Article

Mechanistic Understanding of Delayed Oil Breakthrough with Nanopore Confinement in Near-Critical Point Shale Oil Reservoirs

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Abstract: Recently, significant breakthroughs have been made in exploring northeast China’s shale part of the Q formation. On-site observation reveals that the appearance of oil at the wellhead is only seen a long time after fracturing in many wells. Strong coupling between phase behavior and relative permeability curves in the reservoir with the near-critical point initial condition restricts the efficient development of this kind of shale oil. A series of compositional models are constructed to address the issues to reveal the cause of the late oil breakthrough. Nanopore confinement is checked by including this phenomenon in the numerical model. Before the simulations, the work gives detailed descriptions of the geology and petrophysics background of the target formation. Simulation results show that the delayed oil breakthrough is highly related to the coexistence of three phases at the beginning of production, which is not seen in common reservoirs. The extended period of purely water production complicates subsurface flow behavior and hinders the increase of medium- and long-term oil production. Early-time production behavior in such reservoirs is associated with the gas–liquid relative permeability curves and initial water saturation. Oil–water relative permeability curves affect the water-cut behavior depending on wetting properties. The potential oil-wet property slows down oil breakthroughs. Conceivably, purely gas and water phases exist due to the nanopore confinement of crude oil phase behavior; thus, the late oil production is barely related to the gas–liquid relative permeability curves.

Keywords: shale oil; phase behavior; relative permeability; nanopore; water cut

1. Introduction

The Q formation is widely developed in Songliao Basin, where the oil and gas generation indexes are promising. Few tectonic movements and few large-scale fault zones occur in this area, making it suitable for oil and gas exploration and development [1,2]. The first member of the Q formation contains high-quality source rocks and high hydrocarbon generation potential. The shale formation strata are similar to the Forth Worth Basin and Barnett shale stratigraphic assemblage in the United States, containing miscellaneous mineralogies. The shale of the Q formation is felsic; generally, the first member has less clay than the second and third members [3].

During the development of G Sag in the first member, deferred oil production appears in many horizontal wells after fracturing. The effluent at the wellhead continues to be pure water phase for a long time. This phenomenon did not occur in Longdong shale oil, Mahu tight oil, Jimusar shale oil, and Changqing tight oilfields in China [4,5]. For example, the average oil appearance time in the demonstration area of three-dimensional development of the Mahu tight conglomerate reservoir is just 1.67 days, and the initial production increased rapidly. Similarly, according to our literature survey, such postponed oil production has not been recorded in shale reservoirs in the United States. The late and long-term water production occurrence means that complex phase transition and multiphase flow behavior have occurred in the subsurface, regulating medium- and long-term oil and gas production.
The late appearance of oil is closely related to the initial state of the reservoir at the critical point of the phase envelope and the three-phase flow of oil, gas, and water. Generally speaking, the initial pressure of the reservoir is much higher than the saturation pressure [6], so the oil and water phases coexist, and the oil phase is in a single-phase state [7]. However, for many light crude oils in G sag, the initial state of the reservoir is close to the critical temperature and critical pressure point, implying that oil, gas, and water might coexist initially, leading to the unique production flow circumstance after fracturing. Therefore, the associated production behavior might be unique as well. However, this kind of study has not been attempted in previous research.

Based on the discussions above regarding the necessity to clarify the oil production mechanism and the root cause, the objectives of this study are:

1. Provide a relatively comprehensive background petrophysical delineation of the Q formation.
2. Establish exploratory reservoir simulation models based on PVT data to interpret the delayed oil production where the inceptive models are compositional with deep insight into the coupled phase behavior and relative permeability curves.
3. Investigate possible nanopore confinement on the relative permeability interpretation.

2. Materials and Methods

To achieve the goals listed above, the methodology of this work is structured as below. The geological background will be given for the target formation, including the mineral percentages. Then, the crude oil phase behavior of the target formation will be compared with other places in the world. Based on geological information and the crude oil phase behavior, a numerical model to solve the problem will be described.

2.1. Geological Background

Q formation consists of the first, second, and third members. Recently, the G sag has been of particular interest due to the significant potential of the original oil in place. The estimated recovery oil in the G Sag is $1.286 \times 10^9$ tons. The first member is richer in organic matter than the other two and is interbedded with clay shale, silty shale, and clastic silty shale. The oxygen-poor environment is more damaged as depth increases; therefore, lithofacies in the second and third members change to organic matter-poor shale [3]. Low temperature and pressure nitrogen adsorption show pore size distributions across several wells in the G sag. Generally, samples from these wells show similar attributes. Pore size below 50 nm dominates with 2 nm and 20 nm peaks in the micro- and mesopore range (2–50 nm) [8]. Another smaller peak appears at about 70 nm in the macropore range (>50 nm) (Figure 1). A mercury intrusion experiment shows that most of the pore diameter ranges from 10–130 nm, which is in reasonable agreement with the nitrogen adsorption experiment as micro-and meso pores are challenging to detect using mercury [9].

Laminated and layered shale are classified based on sedimentary structure; the criterion is that each lamination is smaller or larger than 1 mm, respectively [10]. Figure 2 shows mineralogy distribution in laminated shale and layered shale in the G Sag. The samples tested are from 5 wells (5 sub-columns). Mineralogy distribution agrees in laminated and layered shale, where quartz and clay dominate and calcite percentage is relatively low. Mineralogy results classify lithologies into silty shale, limestone shale, and dolomite shale in this region. Silty and limestone shale contains relatively high calcite, and dolomite shale contains relatively high ankerite. Correlation analysis between different mineralogy shows that organic matter is always accounted for with pyrite and clay, consistent with many scanning electron microscopes (SEMs) and well-logging analyses [11,12]. At the same time, quartz presence is irrelevant with plagioclase and ankerite.
well far away from the center of the basin, the density of degassed oil viscosity increases.

The distribution of shale oil worldwide shows spatial and vertical heterogeneity with complex phase behavior variations. For example, Eagle Ford shale oil density in the United States decreases from northwest to southeast. The types of crude oil change from black oil to volatile oil, condensate gas, and dry gas in turn [13].

Due to different degrees of thermal evolution, crude oil composition changes with lithofacies and organic facies, and oil density decreases vertically. For example, in one horizontal well near the center of the Songliao Basin, the viscosity of degassed crude oil is 2.70 mPa·s, and the ratio of gas to oil is 503.6 m³/m³. In contrast, in another horizontal well far away from the center of the basin, the density of degassed oil viscosity increases to 10.74 mPa·s, and the ratio of gas to oil decreases to 72.66 m³/m³. Correspondingly, the phase diagram of crude oil is also quite different.

A relatively comprehensive comparison of three kinds of Eagle Ford shale oil phase diagram and three kinds of Bakken shale oil shows that crude oil’s initial temperature and pressure are far from the critical point (Figure 3a,b). Three kinds of Bakken crude oil are all black oil. Eagle Ford crude oil can be divided into black oil, volatile oil, and condensate gas. The reservoir temperature is slightly higher than the phase critical point temperature in condensate gas, so the condensate phenomenon occurs.
Due to different degrees of thermal evolution, crude oil composition changes with different lithofacies and organic facies, and oil density decreases vertically. For example, in one reservoir, was set to be ten times the matrix permeability due to the stimulation impact. When the pressure coefficient, defined as the ratio between pore and hydrostatic pressure, 1.4 and 1.5 are assumed, the initial pressure of the volatile reservoir is close to the bubble point pressure, 1.4 and 1.5 are assumed, the initial pressure is still far from the critical pressure. The phase profiles of the two shale oils are quite different. When the pressure coefficients are 1.4 and 1.5, the initial pressure of the volatile reservoir is close to the bubble point curve on the phase envelope. Therefore, with such phase diagram features, oil, gas, and water might coexist before reservoir production. Production data also show that these reservoirs usually have delayed oil appearance.

2.3. Numerical Model Construction

An inceptive conceptual model was constructed based on the geological setting and was used to illustrate the deferred oil production for proof of concept. The compositional model GEM in the CMG software Suite was applied in this study. One stage of hydraulic fractured horizontal wells with one perforation was simulated. A finely meshed system was applied with 10 m $\times$ 10 m $\times$ 10 m for each grid. The grid dimension was checked and verified that simulation results were negligibly disturbed with smaller size. The total grid number of the system was 63 $\times$ 31 $\times$ 5 = 9765. Local grid refinement was applied in the hydraulic fracture cell (Figure 4a).

G Sag shale shows strong anisotropy due to the strong lamination. Matrix horizontal and vertical permeability values were $6.85 \times 10^{-2}$ mD and $5.50 \times 10^{-3}$ mD, respectively, based on gas permeability measurement after slippage correction using pulse-decay experiments [16]. The stimulated reservoir volume (SRV) region, located at the center of the reservoir, was set to be ten times the matrix permeability due to the stimulation impact. The detailed phase envelope of the volatile oil with different gas mole fractions is provided in Figure 4b for compositional simulation.

Water saturation was initially set to be 0.4, an averaged value based on logging of the target formation. The later saturation sensitivity analysis was checked on the oil delayed production (Table 1). Figure 5 shows near-critical point volatile and black oil components. The volatile oil had higher percentages in C1 to C4, and for black oil, percentages were higher in fractions above C5.

![Figure 3](image_url)

**Figure 3.** (a) Three kinds of phase diagrams of the Eagle Ford shale oil: black oil, volatile oil, and gas condensate [14]; (b) three kinds of Bakken oil: all black oil [15]; (c) near-critical volatile oil near the center of the Songliao Basin in the G Sag; and (d) black oil far away from the center of Songliao Basin. Different lines in (a,b) denote different oil samples in the specified shale.
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The detailed phase envelope of the volatile oil with different gas mole fractions is provided in Figure 4b for compositional simulation. The volatile oil had higher percentages in C1 to C4, and for black oil, percentages were higher in fractions above C5.

The mole percentage of volatile oil (horizontal well in the basin center) and black oil (horizontal well away from the basin center) components are shown near-critical point volatile and black oil components.

For general black oil reservoirs such as Figure 3d, the initial reservoir pressure is higher than the bubble point curve on the phase envelope. Gas remains dissolved into the oil phase without separation. Therefore, the case will be two phases only, which is straightforward,
and oil breakthroughs could be explored using merely the oil–water relative permeability curves at the early production period.

In the center of the basin, where volatile oil is more frequently reported with late oil production, the volatile oil case is exclusively explained in this study, as shown in Figure 3c. The initial reservoir condition is very close to the phase envelope with 100% gas mole fraction. The flashed results deviate from the phase slightly as there is uncertainty about the phase percentage near the critical point. At the initial reservoir condition, the flashed saturation fraction of each phase is 0.35, 0.4, and 0.25 for gas, water, and oil.

Under the initial reservoir temperature of 118 °C and initial pressure of 35.0 MPa (pressure coefficient 1.4), the compositional model was calibrated based on a series of phase behavior experiments, including differential liberation and constant volume depletion.

Since the scenario is unlike most reservoirs where oil and water exist alone, three phases, including gas, all exist. Therefore, flow behavior is complicated, and it is insufficient to consider water–oil relative permeability only. We investigated the gas–liquid relative permeability curves first. The liquid phase takes account of both oil and water. Because of the difficulty of obtaining valid relative permeability curves, especially for ultra-low permeability porous media, conceptual water–oil, and gas–liquid relative permeability curves were constructed. Capillary pressure curves were imposed based on the Leveret J function for water–oil and gas–liquid. For the well control part in the model, a well with a constant pressure drop rate of 41 KPa/day was used. Extra simulation efforts proved that the bottom hole pressure drop rate impacted the water-cut behavior in the late production time, while this study primarily focuses on the early production behavior and aims to ascertain decisive parameters in the relative permeability curves and reservoir conditions; therefore, the bottom hole drop rate was kept constant throughout the study.

3.1. Gas–Liquid Relative Permeability Curves

This study follows the philosophy of tracing the relative permeability behaviors according to late oil breakthrough field observations combined with compositions model and geologic settings.

The first target is to show the impact of critical gas saturation on water-cut behavior. Critical gas saturation, \( S_{gc} \), is the threshold value when gas starts to become mobile to flow. Oil–water relative permeability curves are settled at this step. The relative permeability curves were constructed starting at the initial gas saturation of 0.35. Then, \( S_{gc} \) increased to 0.45 step-by-step (Figure 6a). The criterion to determine the breakthrough day is that the fraction of phases other than water is higher than \( 8 \times 10^{-6} \). Figure 6b shows that the oil breakthrough time increases as this threshold value increases. When \( S_{gc} \) is below 0.35, oil breaks through as soon as the wellhead is opened without any delay. When \( S_{gc} \) is 0.37, the oil breakthrough time is 53 days. Actual production data show that the oil rate becomes noticeable at about 170 days. When tuning the \( S_{gc} \) to 0.45, the gas relative permeability curve moves left, and the oil breakthrough time is about 196 days (Figure 6d). Therefore, it could be inferred that the late oil production phenomenon is closely related to the high critical gas saturation.

The oil breakthrough time could be explained using the oil saturation hump formed in the saturation vs. time plots in the grid of \((16,32,3)\). Saturations of three phases are plotted with four scenarios of different \( S_{gc} \) in Figure 6c. Gas is easier to bring to the surface due to the low viscosity and favorable flowability. When gas accumulates in the porous media, pressure decreases in the near-wellbore region, and the gas expands to reach the critical gas saturation to form the continuous flow. Under a lower critical gas saturation, the gas flow will be more favorable; liquid flow will be more unfavorable, consisting of both oil and water flow. Because oil cannot be produced, it will start to accumulate near the wellbore region. Like gas, oil needs to accumulate to reach the level of residual oil saturation to allow it to flow. Therefore, the accumulation phenomenon is favorable for an oil saturation increase, and the oil will increase in flowability.
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In contrast, with a higher critical gas saturation, gas will accumulate to increase occupancy in the pore space. Therefore, overall liquid production will be impaired. So, under the same set of oil–water relative permeability curves, the oil flow rate into the wellbore region will also be weakened as the humping phenomenon gradually disappears, as shown in the dashed circles. Therefore, reaching the residual oil saturation will become more difficult, leading to delayed oil breakthrough.

3.2. Oil-Wet vs. Water-Wet

The critical gas saturation and the wettability properties of shale are explored in this section to examine the oil–water relative permeability-dependent oil breakthrough.

In addition to the end points that control multiple phase flow behavior, a water-wet or an oil-wet characteristic is also essential, marked by features such as the crossing saturation, relative permeability curves’ curvature, and maximum points. Generally speaking, for a water-wet porous media system, the crossing point moves relatively right when water saturation is imposed as the $x$-axis. The maximum point on the water relative permeability curve is lower than the oil-wet system. The lacustrine shale oil formation in this study could be considered oil-wet with a rich organic matter content. Still, minerals such as quartz and calcite may also render the media water-wet peculiarity. Some articles suggest the shale
could be considered a “mix-wet” porous media though that is not the main research target in this work [17–19].

The water-wet relative permeability curve with a residual oil saturation of 0.52 is adjusted to be more water-wet by reducing the water curve maximum point from 0.8 to 0.5; therefore, the crossing point moves right, from 0.43 to 0.44 and 0.45, respectively (Figure 7). The lowered water relative permeability, while it does not change the oil relative permeability, implies enhanced oil flowability. Gas relative permeability increased slightly, but to a lesser extent than oil. This is because the water, being only a part of the liquid, is impaired. For more water-wet cases of the 0.44 and 0.45, gas and oil breakthrough immediately after the well is opened without any delay. Further comparison of 0.44 and 0.45 shows that the oil flow rate increases more efficiently in the more water-wet scenario. Still, the gas flow rate is lowered as the easier flow cannot compete against oil.

Therefore, for the porous media initially saturated with three phases, both the gas–liquid and water–oil relative permeability curves will influence the flow behavior of each phase. Higher critical gas saturations delay oil and gas breakthroughs, while the gap between oil and gas will be shortened. Late oil breakthrough is rarely seen in a water-wet system as the hydrocarbon flows relatively easily compared with water. The flowability of two kinds of hydrocarbons, gas, and oil, also need to compete to see the flow rate. Gas is naturally easy-flowing due to its low viscosity and high compressibility, described as dynamic properties. Static properties, the porous media’s water- or oil-wet nature, are equally crucial in governing fluid flowability.

### 3.3. Post-Fracturing Water Saturation Effect

Some fracturing fluids are forced back to the surface during the flowback process, but some water remains blocked in the subsurface. Therefore, water saturation higher than the original value might be seen. The higher water saturation in the SRV region was also investigated by manually increasing the centered reservoir water saturation.

Previous proof-of-concept simulations assumed the water saturation was homogenous with a value of 0.4. In our study, the value increased to 0.45 and 0.5 to study the impact of hydraulic fracturing operations. The SRV region accounted for 21% of the total area. Oil breakthrough time did not change noticeably for the three cases, all around 105 days, which is not surprising because water saturation close to the wellbore and perforation region can quickly decrease as gas accumulates to the breakthrough point to start flowing (Figure 8). However, as fluids continue to produce away from the wellbore, the water-cut behavior
becomes different. The water cut in a lower initial water saturation case drops more quickly. Therefore, it could be reasonably argued that a large-scale stimulated reservoir with high water saturation blocked is also one of the reasons for the long-time water production.

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The typical in-house experiments’ direct phase behavior output parameters for crude oil mixtures are unconfined without the porous media impact. The pore confinement is negligible in conventional reservoirs because molecule size could be insignificant compared with pore size. In unconventional shale reservoirs, molecule size rises compared to pore size.

Different approaches have been proposed to adjust thermodynamic properties to construct the crude oil phase envelope. The general belief is that the phase envelope will shrink due to the pore confinement; the smaller the pore size, the smaller the phase envelope. The bubble point pressure curve will decrease, and the dew point pressure curve might increase or decrease [22]. This work applies the approach by Ma and Jin [23,24] to estimate shifts in critical temperature and pressure. The eight subcomponents shifted critical temperature, and pressure was calculated to reconstruct the phase envelope, taking representative pore sizes of 5 nm, 10 nm, 20 nm, and 50 nm, respectively. In Equations (1)–(3), $\Delta T$ and $\Delta P$ are temperature and pressure deviations, respectively; $D$ and $\sigma$ are pore size and effective molecule size, respectively. The “c” and “cb” subscripts for temperature and pressure denote confined and bulk phases. The effective molecule size is estimated based on Haider’s relationship between the critical temperature and critical pressure [25].

$$\Delta T_C = \frac{T_C - T_{Cb}}{T_C} = 1.1775 \left( \frac{D}{\sigma} \right)^{-1.338} \quad \text{when} \quad \frac{D}{\sigma} \geq 1.5 \quad (1)$$

$$\Delta T_C = \frac{T_C - T_{Cb}}{T_C} = 0.6 \quad \text{when} \quad \frac{D}{\sigma} \leq 1.5 \quad (2)$$

$$\Delta P_C = \frac{P_C - P_{Cb}}{P_C} = 1.15686 \left( \frac{D}{\sigma} \right)^{-0.783} \quad (3)$$

Figure 9 summarizes each component’s thermodynamic properties with varying pore sizes. Flash calculations were performed to reconstruct the phase envelopes. Critical points are labeled on each phase envelope using solid dots. The initial condition with the pressure coefficient 1.4 is illustrated to show the position compared to the phase curves. It is intuitively thought that the vertical line crossing the critical point separates the pure gas.
phase and pure liquid phase outside the envelope. This is not strictly correct, however, due to the blurred saturation situation represented by the shadowed area. After running all the models and flashing the vapor and liquid, trial and error results show that in all the conditions with nanopore confinement, only gas and water exist, i.e., gas saturation is 0.6, and water saturation is 0.4.

\[
\Delta T = \frac{T_c - T}{T_c} = 1.1775 \left( \frac{\sigma}{3} \right)^{-0.77} \quad \text{when} \quad \left( \frac{\sigma}{3} \right) < 1.5 \quad (1)
\]

\[
\Delta p = p_c - p = 1.15686 \cdot D^{0.77} \quad (3)
\]

Figure 9 summarizes each component's thermodynamic properties with varying pore sizes. Flash calculations were performed to reconstruct the phase envelopes. Critical points are labeled on each phase envelope using solid dots. The initial condition with the pressure coefficient 1.4 is illustrated to show the position compared to the phase curves. It is intuitively thought that the vertical line crossing the critical point separates the pure gas phase and pure liquid phase outside the envelope. This is not strictly correct, however, due to the blurred saturation situation represented by the shadowed area. After running all the models and flashing the vapor and liquid, trial and error results show that in all the conditions with nanopore confinement, only gas and water exist, i.e., gas saturation is 0.6, and water saturation is 0.4.

Therefore, if the plausible altered phase envelope does occur, all the hydrocarbon species are in the vapor phase after the vapor–liquid flash calculation. The delayed oil appearance could be simply attributed to the gas–liquid permeability curves that reside in the region below the critical gas saturation. Oil initially appears because of gas condensation at the wellbore and the surface. Critical gas saturation from 0.58 to 0.62 with a step of 0.01 is examined to locate the oil. The first day the oil appears changes between 72 and 122 days in the span of \( S_{gc} \) change. As the reservoir pressure is depleted, gas saturation decreases as if flows to the surface. The appearance of liquid hydrocarbon might be heterogeneous across the near-wellbore region because each hydrocarbon's concentration distribution is uneven. Some mixtures could flash to the liquid phase in the pressure depletion process, but some cannot. The flow will become the three-phase flow scenario when liquid hydrocarbon appears subsurface, but the oil starting time of appearance of oil depends solely on the gas–liquid relative permeability curve.

4. Conclusions

In this study, we addressed the problem of intense phase behavior and relative permeability curves’ coupling in the shale formation of Songliao Basin and found the cause of the unique flow behavior. Detailed qualitative comments are listed below, corresponding to the three objectives listed.

- This work first gives the geology and petrophysics background of the shale oil reservoir in northeast China. The target shale reservoir is characterized by laminated sequence with strong anisotropy. Micropores and mesopores dominate. Pyrite is found to be closely related to the presence of organic matter.
- The models built in this study are to understand the mechanisms of the unusual water-cut behavior for interpretative purposes. The volatile oil frequently associated with the late appearance of oil is discussed. Unlike most conventional reservoirs, the compositional model showed that the target shale reservoir is initially saturated with
three phases: gas, water, and oil. The impacts of gas saturation, water saturation, and wetting properties are described below.

Simulation results show that the initial subsurface gas presence causes the late oil breakthrough. Both the water–oil and gas–liquid relative permeability curves govern the oil breakthrough time. The higher the critical gas saturation is, the later the oil breakthrough time. High water saturation in the SRV region after fracturing was not a controlling factor for oil breakthrough time with fixed relative permeability curves, as the flow period was relatively fast in the near-wellbore region. However, water-cut behavior will differ as fluids continue to be produced away from the wellbore region.

Water-wet porous media makes oil and gas flowable, accelerating hydrocarbon breakthrough time. Therefore, the issue of the late appearance of oil tends to arise in the oil-wet system, following the organic-matter-rich sedimentary environment of the target shale oil formation.

- Nanopore confinement on crude oil phase behavior was analyzed with representative pore sizes from 5 nm to 50 nm. The initial reservoir point will be outside the phase envelope but close to the critical temperature. Based on the flashed evaluation, the initial reservoir will be only gas and water; therefore, the oil breakthrough time will depend entirely on the gas–liquid relative permeability curves.

Funding: The author is grateful to the Science Foundation of China University of Petroleum, Beijing (ZX20210070).

Data Availability Statement: Not applicable.

Acknowledgments: Part of the article was presented at the SPE Eastern Regional Meeting. The authors are grateful for the software donation from Computer Modelling Group Ltd.

Conflicts of Interest: The authors declare no conflict of interest.

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