

Experimental Study on Damage Mechanism of Fracturing Fluid in CBM Reservoir of Eastern Yunnan and Western Guizhou

LIU Naizhen¹, GAO Qing-chun¹

Changcheng Drilling Engineering Limited Company, China Petroleum Groups

E-mail: qc_gwdc@163.com

Abstract: The fracturing of CBM reservoirs can bring some damage because of the loss, intrusion reservoirs and difficult flowback of fracturing fluid. In this paper, damage evaluation experiment of different fracturing fluid on different structure of coal were conducted adopting active water and polymer fracturing fluid which were commonly used in the field. The microstructure of coal sample treated with different fracturing fluid was investigated by scanning electron microscopy (SEM). The microscopic mechanism of fracturing fluid damage to coal fractures and matrix pores was discussed. The results of these tests have shown that the damage of the active water fracturing fluid to the pore structure of coal sample is larger than that of polymer fracturing fluid, but the damage is generally less than 40%. The damage of polymer to fracture is higher than that of matrix. The effect of fracture fluid inorganic salt crystallization, polymer adsorption and water wet expansion can make the matrix pore and fracture decrease, thus damaging the seepage structure and reducing the coal permeability. But the gap between the original structure of microcracks and matrix pores is large, the impact of pollution on them is also quite different. The conclusion of this study is helpful to evaluate the effect of fracturing fluid on coalbed methane mining comprehensively and has a positive significance for the optimization of fracturing fluid formulation.

1. Introduction

Compared with conventional natural gas reservoirs, CBM reservoirs are a typical dual porosity system that includes micropores and mesopores in the coal matrix and fractures surrounded by matrix blocks. The typical characteristics of low permeability, low permeability, gas adsorption, desorption-diffusion and percolation in CBM reservoirs make the natural productivity of CBM wells low. Therefore, CBM wells need to undergo fracturing to achieve good development results, and their production capacity is closely related to fracturing cracks^[1]. During hydraulic fracturing, the injected fracturing fluid first fills and fractures the fractures to form new flow channels, and these liquids then spontaneously enter the matrix pores and fractures. In addition, field tests indicate that a significant portion of the injected fracturing fluid cannot be effectively discharged (approximately 20% to 30%), but remains in the pore-fractured system of coal reservoirs, and these will have an adverse effect^[2] on CBM reservoirs.

The influence of fracturing fluids on the seepage characteristics of coalbed methane reservoirs has been extensively studied. The study of fracturing fluid damage to reservoirs was carried out early in foreign countries[3] [4]. In China, In 2014, Han Jinxuan[9] discovered that the special engineering geological characteristics of Beishen deep coal seam in Shizhuang were more susceptible to fracturing fluid contamination during fracturing. Compared with shallow coal seams, the deep coal seam pollution mechanism is mainly due to adsorption damage and particle clogging. In 2014, KangYili[10] et al. used different fracturing fluids to carry out damage evaluation experiments on tight coal rocks in



the Ningwu Basin. It was believed that the main mechanism of damaging coal reservoirs was the swelling of coal and rock matrix caused by hydrophilic fracturing fluid adsorption. Weak acid clean fracturing fluids have been found to cleave coal seam natural fracture fillers to increase reservoir permeability. In 2015, Gao[6] et al. used 15# coal rock of the Taiyuan Formation in the Qinshui Basin as a target. From the microscopic surface and structure, it was analyzed that the physical and chemical effects of the low-loss active fracturing fluid on the CBM reservoir damaged the CBM reservoir percolation structure and leads to a decrease in macroscopic permeability. In 2016, Kang[7] studied the damage mechanism of water-based fracturing fluids in the multi-scale migration processes such as adsorption, desorption, diffusion, and seepage of anthracite in the Qinshui Basin.

In this paper, the coal samples from the west of eastern Yunnan area were taken as the research object, and the non-steady-state permeability measurement method was used to measure the coal rock permeability before and after the fracturing fluid contamination. The matrix and fracture permeability damage experiments were carried out before and after different types of fracturing fluids acting on coal and rock samples. Scanning electron microscopy (SEM) was used to analyze the changes of micro-structures such as pores and fractures in the coal rock before and after fracturing fluid dyeing, and to reveal the damage mechanism of fracturing fluid to different structures of the reservoir from a microscopic point of view. This research will help us to understand the damage mechanism of the fracturing fluid reservoir and evaluate the impact of the fracturing fluid on the coalbed methane production in the west of eastern Yunnan area, providing some basis for reservoir protection, fracturing design, work system determination.

2. Fracture fluid damages coal rock experiment

2.1. Experimental sample

The eastern Yunnan area is a Late Permian Upper Yangtze basin. The coal-bearing strata are the Changxing Formation and the Longtan Formation. The main recoverable coal seams are located in the lower part of the Changxing Formation, the lower middle section of the Longtan Formation, and the central part of the Longtan Formation. Its coalbed methane resources amount to $49117.9 \times 10^8 \text{m}^3$, accounting for 13% of the country's coalbed methane resources. The fractures in coalbed methane reservoirs developed, and the microfissure density of coal samples was $3\text{--}6/\text{cm}^2$. The porosity and permeability were generally low, and the heterogeneity of the reservoirs was obvious[12][13]. The coal rock and standard coal core in the eastern Yunnan area of eastern Yunnan and western Guizhou are shown in Figure 1.



(a)Original coal rock sample map (b) Experimental Coal Heart Made by Wire Cutting
Fig. 1 Samples of coal-rock in eastern Yunnan and western Guizhou

In order to study the fracturing fluid contamination of different types of coal rocks, the experiment choose the standard coal cores of 3# which have no obvious cracks in the Tianjing Mining Area and the 15# in the Songhe Mining Area with micro-cracks in the eastern Yunnan and western Guizhou area. They are used to represent fissures and matrix porosity coal rocks, respectively. The microscopic components of coal and rock are mainly semi-dark coal and semi-bright coal, followed by gloomy coal, and bright coal is rare. The microscopic type is mainly composed of a vitrinite group and an inert

group. The vitrinite emissivity value of coal seams in Songhe coalfield is generally above 1.5%, which is medium-rank coal and high-rank coal [14]. Due to the soft and brittle nature of coal, conventional coring is difficult. In this experiment, a standard cylindrical core (D=25mm, L=50mm) was drilled using a wire-cutting method, and the flatness of the end face was not more than 0.2mm. It is more representative and persuasive to study the damage of fracturing fluid to the pore structure of the original coal and rock. The basic physical parameters of the raw coal samples are shown in Table 1.

Table 1: Basic physical parameters of coal sample

type	Number	Location	Deep / (m)	Density / (g/cc)	Diameter / (mm)	Long / (mm)	Porosity/ (%)	permeability / (mD)
matrix	SH--1	Song He 15#	320	1.33	25	50.1	0.9305	0.0054
	SH--2			1.35	25.4	50.6	0.8712	0.0043
	TJ--1			1.38	24.9	49.8	1.6233	0.4427
fracture	TJ--2	Hong Guo3#	400	1.42	25	50.1	1.8795	0.3855

According to the type of fracturing fluid used in the gas crack production site of the coal seam in the eastern Yunnan and western Guizhou area. The active water and polymer with higher proportions were chosen as the fracturing fluid in this experiment to treat the coal samples. In particular, the active water fracturing fluid has been widely applied to the hydraulic fracturing of CBM wells. Two fracturing fluid formulations are as follows (mass fraction):

(1) Formula for active water fracturing fluid: 2% potassium chloride + 0.1% caustic agent + 0.05% biocide

(2) Polymer fracturing fluid formula: 0.23% silica gel + 100,000 capsules + gel breaker + 0.36% ammonium chloride + 1.5% acetic acid + 2% sodium borate

2.2. Experimental procedure

Using the COREVAL-700 non-steady-state clad pressure penetrator of VINCI company in France, nitrogen gas was used as the experimental fluid at room temperature to measure the gas permeability of the coal rock before and after the fracturing fluid damage. The experimental flow chart is shown in Figure 2. The equipment is based on the pressure decay data of coal samples and one-dimensional unsteady flow theory, with a minimum permeability of 0.0001 mD. The key factors affecting the gas permeability measurement of coal and rock are stress, gas desorption, gas slippage, and fracturing fluid damage. In order to evaluate the change in gas permeability measured by fracturing fluid damage, experiments using nitrogen and high pore pressures of 2 MPa were used to reduce the effects of gas desorption and slippage. In order to avoid stress sensitivity, the net confining pressure of the entire system was kept constant (the difference between confining pressure and pore pressure) by a computer control system of 3 MPa. During saturation of the saturated gas and pressure, the confining pressure is adjusted to ensure that the effective stress is constant.

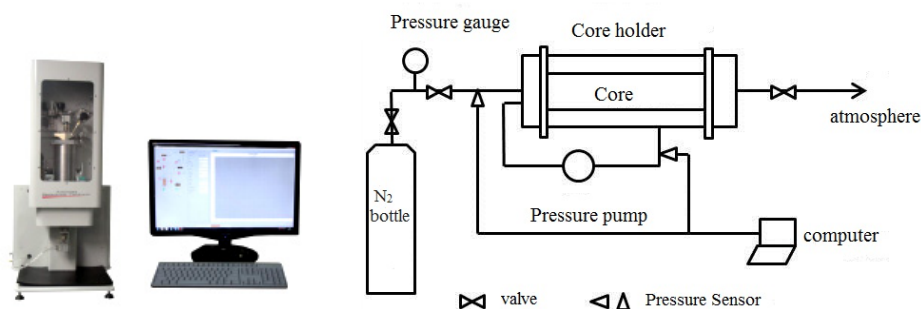


Fig. 2 COREVAL 700 unsteady overpressure permeameter and measurement flow chart

Refer to the “SYT 5358-2010 Reservoir Sensitivity Flow Test Evaluation Method” to carry out

coalmine permeability damage test before and after fracturing fluid contamination.

2.3. Experimental evaluation and analysis

The permeability *damage* rate D_k and the residual resistance factor F_{rr} are selected according to the permeability changes of different types of coal cores affected by different types of fracturing fluid at different times. The degree of permeability damage caused by the coal rock fracturing fluid pollution in the west of eastern Yunnan and western Guizhou area was judged, and the results of the permeability damage of each coal sample were shown in Table 2.

(1) Permeability damage rate reflects the change of permeabilities before and after coal reservoir fracturing fluid contamination, and characterizes the unrecoverable degree of coal reservoir permeability caused by fracturing fluid contamination, expressed as a percentage. Calculate the permeability damage rate D_k caused by fracturing fluid pollution according to equation (1), eg,

$$D_k = \frac{K_{gb} - K_{ga}}{K_{gb}} \times 100\%$$

(2) Residual resistance factor F_{rr} refers to the ratio of coal rock permeability to post-contamination permeability before fracturing fluid contamination, and it is used to express the drop in gas permeability permeability caused by fracturing fluid contamination [15].

$$F_{rr} = \frac{K_{gb}}{K_{ga}}$$

In the formula, D_k is the permeability damage rate before and after fracturing fluid contamination; F_{rr} is the residual resistance factor; K_{gb} is the coal sample permeability before fracturing fluid contamination, mD; K_{ga} is the coal sample permeability after fracturing fluid contamination ,mD.

Table 2 Experimental results of fracturing fluid damage to matrix and fissure coal

Coal core structure	Fracturing fluid type	Number	K_{gb}/mD	Pollution time /d	K_{ga}/mD	$D_k/\%$	F_{rr}
Matrix pores	Active water	SH--1	0.0054	1	0.0047	12.96	1.15
				2	0.0044	18.52	1.23
				5	0.0038	29.63	1.42
				9	0.0035	35.19	1.54
	polymer	SH--2	0.0043	1	0.0041	4.65	1.05
				2	0.0039	9.30	1.10
				5	0.0036	16.28	1.19
				9	0.0073	-69.77	0.59
Fracture system	Active water	TJ--1	0.4427	1	0.4123	6.87	1.07
				2	0.3696	16.51	1.20
				5	0.3227	27.11	1.37
				9	0.2994	32.37	1.48
	polymer	TJ--2	0.3855	1	0.2464	36.08	1.56
				2	0.1656	57.04	2.33
				5	0.1194	69.03	3.23
				9	0.0493	87.21	7.82

From Table 2, it can be seen that the initial permeability of fractured coal rocks is significantly greater than that of matrix coal samples, with a difference of two orders of magnitude. The damage rate of coal fractures and fractures with two kinds of fracturing fluids will increase with the increase of pollution time. Polymer fracturing fluids will damage the fracture permeability far more than active water. For the coal and rock matrix, with the increase of the pollution time, the degree of damage to the matrix permeability of the active water fracturing fluid will also increase, but the matrix-type coal core will increase after the ninth day of polymer fracturing fluid contamination. Different types of fracturing fluid will cause different degrees of damage to the coal core. The damage rate of active water fracturing fluid to coal and rock is generally lower than 40%, and the damage caused by polymer fracturing fluid is not uniform. After tests of different types of fracturing fluids contaminated for the same period of time, it was found that the damage rate of active coal to the permeability of coal and rock matrix is greater than the damage rate of polymer to the matrix, while the polymer fracturing fluid is just the opposite.

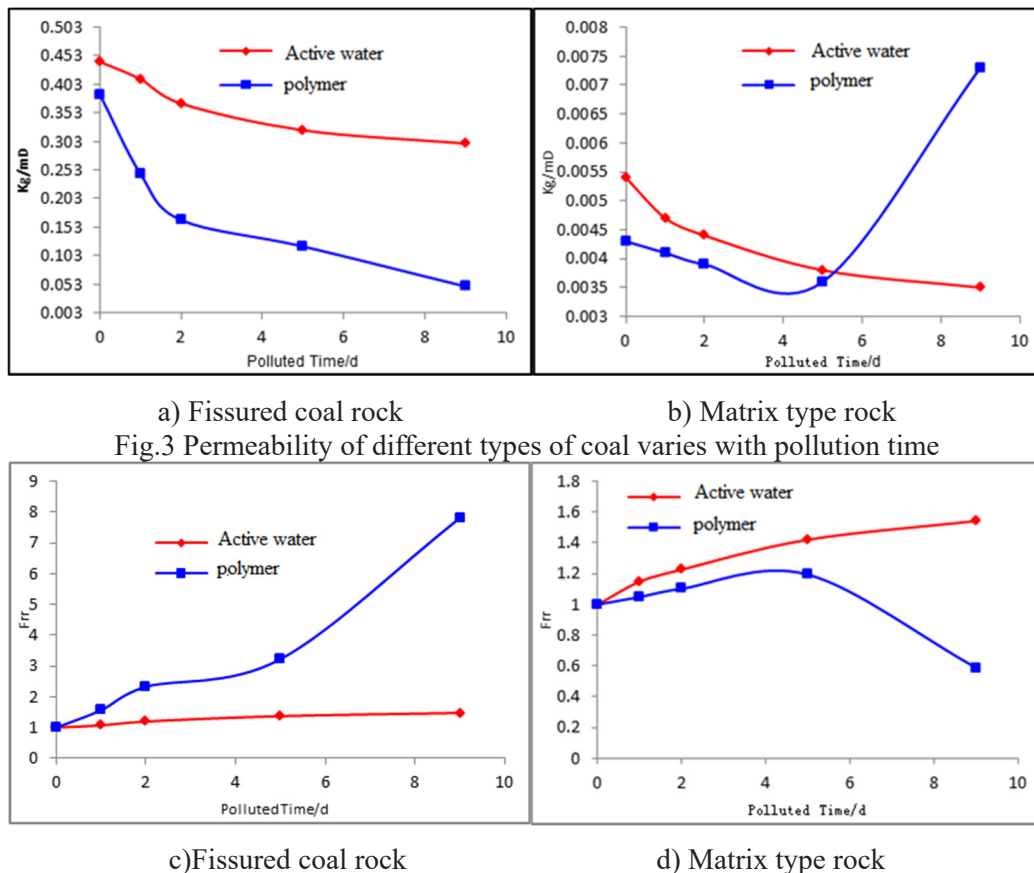


Fig.4 Residual resistance factor of different types of coal varies with pollution time

From Figures 3 and 4, it can be seen that with the increase of pollution time, the permeability of different types of coals shows a decreasing trend, and the permeability damage caused by active water fracturing fluid is small. The initial stage of polymer fracturing fluid damages the fracture permeability, and the damage rate is as high as 90%, which seriously damages the coal rock reservoir. In the later stage of pollution, the permeability of the two types of fracturing fluid to the coal and rock matrix damage decreased less. However, when the contamination occurred on the 9th day, the damage rate of the polymer fracturing fluid to the matrix was not only not increased, but the phenomenon of increased permeability occurred. The Frr values show that the polymer fracturing fluid has very serious fissures on the cracks and less on the substrate. This is because the polymer easily enters into cracks with large size and causes adsorption and blockage, which leads to a large decrease in permeability. Polymers of macromolecules are difficult to enter the pores of the matrix, and only a certain degree of contamination is formed on the surface of the coal sample. Although the active water fracturing fluid can enter the pore cracks, the matrix pores are smaller than the cracks and the permeability is very small, so the damage to the matrix is more obvious and the impact on the cracks is weaker.

In summary, after the intrusion of fracturing fluid, the gas permeability of different types of coal has been significantly damaged. It is mainly attributed to the fact that the substances in the fracturing fluid are adsorbed on the surface of the pore cracks, the adsorption of the fracturing fluid mixture narrows the flow channel, and the absolute permeability is reduced. Previous studies have confirmed that coal swell, adsorption winding, and surface modification are the basic mechanisms that lead to a reduction in the relative permeability of gas measurements [16]. In addition, hydraulic fracturing is more likely to produce pulverized coal in microfractures rather than infiltration pores. Particle transport blocks flow channels, resulting in a decrease in permeability [17]. In order to better reveal the damage mechanism of fracturing fluid to coal rock permeability and scanning electron microscopy (SEM) was applied to the coal rock before and after the pollution to observe the change of coal

microstructure and its correlation with the permeability change.

3. Fracturing fluid damage mechanism of coal reservoir

The study of microscopic pore structure is of great significance for revealing the migration characteristics of coalbed methane. The SEM images before fracturing fluid contamination indicate that a large number of mesopores, micropores and microfissures are present in the raw coal, and the characteristics of coal-rock pore-crack dual media are very obvious. The pore-fissure system not only provides potential sites for adsorption, but also provides channels for gas flow. After the fracturing fluid was contaminated, a large amount of foreign substances were observed on the surface of the pores and fracture structures of coal and rock, including adsorbed organic matter and crystallized inorganic particles. The gas-permeability of cracked and matrix pore-type samples is significantly compromised by the fracturing fluid.

3.1. Experimental device and sample preparation

Scanning electron microscopy (SEM) is an effective method for observing the surface structure of coal samples which can directly measure the pore fracture structure size. The Quanta 200F field emission scanning electron microscope was used to quantitatively characterize pore fractures and surface features of coal samples which has a resolution of 1.2 nm and a magnification ratio of between 200 and 250,000 times, enabling surface morphology and composition analysis of various materials such as mineral rocks and cermet.

On the coal rock samples near the above-mentioned coring borehole, use small scalpel hammers to gently tap small clean coals with a size of 1 to 2 cm³ (as shown on the right of Fig. 5) in order not to damage the original structure of coal rock, and can have a better response to permeability changes. Use the ear syringe to blow off the debris on the surface of the coal block as an observation sample, use the flat side of the sample as the observation surface, and clean the coal sample with alcohol: The process of sample preparation should not be in contact with liquids and the body to avoid changes in the observation surface caused by external factors.



Fig.5 Quanta 200F FESEM and coal sample preparation

3.2. Experimental results and analysis

3.2.1. Fracture fluid damage analysis of coal cracks

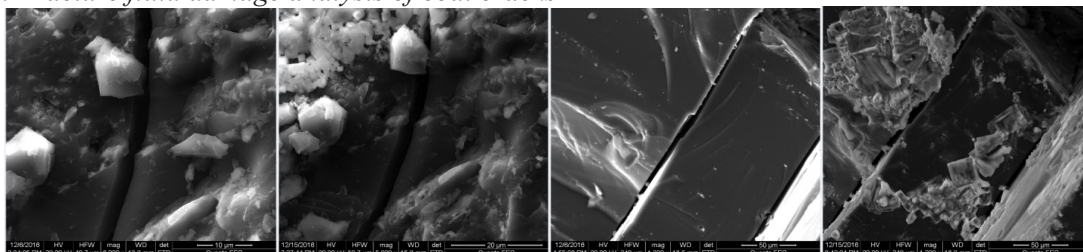
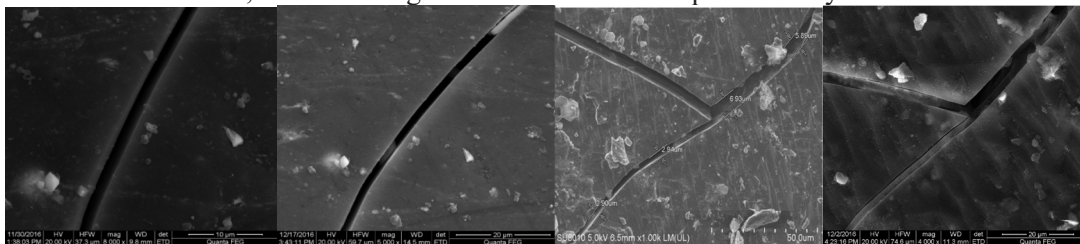


Fig. 6 Fracture structure diagram of Tianjing3# coal sample before and after the pollution by active water

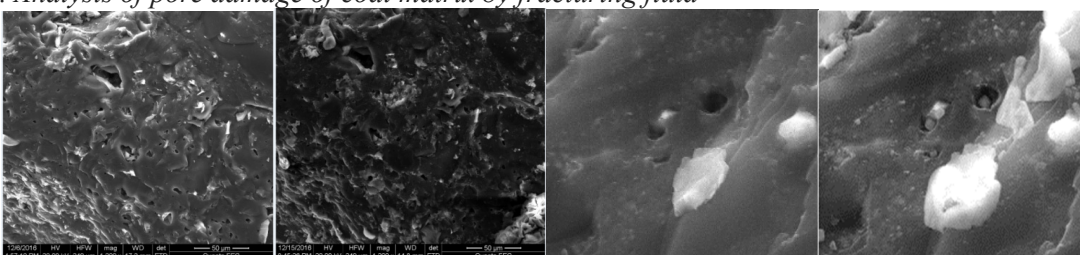
Combining scanning electron microscopy images with the properties of the fracturing fluid, it can be seen that after soaking in the activated water fracturing fluid, a small amount of crystals are attached to the wall of the fracture, the hydrophilicity of the coal is enhanced, the coal rock expands, but the gap is larger, also the attachment of crystals to the crack damage phenomenon is very light, microscopic flow channel structure basically no change, less damage to the permeability. From the polluting time point of view, there were significantly more crystalline deposits contaminated for 5 days than those polluted for 3 days. It can be speculated that as the pollution time increases, the number of fissure clogging becomes more and more, and the permeate flow rate of the coal bed gas becomes smaller, leading to a further decrease in permeability. However, blockages cause subsequent contamination to be difficult to enter the coal core, and the damage speed is getting smaller and smaller. In addition, the crystals adhere to the surface of the matrix, and the plugging of the pores of the matrix is more serious, which has a greater influence on the permeability.



Before pollution Two day after pollution Before pollution Nine day after pollution
Fig. 7 Fracture structure diagram of Tianjing3# coal sample before and after the pollution by polymer

Similar analysis shows that the polymer fracturing fluid blocks the fracture much more severely, and it can be clearly seen that the foreign matter is clogged in the fracture. As the pollution time increases, the fissure channel becomes narrower. Through measurement, it was found that the width of the fissured clogging of 3# coal sample in Yunnan Tianjing reached 7/9 of the crack width. The change in microstructure is basically consistent with the measurement of macroscopic permeability. This is because coal has strong adsorption of organic components in polymer fracturing fluids, and the polymers in the fracturing fluid can easily enter the micro-fractures of coal rock, therefore, the micro cracks in the coal seam are narrowed or even blocked, and the flow resistance of the gas is increased, resulting in a significant decrease in the permeability.

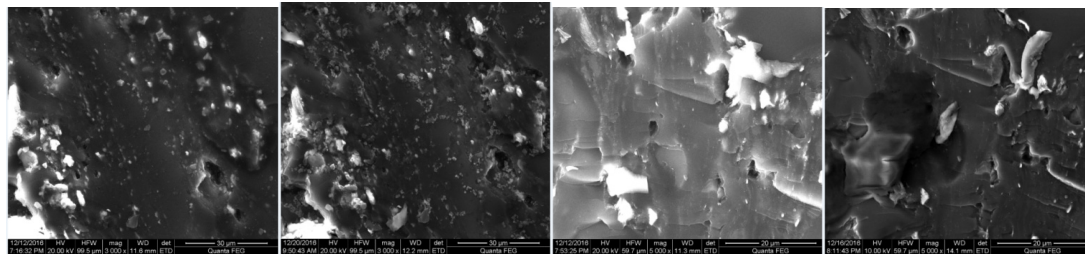
3.2.2. Analysis of pore damage of coal matrix by fracturing fluid



Before pollution One day after pollution Before pollution Five day after pollution
Fig. 8 Pore structure diagram of Songhe15# coal sample before and after the pollution by active water

After the coal rock was polluted by the active water fracturing fluid for 1 day, the basic morphology of the matrix pores did not change much, and there were few foreign crystals in the pores. After polluted for 5 days, the activated water fracturing fluid crystals were massively adsorbed in the pores, blocking the pore structure, which hinders the flow of the fluid and results in a large decrease in permeability. According to the analysis, the pore structure of coal and rock accounts for a large proportion, the adsorption force is strong, the hydrophilicity of the active coal hydrofracturing fluid increases, and the coal rock matrix will expand [18]. Therefore, the gas seepage hole is severely occupied, and at the same time, the low viscosity and high capillary pressure easily enter the pores.

The crystals are easily attached to the surface of the pores, and the pore structure of the coal is very small. A small amount of external damage will significantly change the structure of the percolation channels, thereby reducing the porosity of the matrix. On the contrary, the effect on cracks is not obvious.



Before pollution Two day after pollution Before pollution Nine day after pollution
Fig. 9 Pore structure diagram of Songhe15# coal sample before and after the pollution by polymer

After soaking in the polymer fracturing fluid, some of the pores retained more residual fracturing fluid. This is because coal is an organic rock made of soluble salts and colloidal minerals. When the polymer fracturing fluid comes into contact with coal and rock, the polymer will produce physical adsorption, chemical adsorption and other processes, so that the residue of the fracturing fluid will be adsorbed and stayed, resulting in the smaller pore channels becoming smaller and blocking the pore channel cause a decrease in permeability. However, it can be seen from the scanning electron microscope image that a large amount of polymer residue remains on the surface of the coal sample, the polymer can only block the pores of some of the larger pores, but cannot completely enter the interior of the pore, the damage is hard to penetrate in depth, and the impact on the nanometer-scale pores is limited. However, Songhe coal samples have more acid-soluble minerals. Acid components in polymer fracturing fluids erode these minerals and increase the macroscopic permeability of the seepage channels [19], but it is not very obvious in the microscopic seepage structure.

4. Conclusions

(1) The results of the pollution experiment showed that the permeability of different types of coal after the pollution showed a downward trend, the damage rate of active water fracturing fluid to the permeability of coal was generally lower than 40%, and the degree of damage was small. Polymer fracturing fluids, on the other hand, are very injurious to the fracture permeability of coal and rock, but have little damage to the pores.

(2) The hydrophilicity of the coal sample and the fracturing fluid increases, the moisture in the fracturing fluid will adsorb on the surface of the coal rock, and the coal and rock structure will expand, resulting in a smaller pore fracture structure, resulting in a slight decrease in the permeability. However, relative to the pore fracture structure, the damage to the matrix pore is greater than the damage to the fracture permeability.

(3) The crystals produced by the active water fracturing fluid damage have less damage to the cracks with larger gaps, and the pores that adhere to the pore wall can block the pores and block the pores of the nanometer pores, leading to a significant decrease in the permeability of the matrix pores.

(4) The polymer fracturing fluid will undergo physical-chemical adsorption and other processes with the coal rock, the macromolecular polymer will enter the fissures to narrow the gap, and the fracture permeability will decrease greatly. The higher viscosity makes it difficult to enter the interior of the matrix pores, only partially blocking the voids or surfaces, affecting the subsequent contamination in depth, and having less damage to the pore permeability. The assessment of pollution damage must not only consider the structure of the seepage flow and the formulation of the fracturing fluid, but also the compatibility of the coal rock composition with the fracturing fluid.

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