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Experimental Evaluation of Working Fluid Damage to Gas Transport in a High-Rank Coalbed Methane Reservoir in the Qinshui Basin, China

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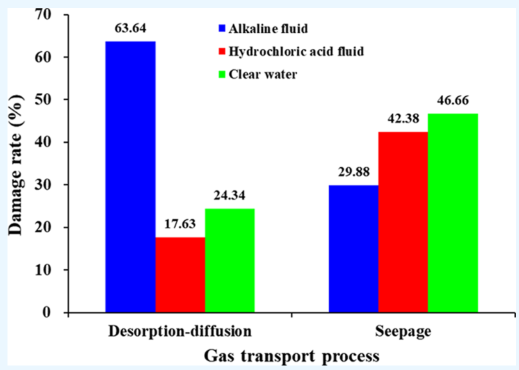
ABSTRACT: Formation damage induced by the injected working fluid runs through the whole life cycle of coalbed methane (CBM) extraction and ultimately reduces the production of CBM wells. The conventional method uses permeability as a parameter to evaluate the formation damage severity to coal by working fluids containing solids. However, less attention has been attracted to the formation damage of the pure liquid phase of the working fluid on the multiscale gas transport process of CBM. Therefore, we present a multiscale working fluid filtrate damage evaluation method considering the desorption, diffusion, and seepage and use it to evaluate high-rank coal in the Qinshui Basin of China. The results show that pure liquids with different pH values and salinities significantly damage the desorption−diffusion and seepage ability of CBM. The damage rates of alkaline fluid, hydrochloric acid fluid, and clear water on the methane desorption capacity of coal are 63.64, 17.63, and 24.34%, respectively, while those on the permeability of coal are 29.88, 42.38, and 46.66%, respectively. The formation damage severity in the seepage process is higher than that in the desorption−diffusion process, which proves the necessity of multiscale working fluid damage evaluation on CBM. Effective channel reduction and resistance increase in gas transport are the mechanisms of working fluid filtrate-induced formation damage, which are caused by water blocking, sensitive mineral swelling and clogging, and strengthened stress sensitivity. In addition to controlling the solid damage of the working fluid, reducing the invasion of the working fluid filtrate and maintaining its compatibility with the coal and formation fluids are even more important to protect the coal reservoir.



1. INTRODUCTION

CBM is an unconventional natural gas with a total global reserve of over 260×10^{12} m^3.[1] Because of its huge resource potential and cleanability, CBM is considered an important source for meeting China's growing energy demand. Unlike Australian and American countries that extract CBM from low-rank CBM reservoirs, high-rank coal is the major contributor to CBM production in China, accounting for more than 90% of total production.[2−4] However, after decades of development as China's first commercially exploited CBM field, production in the southern Qinshui Basin has yet to meet expectations. Therefore, increasing production is the current priority for CBM extraction in China.

In the life cycle of a CBM well, large amounts of extraneous working fluids (drilling, completion, acidifying, fracturing fluids, etc.) are successively injected into the coal formation. During the working fluid injection, the extraneous fluid first invades and fills the fractures under overbalanced pressure. The invading fluids then spontaneously imbibe from the fracture surfaces into the matrix pores under capillary pressure.[5] These fluids can hardly flow back completely. The liquid phase trapped is mostly filled in the pores, throats, and fractures of the CBM reservoir.[6] Original in situ environmental conditions, including the fluid, pressure, and temperature, change after injecting the working fluid. The extraneous working fluids are usually not perfectly compatible with the formation fluid in coal.[7] Incompatible interactions occur between the extraneous working fluid and coal or formation water. These factors reduce the pore structure available for gas transport by means of pore clogging, clay mineral swelling, water blocking, etc.[8,9] As a result, formation damage occurs in the CBM reservoir. History matching of a horizontal CBM well in Australia shows a drastic decline in the bottomhole pressure when pumping began and a high skin factor, which proved

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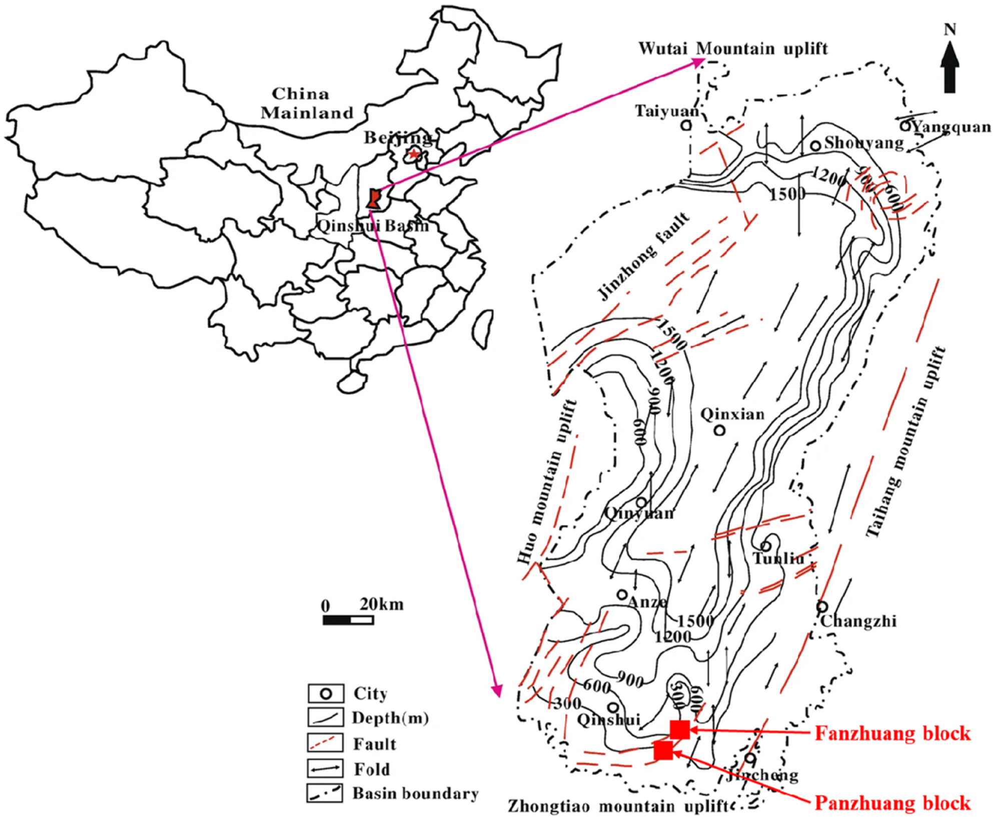

Figure 1. Geological map of the Qinshui Basin and location of the Fanzhuang and Panzhuang areas.[19] Adapted with permission from [He, H.; Tian, C.; Jin, G.; Han, K. Evaluating the CO_2 geological storage suitability of coal-bearing sedimentary basins in China. Environ. Monit. Assess. 2020, 192, 1−13]. Copyright [2020] [Springer].

high formation damage after drilling.[10] A vertical well case in the southern Qinshui Basin also shows a gas production reduction of 78% associated with fracturing fluid leakage from an adjacent well.[11]

Unlike conventional natural gas, which is mainly free gas, CBM exists mainly as adsorbed gas.[12,13] The production of CBM is mostly subject to the gas multiscale transport process: (1) methane desorption from the coal matrix surface, (2) diffusion from the matrix to mesopores, macropores, and fractures under a concentration gradient, and (3) seepage in pores and fractures under a pressure gradient.[14] The methane transport in coal is controlled conjointly by the desorption, diffusion, and seepage capacity. Therefore, formation damage induced by the extraneous working fluid on any part of the multiscale methane transport process may significantly reduce the ultimate production of CBM. However, most formation damage evaluations of coal only focus on seepage. The permeability reduction in the flow test before and after working fluid treatment is used to evaluate the damage severity.[15,16] In fact, working fluids can invade the pores, throats, cleats, and fractures of coal through lost circulation, filtration, and spontaneous imbibition. They change the methane transport channels and damage the corresponding methane transport process. Damage to permeability is not sufficiently representative of damage to the entire methane transport process. In recent years, the effect of working fluids on coal adsorption, desorption, and diffusion has gradually attracted the interest of researchers,[15,17,18] but damage evaluation considering the multiscale methane transport process in coal is still rarely reported.[8,9] These results all proved significant formation damage after various drilling fluids, fracturing fluids, and water. Despite the shallow pore clogging caused by the working fluid containing solids, formation damage caused by filtrate that deeply invades and difficult flowbacks needs more attention.

In this paper, a formation damage evaluation method considering multiscale methane transport was proposed to investigate the working fluid filtrate-induced formation damage of the high-rank CBM reservoir of the Qinshui Basin in China. The formation damage severity of the working fluid filtrate on each link of the methane transport process was quantitatively evaluated. The effects of the working fluid filtrate on the multiscale gas transport of CBM were investigated. The mechanism of working fluid filtrate-induced damage on multiscale gas transport of CBM was also discussed.

2. GEOLOGICAL CHARACTERISTICS OF THE 3# HIGH-RANK COAL RESERVOIR IN THE QINSHUI BASIN

The Qinshui Basin is located in southeastern Shanxi Province. It is the largest CBM field in China (Figure 1). The 3# coal in the southern Qinshui Basin is a major coal formation. It is characterized by a tight matrix, low pore pressure, low gas saturation, strong heterogeneity, and rich in sensitive minerals. The pores in the matrix are mainly micropores, transitional pores, and mesopores, with rare macropores. The average pore throat radius is 2−10 μm. Only approximately 10% of pores are larger than 1000 μm in diameter. The effective porosity ranges from 1.15 to 7.69% and is mainly distributed within 5%. The contribution of pores to the permeability is very limited. Low reservoir pressure prevails in CBM reservoirs. Their pressure coefficients are mostly between 0.76 and 0.98. The Fanzhuang and Panzhuang areas have low average gas saturations of 71.8 and 69.7%, respectively. However, they have relatively high gas saturations in the Qinshui Basin. Because of multiple phases of tectonic movements in the Qinshui Basin, the CBM reservoirs have a high degree of thermal evolution, resulting in strong heterogeneity. The matrix is tight while cleat and fracture systems are developed, and some CBM reservoirs even have fracture zones. The content of clay minerals ranges from 4 to 9%, with an average content of 6.71%. The relative contents of illite, kaolinite, mixed-layer illite and smectite (I/S), and chlorite are 44.86, 26, 17, and 12.14%, respectively.[15] Quartz, calcite, anorthose, siderite, hematite, pyrite, iron, ankerite, and tobelite also exist with small contents. The above characteristics make coal vulnerable to formation damage.

3. METHODOLOGY

CBM production is a multiscale methane transport process that includes desorption, diffusion, and seepage. A reasonable formation damage evaluation of working fluids should consider all these processes. In this paper, the desorption ratio was used to evaluate the formation damage severity of desorption and diffusion at the nanometer scale, and the permeability recovery rate was used for the formation damage severity of seepage at the micrometer–millimeter scale.

3.1. Experimental Sample. The experimental samples used in this study were from the 3# coal in Qinshui Basin, Shanxi Province, China. Cylindrical cores were drilled from a natural coal block for permeability measurement. The cores have a diameter of 2.5 cm and a length of 4–5 cm. Shredded coal samples were ground into particles for methane desorption measurement. Approximately 100 g of 60–80 mesh coal particles were required for desorption measurement. The working fluid filtrate used for the experiments was simulated formation water, brine, hydrochloric acid (12 wt % HCl), and alkaline fluid. The alkaline fluid was made up of a sodium hydroxide and potassium chloride solution with the same salinity as the simulated formation water. By changing the concentration of sodium hydroxide, the pH value of the alkaline fluid was adjusted to 7.4, 8.5, 10, 11.5, and 13.

3.2. Multiscale Working Fluid Damage Evaluation Method. *3.2.1. Measurement of Desorption Ratio (J_x).* The desorption ratio is the ratio of the methane desorption volume of the working fluid filtrate-treated coal sample to that of the simulated formation water-treated coal sample. Therefore, the volumetric method was used for desorption measurement.[20,21] This method proved to be a reasonable method for measuring the gas adsorption and desorption capacity of a rock.[8,15] A lower desorption ratio indicates more serious damage to the methane desorption ability by the working fluid. The desorption ratio is a comprehensive characterization parameter of the desorption gas volume and desorption rate of the coal matrix. The specific steps of the experiment are as follows: (1) approximately 60–80 mesh sieved coal particles were selected to fill the coal sample cylinder. (2) The coal particles were dried at 40 °C and then vacuum degassed. (3) The coal particles were soaked in a working fluid filtrate for 1 h, and the working fluid filtrate was drained. (4) The adsorption module of the adsorption desorption instrument (Figure 2) is turned on after the coal sample is subjected to vacuum degassing until adsorption equilibrium is reached. (5) The methane desorption volume Q_{ei} was recorded at time t_{ei} until the increment of methane desorption volume at adjacent measuring times was less than 2%. (6) Change the working fluid filtrate and repeat steps (1–5). (7) Calculate the methane desorption ratio J_x

Figure 2. Schematic of the methane adsorption and desorption measurement apparatus for coal.

$$J_x = \frac{Q_{e,max}}{Q_w} \times 100\% \tag{1}$$

where $Q_{e,max}$ is the equilibrium methane desorption of the coal particles soaked with working fluid filtrate, cm^3/g, and Q_w is the equilibrium methane desorption of coal particles soaked with deionized water, cm^3/g.

3.2.2. Measurement of Permeability Recovery Rate (R_p). The permeability recovery rate is the percentage of the permeability of the damaged coal core to its initial permeability. The smaller the rate is, the worse the severity of damage by the working fluid on seepage. The permeability of coal was measured by the apparatus and method recommended in Chinese oil industry standard SY/T 5358-2010 (formation damage evaluation by flow test).[22] A high-accuracy flowmeter with a measurement range of 1.67×10^{-11}–8.33×10^{-9} m^3/s was used to measure flow for calculating permeability. The experimental system was held at 25 °C, and the confining pressure was applied and maintained at 3–5 MPa. The initial permeability (K_0) of the saturated core is determined using simulated formation water at an injection pressure maintained at approximately 1.5 MPa. After the initial permeability measurement, the working fluid filtrate was reversely displaced into the core at the same injection pressure. The displacement ceased when the working fluid filtrate with two times the pore volume of the core was displaced into the core. Then, the working fluid filtrate was kept in the core to interact with the core for 1 h under an unchanged confining pressure and temperature. The permeability (K_d) of the core was determined again using simulated formation water at the same injection pressure as that of the initial permeability. The permeability recovery rate can also be calculated by eq 2

$$R_p = \frac{K_d}{K_0} \times 100\% \tag{2}$$

where R_p is the permeability recovery rate, %; K_d is the permeability of the coal core determined in the flow back process, mD; and K_0 is the initial permeability of the coal core determined by the simulated formation, mD.

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4. RESULTS AND DISCUSSION

4.1. Working Fluid Damage to the Desorption, Diffusion, and Seepage of CBM. *4.1.1. Working Fluids Damage Methane Desorption.* Figure 3 shows the methane desorption volume of the raw, simulated formation water, alkaline fluid (pH = 13), hydrochloric acid fluid, and clear water-treated coal samples. Clear water can be considered a special brine with a salinity of zero. The raw and simulated formation water-treated coal samples have methane desorption volumes of 21.28 and 19.06 cm^3/g in 120 min, respectively. This indicates that the water content impairs the methane desorption ability of coal. The alkaline fluid, hydrochloric acid fluid, and clear water-treated coal samples desorb 6.93, 15.70, and 14.42 cm^3/g methane in 120 min, respectively. This result is consistent with the methane adsorption volume of the working fluid filtrate-treated coal sample. The raw, simulated formation water, alkaline fluid, hydrochloric acid fluid, and clear water-treated coal samples adsorb 27.29, 23.30, 7.72, 18.7, and 17.5 cm^3/g methane, respectively.

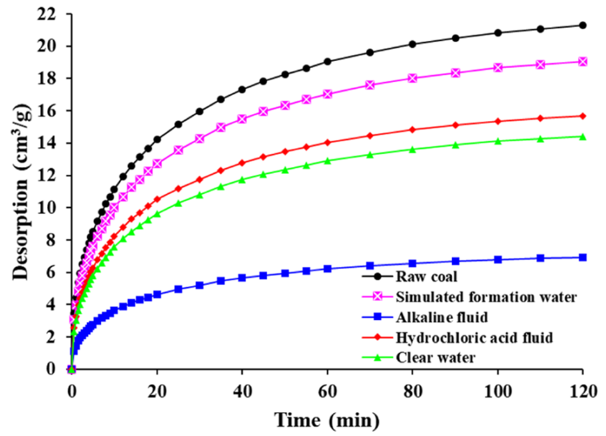

Figure 3. Time-dependent curve of methane desorption of the raw, simulated formation water, alkaline fluid (pH = 13), hydrochloric acid fluid, and brine-treated coal samples.

The desorption ratios of alkaline fluid, hydrochloric acid fluid, and a brine-treated coal sample are 36.36, 82.37, and 75.66%, respectively (Figure 4). Alkaline fluid seriously damages the methane desorption ability of coal, while that of hydrochloric acid fluid is the weakest. Brine damage to the methane desorption ability of coal is medium compared to alkaline fluid and hydrochloric acid fluid. They all show more serious damage to the desorption of coal than the simulated formation water.

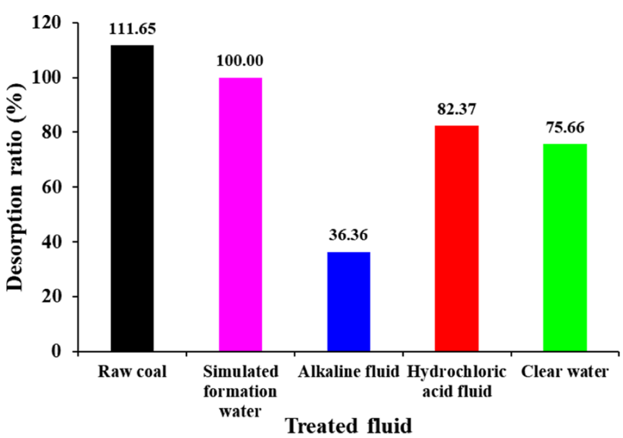

Figure 4. Methane desorption ratio of the raw, simulated formation water, alkaline fluid (pH = 13), hydrochloric acid fluid, and brine-treated coal samples.

4.1.2. Working Fluids Damage Permeability. Figure 5 shows the permeability recovery rate of coal cores treated by alkaline fluids. The permeability recovery rate of the coal core decreases as the pH value of the alkaline fluid increases. This indicates that the higher the pH of the alkaline fluid is, the more severe the permeability damage. However, the overall alkaline fluid-induced permeability damage is relatively weak. The permeability recovered by 70.12% when the pH value reached 13 (strong alkaline fluid).

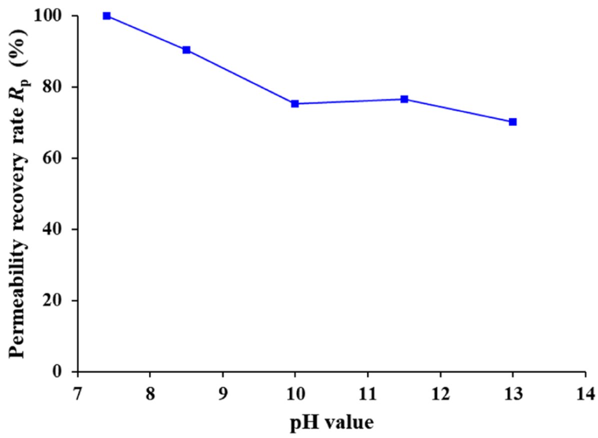

Figure 5. Permeability recovery rate (R_p) of coal cores damaged by alkaline fluid.

Figure 6 shows that the permeability recovery rate of the coal core declines with the continuous injection of hydrochloric acid fluid. The permeability recovery rate decreased slowly after 10 pore volumes of hydrochloric acid fluid were injected. Finally, a permeability recovery rate of 57.62% was obtained after 15 times pore volumes of hydrochloric acid fluid were injected.

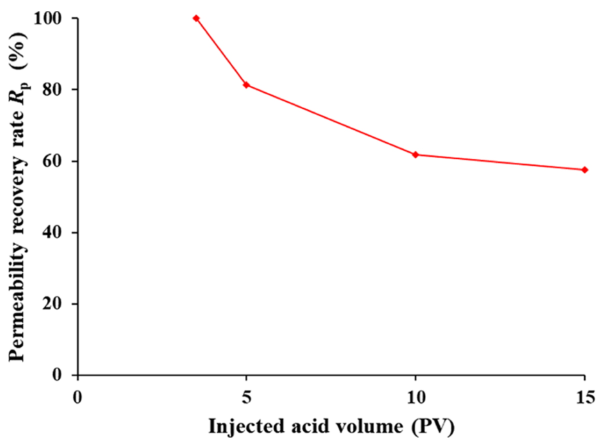

Figure 6. Permeability recovery rate (R_p) of a coal core damaged by hydrochloric acid fluid.

Figure 7 shows the permeability recovery rate of coal cores treated with brine. The permeability recovery rate decreases with decreasing brine salinity. The permeability recovery rate

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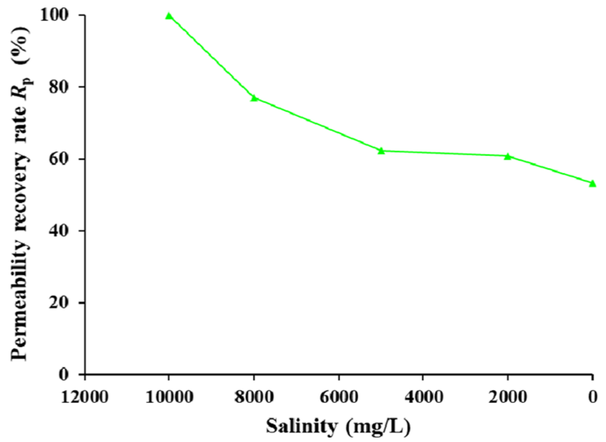

Figure 7. Permeability recovery rate (R_p) of coal cores damaged by brine.

decreases to 53.34% as the salinity decreases to 0. That is, clear water causes the greatest damage to the coal core permeability. When salinity declines to 5000 mg/L, the severity of damage to coal core permeability by brine is stronger than that of alkaline fluid and hydrochloric acid fluid. Obviously, clear water damages coal core permeability more seriously than strong alkaline fluid and strong acid fluid.

4.2. Working Fluids Damage Multiscale Gas Transport in CBM.

A damage rate is used to evaluate the severity of working fluid filtrate-induced damage on desorption and permeability (Figure 8). It is defined as the ratio of the loss desorption volume or permeability of the fluid-treated coal sample to that of the simulated formation water-treated coal sample. The damage rates of the desorption of alkaline fluid-, hydrochloric acid fluid-, and clear water-treated coal samples are 63.64, 17.63, and 24.34%, respectively. The order of the damage severity of the desorption ability is alkaline fluid, clear water, and hydrochloric acid fluid. The damage rates of the permeability of the alkaline fluid-, hydrochloric acid fluid-, and clear water-treated coal samples are 29.88, 42.38, and 46.66%, respectively. The order of the damage severity of permeability is clear water, hydrochloric acid fluid, and alkaline fluid.

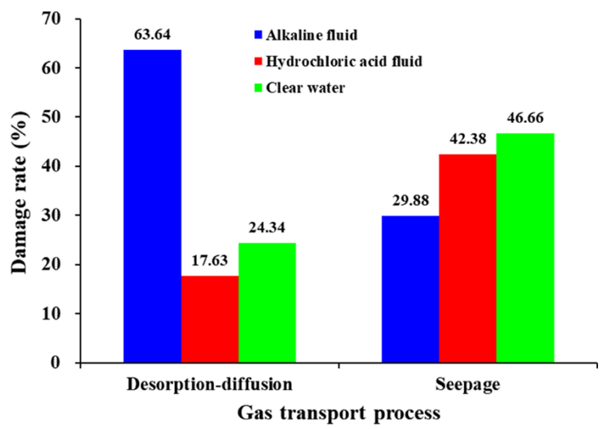

Figure 8. Formation damage rate of working fluid filtrate-treated coal samples.

There is a significant difference between the formation damage rates of the permeability and desorption ratios. For alkaline fluid-treated coal samples, the damage severity of desorption is much higher than that of permeability. For hydrochloric acid fluid- and clear water-treated coal samples, the damage severity of desorption is lower than that of permeability. These differences indicate that the same working fluid filtrate has different effects on different gas transport processes. In general, the seepage process is more sensitive to working fluid filtrates than the desorption−diffusion process.

4.3. Mechanisms of Working Fluid Damage to Multiscale Gas Transport of CBM.

4.3.1. Water Blocking. The simulated formation water-treated samples show a higher formation damage rate than the dry samples, which is mainly attributed to water blocking. The water blocking of coal is mainly influenced by the pore size and original water saturation of coal and the contact angle and surface tension of fluids.[6] The pore throat radius of the 3# coal mainly lies between 2 and 10 μm. The 3# coal has a high surface tension and a low contact angle (61.4−82.1°) of the water.[23] The above-mentioned factors result in a high capillary pressure, which drives the working fluid filtrate to invade the coal pore and throat system through spontaneous imbibition. The filtrate can invade much deeper under an overbalanced pressure condition, such as drilling and hydraulic fracturing operations. The filtrate can invade into nanometer pores under an overbalanced pressure of 4.5 MPa.[18] Then, gas transport must overcome the capillary resistance caused by the Jamin effect and the capillary effect.[7,15,24] They essentially clog the continuous liquid phase and discontinuous gas phase in tiny pore throats. The effective gas transport channels are narrowed, and the gas transport resistance is increased.

The invaded working fluid filtrates can hardly completely flow back. A certain amount of liquid is eventually trapped in the pores and results in a higher irreducible water saturation.[23] In addition, as trapped water occupies the pore, throat, and microfracture space, the gas transport channels become narrower. This leads to a higher gas residual percentage during desorption and a lower relative permeability of gas after working fluid filtrates invade.[18] The initial desorption rate and the decay rate both decreased as the water content increased.[25] The trapped water absorbs on the surface of pores, throats, and microfractures in the form of a water film. OH^- ions adsorbed on the coal surface increase with an increasing pH of alkaline fluid. The increasing OH^- ions strengthen the hydrophilicity of the coal surface and consequently thicken the water film. The effective gas transport channels narrow, and the capillary resistance increases. The desorption process mainly occurs in the micropores, where the places invading filtrates remain. This is consistent with the experimental results of the significantly higher damage rate of alkaline fluid than that of acid and clear water during desorption and diffusion.

4.3.2. Sensitive Mineral Swelling and Clogging. The 3# coal has an average total mineral content of 14.86% and a clay mineral content of 6.71%.[15] The clay minerals are present as pore lining, pore filling, and pore bridging deposits in the pores and throats (Figure 9). They can be directly exposed to the working fluid filtrates. On the one hand, the clay minerals swell to narrow the pores, throats, cleats, and fractures of coal. On the other hand, the clay minerals disperse and migrate to clog the pores, throats, cleats, and fractures of coal. These changes in pore structure are confirmed by the following experimental results: (1) linear swelling rates of the 3# coal between 0.45 and 3.78% after working fluid treatment.[15] (2) An increase in the specific surface area and pore volume and a reduction in the average pore radius after working fluid treatment in low-temperature nitrogen adsorption.[8,9] As a result, the effective

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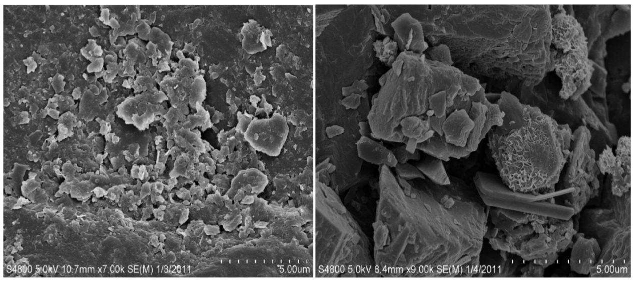

Figure 9. Scanning electron microscope images of kaolinite (left, ×7000) and chlorite (right, ×9000) in the 3# coal.

gas transport channels are narrowed and reduced, and the gas transport resistance is increased. Therefore, all coal samples show a reduction in the permeability after working fluid filtrate treatment. The reduced permeability slows the pressure reduction rate in coal, which decreases the desorption and diffusion rates of coal.

Table 1 gives the sensitive clay mineral contents of the 3# coal. Kaolinite is velocity-, alkali-, and salinity-sensitive, with a relative content of 26%. It disperses stably at pH > 8 or low salinity. The mixed-layered illite/smectite (I/S) has a highly illitic nature. They are alkali- and salinity-sensitive, with a total relative content of 62%. Illite is already highly dispersed, and it easily detaches from coal by fluid movement at a pH above the zero net charge of illite. The migration of fine illite is more likely to occur than that of more coarse-grained kaolinite. Chlorite is not generally reactive to aqueous fluids, but it is susceptible to acid dissolution and decomposes to yield siliceous or ferruginous gelatinous masses.[26] The hydrochloric acid fluid also interacts with minerals to produce chemical precipitates and dissolves other minerals (calcite, etc.) to release fines.

Table 1. Sensitive Clay Minerals of the 3# Coal Reservoir of the Qinshui Basin in China

clay mineral type	relative content of minerals (%)	sensitivity	sensitivity mechanisms
illite	44.86	water/salinity and alkali sensitive	swelling, dispersion migration
kaolinite	26	velocity, alkali, and salinity sensitive	dispersion migration
mixed-layered illite/smectite (I/S)	17	water/salinity and alkali sensitive	swelling, dispersion migration
chlorite	12.14	acid sensitive	precipitate, dissolution migration

The total sensitive clay mineral content in the 3# coal is low. This is consistent with the relatively weak formation damage rate in desorption and permeability observed in experiments. The swelling and dispersion of clay minerals are controlled by the pH and salinity of the working fluid filtrate. A higher pH and lower salinity cause more swelling and dispersion of clay minerals.[26] Among all the experimental working fluid filtrates, the pH and salinity of the alkaline fluid are the highest. The opposite effects of high pH and high salinity neutralize the damage to clay minerals. This makes the permeability reduction of alkaline fluid significantly lower than that of clean water with zero salinity. The permeability reduction of hydrochloric acid fluid is a combined effect of the lowest pH, the moderate salinity, and the precipitation and fines of acid-sensitive minerals. Thus, it is slightly lighter than the permeability reduction of clean water.

4.3.3. Working Fluids Strengthen Stress Sensitivity. The tiny pores and throats contribute to limited permeability, while the developed cleats and microfractures dominate the permeability of CBM (Figure 10). Therefore, the cleats and microfractures are the first spaces where working fluid filtrates invade. Thereafter, they invade the matrix through the surfaces of cleats and microfractures. The invading working fluid filtrate influences the mechanical properties of coal. A significant reduction in the compressive strength and Young's modulus of coal was observed after working fluid filtrate treatment.[8] The working fluid filtrate-treated coal becomes weaker and more ductile than raw coal. Due to the high content of organic matter in high-rank coal, the permeability of microfractures was reported to be quite sensitive to effective stress variation.[27−29] The invading working fluid filtrate strengthens the stress sensitivity of coal. Therefore, the cleats and microfractures are easier to close under the same effective stress loading. This ultimately reduces the permeability of coal and the productivity of CBM. The cleats and fractures in high-rank coal reservoirs are more developed than those in low-rank coal reservoirs. The working fluid filtrate strengthened stress sensitivity, which is more pronounced on multiscale gas transport of high-rank CBM. The permeability of coal is mainly contributed by macropores, cleats, and fractures. The strengthened stress sensitivity may be one reason working fluid filtrate damage on the seepage process is more serious than that of the desorption−diffusion process.

Figure 10. Cleats and fractures of a coal cast thin section under 25× magnification.[15] Reprinted with permission from [Huang, W. A.; Lei, M.; Qiu, Z. S.; Leong, Y. K.; Zhong, H. Y.; Zhang, S. F. Damage mechanism and protection measures of a coalbed methane reservoir in the Zhengzhuang block. J. Nat. Gas. Sci. Eng. 2015, 26, 683−694]. Copyright [2015] [Elsevier].

5. CONCLUSIONS

(1) The 3# high-rank coal reservoirs in the southern Qinshui Basin are characterized by a tight matrix, developed cleats and fractures, low pore pressure, low gas saturation, strong heterogeneity, and rich sensitive minerals. The formation damage potential is considered after the working fluid filtrates have invaded.

(2) Pure liquids with different pH values and salinities significantly damage the desorption−diffusion and seepage ability of CBM. The damage rates of alkaline fluid, hydrochloric acid fluid, and clear water on the desorption of coal are 63.64, 17.63, and 24.34%, respectively, while those of the permeability of coal are

29.88, 42.38, and 46.66%, respectively. The significant difference between the damage rate of the desorption ratio and permeability demonstrates the necessity of multiscale working fluid damage evaluation on CBM.

(3) The working fluid filtrate damages the desorption, diffusion, and seepage of CBM in the mechanisms of water blocking, sensitive mineral swelling and clogging, and strengthened stress sensitivity. They reduce the amount and size of effective gas transport channels and increase the resistance of gas transport. This eventually leads to a non-negligible decline in the gas transport ability of CBM.

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Notes

The authors declare no competing financial interest.

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