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Spontaneous Imbibition and Core Flooding Experiments of Enhanced Oil Recovery in Tight Reservoirs with Surfactants

Shaojie Zhang [1,2,3,4,*], Feng Zhu [1,2], Jin Xu [1,2], Peng Liu [1,2], Shangbin Chen [3,*] and Yang Wang [3]

1 State Key Laboratory of Shale Oil and Gas Enrichment Mechanisms and Effective Development, Beijing 102206, China
2 SINOPEC Key Laboratory of Petroleum Accumulation Mechanisms, Wuxi 214126, China
3 School of Resources and Geoscience, China University of Mining and Technology, Xuzhou 221116, China
4 Department of Petroleum Engineering, University of North Dakota, Grand Forks, ND 58202, USA
* Correspondence: shaojiezhang@cumt.edu.cn (S.Z.); shangbinchen@163.com (S.C.); Tel.: +86-16633988309 (S.Z.); +86-13585390796 (S.C.)

Abstract: Despite the implementation of hydraulic fracturing technologies, the oil recovery in tight oil reservoirs is still poor. In this study, cationic, anionic, and nonionic surfactants of various sorts were investigated to improve oil recovery in tight carbonate cores from the Middle Bakken Formation in the Williston Basin. Petrophysical investigations were performed on the samples prior to the imbibition and core-flooding experiments. The composition of the minerals was examined using the XRD technique. To investigate the pore-size distribution and microstructures, nitrogen adsorption and SEM techniques were applied. The next step involved brine and surfactant imbibition for six Bakken cores and two Berea sandstone cores. The core samples were completely saturated with Bakken crude oil prior to the experiments. The core plugs were then submerged into the brine and surfactant solutions. The volume of recovered oil was measured using imbibition cells as part of experiments involving brine and surfactant ingestion into oil-filled cores. According to the findings, oil recovery from brine imbibition ranges from 4.3% to 15%, whereas oil recovery from surfactant imbibition can range from 9% to 28%. According to the findings, core samples with more clay and larger pore diameters produce higher levels of oil recovery. Additionally, two tight Bakken core samples were used in core-flooding tests. Brine and a separate surfactant solution were the injected fluids. The primary oil recovery from brine flooding on core samples is between 23% and 25%, according to the results. The maximum oil recovery by second-stage surfactant flooding is approximately 33% and 35%. The anionic surfactants appear to yield a better oil recovery in tight Bakken rocks, possibly due to their higher carbonate mineral concentrations, especially clays, according to both the core-scale imbibition and flooding experiments. For studied samples with larger pore sizes, the oil recovery is higher. The knowledge of the impacts of mineral composition, pore size, and surfactant types on oil recovery in tight carbonate rocks is improved by this study.

Keywords: tight oil reservoirs; enhanced oil recovery; surfactant; spontaneous imbibition; core flooding

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1. Introduction

Tight oil and gas are playing a bigger role in the energy mix in the United States and China [1,2]. The Bakken deposit is one of the biggest unconventional reservoirs in terms of its oil and gas reserves. The Middle Bakken Formation is primarily composed of tight carbonate rocks. Tight shale deposits in the Upper and Lower Bakken members are the Middle Bakken member's hydrocarbon sources. The Middle Bakken formation's productivity is increasing owing to horizontal drilling and multistage hydraulic-fracturing technologies. The permeability and porosity of the reservoirs are so low that they still have low recovery coefficients. According to a variety of recent studies, the Middle Bakken component is a typical tight layer with extremely low porosity [3,4]. The observed diameters of the microstructures range between nanometers and micrometers, and they frequently have some natural fractures [5]. In addition, Cho et al. (2016) examined the Bakken rocks' pore size distributions [6]. They discovered that unimodal and bimodal pore-size distribution curves can be seen in the Middle Bakken samples. Additionally, they used X-ray diffraction (XRD) technology to investigate the composition of minerals. The findings showed that quartz, dolomite, and calcite were the three most common minerals.

Enhanced oil recovery methods are of great interest to both the petroleum industry and academia because of the extremely low oil recovery factors in tight oil reservoirs. One of the most frequently utilized methods is surfactant-enhanced oil recovery. The surfactant can attach to the rock's surface and convert the rock surface from oil-wet to water-wet. Surfactants, which are amphiphilic chemicals with both hydrophobic and hydrophilic groups, can change the wettability of rock surfaces and decrease the oil–water IFT [7–9]. In fractured reservoirs, imbibition is an important mechanism to recover oil from the tight matrix [10–12]. At the presence of capillary pressure, the wetting phase can displace the non-wetting phase, and this process is the imbibition. The counter-current spontaneous imbibition is the most important imbibition type in fracture reservoirs [13–15]. Therefore, it is of great importance to understand the spontaneous-imbibition mechanisms in tight reservoirs to improve the oil recovery [16]. A large number of studies recently have shown that surfactant imbibition might considerably improve oil recovery in unconventional reservoirs. Wang et al. (2014) conducted spontaneous-imbibition studies using various anionic and nonionic surfactants on a large number of Bakken rock samples [17]. The results indicated that the nonionic surfactant (SNA) can greatly increase the final oil recovery. Additionally, surfactant-imbibition studies were conducted on Middle Bakken rocks by Olatunji et al. (2018) [18]. According to their findings, during brine imbibition, there was very little oil recovery, whereas approximately 30–45% of the oil was recovered through surfactant imbibition.

The effectiveness of surfactant-enhanced oil recovery in tight oil reservoirs is frequently investigated using water and surfactant core flooding [19,20]. Through micelle formation and adsorption at the oil–water interface, the surfactant flooding can reduce the interfacial tension and mobilize the oil flow toward the continuous water phase. The lower IFT expands the capillarity number and enhances the region of sweep for the water floods from a microscopic pore-scale perspective [21]. Alkali-surfactant-polymer (ASP) flooding has been a hot topic for enhanced oil recovery in various oil reservoirs around the world [22,23]. However, ASP flooding still has some challenges, such as limited pore size and high salinity in tight carbonate reservoirs [24]. Core flooding and imbibition on carbonate core samples were carried out by Zallaghi and Khazali (2021) utilizing cationic and nonionic surfactants, along with diluted seawater [25]. As a result of IFT decrease and wettability alternation, their findings demonstrate that the surfactant and diluted saltwater exhibit considerable synergy for improving oil recovery in carbonate reservoirs. Through surfactant flooding, the microemulsion is also a crucial component of increased oil recovery. According to laboratory experiments, the oil recovery of the synthesized emulsion system is 5% higher than that of no emulsions [26]. Numerous numerical simulations of surfactant-enhanced oil recovery have been conducted [27,28]. Surfactant-enhanced oil recovery simulation is complex because models must account for a variety of factors, such as rock mechanics, surfactant adsorption, relative permeability, diffusion, viscosity, interfacial tension, phase behaviors, and so on [29–32].

Numerous studies have been done on the use of various surfactants to enhance oil recovery in tight rocks. However, there is very little research that investigates how pore structure and mineral composition affect surfactant EOR. On samples of rock from the Bakken and Berea in this study, studies on spontaneous imbibition and core flooding with various surfactant solutions and brine were conducted. Bakken samples' microstructures and pore-size distributions were discovered using nitrogen adsorption and SEM techniques. Additionally, the XRD method was used to determine the samples' mineralogical compositions. On oil recovery, the effects of pore-size distribution, mineralogy, and surfactant types were examined.

2. Materials and Methods

2.1. Core Samples and Surfactants

To conduct research on core flooding and spontaneous imbibition, ten core plugs were used. The Williston Basin's Middle Member of the Bakken Formation yielded eight Bakken cores. Samples B5 and B6 are obtained from well #1 at the depth of 11,096 ft. Samples B1–B4 are obtained from well #2, with a depth of approximately 10,791–10,801 ft. Samples B7 and B8 are obtained from well #3, with a depth of 10,636 ft. Figure 1 displays images of three Bakken samples. The color of the Bakken samples ranges from grey to dark grey. The Middle Bakken member is the primary oil-producing member in the Bakken formation, and the lithology of Middle Bakken samples was described as siltstones. Two Berea cores, A1 and A2, are sandstone and were drilled from Berea outcrops in Cleveland Quarries, Ohio. The Bakken samples were drilled from the whole core and then cut into plugs, with lengths varying from 2.37 to 5.53 cm. The Berea samples were drilled from a rock cube cut from the outcrop. The Bakken samples were extracted with toluene and methanol in a Soxhlet extractor for 20 days to fully remove the residual oil. Then, those samples were dried in an oven at 100 °C for seven days to remove the residual water. Diameter length, porosity, and permeability tests were conducted on the core samples following cleaning and drying. Table 1 displays the petrophysical characteristics of the examined samples.

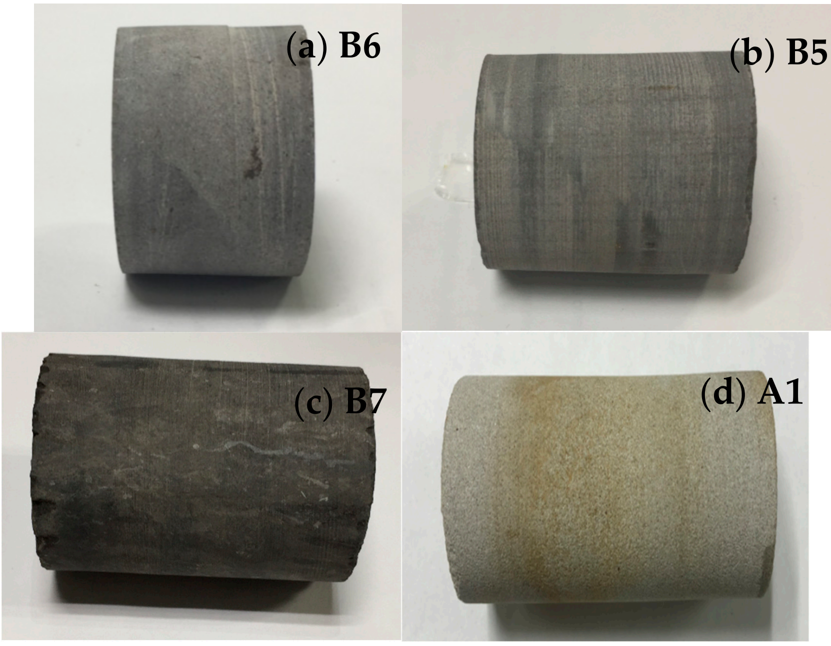

Figure 1. Pictures of studied Bakken and Berea core samples.

Table 1. Petrophysical properties of studied samples.

Samples	Length (cm)	Diameter (cm)	Permeability (mD)	Porosity (%)
B1	3.19	3.96	0.004	0.042
B2	3.17	3.95	0.002	0.038
B3	3.14	3.96	0.002	0.036
B4	3.32	3.95	0.005	0.068
B5	4.07	3.75	0.051	0.053
B6	2.37	3.78	0.064	0.057
B7	5.53	3.83	0.030	0.065
B8	5.26	3.84	0.030	0.065
A1	6.68	3.96	57.60	0.200
A2	6.94	3.95	64.10	0.190

The eight Middle Bakken samples are tight rocks with low porosity and permeability, as illustrated in Table 1. The Bakken samples have air permeabilities that range from 0.002 to 0.064 mD. The porosity of these samples was tested using nitrogen. The Bakken sample porosity ranges from 0.036 to 0.068. The permeability and porosity of the Berea samples are significantly higher than those of the Bakken samples.

2.2. SEM and XRD Analyses of Samples

Samples from the Bakken were examined for microstructures using the SEM technique. Figure 2 shows SEM pictures of the examined samples.

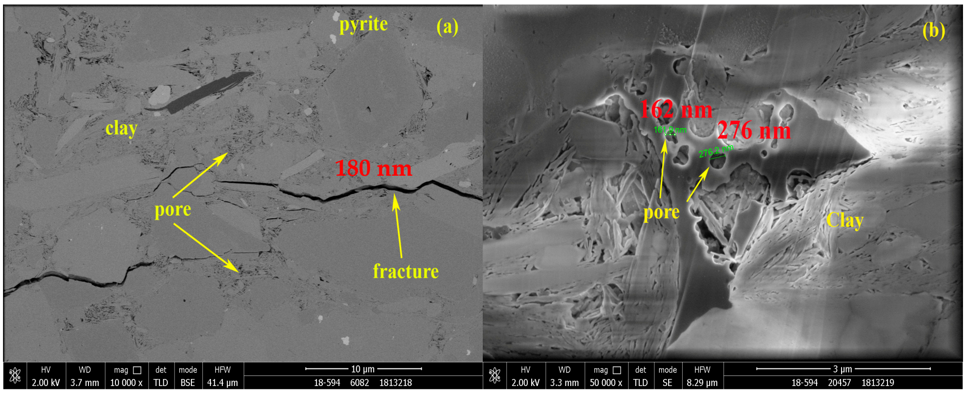

Figure 2. SEM images of the Middle Bakken samples. (**a**) B5 and B6. (**b**) B7 and B8.

According to the SEM pictures (Figure 2), B5 and B6 are characterized by fractures, clays, and numerous nanometer-sized pores. The pores' and fractures' widths vary from nanometers to micrometers, and 180 nm-wide natural fractures of considerable size were seen. Large numbers of micro- and macro-pores could be observed inside the clay minerals in samples B7 and B8. Samples B7 and B8 showed no apparent fractures.

2.3. Imbibition and Core-Flooding Experiments

Figure 3 shows a schematic of experimental setup. The samples were completely saturated in Bakken oil prior to the tests. The core samples were vacuumed using the ZYB-II Vacuum Saturation Device. The experimental steps for the core-saturation processes are as follows: (1) To remove the air from the core samples, they were vacuumed for 24 h. (2) The Bakken and Berea samples were saturated with crude oil for 20 days at a pressure of 20 MPa. Until the pressure does not change, the cores are fully saturated. (3) Prior to the imbibition and core-flooding tests, core samples were aged in Bakken crude oil in the oven at 60 °C for a month. (4) The cores were placed at the bottom of the imbibition cells with an accuracy of 0.05 mL. Then, the imbibition cells were filled with brine solution and different surfactant solutions. (5) The displaced oil volume was recorded with time until no oil was displaced out. All studies were carried out in an oven set at 60 °C, with all-face-open boundaries. The reservoir temperature of the Middle Bakken formation could range from 90 to 120 °C [33,34]. To avoid overheating and assure the stability of the surfactant solution at atmospheric pressure, the experimental temperature for spontaneous imbibition was set at 60 °C. The experimental temperature of the core flooding was also set at 60 °C.

The experiments' brine solution contains 2 wt% NaCl. Commercial surfactants were utilized in the core-flooding and imbibition studies. The nonionic surfactant MERPOL-HCS (HCS) was supplied by Stepan Company. The anionic surfactants include SOLOTERRA-964 (964) and sodium dodecyl sulfate (SDS). The cationic surfactants include cetyltrimethylammonium bromide (CTAB). The surfactant solutions are composed of 2 wt% NaCl and 0.1 wt% surfactants. The low-salinity brine and surfactant solutions were used in this research potentially because the low-salinity conditions could favor the recovery of oil over high-salinity conditions [35]. A surfactant concentration of 0.1 wt% was selected, considering the effectiveness of surfactant-assisted oil recovery. Research has shown that with a concentration of 0.05–0.2% surfactant, the final oil recovery in the Bakken samples was significantly improved [33].

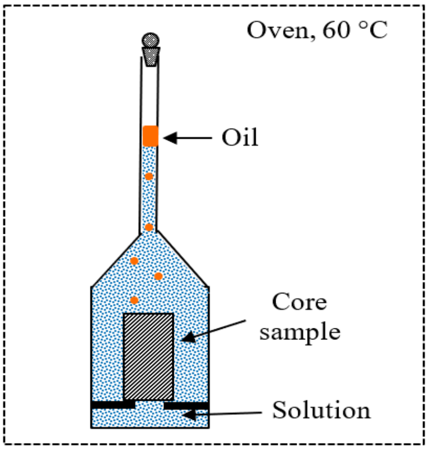

Figure 3. Image of spontaneous imbibition experiments setup. Experiments were conducted in an oven at 60 °C.

Figure 4 depicts the schematic of the core-flooding system used in this investigation. In this section, two different samples of fully saturated Bakken crude oil were filled into the core holder at a confining pressure of around 2000 psi at 60 °C. At a rate of 0.1~0.3 mL/h, the brine and surfactant solutions were injected. A measuring cylinder was used to collect the water and oil that were generated. The volumes of the injected solution and generated oil were measured.

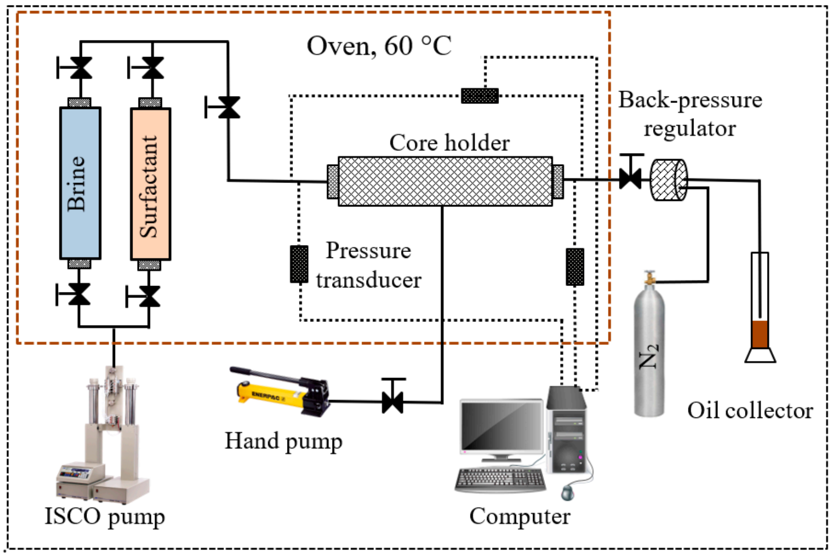

Figure 4. Schematic of core-flooding setup.

3. Results and Discussion

3.1. Rock Characterizations

The porous size distribution (PSD) could have great impacts on the oil recovery rate from tight reservoirs. Nitrogen adsorption was commonly used to obtain the PSD characteristics of tight rocks. Figure 5a displays the PSDs of the studied Bakken samples. However, the Berea samples are high-permeability and high-porosity rocks. The mercury-intrusion method is frequently used to measure the PSD of high-permeability samples. Figure 5b displays the PSD of the Berea samples by Cardoso and Balaban [36]. By analyzing the pore size distributions, the following characteristics were observed: (1) The Middle

Bakken samples have a high concentration of nanopores with widths of approximately 3~200 nm. The diameter of the Berea samples, however, varies from 0.5 to 100 m. (2) B5 and B6 display a unimodal PSD, whereas B1~B4 display bimodal pore size distributions that are similar to the results of Cho et al. (2016) [6]. (3) The average pore width of samples B5 and 6 from well #1 is bigger than that of samples B1~B4 from well #2. The Bakken samples B1~B4 have more mesopores (3–20 nm) than Bakken samples B5 and B6. The two-phase flow mechanisms in micropores and mesopores could differ significantly. The flow in mesopores could be described by the Navier–Stokes equation and Darcy's Law. However, molecular dynamic simulations have revealed that the water–oil flow in nano- and micropores is more complex due to strong liquid–wall interfacial iteration, water and hydrocarbon adsorption on the pore wall, and strong electrostatic forces that cause a water film on the pore wall [37–39].

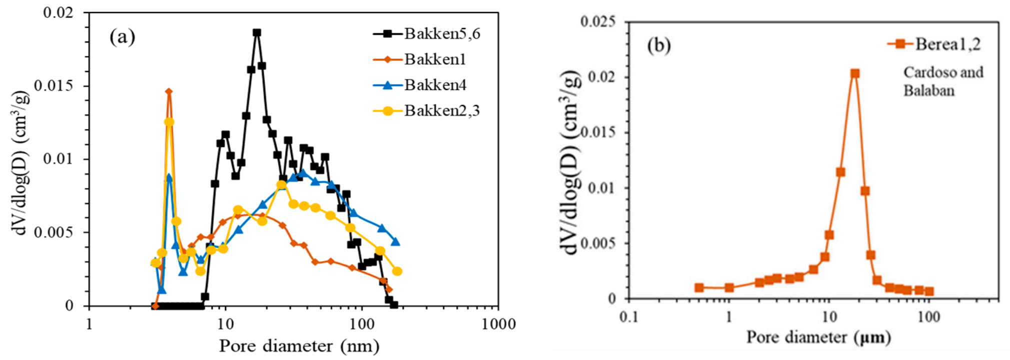

Figure 5. Pore size distribution of Bakken and Berea samples. (**a**) Bakken samples. (**b**) Berea samples from mercury intrusion by Cardoso and Balaban [36].

The results of the X-ray diffraction (XRD)-based mineralogical investigation for the core samples are displayed in Table 2. The quartz, dolomite, feldspar, and calcite are the main minerals found in the Middle Bakken samples, as shown in Table 2. Limestone in the Middle Bakken cores is pale gray. The Berea cores, on the other hand, are quartz sandstone with fine-grained clay cement. Quartz is the main mineral found in Berea cores.

Table 2. XRD analyses of the studied Middle Bakken and Berea cores.

Samples	Kaolonite (wt%)	Illite (wt%)	Mx I/S (wt%)	Chlorite (wt%)	Quartz (wt%)	Calcite (wt%)	Dolomite (wt%)	Feldspar (wt%)	Pyrite (wt%)
B1	-	7	1	-	36	20	20	15	1
B2, B3	-	16	1	1	33	3	24	19	3
B4	-	6	1	1	42	5	25	19	1
B5, B6	11	2	1	30	14	28	12	2	11
B7, B8	12	2	1	29	11	25	18	2	12
A1, A2	5	-	-	-	88	-	2	5	-

3.2. Enhanced Oil Recovery by Surfactant Imbibition

In total, 4 groups of spontaneous imbibition were conducted using brine, 0.1 weight percent SDS (anionic), 0.1 weight percent CTAB (cationic), and 0.1 weight percent HCS (nonionic). The samples used in the experiments are all from the same well and are all Bakken samples with limited depth variation (10,791~10,801 ft).

The oil recovery is shown as a percentage of the initial oil in core samples. The oil recovery is expressed as $R = V_{op}/V_{oi}$, where V_{op} is the volume of produced oil in the oil collector and V_{oi} is the initial oil volume in cores, representing the total oil contained in the core sample at the beginning. Figure 6 shows that during the spontaneous-imbibition experiment, the brine alone was not capable of producing much oil. Only 4.3% is the brine imbibition's total recovery factor. However, compared to brine imbibition, surfactant-assisted imbibition has a substantially larger recovery factor. The recovery factor of the anionic surfactant (SDS) is three times that of brine. The recovery factors of nonionic (HCS) and cationic (CTAB) are around two times those of brine. The reason anionic surfactants yield the higher oil recovery, despite the fact that the concentration of those surfactants is the same, is presumably due to their better efficiency in the wettability alternation. Two main mechanisms responsible for the wettability alteration are ion-pair formation and adsorption of surfactant molecules through interactions with the adsorbed crude oil components on the rock surface [40]. Results from the literature show that the cationic surfactant CTAB can increase the zeta potential of carbonate minerals and change the negatively charged surface into positively charged surface through desorption of stearic acid from the mineral surface via ionic interaction [41]. Experimental results also confirmed that the surfactant HCS can significantly decrease the contact angle of water on the rock surface [42]. In addition, the oil recovery could also be significantly impacted by interfacial tension (IFT). The wettability, however, is what ultimately determines how much oil can be recovered from tight rocks [43,44]. Surfactant adsorption at the rock surface and oil–water contact is the primary source of the wettability alternation. Due to the substantially higher hydroxyl density on its surface compared to other minerals, clay is able to adsorb a significant amount of surfactants by hydrogen bonding [45]. The wettability alternation is a crucial process for the increased oil recovery employing low-salinity water flooding in carbonate reservoirs, according to earlier studies [46]. The PSDs in the Bakken samples are comparable. In light of the mineralogical composition, they differ significantly. According to Table 2, Bakken samples B2 and B3 have substantially higher clay content than samples B1 and B4. As a result, the high oil recovery factors of B2 and B3 can be attributed to the wettability alternation brought on by more surfactant adsorption at the Middle Bakken minerals, particularly clays.

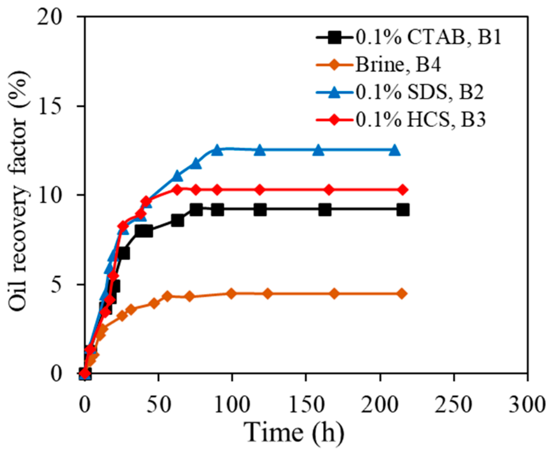

Figure 6. Oil recoveries of B1~B4 (well #1) through spontaneous imbibition experiments with and without various surfactants added.

Spontaneous-imbibition experiments were performed on the Bakken and Berea sandstones to examine the effects of pore size on oil recovery. In comparison to Bakken samples, the pore diameters of Berea samples are substantially bigger (Figure 5). Because A2′s average pore size is bigger than that of B6 and B4, it recovers 47% more oil by brine imbibition than B6 and B4. Berea cores typically have pores that are 21 μm in diameter. Contrarily, the typical pore sizes of B6 and B4 are about 40 and 28 nm, respectively. The bimodal distributions can be seen in the PSDs of sample B4, indicating that there are a number of pores that are extremely small, about 4 nm in diameter. B6 has a 15% final oil recovery (Figure 7a), which is higher than B4′s 4.3% recovery from brine imbibition. In the presence of capillary pressure, the non-wetting phase was primarily displaced out of the larger pores, and the wetting phase flows into smaller pores before the larger pores [47]. The larger pores in Berea samples provide a large number of preferred pathways for the oil phase, thus yielding a higher ultimate recovery factor than B6 and B4. In addition, due to the significant percentage of pores that are nanometer-sized, the oil recovery rate of B4 from brine imbibition is quite lower than that of sample B6. Surfactant spontaneous-imbibition tests using the 0.1 wt% anionic surfactant (964) were carried out on B5 and Barea sample A1. As shown in Figure 7b, the surfactant solution significantly increases the oil recovery in sample B5. Similarly, the surfactant imbibition A1 yields higher oil recovery than B5 due to its much larger pore size. However, B5 recovers about twice as much oil as B6, while oil recovery in Berea sample A1 is slightly improved. This is probably because the Berea samples are primarily composed of quartz, while B5 and B6 have much larger clay content than the Berea samples (Table 2). The anionic surfactant (964) can alter B6′s wettability from oil-wet to water-wet, leading to markedly better oil recovery because of its high adsorption content on clay surfaces.

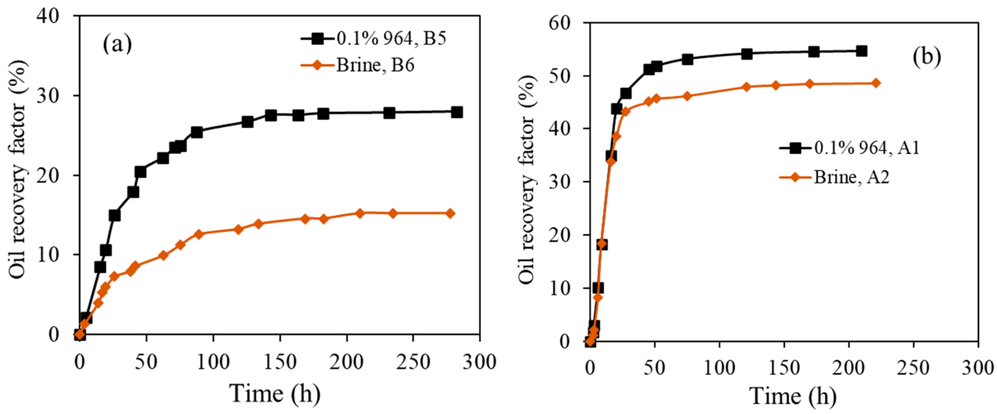

Figure 7. Oil recovery of Middle Bakken and Berea core samples with different pore sizes. (**a**) B5, B6 from well #1. (**b**) A1 and A2.

3.3. Enhanced Oil Recovery by Core Flooding

Figure 8 displays the oil recovery factors from the brine flooding and surfactant flooding. The same whole core sample is used to drill samples B7 and B8. Thus, these two core plugs were assumed to have similar pore size distributions and similar mineral compositions. In this section, the primary recovery was obtained through the use of brine flooding, and the secondary recovery was obtained by the use of surfactants. To ensure that the most oil was recovered, the maximum volume of brine and surfactant solutions injected was 3PV. The brine and surfactant solution concentrations are essentially the same as those for spontaneous imbibition. For those core-flooding experiments, the cationic surfactant CTAB and the anionic surfactant SDS were used, and their concentrations are 0.1 weight percent. Figure 8a shows that samples B7 and B8 have a primary oil recovery of about 23% and 25%, respectively. Compared to spontaneous imbibition, which only produces a maximum recovery of 5~15%, brine flooding gives a substantially higher oil recovery. Therefore, compared to spontaneous imbibition, the core-flooding studies likely yield a higher sweep efficiency.

On these tight core samples, brine flooding experiments were followed by surfactant flooding tests. Both cationic and anionic surfactants were used to examine the impact of the surfactant on oil recovery. The ultimate oil recovery applying SDS is around 35.1%, as shown in Figure 8b, whereas the oil recovery using CTAB is approximately 33.4%. While the CTAB only increased oil recovery from 25.1% to 33.4%, the SDS raised the ultimate oil recovery from 23% to 35%. In comparison to cationic surfactants, anionic surfactants produce a better efficiency of increased oil recovery. Figure 6 illustrates similar results for oil recovery using an anionic surfactant. The charges of surfactants and rock surfaces may both have an impact on the oil recovery. Results from previous studies also support the hypothesis that better oil recovery on carbonate rocks may be achieved by injecting an anionic surfactant into them [48]. The surfaces of carbonate minerals, such as dolomite, are often positively charged [49], which probably facilitates surfactant adsorption on the pore surfaces and results in a significant variation in the wettability of rocks.

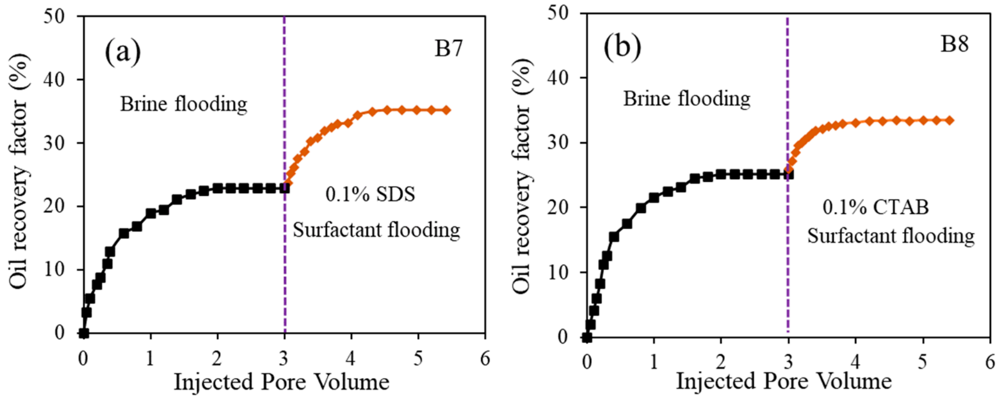

Figure 8. Enhanced oil recovery in two Bakken tight rocks samples by brine and surfactant flooding. (**a**) Flooding with 0.1% SDS. (**b**) Flooding with 0.1% CTAB.

4. Conclusions

This study carefully examined the effectiveness of anionic, nonionic, and cationic surfactants on the oil recovery from various tight Bakken and Berea core samples through spontaneous imbibition and core-flooding experiments.

The SEM and nitrogen adsorption results show that the Bakken samples contain a significant number of nanopores. Large natural fractures were visible in the study samples' SEM pictures. According to measurements on pore size distribution, the majority of the pore diameters in the Bakken samples fall between 3 and 200 nm. Quartz dominates the Middle Bakken sample's mineral composition, followed by dolomite and calcite. The quartz percentage of the Berea samples is higher than 88%, whereas the amounts of clay and feldspar are incredibly low.

The oil recovery in tight Bakken samples by brine imbibition is approximately 4.3~15%, which is very low. The oil recovery from surfactant imbibition is approximately 9~28%, indicating that those surfactants can greatly improve the oil recovery in tight reservoirs through the imbibition process. The results also suggest that the anionic surfactants have a higher ability to improve oil recovery than cationic and nonionic surfactants in Bakken rocks. Due to their strong capacity for wettability alternation, anionic surfactants can significantly improve oil recovery. In addition, the pore sizes also have substantial effects on the oil recovery. The results show that recovery factors increase with increasing pore size. Compared to tests using the imbibition experiment, core flooding with brine and surfactants results in a higher oil recovery of 33~35%. The anionic surfactant favors the recovery of oil in tight Bakken rocks, as demonstrated by studies at the core scale with flooding.

This research experimentally explored the factors, such as pore size, surfactant charge, and mineral composition, that affect the oil recovery in tight rocks. The results could have important applications in the development of unconventional oil reservoirs.

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