indiana Limestone and 0.35-md Lueders Limestone (Liang et al. 2017a, 2017b, 2018). In this study, the capillary number (N_c) was kept at the same order of magnitude as previous studies to ensure the success of observing both forms of water blockage in the micromodel; the chosen N_c was approximately 4.6×10^{-8} in the baseline case, which was in the same range as tight oil production (10^{-6} to 10^{-9}) (Longoria et al. 2017). Meanwhile, considering the limitation of the current experimental setup, the superficial velocity was chosen at 2.17 ft/D (7 μ L/h within the chip) in Step 3 of the chipflood sequence. This hydrocarbon velocity is larger than that in the tight rocks during the flowback stage, where the water blockage is longer and severer (Longoria et al. 2017; Liang et al. 2018). Nevertheless, the appropriateness of the chosen velocity depends on whether both forms of water blockage can be observed in the micromodel before a surfactant is used, and this was examined before the comparative experiments started.

Before starting the three-step chipflood sequence, the glass chip was rinsed with DI water, then with ethanol, and finally with the hydrocarbon that would be tested (i.e., *n*-hexane or *n*-pentane). When this had been done, the chip was dried at 100–120°C. Afterward, the hydrocarbon and the mimicked fracturing fluid were injected into the glass chip from opposite ports in different steps of the sequence, described as follows.

Step 1: Reservoir Saturated with Hydrocarbon. After the glass chip cooled down, the hydrocarbon (either *n*-hexane or *n*-pentane) was injected from Port A (see Fig. 1). In the beginning, Port B and Port C were open for purging. Once the glass chip and all tubings were fully saturated with hydrocarbon, Port A was closed and Port B was connected to the water-injection pump.

Step 2: Fracturing-Fluid Invasion. The mimicked fracturing fluid (either pure DI water or 1 wt% surfactant at different salinities, all dyed blue) was injected into the glass chip from Port B toward Port C. This step was stopped after the fluid swept two-thirds of the matrix (i.e., the invasion depth was kept constant for DI water and the surfactant solution, not the invasion volume). The injection rate was 30 $\mu\text{L/h}$ during this step, which was approximately four times that of Step 3; this matched the procedure followed in the study by Liang et al. (2017a). In brief, this step simulates the loss of fracturing fluid into the matrix as well as its interaction with hydrocarbon before flowback and production starts. The water-invasion depth was maintained the same in all chipflood tests for comparing the influence of different surfactants.

Step 3: Fracturing-Fluid Flowback during Hydrocarbon Production. Once the invasion process was finished, Port B was switched to the outlet and Port C was sealed. Hydrocarbon (either *n*-hexane or *n*-pentane) was injected from Port A at the constant flow rate of 7 $\mu\text{L/h}$ (2.17 ft/D) for at least 2.5 pore volumes (PV) for the microscopic observation. In brief, this step simulates the hydrocarbon production from the deeper region of the formation rock toward the open fracture.

In tight rocks, water can accumulate at the fracture bottom because of to gravity and the water blockage in the near-fracture region of the matrix cannot be mitigated until the water in the fracture is cleaned up (Sharma and Agrawal 2013). However, the influence of gravity is insignificant on water blockage within the matrix of tight rocks where the capillary pressure is large; this was observed in previous studies using CT-monitored corefloods (Liang et al. 2017a, 2018). Therefore, all chipflood tests were conducted horizontally and the influence of gravity was not considered in this study.

Results and Discussion

Before testing the surfactant additives, dyed DI water was used as the mimicked fracturing fluid and tested in the micromodel to establish a baseline. All the tests followed the three-step chipflood sequence introduced above. For a simpler comparison, the results obtained from Step 2 are presented in the Simulation of Fracturing-Fluid Invasion (Step 2) subsection, while the results obtained from Step 3 are presented in the Simulation of Fracturing-Fluid Flowback (Step 3) subsection.

Simulation of Fracturing-Fluid Invasion (Step 2). Step 2 simulated the invasion of the fracturing fluid into the rock matrix during hydraulic fracturing. In this step, the mimicked fracturing fluid (either DI water or 1 wt% surfactant at different salinities, all dyed blue) was injected at a constant flow rate into the hydrocarbon-saturated micromodel from the right-hand side (Port B) to the left-hand side (Port C).

Water Invasion without Surfactant. This subsection shows the results of the baseline measurement, in which DI water invaded the *n*-hexane-saturated micromodel. **Fig. 3** (left column) shows the change in phase saturations within 20 seconds (i.e., 0.024 PV) during this step. It can be observed that water (blue fluid) migrated along the wall/edges of pore bodies and pore throats, resulting in bypassed oil (white fluid) in the center of every pore body. Although the local invasion profile was irregular, the overall invasion front of water was almost uniform from a macroscopic view; this agrees with the viscosity ratio (mobility ratio) that generates a favorable displacement (i.e., piston-like displacement), where the mobility ratio was less than 0.05.

Since this micromodel was water-wet, spontaneous imbibition could happen and drive water to migrate along the pore wall/edges, leading to a similar invasion profile. To test the impact of this driving force, the pump was turned off after the designed volume of water invaded the matrix, while the water source was still connected to the injection port; this allowed the spontaneous imbibition to occur by itself within the porous medium. **Fig. 3** (right column) shows the change in phase saturations during this spontaneous imbibition within 20 seconds. Although water migration was also observed at the local water invasion fronts, its speed was much slower than that of the invasion in Step 2 (left column of **Fig. 3**). Therefore, the imbibition profile observed in Step 2 was mainly driven by the viscous force (i.e., injection pressure), and it was not an artifact of spontaneous imbibition in the water-wet porous medium.

Water Invasion with Surfactant under Winsor-I Conditions. After the baseline measurement, the identical chipflood scheme was repeated except that the mimicked fracturing fluid was changed to 1 wt% surfactant under Winsor-I conditions. **Fig. 4** shows the change in phase saturations when this surfactant solution invaded the *n*-hexane-saturated micromodel. Similar to the baseline, the invasion front migrated uniformly with time from the right-hand side to the left-hand side of the micromodel. However, residual oil saturation was significantly reduced compared to the baseline case (**Fig. 3**). Meanwhile, it was also observed that numerous small oil droplets were formed within the invaded region, among which the droplets near the invasion front (i.e., near the oil–water interface) had fuzzier edges than those near the invasion tail (i.e., near the water injection port). A close-up view of oil droplets within the near-invasion-front region is shown in **Fig. 5**. Submicrometer-sized oil droplets (i.e., diameter < 1 μm) can be observed as well as the blurred interface between the oleic phase (in white) and the aqueous phase (in blue); both are likely the evidence showing the formation of Winsor-I microemulsions. This agrees with the expectation that the microemulsion forms at the invasion front where the oleic phase and the aqueous phase initially mix with each other (Lu and Pope 2017).

Water Invasion with Surfactant under Winsor-III Conditions. The chipflood scheme was repeated for 1 wt% surfactant under Winsor-III conditions, and **Fig. 6** shows the change in phase saturations when this surfactant solution invaded the *n*-hexane-saturated micromodel. It was observed that the invasion front also migrated uniformly with time from the right-hand side to the left-hand side of the micromodel. Like Winsor-I conditions (**Fig. 4**), reduced residual oil saturation and numerous small oil droplets were both observed within the invaded region. However, unlike Winsor-I conditions, small oil droplets were found only within the first two-thirds of the region behind the invasion front; in the region near the matrix inlet (i.e., the invasion tail), an elongated mixed phase was observed. **Fig. 7** provides a close-up view of this mixed phase within the near-invasion-tail region. It can be clearly seen that the blurred mixed phase (in light blue) was elongated with the flowing aqueous phase (in blue), forming a belt-like pattern. Accompanied by this mixed phase, submicrometer-sized oil droplets were also observed within this region. These observations likely indicate that the Winsor-III microemulsion forms near the invasion tail where the mixing was sufficient. This is different from the expectation that a microemulsion forms at the invasion front where the oleic phase and the aqueous phase initially mix with each other (Lu et al. 2014; Lu and Pope 2017).

To explore this phenomenon more deeply, an identical experiment was repeated with a special focus on the phase change within the invasion tail. As pointed out by red arrows in **Fig. 8**, residual oil droplets were observed behind the solid grains of the micromodel (closed transparent circles) after approximately 0.3 PV of 1 wt% surfactant flood (top left figure). After another 0.3 PV of the flood, these oil droplets were gradually sheared by the flowing surfactant solution and eventually disappeared; meanwhile, the belt-like mixed

phase was formed. These observations indicate that the formation of Winsor-III microemulsion is likely both time dependent and shear dependent; it does not form right after the initial mixing of the surfactant solution and the hydrocarbon. At the end of this flood, the residual oil saturation, in the form of either residual oil droplets or the mixed phase, was almost zero; this was expected for such an ultralow-IFT condition.

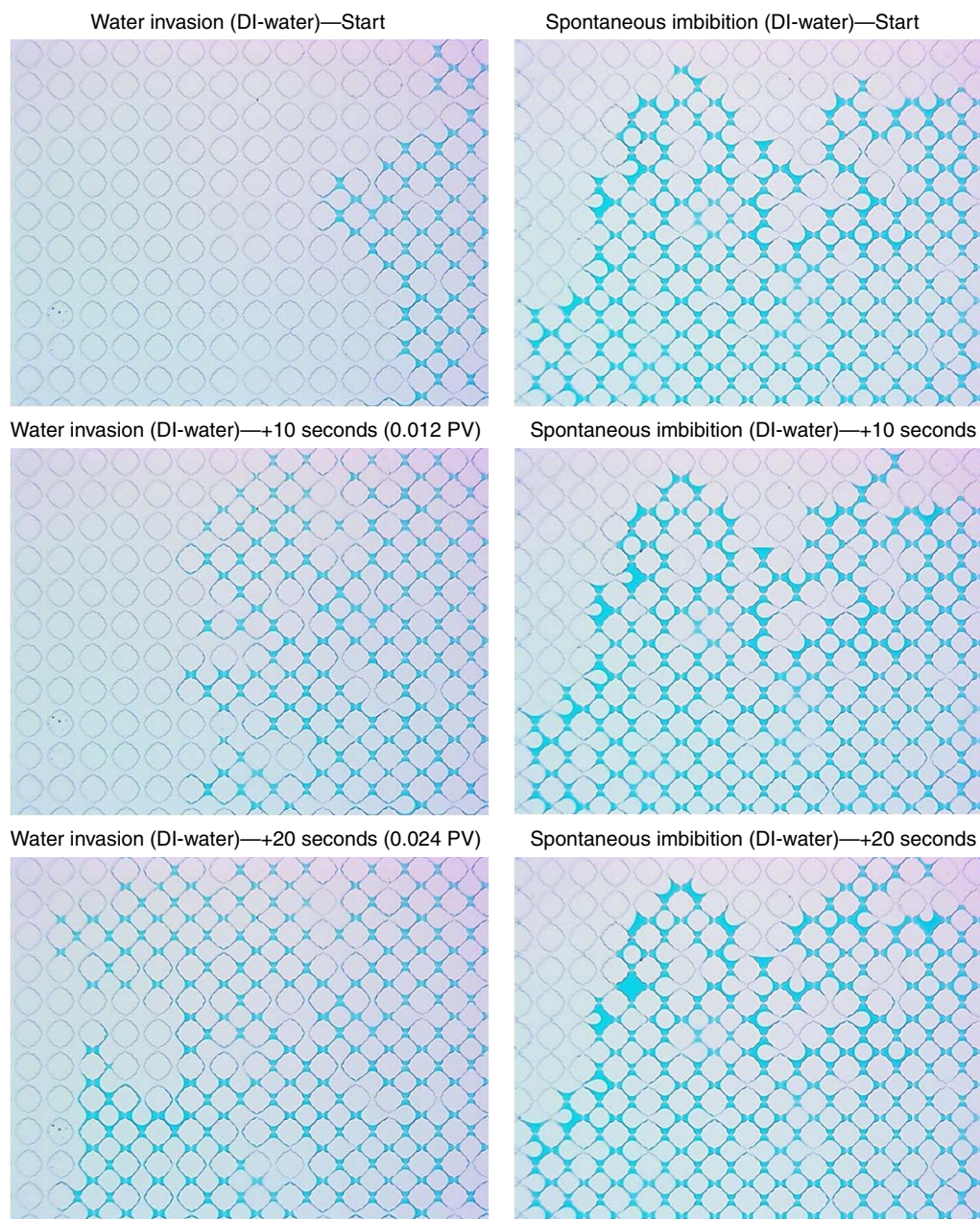


Fig. 3—Change in phase saturations during water invasion (left column) or pump shut-in (right column) within 20 seconds. Closed transparent circles are solid grains of the micromodel, and the remainder of the space is the porous medium. Within the pore space, water is blue and *n*-hexane is white. In this step, water is applied from the right-hand side.

Water Invasion with Surfactant under Winsor-II Conditions. Fig. 9 shows the change in phase saturations when 1 wt% surfactant under Winsor-II conditions invaded the *n*-pentane-saturated micromodel. It was also observed that the invasion front migrated uniformly with time from the right-hand side to the left-hand side of the micromodel. However, unlike the Winsor-I and -III conditions (Figs. 4 and 6), an extensive mixed phase (in light blue) immediately formed behind the invasion front. Fig. 10 provides a close-up view of the formation of this new phase during the invasion. As can be seen in this figure, right after 1 wt% surfactant invaded the hydrocarbon-saturated pores, two fluids mixed with each other and formed a relatively homogeneous phase. Compared with the previous two surfactant conditions, this new phase is very likely the microemulsion phase (i.e., Winsor type-II microemulsion), and it almost spread across the entire invaded region. In this condition, small scattered oil droplets were not observed within the invaded region; residual oil was only present as the microemulsion phase. With a decrease of the volume percentage of the microemulsion phase from the invasion front to the invasion tail, residual oil saturation also decreased. This trend agrees with the CT results of corefloods shown in the study by Liang et al. (2017a).

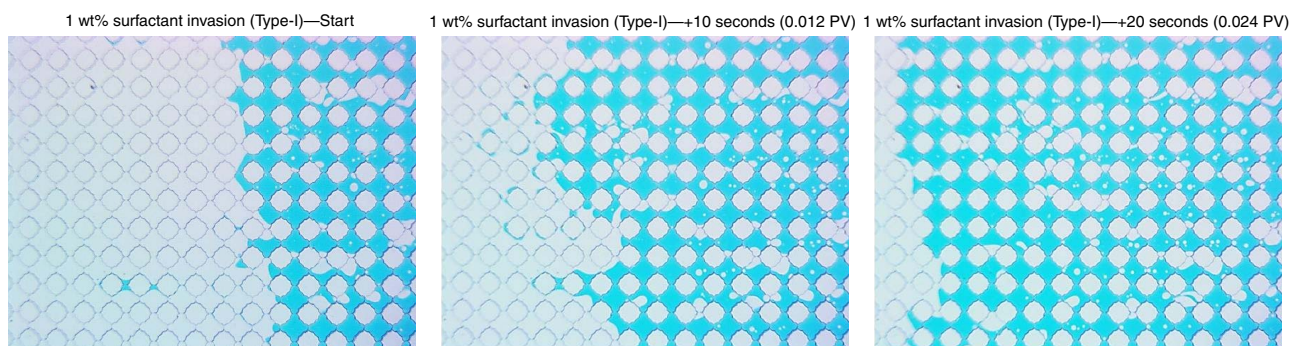


Fig. 4—Change in phase saturations during the invasion of 1 wt% surfactant under Winsor-I conditions within 20 seconds. Closed transparent circles are solid grains of the micromodel, and the remainder of the space is the porous medium; dyed DI water is blue, while *n*-hexane is white.

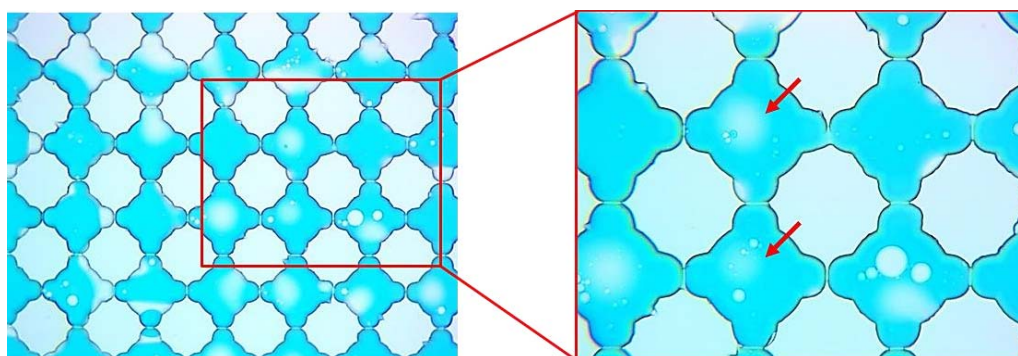


Fig. 5—Close-up view of the near-invasion-front region after 1 wt% surfactant under Winsor-I conditions invaded the micromodel (end of Step 2). Red arrows point to the fuzzy clusters that are likely Winsor-I microemulsions.

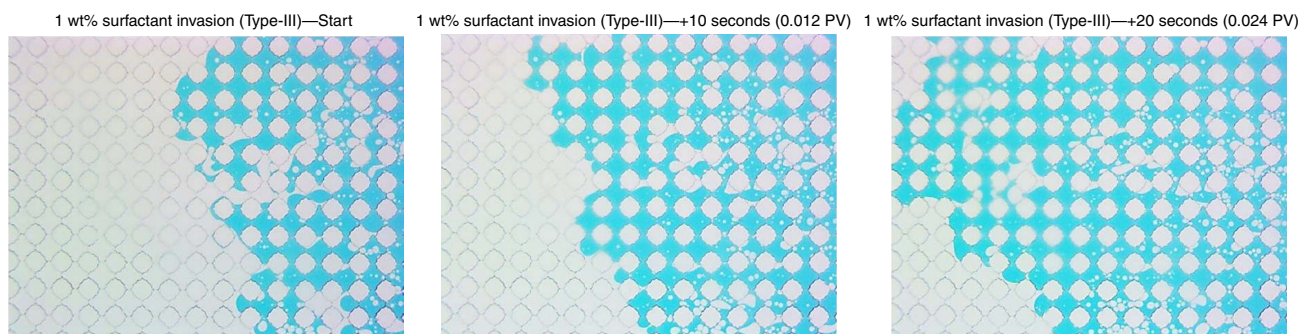


Fig. 6—Change in phase saturations during the invasion of 1 wt% surfactant under Winsor-III conditions within 20 seconds. Closed transparent circles are solid grains of the micromodel, and the remainder of the space is the porous medium; dyed DI water is blue, while *n*-hexane is white.

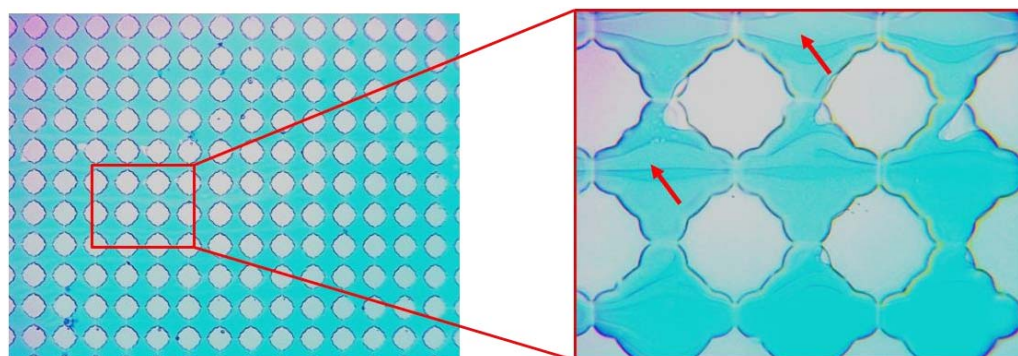


Fig. 7—Close-up view of the near-invasion-tail region after 1 wt% surfactant under Winsor-III conditions invaded the micromodel. Red arrows point to the fuzzy clusters that are likely microemulsions.

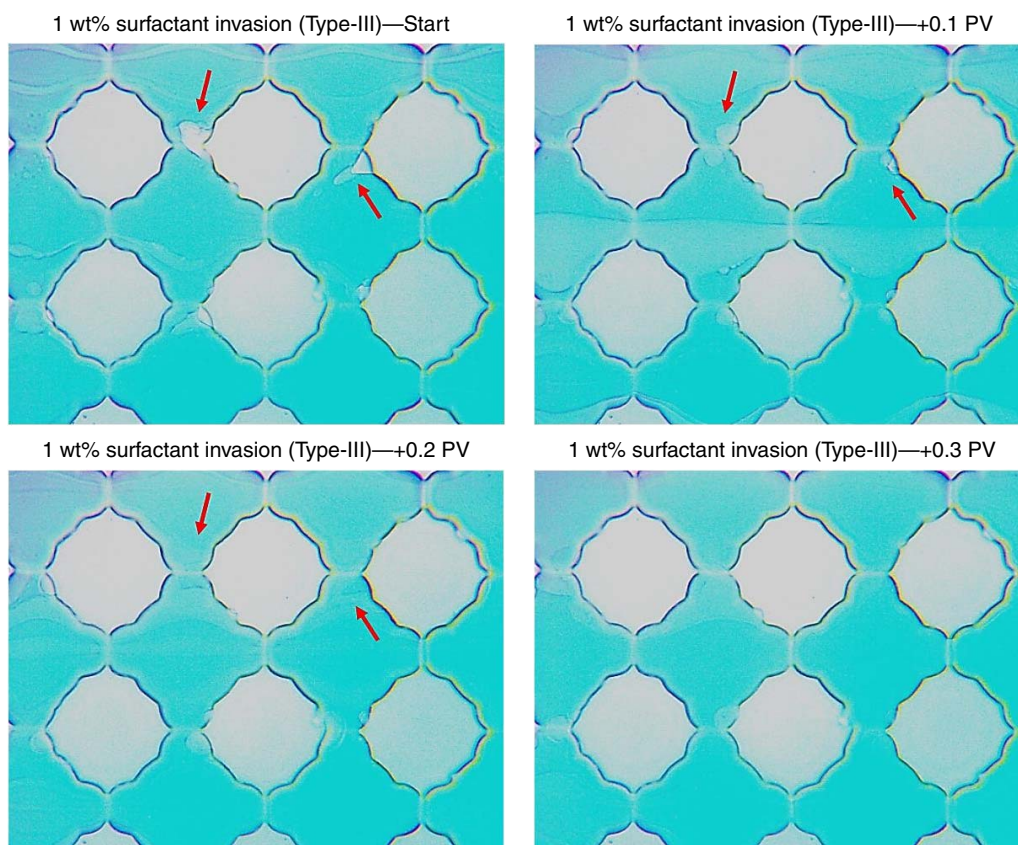


Fig. 8—Close-up view of the formation of Winsor-III microemulsions. Red arrows point to the residual oil droplets that were gradually sheared to form microemulsions and carried away by the surfactant solution.

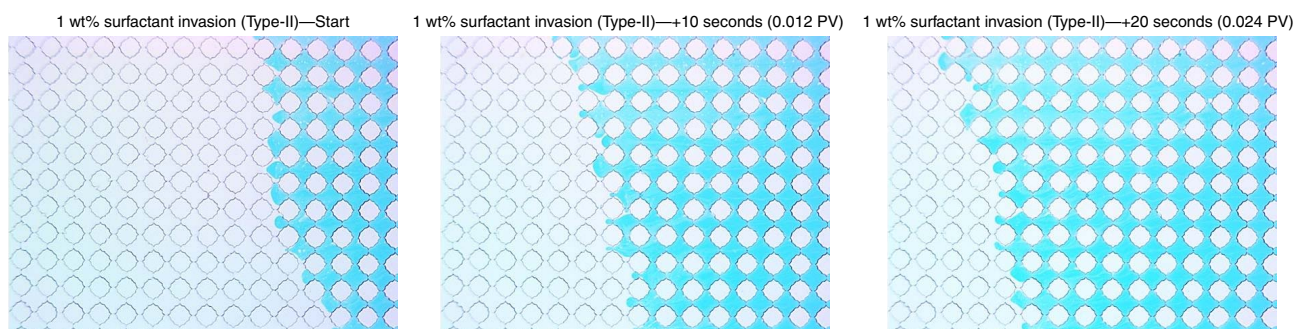


Fig. 9—Change in phase saturations during the invasion of 1 wt% surfactant under Winsor-II conditions within 20 seconds. Closed transparent circles are solid grains of the micromodel and the remainder of the space is the porous medium; dyed DI water is blue, while *n*-pentane is white.

Simulation of Fracturing-Fluid Flowback (Step 3). After the water invasion (Step 2), hydrocarbon was injected at a constant flow rate into the micromodel from the left-hand side (port A) to the right-hand side (port B). As introduced in the “Chipflood” schemes section, Step 3 simulated the flowback and hydrocarbon production through the water-invaded rock adjacent to the open fracture.

Flowback without Surfactant. This section shows the results of the baseline measurement, in which *n*-hexane flowed through the DI water-invaded micromodel at a constant flow rate. **Fig. 11** shows the change in phase saturations within 180 seconds (i.e., 0.05 PV) during this step. It can be observed that *n*-hexane (white fluid) fingered through the invading water (blue fluid) and resulted in a significant amount of water being bypassed. This agrees with the expectation for the low viscosity fluid (*n*-hexane) displacing the high viscosity fluid (water), where the mobility ratio was larger than 20 in this case.

After 2.5 PV of *n*-hexane flood, numerous bypassed/trapped water islands were observed in the original water-invaded region. They were likely to remain trapped because *n*-hexane had broken through the water-invaded region and created the preferred flow paths. Moreover, it was also observed that an extra volume of water was trapped near the outlet of the micromodel matrix because of the matrix–fracture interaction (**Fig. 12**, left). This was attributed to the capillary discontinuity at the outlet of the matrix that prevented water from leaving (i.e., capillary end effect at the fracture face). Therefore, the hydrocarbon needed to snap off through this layer of water and then flow into the open space that mimicked the fracture (**Fig. 12**, right). A short video that shows the impediment of this form of water blockage to hydrocarbon flow, the degree and time span of which have been detailed in previous studies (Liang et al. 2017b, 2018) is available at https://v.youku.com/v_show/id_XNDQ0NjcyODU4OA==.html?spm=a2h3j.8428770.3416059.1.

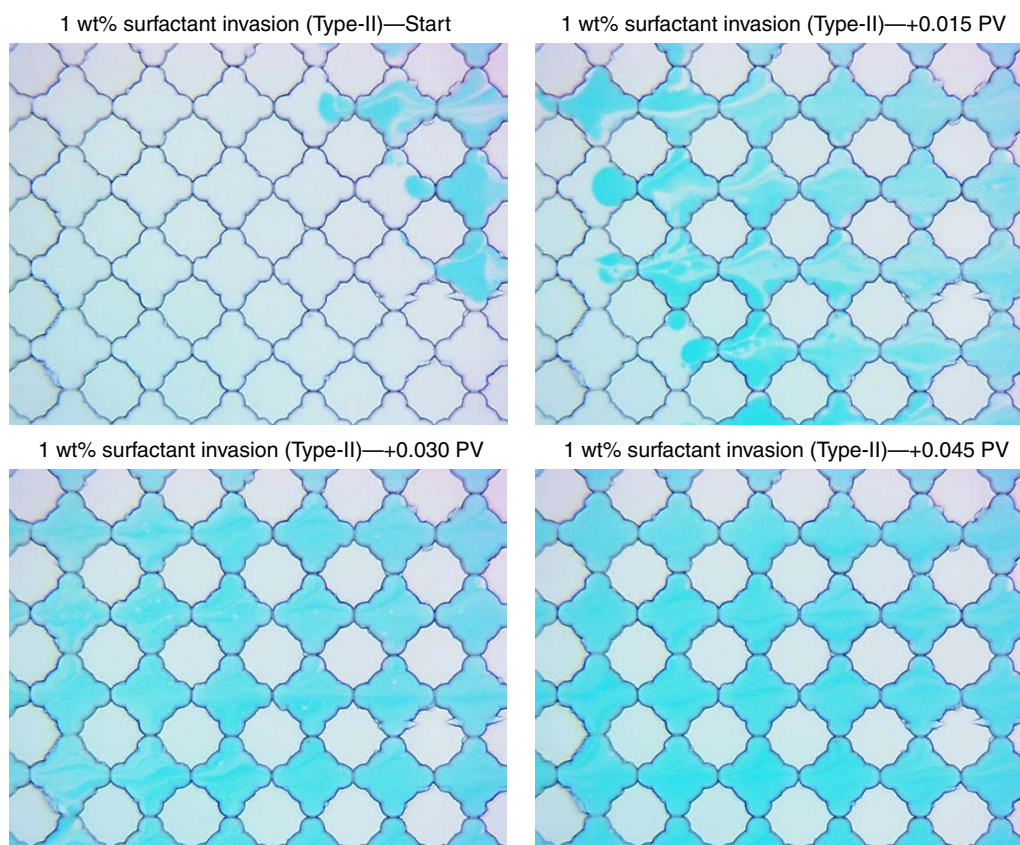


Fig. 10—Close-up view of the formation of Winsor-II microemulsions during the initial mixing at the invasion front. Dyed DI water is blue, while *n*-pentane is white; the light-blue fluid formed during mixing is very likely a Winsor-II microemulsion.

Flowback with Surfactant under Winsor-I Conditions. Fig. 13 shows the change in phase saturations within 360 seconds (i.e., 0.10 PV) when *n*-hexane flowed through the micromodel that was invaded by 1 wt% surfactant under Winsor-I conditions. Unlike the baseline case (Fig. 11), the majority of the trapped water was mobilized by the hydrocarbon. With the decrease of water saturation in the invaded region, the form of hydrocarbon changed from small spherical droplets scattered in water to bigger droplets wrapped in water. These droplets kept growing with time when less water was left behind in the form of droplet films. In this case, viscous fingering was not observed, and most droplets were mobile before the breakthrough of hydrocarbon.

Fig. 14 shows the change in phase saturations across the lateral space of the entire invaded region after 0.5 PV of *n*-hexane was flooded through the model: from the original invasion front (top figure) to the middle range of the invasion body (center figure), and then to the invasion tail that was now at the outlet of the micromodel matrix (bottom figure). As shown in the top figure, residual water in the region near the original invasion front was in a form similar to that observed in the baseline case (Figs. 11 and 12). This was likely from adsorption of surfactants during the invasion process, resulting in a trace of surfactant at the invasion front; the same phenomenon has been reported from CT measurements (Liang et al. 2017a). Toward the matrix outlet, oil droplets were observed, and the form of residual water changed to thin films between these oil droplets. Although the sizes of oil droplets might be bigger than the pore throats, they could easily squeeze through the matrix without phase separation (i.e., droplet bursting) (bottom figure). Water trapped because of the matrix–fracture interaction was not observed near the matrix outlet. This is expected for a reduction in IFT that eliminates the capillary discontinuity from the matrix outlet to the fracture.

During the flowback, several oil droplets were bypassed and trapped within the matrix; meanwhile, droplet bursting was also observed from the near-invasion-front region toward the matrix outlet. Fig. 15 shows the changes in trapped droplets within 108 seconds (i.e., 0.03 PV). It can be observed that the bypassed/trapped droplets started to burst from the contact where a lump of droplets encountered the flowing hydrocarbon (pointed out by red arrows in figures). These droplets could block the flow of hydrocarbon; meanwhile, they were likely to experience the force from hydrocarbon that could cause them to burst. This bursting was unlikely caused by the solubilization/loss of surfactants into the hydrocarbon; this can be found out compared to the case under Winsor-III conditions (Fig. 16), as detailed in the following subsection. Moreover, along with the bursting of oil droplets, the aqueous phase and the oleic phase separated and generated small water droplets. The majority of these water droplets could be mobilized by hydrocarbon and were eventually recovered from the micromodel matrix (see Fig. 15).

In summary, using a surfactant additive under Winsor-I conditions can effectively remove the water blockage caused by the invasion of the fracturing fluid.

Flowback with Surfactant under Winsor-III Conditions. Fig. 17 shows the change in phase saturations within 360 seconds (i.e., 0.10 PV) when *n*-hexane flowed through the micromodel that was invaded by 1 wt% surfactant under Winsor-III conditions. Similar to Winsor-I conditions (Fig. 13), the majority of the trapped water was mobilized by hydrocarbon and the form of hydrocarbon changed from small spherical droplets scattered in water to bigger droplets wrapped in water with the decrease of water saturation (i.e., from the original invasion front to the matrix outlet). However, unlike Winsor-I conditions, oil droplets were less homogeneous under this condition, where big lumps of oil were observed. Moreover, water films between these oil droplets were also thicker than those under Winsor-I conditions.

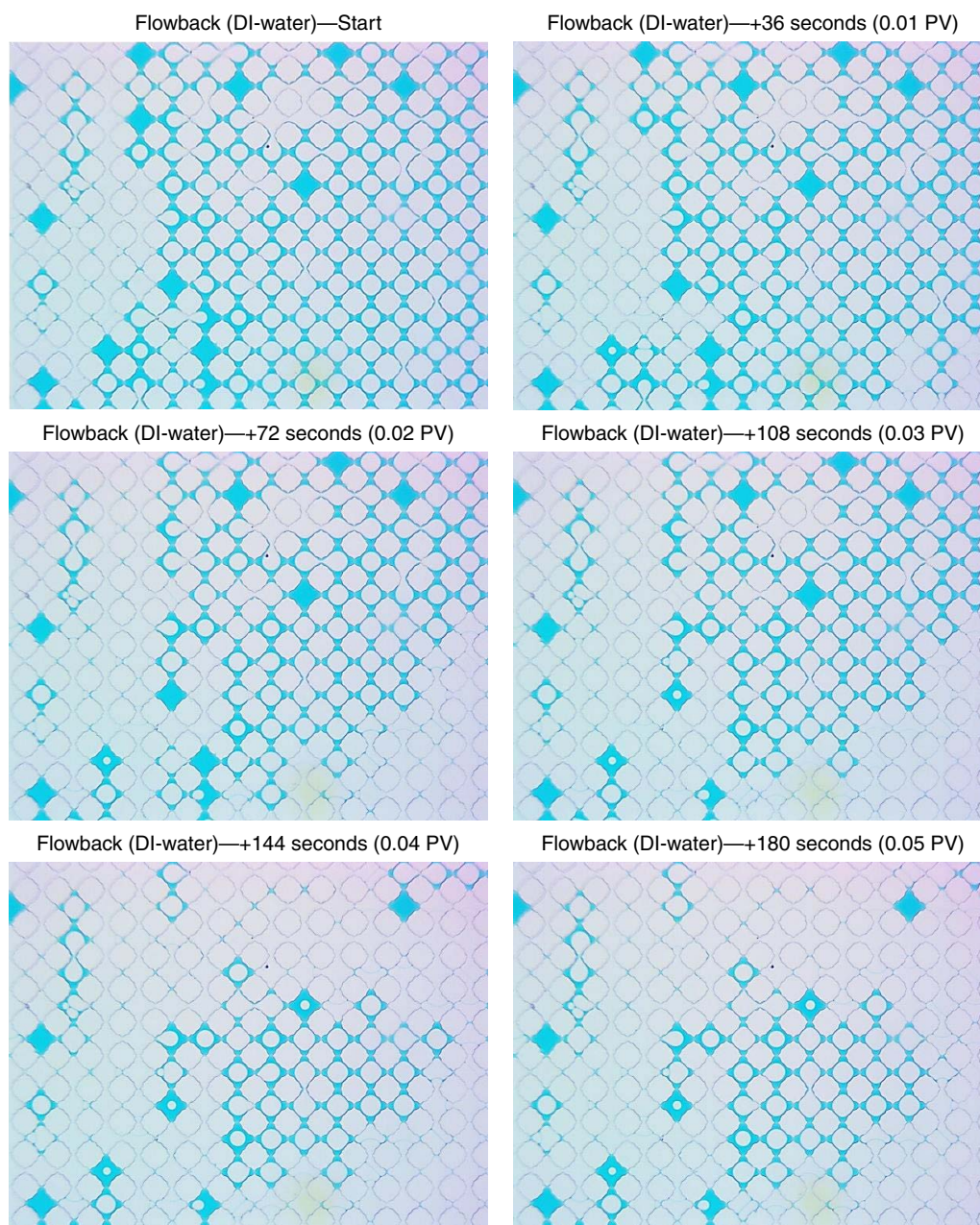


Fig. 11—Change in phase saturations during flowback and hydrocarbon production from the DI-water-invaded micromodel for 180 seconds (0.05 PV). Closed transparent circles are solid grains of the micromodel, and the remainder of the space is the porous medium; dyed DI water is blue, while *n*-hexane is white.

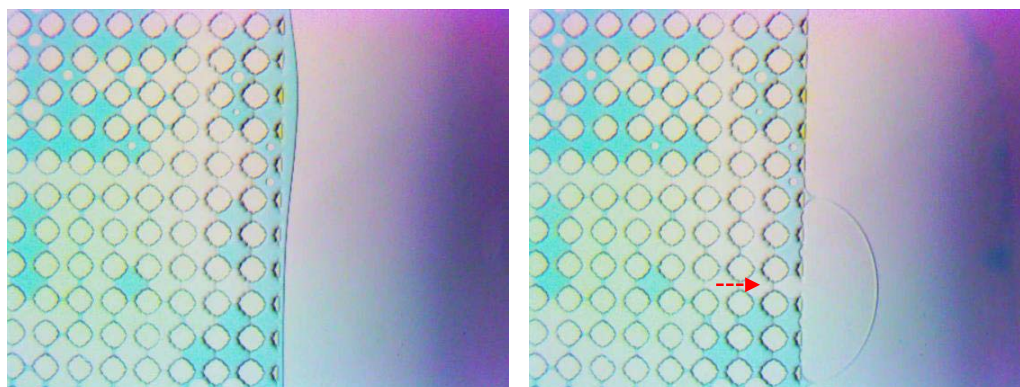


Fig. 12—Water trapping near the outlet of the micromodel matrix (*n*-hexane flowed from left to right) due to the matrix—fracture interaction (left figure). Snap-off of *n*-hexane through the layer of trapped water into the mimicked fracture (right figure); the red arrow points a temporary flow path opened for *n*-hexane.

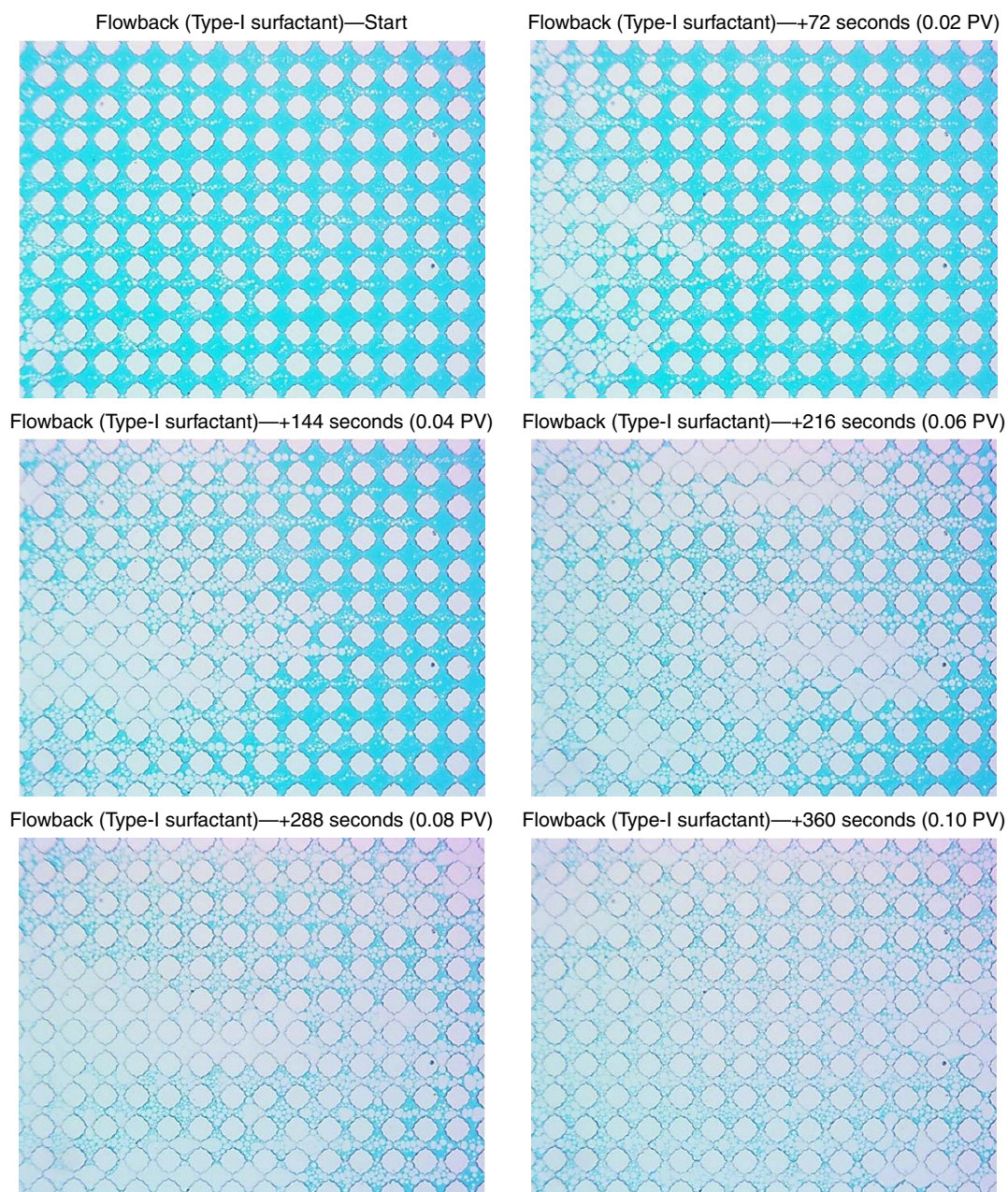


Fig. 13—Change in phase saturations during flowback and hydrocarbon production from the micromodel invaded by 1 wt% surfactant under Winsor-I conditions for 360 seconds (0.10 PV). Closed transparent circles are solid grains of the micromodel, and the remainder of the space is the porous medium; dyed DI water is blue, while *n*-hexane is white.

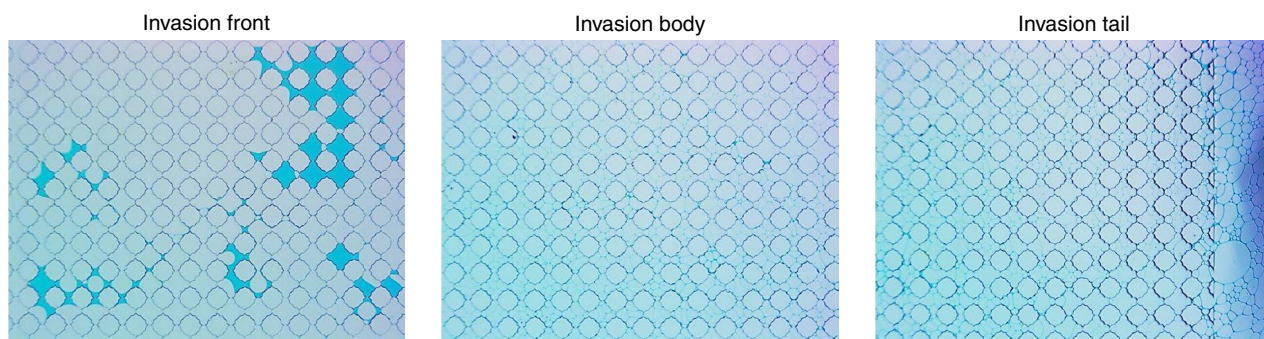


Fig. 14—Change in phase saturations at the invasion front (top), within the middle range of the invasion body (center), or at the invasion tail (bottom) after 0.5 PV of *n*-hexane flooded through the micromodel invaded by 1 wt% surfactant under Winsor-I conditions.

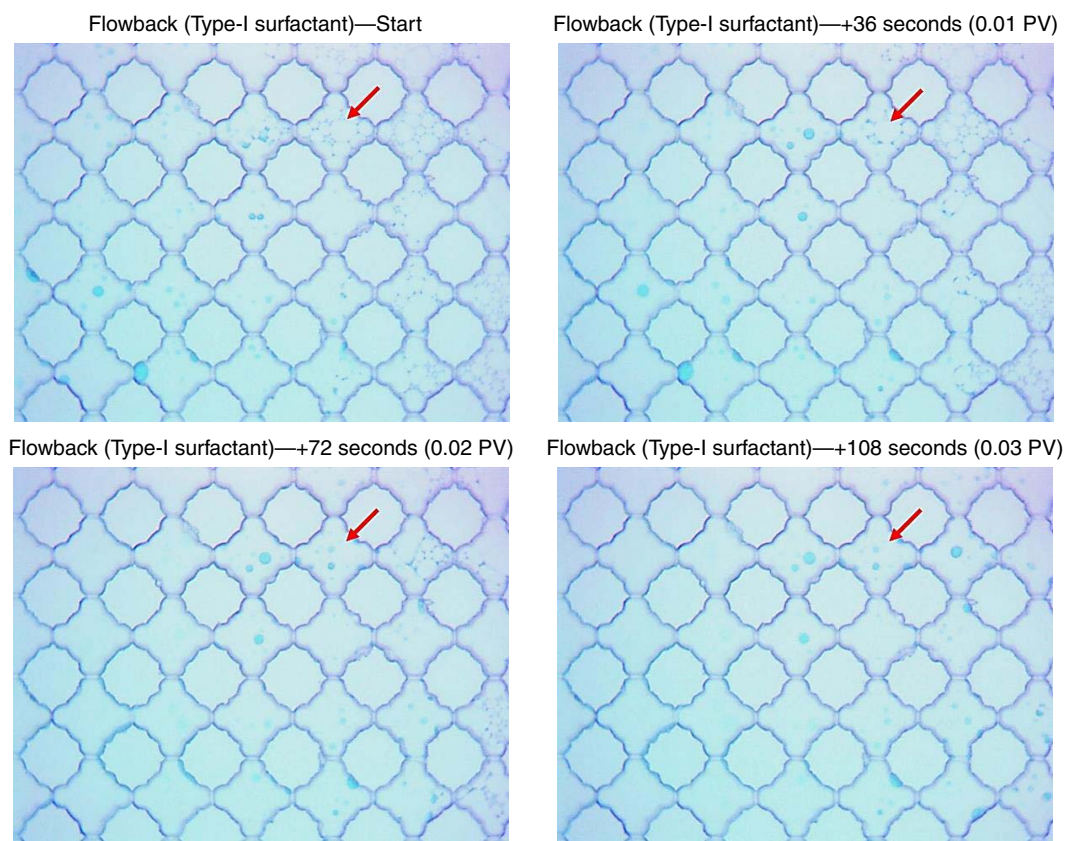


Fig. 15—Changes in bypassed/trapped droplets in 108 seconds (0.03 PV) after 0.5 PV of *n*-hexane flooded through the micromodel invaded by 1 wt% surfactant under Winsor-I conditions. Red arrows point to the change within the same pore at different times.

Fig. 18 shows the change in phase saturations across the lateral space of the entire invaded region after 0.5 PV of *n*-hexane flooded through the model: from the original invasion front (top figure) to the middle range of the invasion body (center figure), and then to the invasion tail which was now at the outlet of the porous medium (bottom figure). Like Winsor-I conditions, residual water attached to the walls/edges of pores in the region near the original invasion front by means of the adsorption loss of surfactant (top figure). Toward the matrix outlet, water was in the form of films between oil droplets; however, in this case, more water was trapped between the oil droplets, resulting in a slower migration of oil droplets toward the matrix outlet. Along with the migration, bursting of oil droplets was also observed; however, the speed of bursting was much slower than that under Winsor-I conditions. Fig. 16 shows the changes of trapped oil droplets within 432 seconds (i.e., 0.12 PV). Unlike Winsor-I conditions, some droplets became smaller with higher water/oil ratios, and the phase separation (i.e., droplet bursting) was not ubiquitous. This was likely caused by the ultralow oil–water IFT under Winsor-III conditions where the mixed phase was stable regardless of hydrocarbon flow. Therefore, the relatively fast bursting speed of droplets observed under Winsor-I conditions (Fig. 15) can be attributed to the weakly stabilized oil–water interface, rather than to the loss of surfactants in hydrocarbon.

Fig. 19 shows the change in phase saturations across the lateral space of the invaded region after 2.5 PV of *n*-hexane flooded through the micromodel. In Region (a), where the surfactant concentration was low because of the adsorption loss of surfactant, residual water remained attached to the walls/edges of pores (Fig. 19a). Without changing the viscous force or the pressure drawdown, this trapped water is unlikely to move with hydrocarbon. From the rear section of Region (a) to the front section of Region (c), tiny water droplets from the burst oil droplets and small unbroken oil droplets without phase separation were both observed (Figs. 19a through 19c); the phase separation was likely to continue, although it happened slowly because of the ultralow oil–water IFT that stabilized the mixed phase. In the following regions [i.e., the rear section of Region(c) to Region(f)], unbroken droplets were observed (Figs. 19c through 19f). This poor efficiency was likely caused by the high water/oil ratios of the droplets that increased their viscosity, and thus slowed down their migration velocity; this agrees with the measurement result that Winsor-III microemulsion has a higher viscosity than Winsor-I microemulsion (Lu et al. 2013; Tagavifar et al. 2018). Furthermore, the observation also agrees with the CT results of Liang et al. (2017a): water saturation decreased almost uniformly with time from the original invasion front to the rock outlet under type-III conditions. In the region near the matrix outlet, like Winsor-I conditions, water trapping resulting from the matrix–fracture interaction was not observed because of the IFT reduction (Fig. 19f).

In summary, a further reduction in oil–water IFT (Winsor-III conditions) does not improve the efficiency of mitigating water blockage. In fact, it creates a stable/long-lasting mixed phase that impedes the flow of hydrocarbon.

Flowback with Surfactant under Winsor-II Conditions. **Fig. 20** shows the change in phase saturations within 360 seconds (i.e., 0.10 PV) when *n*-pentane flowed through the micromodel that was invaded by 1 wt% surfactant under Winsor-II conditions. Like Winsor-I and -III conditions, the invading water (blue fluid) and the previously formed microemulsion (light-blue fluid) could both be mobilized toward the matrix outlet where water saturation decreased, and the form of residual water changed into thin films between oil droplets. However, unlike the other two conditions, preferred flow paths were observed where the oil droplets burst relatively rapidly when they came into contact with the flow of hydrocarbon. When the hydrocarbon broke through the micromodel matrix through these preferred flow paths, migration of oil droplets and the flowback of water both stopped; the invading water remained in the matrix in the form of droplet films. **Fig. 21** provides a close-up view of the formation of the preferred flow paths within 108 seconds (0.03 PV) after flooding 0.5 PV of *n*-pentane. As pointed out by two red arrows, preferred flow paths were gradually formed after the bursting of oil

droplets (i.e., phase separation), leaving a lump of oil droplets remaining unchanged between them. The speed of droplet bursting under Winsor-II conditions was faster than under the other two Winsor conditions, and this was expected for such a high-salinity environment where surfactant preferred to stay in oil instead of in water films that stabilized the oil droplets.

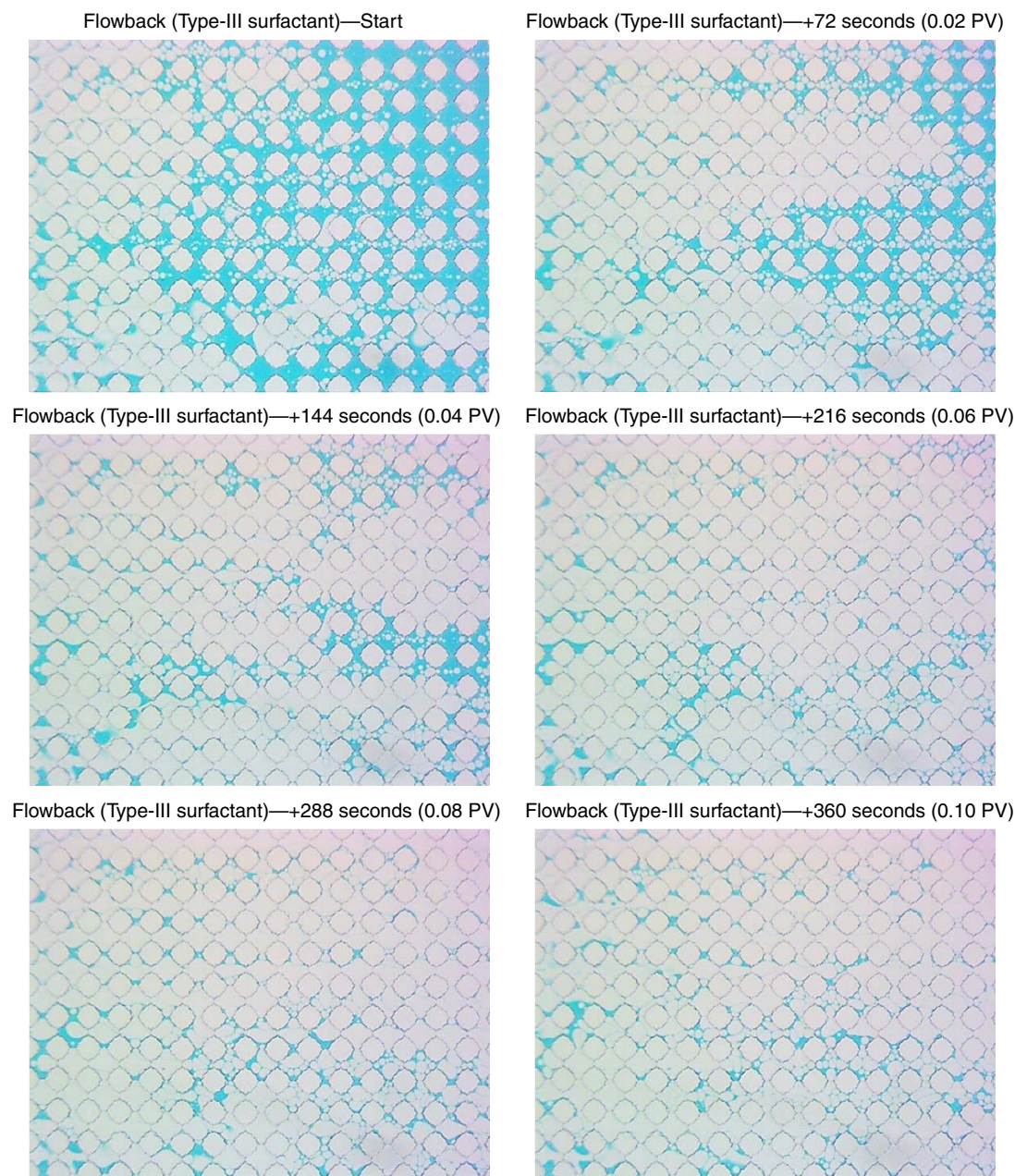


Fig. 16—Changes in bypassed/trapped droplets in 432 seconds (0.12 PV) after 0.5 PV of *n*-hexane flooded through the micromodel invaded by 1 wt% surfactant under Winsor-III conditions. Red arrows point to the change within the same pore at different times.

Fig. 22 shows the change in phase saturations across the lateral space of the entire invaded region after 0.5 PV of *n*-pentane flooded through the model: from the original invasion front (top figure) to the middle range of the invasion body (center figure), and then to the invasion tail, which is now at the outlet of the porous medium (bottom figure). Like Winsor-I and -III conditions, residual water attached to the walls/edges of pores was observed in the region near the original invasion front where the surfactant concentration was low because of adsorption loss (top figure). Toward the matrix outlet, the preferred flow paths and oil droplets were both observed (center figure). It can be noticed that oil droplets under this condition had thin water films like those under Winsor-I conditions (Fig. 14), which were distinct from those under Winsor-III conditions (Fig. 18); this might be because the ultralow oil–water IFT under Winsor-III conditions allows water to coexist with oil, forming a relatively stable mixed phase. In the region near the matrix outlet, like the other two conditions, water trapping because of the matrix–fracture interaction was not observed because of the IFT reduction.

Fig. 23 shows water saturations in six different locations near the matrix outlet after flooding 2.5 PV of pentane. As pointed out by red arrows in the figure, two major preferred flow paths could be observed, which were accompanied by water droplets from the burst oil droplets. For other locations, water remained trapped in the matrix as water films between the oil droplets. Under Winsor-II conditions where salinity was high, surfactants tended to migrate from water into the oil phase, which results in phase separation (i.e., droplet bursting) that creates the preferred flow paths that bypass a significant amount of oil droplets. Therefore, using surfactant additives under Winsor-II conditions resulted in poor efficiency of removing the invading water, possibly even worse than under Winsor-III conditions. This also agrees with the results of the CT-monitored corefloods reported by Liang et al. (2017a, 2017b).

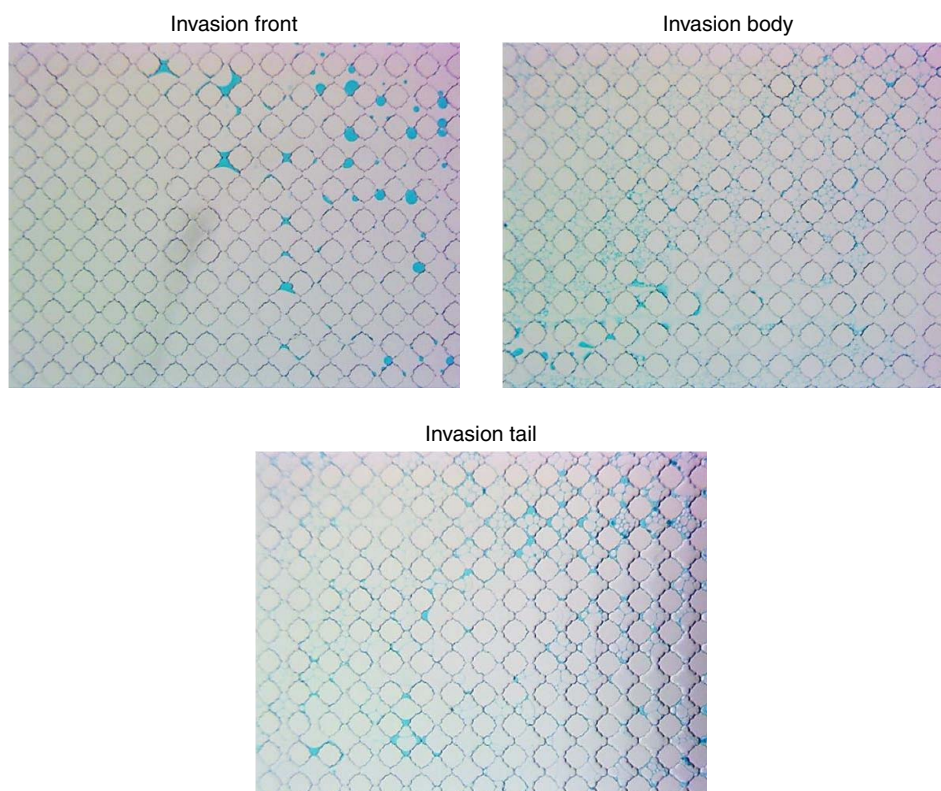


Fig. 17—Change in phase saturations during the flowback and hydrocarbon production from the micromodel invaded by 1 wt% surfactant under Winsor-III conditions for 360 seconds (0.10 PV). Closed transparent circles are solid grains of the micromodel and the remainder of the space is the porous medium; dyed DI water is blue, while *n*-hexane is white.

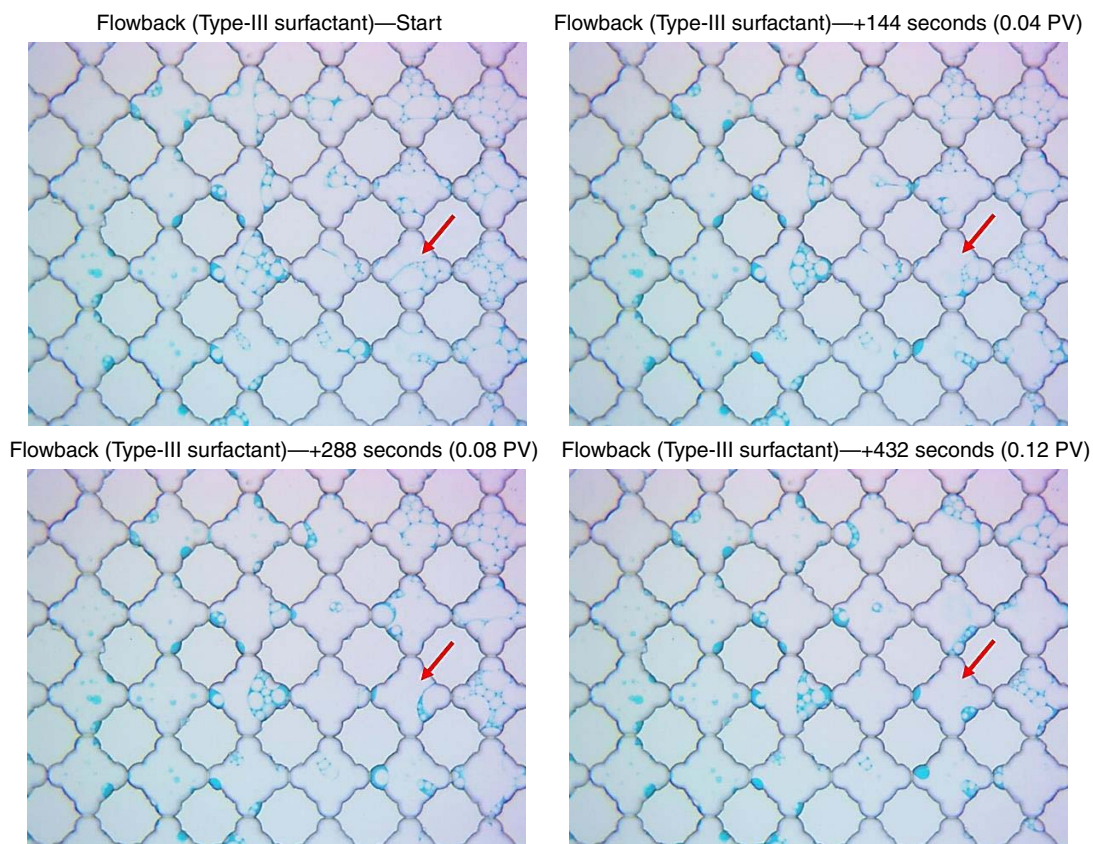


Fig. 18—Change in phase saturations at the invasion front (top), within the middle range of the invasion body (center), or at the invasion tail (bottom) after 0.5 PV of *n*-hexane flooded through the micromodel invaded by 1 wt% surfactant under Winsor-III conditions.

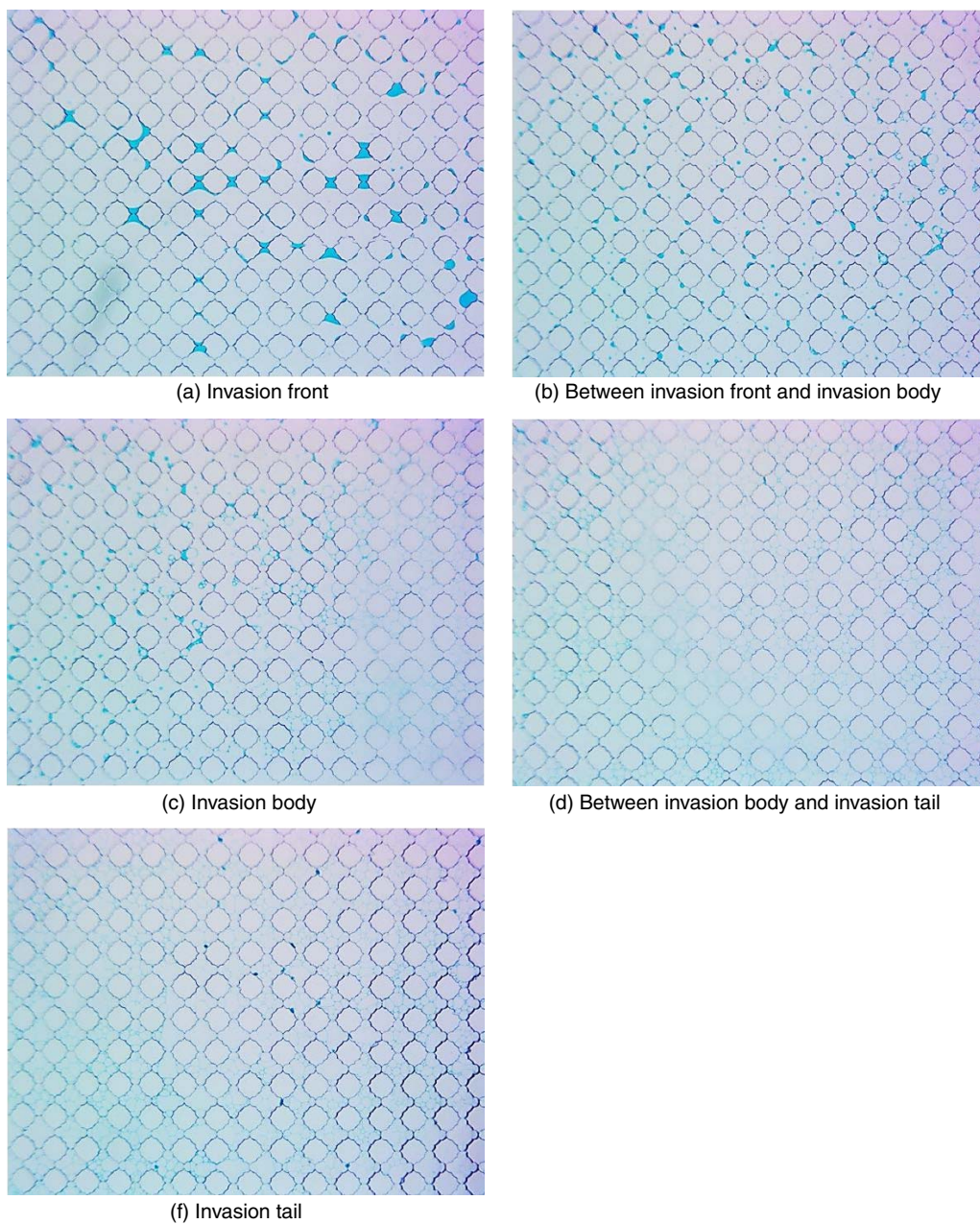


Fig. 19—Change in phase saturations across the invaded region (from the original invasion front to the invasion tail) after 2.5 PVs of *n*-hexane flooded through the micromodel invaded by 1 wt% surfactant under Winsor-III conditions.

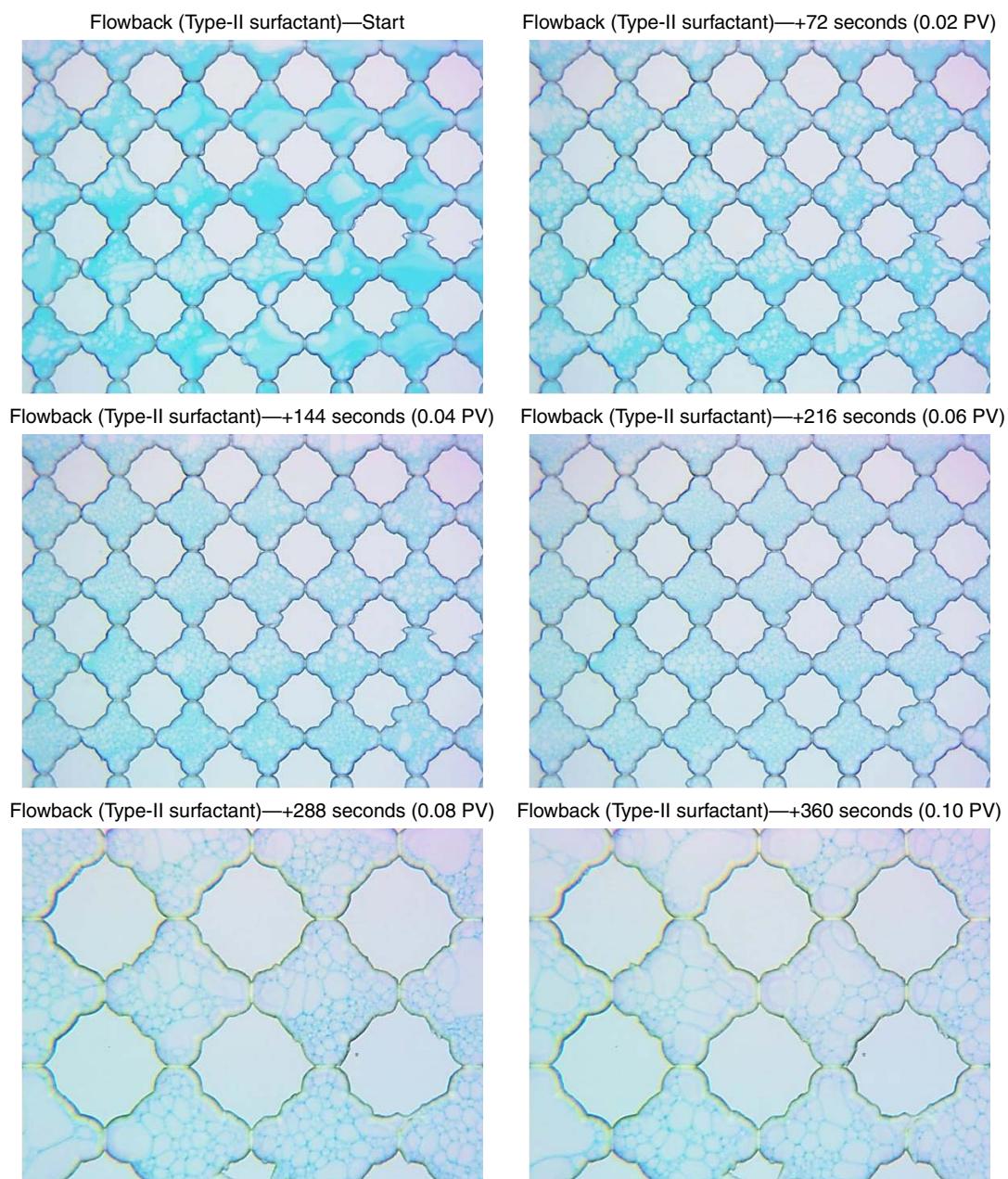


Fig. 20—Change in phase saturations during flowback and hydrocarbon production from the micromodel invaded by 1 wt% surfactant under Winsor-II conditions for 360 seconds (0.10 PV). Closed transparent circles are solid grains of the micromodel, and the remainder of the space is the porous medium; dyed DI water is blue, while *n*-pentane is white.

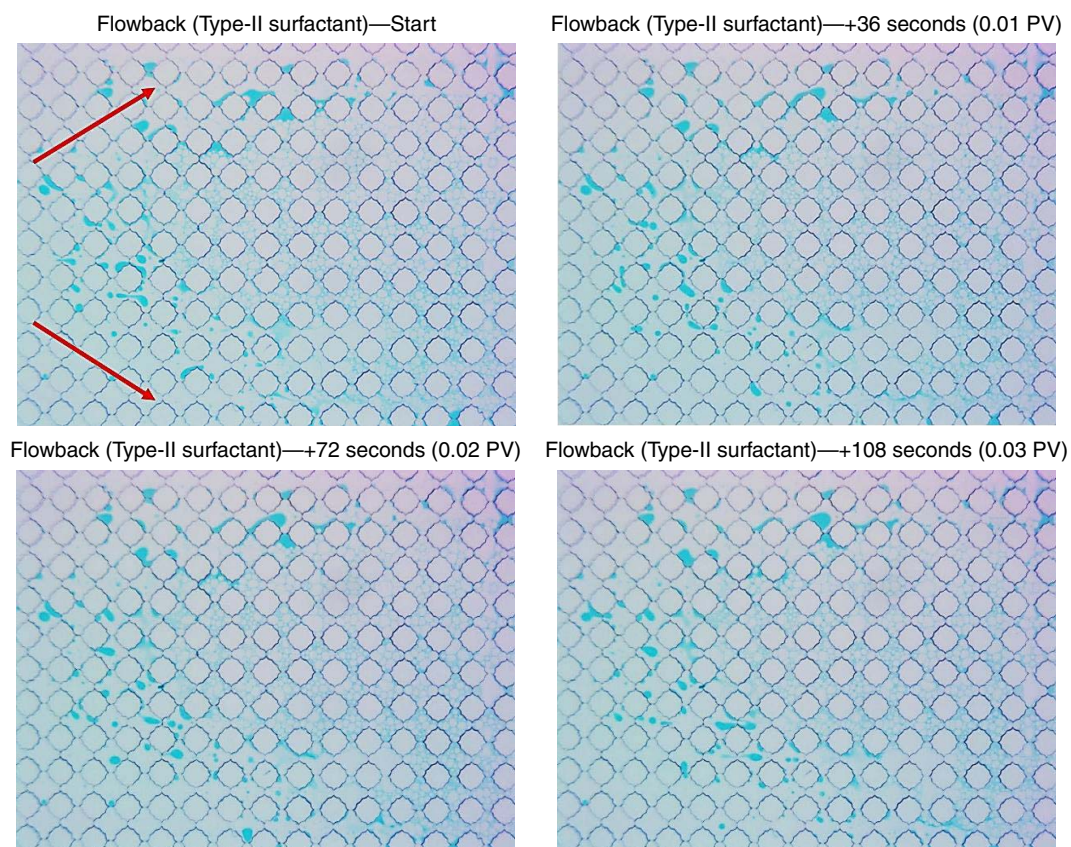


Fig. 21—Changes in the trapped oil droplets for 108 seconds (0.03 PV) after 0.5 PV of *n*-pentane flooded through the micromodel invaded by 1 wt% surfactant under Winsor-II conditions. Red arrows point to the formation of two preferred flow paths with time.

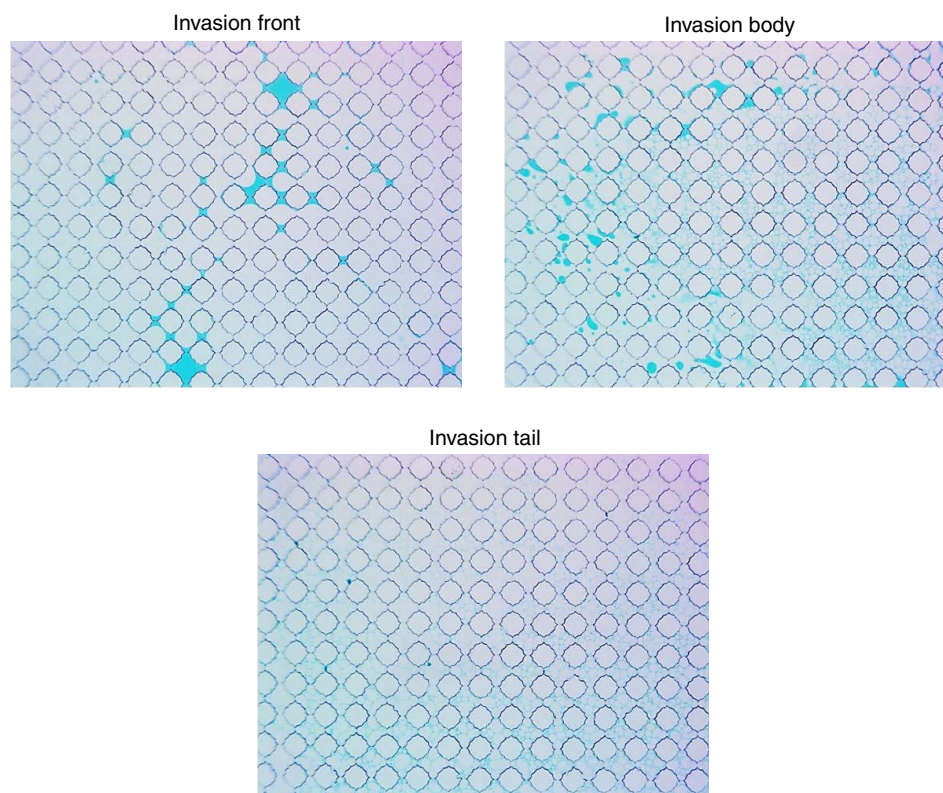


Fig. 22—Change in phase saturations at the invasion front (top), within the middle range of the invasion body (center), or at the invasion tail (bottom) after 0.5 PV of *n*-pentane flooded through the micromodel invaded by 1 wt% surfactant under Winsor-II conditions.

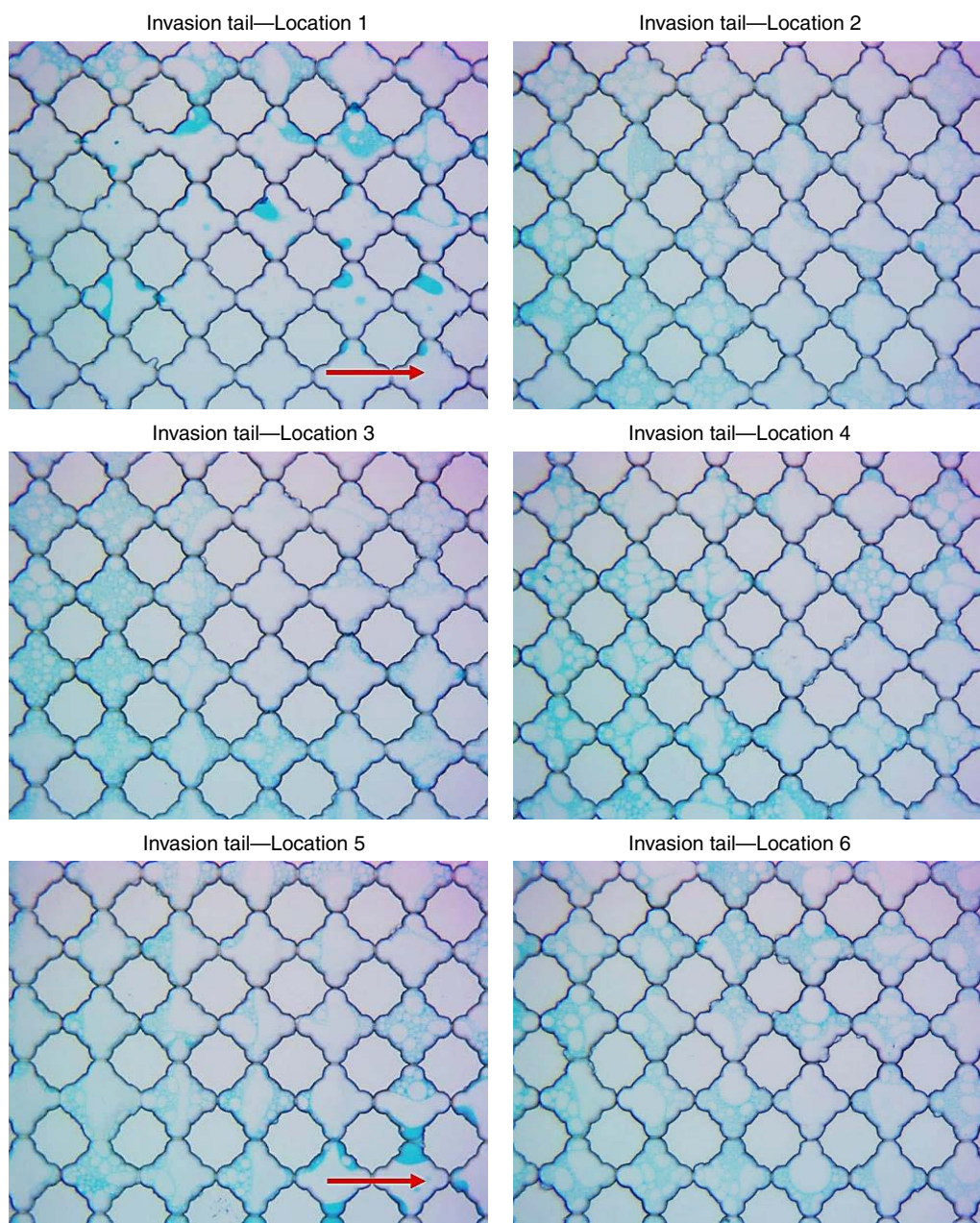


Fig. 23—Water saturations in six different locations near the outlet of the micromodel matrix after flooding 2.5 PV of *n*-pentane. Red arrows point the preferred flow paths.

Conclusions

In this study, a three-step chipflood sequence was conducted on a 2.5D glass micromodel to visualize the form of water blockage caused by the invading fracturing fluid, as well as the effect of different microemulsion-forming surfactants in mitigating such blockage. This glass micromodel can provide a good approximation of real rock and enable the direct observation of in-situ phase changes when oil is mixed with water or a surfactant solution at different salinities within the porous medium.

The major findings of this study are the following:

1. Besides water trapping on the walls/edges of pores, water trapping at the outlet of the micromodel matrix caused by the matrix–fracture interaction was directly observed in the case when no surfactant was used. A thin layer of water was retained at the matrix outlet that the hydrocarbon needed to snap off before reaching the fracture.
2. During the hydraulic-fracturing process, the surfactant-aided fracturing fluid invades the formation and mixes with the reservoir oil to form microemulsions. For the surfactant in the Winsor-I and -II conditions, it was observed that microemulsions mainly formed near the invasion front where the hydrocarbon and the surfactant solution initially mixed. However, for the surfactant under Winsor-III conditions, it was observed that microemulsions formed far behind the invasion front where sufficient mixing was required. This is different from the previous findings from the sandpack observations (Lu et al. 2014; Lu and Pope 2017); thus, a more in-depth study is needed.
3. During flowback and production processes, flowback surfactants under different Winsor conditions performed differently. Under Winsor-I conditions, the mixed phase between hydrocarbon and water (i.e., oil droplets) was mobile and the bypassed/trapped oil droplets could break with time at a relatively fast rate; these gave rise to the best efficiency in removing the invading fluid. Under Winsor-III conditions, thicker water films were observed in the mixed phase, and the bypassed/trapped droplets were relatively

stable because of the ultralow oil–water IFT; both decreased the mobility of the mixed phase and resulted in poor efficiency in removing the invading fluid. However, under Winsor-II conditions, distinct preferred flow paths were observed, and a significant amount of water was left behind in the form of water films of the bypassed droplets among these preferred flow paths; this resulted in the worst efficiency in removing the invading fluid.

4. Unlike CEOR surfactants, flowback surfactants do not require achieving ultralow oil–water IFT that generates the Winsor-III microemulsion. On the one hand, ultralow IFT stabilizes the microemulsion phase, which hinders hydrocarbon flow. On the other hand, the capillary number can be as low as 10^{-9} during flowback in tight oil reservoirs, where even ultralow IFT cannot trigger capillary desaturation of the invading water. Observation suggested that an oil–water IFT of 10^{-2} to 10^{-3} mN/m would be enough to eliminate the matrix–fracture interaction and enhance the oil production.
5. Observation also suggested that besides the adsorption loss of the surfactant onto the rock surface, partitioning of the surfactant in the reservoir oil also needs to be minimized; this allows the surfactant to stay longer with the invading fracturing fluid, thus better mitigating the water blockage.

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