

Experimental study on permeability improvement of different rank coal samples by acidification

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In view of the fact that the coal seam is blocked by filling material, the permeability in the coal seam is low. Therefore, a new method was put forward to enhance the permeability of coal seam by acid injection. The permeability increase effect *via* acid injection in different-rank coal reservoirs was studied. The characteristic changes of surface structure, pore structure, mineral content and permeability of coal samples were analyzed by SEM, ASAP, XRF and permeability measurements. Experimental results showed that: After acidification, the surface crack profile of the coal sample was clearer, the number and width of the cracks increased, the porosity-fracture network connectivity was enhanced, but the dissolution effects of high-rank coal were relatively poor. The specific surface area of coal samples decreased, the pore volume increased, and the changes were mainly concentrated at the pore width of 2~10 nm. Ca, Mg and other mineral content obviously decreased. Acidification did not cause damage to the organic matter of coal. The permeability of coal samples generally increased, the lower the coal rank, the higher were the initial permeability and the carbonate mineral content, the more obvious was the permeability increment. Coal injection technology can effectively improve the pore structure of coal, enhance the diversion capacity, and increase the permeability of coal reservoir, which leads to the improvement of gas drainage rate and gas resource exploitation.

Key words: Coal bed acidification, Chemical permeability improvement, ASAP, XRF.

INTRODUCTION

The permeability of the coal seam can affect the coal bed methane (CBM) recovery factor and gas extraction rate. The permeability of the coal seam in China is generally by 2~ 3 orders of magnitude lower than that in America due to the special geological conditions [1,2], causing difficulty of gas flow in the coal seam and low productivity of CBM. Moreover, with the increase in mining depth, gas content will increase and coal seam permeability will further decrease [3].

A lot of studies have been done for the permeability improvement in low gas permeability coal seams. The hydraulic fracturing technology was improved by using high-voltage pulse pressure [4,5]. On the basis of hydraulic fracturing technology, chlorine dioxide was used as a gel breaker to increase the permeability of coal seam [6]. Based on the fracture mechanics and seepage mechanics theory, the parameters between hydraulic slotting and hydraulic fracturing were optimized [7,8]. The liquid CO₂ blasting technology was used to improve coal seam permeability [9,10]. The coexistence of coal seam permeability improvement and stability of surrounding rock

through controlled crack evolution direction was studied by directional blasting technology [11]. Ultrasonic technology has also been used for low coal seam permeability enhancement [12,13]. Through the acidification experiment of No. 3 coal reservoir in Jincheng mining area, it was proved that hydrochloric acid can effectively enhance the rich calcite coal seam [14]. The effect of microorganisms on enhancing the permeability of different-rank coals was also studied [15]. At present, the permeability of coal reservoir is mainly improved by physical methods, such as hydraulic fracturing, hydraulic slotting, deep hole blasting; there are also several chemical and biological methods, gradually showing a trend of diversification.

The pore-fracture system of the coal reservoir is the channel of material exchange in the coal seam; the development degree and the connectivity of coal seam influence the permeability of the coal reservoir. In the long geological time, a lot of the coal reservoir pore-fracture has been blocked by minerals such as calcite, dolomite, hematite, pyrite, and other impurities. For example, the coal reservoir fractures in North China Permo-Carboniferous coal and Henan Jiaozuo area are mainly filled with calcite [16,17]. For this kind of low permeability coal seams, it is undoubtedly one

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of the better choices to use a chemical method to increase permeability by acid injection in the coal seams.

THEORY

The permeability improvement of coal reservoirs by acid injection is a method which can improve the conductivity of the coal reservoir by injecting one or several kinds of acid into the coal seam. Through the dissolution and corrosion of the acid to the cement and blockage in the coal reservoir pore–fracture system, the permeability of the coal seam is increased [18,19]), as Figure 1 shows.

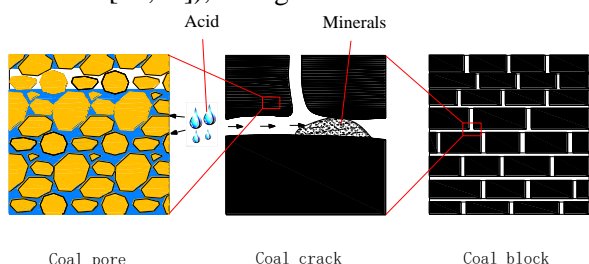
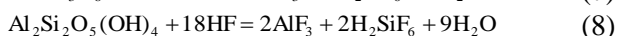
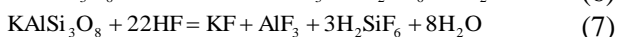
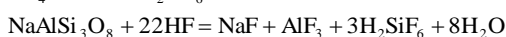
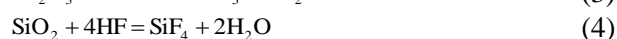
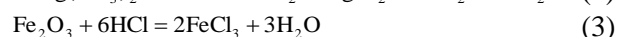
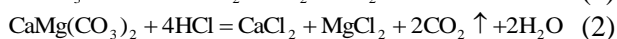
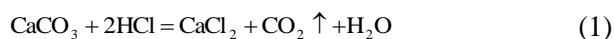


Fig. 1. Principle of coal bed acidification

Chemical reactions

The chemical principles of reactions between different minerals and acids in coal seams are different. According to the common components of minerals in coal seams, the main reaction equations are as follows:



Erosion

When acid is injected into coal seam, the chemical reaction will occur. At the same time, the acid will corrode the coal matrix touched by the acid. Some colloids and blockages in the coal matrix are eroded and dissolved under the action of acid. With the process of acid flowback, the dissolved substance is brought out of the coal body, thus increasing the porosity and connectivity of coal. The basic condition for acid to produce corrosion of coal is that the acid liquid can be fully contacted with coal. The more the coal contacts with the acid solution, the more H^+ is transferred, the reaction rate is faster and the degree of dissolution is higher.

EXPERIMENTAL

Acid system

According to the principle of increasing permeability by injecting acid into the coal seam, in this experiment, the "HCl+HF" mixed acid was chosen to do the acidification experiment. In order to avoid damage of coal seam due to the secondary reaction with HF in acidification process, high concentration of HCl is needed to restrain that. Therefore, the mass fractions of HCl prepared in this experiment were 9%, 12% and 15%, and the mass fraction of HF was 3%. At the same time, it was considered that the non-crystalline kaolinite and other minerals in the fissures of the coal reservoir expanding and blocking the channel during the acidification process, on the contrary, reduce the permeability of the coal seam. So, 2% NH_4Cl (commonly used in petroleum field) was added as an antiswelling agent [20,21]. So, three kinds of acid systems of this experiment were constituted: 9%HCl+3%HF+2% NH_4Cl (Acid I), 12%HCl+3%HF+2% NH_4Cl (Acid II) and 15%HCl+3%HF+2% NH_4Cl (Acid III). Both the acid container and the acidification site were inert plastic bottles.

Experimental program

The experimental coal samples were collected from Xinzhouyao mine of Datong in Shanxi province, Qianjiaying mine of Tangshan in Hebei province and Jiangjiahe mine of Xianyang in Shaaxi province, the coal ranks of which are coking coal, fat coal, non-caking coal. Basic characteristic parameters of the initial coal samples are shown in Table 1. In order to study the permeability increase effect *via* acid injection in different-rank coal reservoirs, four series of experiments, A, B, C and D, were carried out.

Series A was a scanning electron microscope (SEM) observation experiment. First of all, parts of the coal samples in each mine were acidified with acid II for 24 h, and then dried for 3 h. All coal samples were prepared from thin coal of about 1 cm^2 . The surface structure characteristics of different-rank coal samples before and after acidification were comparatively analyzed by a Q45 tungsten filament SEM system.

Series B was a low-temperature nitrogen adsorption experiment. First of all, parts of the coal samples in each mine were acidified with acid II for 24 h, and then dried for 3 h.

Table 1. Characteristic parameters of coal samples

Sample source	Coal seam	$M_{ad}/\%$	$A_{ad}/\%$	$V_{daf}/\%$	$R_{o,max}/\%$	Porosity/%	Coal rank
Xinzhouyao mine	11-2#	2.04	8.62	25.29	1.32	6.94	Coking coal
Qianjiaying mine	12#	1.70	14.11	28.36	1.08	7.23	Fat coal
Jiangjiahe mine	4#	5.03	7.54	30.56	0.87	6.58	Non-caking coal

The coal samples before and after acidification were prepared into 60 ~ 80 mesh, then the surface area and pore structure characteristics of coal samples were measured by a Micromeritics ASAP 2020 physisorption instrument. The experimental procedure is as follows: put about 2 g of the pulverized coal into the sample tube and after degassing, quality test and sample analysis were carried out for each sample. At a correlation coefficient greater than 99.9%, the test results were believed to be reliable.

Series C was a mineral content measuring experiment. Parts of the coal samples in each mine were acidified with acid II for 24 h, and then dried for 3 h. The coal samples before and after acidification were prepared into 200 mesh. The mineral content of coal samples before and after acidification was measured by a XRD6100 test system on a S8 TIGER X-ray fluorescence spectrometer. The test angle range of XRD was 2°~120°.

Series D was a permeability test. In this experiment, the permeability of the coal samples was measured by a self-made coal and rock permeability measurement system (Figure 2) mainly composed of high-pressure gas source, sealing device, pressure chamber, pressure device, pressure stabilizer, gas collecting device and other parts. Assuming that experimental coal sample is a continuous incompressible medium, on the basis of the one-dimensional theory of a single-phase flow, gas state equation and Darcy's law, the formula of coal and rock permeability can be obtained [22]:

$$K = \frac{2\mu P_0 Q L}{A(P_1^2 - P_2^2)} \quad (9)$$

where K is permeability, mD; μ is viscosity coefficient of methane, mPa·s; P_0 is standard atmospheric pressure, Mpa; P_1 and P_2 are the gas pressures of pressure chamber inlet and outlet, MPa, respectively; Q is the gas flow under standard conditions, cm³/s; L is the length of coal sample, cm; A is the cross section area of coal sample, cm².

The coal samples were made into standard specimens of size $\phi 50\text{mm} \times 100\text{mm}$ by a core drilling machine and cutting machine. The permeability of the standard specimens was tested by soaking the specimens in acid I, II, III for 6, 12, 18, 24, 30 and 36 h. The specimens must be dried at

60 °C for 3 hours before testing, to avoid the effect of moisture content on the permeability test. During the test, the confining pressure was 2 MPa, the axial pressure was 1 MPa and the experimental temperature was 25 °C.

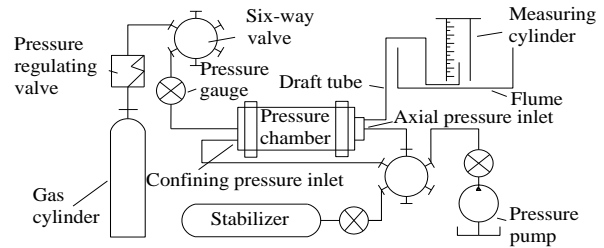


Fig. 2. Coal permeability measurement system

RESULTS AND DISCUSSION

Surface structure characteristics

The surface structures of coal samples before and after acidification are shown in Fig. 3. In the initial state, Xinzhouyao and Qianjiaying coal sample porosity degrees were better in comparison with Jiangjiahe. Some fillings can be observed in the pores and fissures of each coal sample. Among them, scattered minerals (Figure 3e) can be observed in the coal samples of Jiangjiahe. After the acidification, the fillings (such as minerals and colloids) were eroded, the outline of pores was clearer, the length, width and number of cracks increased, and the connectivity of pore-fracture system was remarkably improved, especially, the coal samples of Jiangjiahe.

The results show that the acidification corrosion of the low rank coal samples of Jiangjiahe is obviously better than that of the high-rank coal samples of Xinzhouyao and Qianjiaying under the same conditions. First reason, there are differences in the content of minerals contained in each coal sample. Jiangjiahe coal sample contains more minerals, its acidizing effect is better. Another reason is that high-rank coal has a high degree of aromatic ring polymerization, and less side chains reacting with the acid, so it can greatly resist acid corrosion.

At a larger specific surface area, the higher degree of coal body tortuosity inside leads to higher conductivity to gas storage in the coal seam. However, this is not good for gas storage.

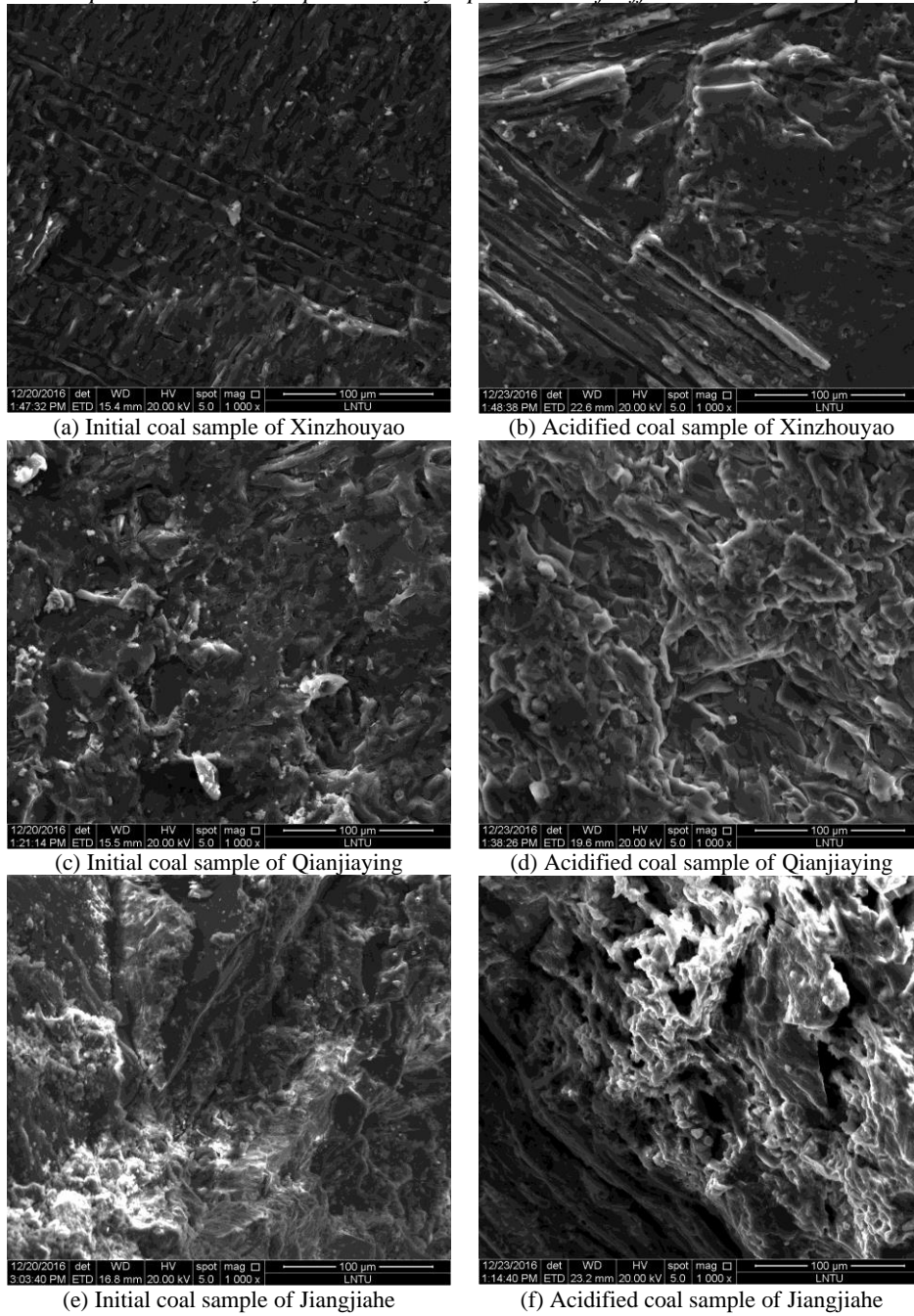


Fig. 3. The surface structure before and after acidification . Magnification 1000×

The greater the pore volume of coal, the higher is the connectivity of coal and the conductivity to fluid migration in the coal seam. The test results of this experiment are shown in Table 2 and Figures 4 and 5.

According to Table 2, before coal sample acidification, the pore structure of coal increases with the increase in coal rank. After the acidification of coal samples, the specific surface area of coal decreased, indicating that the acidification process makes the internal structure of

coal become simpler, and reduces the gas adsorption sites which are conducive to gas desorption. With the development of acidification, the internal space of coal increases, and the total pore volume increases. Whether in specific surface area or pore volume, Jiangjiahe coal samples changes were the most obvious while Xinzhouyao coal samples changes the least obvious. This shows that the effect of acidizing decreases with the increase in coal rank.

Table 2. Comparison of specific surface area and pore volume before and after acidification

Sample source	Initial specific surface area /m ² /g	Specific surface area after acidification/m ² /g	Decrease percentage /%	Initial total pore volume /cm ³ /g	Total pore volume after acidification /cm ³ /g	Increase percentage /%	Coal rank
Xinzhouyao mine	8.1609	7.7085	5.54	0.017018	0.018211	7.01	Coking coal
Qianjiaying mine	6.4111	5.1739	19.30	0.005918	0.011032	86.42	Fat coal
Jiangjiahe mine	4.3567	2.2170	49.11	0.003186	0.007991	150.82	Non-caking coal

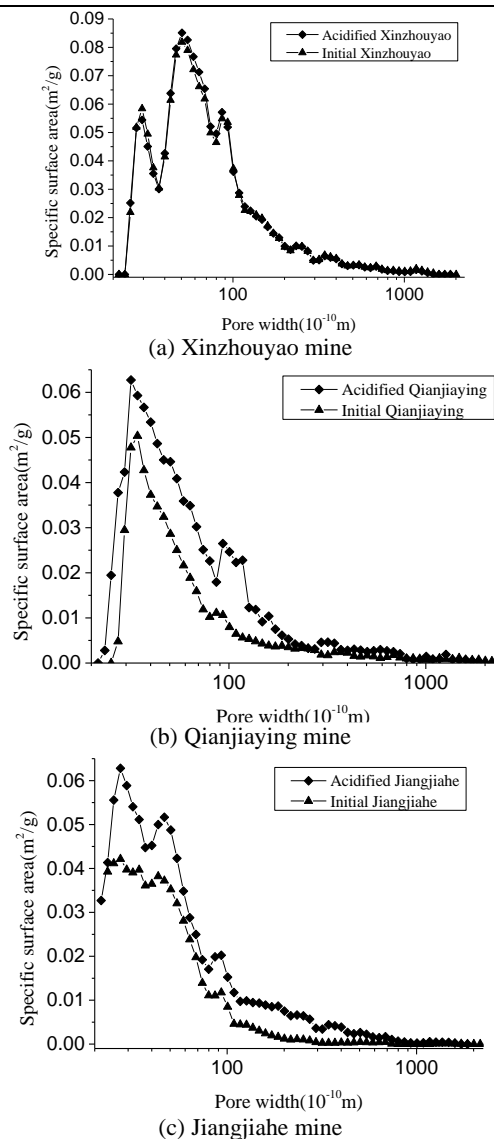


Fig. 4. Relationship between specific surface area and pore width before and after acidification

Figures 4 and 5 show that the specific surface area and pore volume of the coal samples are mainly distributed in the pore width range of 10 nm or less, which indicates that the micropores in the coal are well developed. After acidification, the specific surface area of the coal samples obviously increases within the pore width of 2~10 nm (except Xinzhouyao).

Its distribution regularity is consistent with that of initial coal samples. It indicates that the acid also has a good corrosion effect on the micropores of coal samples.

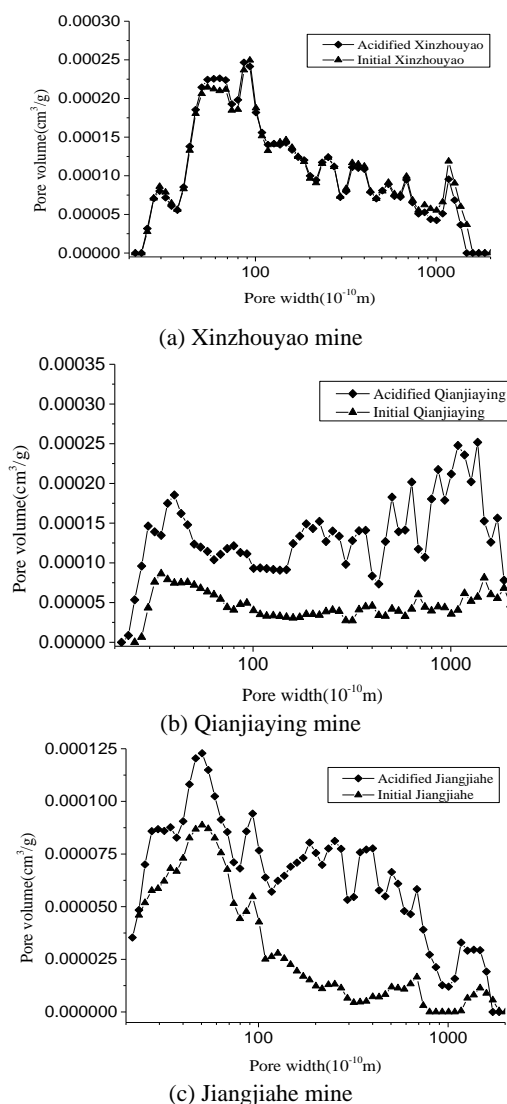


Fig.5. Relationship between pore volume and pore width before and after acidification

The change in pore volume obeys the same law. Moreover, the pore volumes of the coal samples from Qianjiaying and Jiangjiahe mine also obviously increased in the range of 10~100 nm, which indicates that the pores in the scale are filled with more fillers and the fillers are sufficiently corroded, so that the connectivity between the pores is effectively enhanced.

Mineral content

The types and contents of the fillings in the coal samples directly affect its permeability. XRD experiment was mainly intended to determine the composition of minerals in the coal samples.

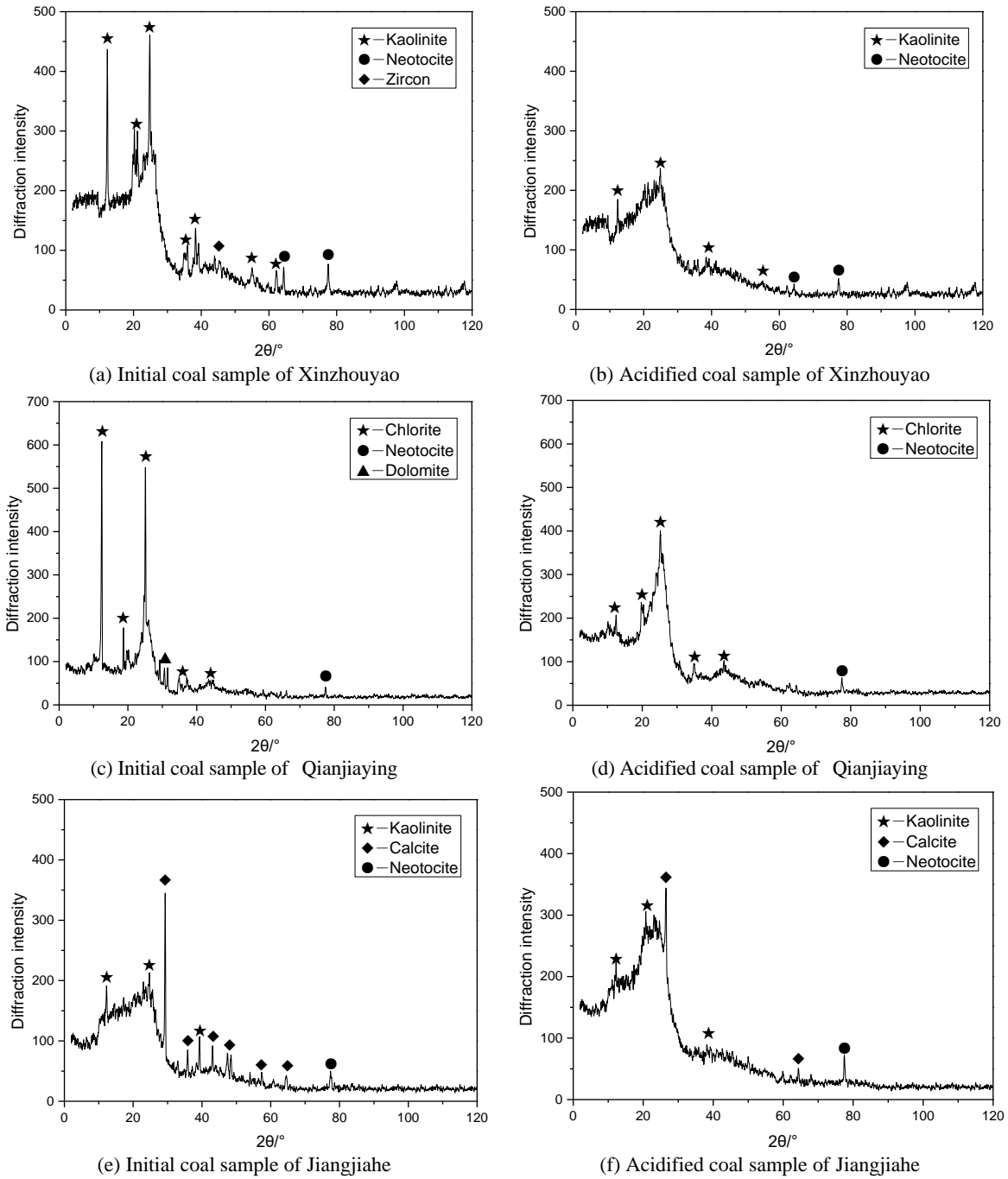


Fig.6. XRD spectra of coal samples before and after acidification

XRD test

After the test, MDI Jade5.0 post-processing software was used for peak search, smooth, integral and other processing, and finally draw the scanning range and the diffraction intensity curve (Figure 6).

As shown in Figure 6, after acidification, the diffraction intensity of dolomite, calcite, kaolinite, chlorite, zircon in each coal sample is reduced, and some diffraction peaks of these minerals have disappeared, but the diffraction peak of neotocite is almost not changed. It can be inferred that the minerals whose diffraction peak is reduced or

disappeared, are effectively dissolved, and the effect of acidification on bannisterite is not obvious.

Organic speciation analysis

Because the acid solution is very corrosive to metals, people are bound to doubt whether the coal sample acidification process affects the coal quality. The organic matter in coal consists of two parts: amorphous and microcrystalline component (Figure 7) [23]. The amorphous component has no fixed structure and cannot form an X-ray diffraction peak; but the diffraction peak of the microcrystalline structure is basically consistent with the 002 and 100 diffraction peaks of the graphite crystal [24,25].

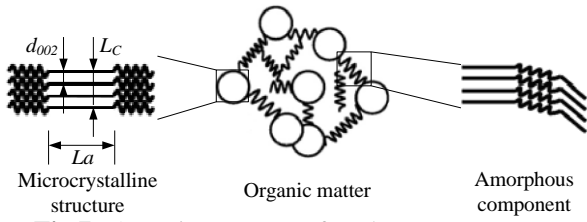


Fig.7. Organic structure of coal

According to the Prague method, the morphology of crystallites in coal can be characterized by the spacing of aromatic carbon layers, the diameter of nucleation lamellae, the height of lamellae and the number of lamellae, equations (10)~(13) [23, 26]. If the above parameters are greatly different before and after acidification, this proves that the acid solution has greater harm to the coal quality; otherwise, the damage is less or not harmful.

$$d_{002} = \frac{\lambda}{2 \sin \theta_{002}} \quad (10)$$

$$L_c = K_1 \frac{\lambda}{\beta_{002} \cos \theta_{002}} \quad (11)$$

$$L_a = K_2 \frac{\lambda}{\beta_{100} \cos \theta_{100}} \quad (12)$$

$$N_c = \frac{L_c}{d_{002}} + 1 \quad (13)$$

where d_{002} is aromatic carbon film spacing, 10^{-10} m; λ is wavelength of X rays, 1.5406×10^{-10} m; θ_{002} , θ_{100} are 002 and 100 peak positions, $^\circ$; L_c is aromatic carbon crystal height, 10^{-10} m; L_a is aromatic carbon nucleus lamellar diameter, 10^{-10} m; K_1 and K_2 are shape factors, $K_1=0.94$, $K_2=1.84$; β_{002} and β_{100} are the half heights of 002 and 100 peaks,

10^{-10} m; N_c is aromatic carbon nucleus lamellar number.

According to the XRD test results before and after acidification of each coal sample, the diffraction angle and half width of 002 peaks and 100 peaks of each coal sample were extracted, then the microcrystalline structure parameters of each coal sample were calculated. The calculation results are shown in Table 3.

From the table above, it is seen that the microcrystalline structure parameters of coal samples before and after acidizing are basically unchanged, which indicates that the acidification process does not cause obvious damage to the organic matter in coal. In addition, the acid system is inorganic. According to the principle of similarity and compatibility, the solubility of organic matter in coal is very limited.

XRF test

The final test results of XRF are expressed as oxide contents. The test results of this experiment are shown in Table 4.

Table 4 shows that the composition and content of minerals in coal samples are related to the depositional environment. The coal samples of Qianjiaying are mainly composed of Si and Al minerals, the coal samples of Jiangjiahe are dominated by Ca mineral, and the mineral content of coal samples from Xinzhouyao is low.

The mineral content of the coal samples after acidification was greatly reduced, and the acidizing effect was obvious. Among them, the percentage of CaO in Jiangjiahe coal sample decreased from 14.61% to 0.32%, which is the reason why the coal sample produced a large number of bubbles during the acidification reaction.

Table 3. Microcrystalline structure parameters of coal samples

Sample source	Type	$(^\circ)2\theta_{002}$	$(^\circ)2\theta_{100}$	$(\text{\AA})d_{002}$	$(\text{\AA})L_c$	$(\text{\AA})L_a$	$(\text{\AA})N_c$
Xinzhouyao mine	Initial	24.87	43.68	3.56	8.21	6.62	3.31
	Acidized	24.65	43.75	3.59	8.20	6.87	3.28
Qianjiaying mine	Initial	23.91	43.02	3.73	14.82	6.08	4.97
	Acidized	23.42	42.51	3.74	14.79	6.07	4.95
Jiangjiahe mine	Initial	25.67	43.80	3.47	4.16	5.38	2.20
	Acidized	25.49	43.69	3.50	4.14	5.40	2.18

Table 4. Content of mineral before and after acidification

Sample name	SiO ₂ %	Al ₂ O ₃ %	TFe%	CaO%	MgO%
Initial coal sample of Xinzhouyao	2.17	0.82	0.11	0.088	0.066
Acidified coal sample of Xinzhouyao	0.66	0.32	0.07	0.042	0.012
Initial coal sample of Qianjiaying	5.71	5.37	1.47	1.25	0.39
Acidified coal sample of Qianjiaying	1.93	1.83	0.79	0.28	0.10
Initial coal sample of Jiangjiahe	2.32	1.65	1.07	14.61	1.30
Acidified coal sample of Jiangjiahe	0.78	0.56	0.57	0.32	0.16

Compared with the dissolution of different minerals, the contents of Ca and Mg minerals are larger than those of Si, Al and Fe minerals. This is because the former mainly exist as carbonates, relatively easy to react with acid solution, the latter mainly exist as oxides, the reaction rate is relatively slow, and the reaction conditions are relatively harsh. Therefore, the greater the content of Ca and Mg minerals in the coal seam, the more conducive is the latter to the acidification reaction and the better is the acidizing effect, for example, the Jiangjiahe coal mine.

Permeability

The test results of the permeability changes of coal samples in different acids after different acidification times are shown in Table 5 and Figure 8. From the test results, the permeability characteristics of different acidified coal samples can be obtained:

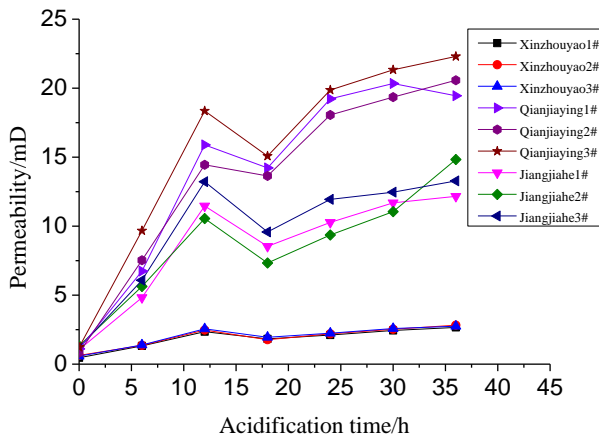


Fig. 8. Relationship between coal sample permeability and acidification time

1) At the beginning of acidification, the permeability of coal samples increased rapidly, but after acidification for 12 h, the permeability slightly decreased. The reason is that the coal sample contains clay minerals. After a period of immersion,

the clay minerals expand and plug the pore-fracture channel, which leads to a decrease in permeability. However, due to the limited content of clay in the coal sample and the anti-swelling effect of ammonium chloride, the permeability slightly decreased. The extent and depth of the corrosion are larger with the acidification proceeding. The permeability gradually increases and eventually reaches a stable value. With the continuation of acidification, the extent and depth of corrosion increase, the permeability gradually increases, and finally tends to be stable.

2) The permeability increase effect *via* acid injection in Xinzhouyao coal samples with high-rank coal is obviously inferior to that of low-rank coal samples. The contents of Ca and Mg minerals and the initial permeability in Jiangjiahe and Qianjiaying coal samples are high, which provided more reactants and reaction conditions for the acidification reaction. More pores and fissures were connected and the permeability was greatly increased.

3) In the same acidification time, the higher the acid concentration, the faster increased the permeability. However, due to the excess acid in the experiment, the filler in the coal sample pores can be fully corroded, thus the final permeability improvements of different acid systems are not very different.

DISCUSSION

According to the four series of experiments, the effect of permeability increase of coal seam by acid injection was analyzed from different emphases. Under the same conditions, the coal samples of Xinzhouyao showed the worst effect on permeability improvement by acid injection, and Jiangjiahe and Qianjiaying were gradually weakened.

Table 5. Permeability changes of coal sample before and after acidification

Sample name	Acid system	Permeability /mD						
		Acidified 0 h	Acidified 6 h	Acidified 12 h	Acidified 18 h	Acidified 24 h	Acidified 30 h	Acidified 36 h
Xinzhouyao 1#	I	0.46	1.32	2.35	1.83	2.11	2.44	2.66
Xinzhouyao 2#	II	0.63	1.36	2.47	1.77	2.20	2.53	2.82
Xinzhouyao 3#	III	0.58	1.39	2.56	1.95	2.24	2.58	2.75
Qianjiaying 1#	I	1.05	4.82	11.46	8.54	10.27	11.69	12.16
Qianjiaying 2#	II	1.32	5.63	10.55	7.33	9.36	11.05	14.83
Qianjiaying 3#	III	1.18	6.08	13.22	9.58	11.94	12.46	13.28
Jiangjiahe 1#	I	1.10	6.74	15.89	14.22	19.23	20.34	19.45
Jiangjiahe 2#	II	0.82	7.53	14.45	13.64	18.06	19.35	20.57
Jiangjiahe 3#	III	1.23	9.67	18.35	15.08	19.87	21.33	22.30

The distribution of the permeability improvement was the same as the distribution of mineral content in the coal sample, that is, the amount of the mineral component and the content of the coal sample are the keys to the permeability increase of coal seam by acid injection. Moreover, the initial permeability of Qianjiaying coal sample was higher than that of Xinzhouyao, the higher initial permeability is favorable for the acid to seep into the coal seam and promote acidification. In addition, the results of microscopic observation and low-temperature nitrogen adsorption test showed that the corrosion of acid for the medium-rank coal samples of Jiangjiahe and Qianjiaying is better than for the high-rank coal samples of Xinzhouyao. It can be seen that under the same acid system, the permeability increase of coal samples by acid injection is influenced by the mineral content, initial permeability, coal rank and other factors.

CONCLUSIONS

1) A lot of pore channels are blocked in the coal seam, which leads to the difficulty of gas migration and the low permeability of the coal seam. Therefore, a chemical method for increasing permeability of coal seam by acid injection was put forward. After acidification, the filling materials such as minerals and colloid were corroded, and the fracture connectivity of the coal sample surface was obviously improved. The lower the coal rank, the lower was the degree of polymerization of the aromatic ring, the more easily it was corroded.

2) After acidification, the specific surface area and the total pore volume increased, and the specific surface area and total pore volume mainly increased within pore widths of 2 ~ 10 nm. The porosity parameters of middle- and low-rank coal samples were larger than those of high-rank coal samples, which were corroded easily.

3) Acidification does not cause damage to the organic matter of coal. After acidification, the contents of Si, Al, Fe, Ca, Mg and other minerals in the coal samples all decreased, especially Ca and Mg minerals, indicating that the higher the content of these minerals, the better is the permeability of coal seam by acid injection.

4) The permeability of coal samples increased after acidification. The higher the initial permeability of coal samples, the higher were the mineral contents of Ca and Mg, and the better was the permeability of the coal seam by acid injection. The higher the concentration of acid, the greater was the rate of increase in permeability.

5) It is a good method to increase the permeability of the coal body by using acid solution to dissolve and corrode the fillings in the pores and

fractures of the coal seam. However, how to control the concentration and dosage of acid in different coal seams needs to be solved in the further research.

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