Experimental study on gas slippage of Marine Shale in Southern China

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1. Introduction

As shale gas reservoirs are increasingly becoming major fossil energy in today's world, understanding the gas flow mechanism in these unconventional gas resources is crucial. The dynamics of gas flow in shale gas reservoirs has become an important research topic during the current decade in the oil and gas industry [1].

There are four types of porous media of different length scales in shale gas reservoirs: nanopores, micropores, natural fractures and hydraulic fractures [2]. Fig. 1 shows the pores in conventional and shale gas reservoirs. As presented in the schematic figure, shale gas reservoirs contain more nanopores than conventional reservoirs. Radius of pore throats in gas shale sediments generally ranges from a few nanometers to a few micrometers [3]. These matrix pores make up the significant portion of reservoir space of natural gas. The complex fracture system composed of natural fractures and hydraulically induced fractures connects matrix pores, which forms a high-permeability network in gas shale.

When gas flows from nanopores to fractures and from fractures to wellbore, the flow mechanism varies due to the different length scales of flow channels. Gas flow in larger pores, throats and fractures generally follows Darcy equation, and in nanopores, slip flow and diffusion dominate [4]. The Knudsen number, a significant dimensionless parameter, is used to classify the flow regimes in porous media of different length scales. It is defined as the ratio of the gas mean free path \( \lambda \) (m) and the pore radius \( r \) (m) [3].

\[
Kn = \frac{\lambda}{r}
\]
The gas mean free path \( \lambda \) is defined as

\[
\lambda = \frac{K_B T}{\sqrt{2 \pi \delta^2 p}}
\]

in which \( K_B \) is the Boltzmann constant, \( 1.3805 \times 10^{-23} \text{ J/K} \); \( T \) is temperature, K; \( p \) is pressure, Pa; \( \delta \) is the collision diameter of the gas molecule. For methane, \( \delta = 0.4 \times 10^{-8} \text{ m} \).

Different Knudsen numbers correspond to different flow regimes [5,6]: continuum flow \( (Kn < 0.001) \), slip flow \( (0.001 < Kn < 0.1) \), transitional flow \( (0.1 < Kn < 10) \), and free molecular flow \( (Kn > 10) \). Fig. 2 presents the Knudsen number as a function of pressure with various pore sizes ranging from 1 nm to 5 \( \mu \text{m} \).

The gas slippage typically occurs in the condition where the mean free path of the gas molecules is no more negligible compared to the average effective pore throat radius \( (0.001 < Kn < 0.1) \). The gas molecules would slip on the inner surfaces of the pores. This effect gives apparently higher permeability than the absolute permeability measured using a liquid [7,8].

Klinkenberg (1941) [9] first addressed the gas slippage in porous media and gave a linear correlation between the measured gas permeability and the reciprocal mean pore pressure.

\[
k_a = k_\infty \left(1 + \frac{b_k}{\bar{p}}\right)
\]

where \( k_\infty \) is the absolute permeability, \( mD \); \( k_a \) is the measured gas permeability (apparent permeability), \( mD \); \( b_k \) is the Klinkenberg slippage factor, MPa; \( \bar{p} \) is the mean pore pressure, MPa. Table 1 presents various correlations for gas slippage factor \( b \) proposed in the subsequent work.


\[
k_a = k_\infty \left(1 + \frac{4Kn}{1 - bKn} \right)
\]

\[
\alpha = \frac{128}{15\pi^2} \tan^{-1} \left(4Kn^{0.4}\right)
\]

where \( \alpha \) is the dimensionless sparse coefficient, \( b \) is gas slippage factor and generally \( b \approx 1 \). Civan (2010) [13] improved Beskok and Karniadakis model and demonstrated a simple inverse power-law expression of the sparse coefficient \( \alpha \) as given below

\[
\alpha = \frac{a_0}{1 + AKn^B}
\]

where \( A = 0.170, B = 0.434, a_0 = 1.358 \).

Tang et al. (2005) [15] reported that the second-order term of the Knudsen number \( (Kn^2) \) cannot be neglected for gas flow with relatively high Knudsen numbers. They presented a widely known model given as follows

\[
k_a = k_\infty \left(1 + \frac{A}{\bar{p}} + \frac{B}{\bar{p}^2}\right)
\]

where \( A, B \) are constants that depend on gas properties and pore geometry.

Zhu et al. (2007) [16] also recommended using a higher-order equation which can be written as

\[
k_a = k_\infty \left(1 + Ae^\delta\right)
\]

Fathi et al. (2012) [18] proposed the following equation based on their numerical analysis.
shown that the facility possesses excellent sealing when confining pressure is over 5 MPa. Results of standard core showed that experimental data were within the accepted error range. Fig. 4 shows the diagram of the gas flow experiment.

The core sample was enclosed in a rubber sleeve which was mounted in a core holding unit. We used nitrogen gas as the pore fluid and water as the confining fluid. In all our tests, the

\[ k_a = k_\infty \left[ 1 + \left( \frac{b}{P} \right)^2 \left( \frac{k_{02}}{L} \right) \right] \quad (8) \]

where \( k_{02} \) is a new length scale associated with the kinetic energy of the bouncing-back molecules.

There are several ways [8] to measure shale permeability such as steady-state [1,19] and pressure pulse-decay [14,20] measurements. Experiment study of slippage is based on these measurements in different experimental conditions. Steady-state measurement is widely used to measure the permeability of porous media. Zhu et al. (2015) [21], Yang et al. (2015) [22] and Kang et al. (2015) [19] conducted steady-state flow experiments at constant confining pressure and constant net confining pressure. However, Steady-state measurement usually costs much time. Therefore, Brace et al. (1978) [23] proposed pulse-decay method based on unsteady-state flow mechanism, which greatly reduces the time of measurements. Fathi et al. (2012) [18] conducted a transient permeability measurement using crushed samples. Ren et al. (2015) [24] proposed an unsteady-state measurements considering slippage.

In this paper, we presented a laboratory study on gas slippage by using low permeability shale samples. We characterized two types of cores from Qionghuzi Formation and Longmaxi Formation of Marine Shale in Southern China. Cores with well-tended appearance are used to model shale matrix and cores with penetration fractures are used to model matrix affected by hydraulic fractures or macro natural fractures. We measured gas permeability by changing the pore pressure at constant confining pressure. Then we analyzed the difference of experiment results between two types of cores and showed the gas flow behavior in cores with fractures.

2. Sample description

We extracted eight intact core samples for gas flow experiments from Qionghuzi Formation and Longmaxi Formation of Marine Shale in Southern China. Four cores numbered S1–S4 were with well-tended appearance and cores numbered S5–S8 were with penetration fractures (Fig. 3). The brittleness of shale makes it easy to make fractures. Fractures of cores S5–S8 generated during the process of increasing confining pressure in core holding unit. Then we heated these eight cores to 60 °C to drive off any free fluid for 48 h.

3. Gas flow experiments and experimental setup

We conducted gas flow experiments by using the HA-III-AH2S-CO2 experimental facility of core. Iron core and standard core were tested respectively to verify the sealing and precision of the equipment before the measurements. Results of iron core showed that the facility possesses excellent sealing when confining pressure is over 5 MPa. Results of standard core showed that experimental data were within the accepted error range. Fig. 4 shows the diagram of the gas flow experiment.

Fig. 4. Experimental diagram. 1. Nitrogen gas cylinder. 2. Pressure regulating valve. 3. Pressure sensor. 4. Core holding unit. 5. Temperature control chamber. 6. Confining pressure pump. 7. Soap film flowmeter. 8. Computer.

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**Table 1**

Various correlations for Klinkenberg gas slippage factor.

<table>
<thead>
<tr>
<th>Model</th>
<th>Correlation</th>
<th>Comments</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Klinkenberg (1941)[9]</td>
<td>( b_k = 4.61(\sigma/r)^{0.39} )</td>
<td>( \sigma = 1 ), Air</td>
<td>( h_0 ): psi</td>
</tr>
<tr>
<td>Heid et al. (1950) [10]</td>
<td>( b_k = 11.419(\sigma_k)_{0.33} )</td>
<td>Air</td>
<td>( h_{air} ): mD</td>
</tr>
<tr>
<td>Jones and Owens (1979) [11]</td>
<td>( b_k = 12.639(\sigma_k)_{0.53} )</td>
<td>Air</td>
<td>( \sigma ): psi</td>
</tr>
<tr>
<td>Sampath and Keighin (1982) [12]</td>
<td>( b_k = 13.851(\sigma_k)_{0.53} )</td>
<td>Air</td>
<td>( \sigma ): psi</td>
</tr>
<tr>
<td>Florence et al. (2007) [7]</td>
<td>( b_k = 0.0094(\sigma_k)_{0.5} )</td>
<td>Nitrogen</td>
<td>( h_0 ): Pa</td>
</tr>
<tr>
<td>Civan (2010) [13]</td>
<td>( b_k = 0.026(\sigma_k)_{0.4} )</td>
<td>Nitrogen</td>
<td>( h_{air} ): mD</td>
</tr>
<tr>
<td>Letham and Bustin (2015) [14]</td>
<td></td>
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\( k_a = k_\infty \left[ 1 + \left( \frac{b}{P} \right)^2 \left( \frac{k_{02}}{L} \right) \right] \quad (8) \)
Experimental Data of S8

Experimental Data of S7

Experimental Data of S6

Liner Fitting for S5

Experimental Data of S5

Experimental Data of S4

Experimental Data of S3

Experimental Data of S2

Experimental Data of S1

confining pressure was at 20 MPa and the downstream pressure was atmospheric pressure. The initial upstream pressure was at 0.2 MPa. Two pressure sensors and a flow sensor were used to detect and transmit experimental data so that we could observe and log it on the computer. We recorded the upstream pressure, downstream pressure and flow rate until the data remained stable. Then we increased the upstream pressure and conducted next stage of experiments. For cores S1–S4, we increased the upstream pressure by 0.2 MPa per stage; for cores S5–S8, we increased the upstream pressure by 0.1 MPa per stage. Computer recorded pressure, temperature and flow rate in real time, and it calculated the measured gas permeability by using the equation as follows:

\[ K_g = \frac{2q_0\mu L}{Ap_0(1/c_0^2 - 1/c_1^2)} \times 10^{-4} \]  

(9)

where \( q_0 \) is outlet flow, cm\(^3\); \( \mu \) is gas viscosity, mPa·s; \( p_0 \) is atmospheric pressure, MPa; \( A \) is cross-sectional area of core, cm\(^2\); \( L \) is length of core, cm; \( p_1, p_2 \) are the upstream pressure and downstream pressure, MPa; \( K_g \) is measured gas permeability, mD. The viscosity was corrected during the calculation for it would change with temperature and pressure.

4. Results and discussion

4.1. Gas slippage in S1–S4

Figs. 5 and 6 present gas flow rate as a function of differences of pressure squares. Overall, gas flow rate increases with the increase of differences of pressure squares and presents a convex trend. However, there are obvious differences between the curves. Gas flow rate in Fig. 6 ranges from 0 to 7.2 cm\(^3\)/s while gas flow rate in Fig. 5 ranges from 0 to 0.3 cm\(^3\)/s, which indicates that fractures contributes much to the gas flow rate of cores S5–S8. Then, the trend of convex was related to the absolute permeability [21]. The curves of lower-permeability cores (S1–S4) are more like a straight line while curves of higher-permeability cores (S5–S8) would present an obvious convex trend.

4.2. Gas slippage in S1–S4

Klinkenberg curves of S1–S4 measured in this study are presented in Fig. 7. Four curves all follow a near-linear trend with varying amounts of scatter. Permeability increases linearly with incremental inverse pore pressure, which indicates that the slippage dominates in matrix at the experimental condition \((p_c = 20 \text{ MPa}, p_p < 3 \text{ MPa}, T = 60 \text{ °C})\). Permeability and slippage factor were calculated by comparing the linear fitting data with the Klinkenberg slippage model and we would discuss it in subsequent work.

As presented in Fig. 8, permeability ranges from 0.05 mD to 0.5 mD which is a few orders of magnitude larger than that of S1–S4. The existence of fractures would improve shale permeability obviously. Moreover, Klinkenberg plots of S5–S8 follow an obvious non-linear relation which could be divided into two parts. In the left part of the curves \((1/p_p < 2 \text{ MPa}^{-1})\), there was dramatic increase. Then we saw an approximately linear rise and the slope of the section was close to that of S1–S4 when inverse pore pressure was over 2 MPa\(^{-1}\).

4.3. Flow mechanism in shale matrix and fractures

There was no penetration fractures in cores S1–S4, so gas flow in these samples could be seemed as flow in shale matrix in

Fig. 5. Gas flow rate versus differences of pressure squares of samples S1–S4.

Fig. 6. Gas flow rate versus differences of pressure squares of samples S5–S8.

Fig. 7. Klinkenberg plots of S1–S4.

Fig. 8. Klinkenberg plots of S5–S8.
which gas slippage dominates. Measured gas permeability is as a linear function of inverse pore pressure. For cores S5–S8, existence of fractures complicates the flow mechanism. Fractures opening or closing is related to the effective stress which is defined as the difference between confining pressure and pore pressure. In this study, confining pressure was set as a constant 20 MPa, so effective stress negatively correlated with pore pressure.

Fig. 9 presents different flow mechanisms of different stress condition in fractures. High effective stress (low pore pressure) would lead to closure of the fractures. However, fractures would not close completely for the anisotropy of shale and anti-deformation of filler. The flow mechanism was same as flow in shale matrix that gas slippage dominates and the matrix has higher permeability due to the unclosed fractures. We saw a linear correlation in this pseudo-matrix section. Contrarily, low effective stress (high pore pressure) would open the fractures, which would extend the flowing space. According to Eq. (1), the Knudsen number negatively correlated with pore radius, so extension of flowing space causes a decrease of the Knudsen number, which reduces the contribution of gas slippage effect. Moreover, high pore pressure and wide flowing channels even lead to the occurrence of Forchheimer effect. A sharp decrease appears in this pseudo-fracture section.

4.4. Correlation for slippage factor $b$

Slippage factor $b$ and corrected permeability of measured cores were given by regressing linear experimental data to the Klinkenberg slippage model (Eq. (3)). As presented in Fig. 10, the slippage factor of S1–S4 ranges from 0.0526 MPa$^{-1}$ to 0.3333 MPa$^{-1}$ when permeability ranges from 0.0078 mD to 0.0012 mD. For pseudo-matrix flow (linear section in Fig. 7) of S5–S8, slippage factor ranges from 0.0316 MPa$^{-1}$ to 0.0431 MPa$^{-1}$ when permeability ranges from 0.0348 mD to 0.4167 mD. Fig. 10 indicates that slippage factor $b$ negatively correlates with corrected permeability, which fits the equation as follows: $b = aK^b$. We conducted exponential fit and got the correlation between slippage factor $b$ and corrected permeability: $b = 0.0233k^{0.246}$. When corrected permeability is lower than 0.05 mD, slippage factor $b$ decreases quickly with the increase of permeability [25].

5. Conclusions

Contrast experiments were run by using cores with penetration fractures and no fractures under constant confining pressure. The main conclusions in this paper are as follows:

(1) Gas flow in shale matrix is in the control of gas slippage and follows the Klinkenberg model at the experimental conditions where the confining pressure is 20 MPa, the temperature was 60 °C and the pore pressure is lower than 3 MPa. Slippage factor $b$ ranges from 0.05 to 0.3.

(2) Gas flow in core with fractures presented a two-section characteristic which is composed of pseudo-matrix section and fracture section. In pseudo-matrix section where inverse pore pressure is over 2 MPa, permeability increases linearly and slowly with the incremental inverse pore pressure.

In fracture section where inverse pore pressure is less than 2 MPa, permeability increases rapidly with the incremental inverse pore pressure.
(3) Effective stress affects the opening and closing of fractures, which would have significant effects on gas flow. High effective stress causes fractures closing and enhances gas slippage, while low effective stress opens the fractures and weakens gas slippage.

(4) A correlation between slippage factor $b$ and corrected permeability was built. The correlation indicates that $b$ changes little when permeability is higher than 0.05 mD, while $b$ decreases sharply with the increase of permeability when permeability is lower than 0.05 mD.

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References