

Optimization of Acid Fluid System Suitable for High-Rank Coal

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Abstract - In recent years, acidification technology has been widely applied to increase coal seam permeability to enhance gas drainage, and the acid fluid is the key factor determining the effectiveness of acidification permeability enhancement. To find the optimal acid fluid system suitable for high-rank coal acidification permeability enhancement, taking Jiulishan Coal Mine as an example, low-field nuclear magnetic resonance (LF-NMR) tests were conducted to preliminarily select the acid type suitable for high-rank coal. Subsequently, the orthogonal experimental method was employed to optimize the acid fluid system suitable for high-rank coal. The study shows that after acidification, the number of mesopores and macropores-microfractures, as well as their pore volume proportion in the coal samples, significantly increased. Furthermore, the connectivity between pores and fractures was enhanced, resulting in increased effective porosity and NMR permeability of the coal samples. By comparing and analyzing the changes in effective porosity and NMR permeability of coal samples before and after acidification with three mixed acid solutions, the preferred acid type combination suitable for high-rank coal acidification was identified as HCL and HF. The acid fluid system optimized using the orthogonal experimental method was determined to be 5%HCL+3%HF.

Keywords: Acid fluid system; Effective porosity; Permeability; Orthogonal experiment; Acid optimization

1. Introduction

With the gradual depletion of shallow coal resources, exploiting deep resources has become an inevitable trend in China's coal resource extraction. As mining depth increases, in-situ stress and gas pressure continuously rise, while permeability gradually decreases. Consequently, gas dynamic disasters accompanying deep coal mining become more prominent, and gas drainage is an effective method for preventing these disasters. Due to the low porosity and low permeability characteristics of China's coal seams, efficient gas drainage is restricted [1]. To enhance the permeability of low-permeability coal seams, hydraulic fracturing technology has been widely applied for coal seam modification. However, hydraulic fracturing consumes large amounts of water resources and is influenced by coal and rock characteristics, leading to short fracture propagation lengths [2], small radii of fracturing influence, and limited effectiveness in coal seam modification. The mechanical properties of anthracite coal in the Jiaozuo mining area are relatively close to those of its roof and floor rocks. During hydraulic fracturing, low pressure cannot open the fractures within the coal, while high pressure connects the coal seam with the aquifers in its roof and floor, leading to fracturing failure [3]. Therefore, conventional hydraulic fracturing technology cannot achieve the goal of enhancing permeability to improve gas drainage. Research has found that pores, throats, and fractures in coal are filled with carbonate minerals such as calcite and dolomite [4-6], leading to reduced coal seam permeability. In view of this, it is hoped to adopt a technical method that dissolves the minerals within the pores and fractures of the coal body, thereby increasing the coal's pore volume and porosity, improving pore connectivity, and ultimately enhancing the coal seam's permeability.

Acidification technology involves injecting acid fluid into the formation under conditions below the formation fracturing pressure, allowing the acid to react with and dissolve minerals in the formation, thereby increasing its permeability. In 1985, Vasyuchkov [7] reported the change patterns of porosity and permeability of coal samples before and after treatment with water and hydrochloric acid under different stress loads, proposing that hydrochloric acid treatment can increase coal's porosity and permeability. In 1998, Su Xianbo et al. [8] proposed that in high-rank coal development areas similar to Jiaozuo anthracite, where fractures are primarily filled with calcite, acidification technology is superior to hydraulic fracturing in enhancing coal seam permeability. Currently, domestic and international scholars, by studying the effects of acidification on the porosity, permeability, and surface morphology of coal bodies [9], have

confirmed that acidification can significantly increase the pore volume and porosity in coal, markedly improve the connectivity between pores and fractures, and consequently enhance coal seam permeability. However, the key factor determining the effectiveness of acidification permeability enhancement is the acid fluid system, which includes the acid type and concentration ratio. Existing research literature mostly focuses on using hydrochloric acid, widely applied in oilfield stimulation, as the acidification fluid to analyze its permeability enhancement effect on low-rank coal. However, the geological characteristics and mineral compositions of coal reservoirs are significantly different from those of oilfield carbonate or sandstone reservoirs, thus the acid fluid system suitable for high-rank coal acidification remains unclear. Therefore, finding the optimal acid fluid system suitable for high-rank coal acidification permeability enhancement is a critical issue that urgently needs to be addressed for applying acidification technology to increase coal seam permeability.

To find the optimal acid fluid system suitable for high-rank coal acidification permeability enhancement, this study takes high-rank coal from the Jiulishan Mine in Jiaozuo as an example. Coal columns were soaked in distilled water and three mixed acid solutions (HCL+HBF₄, H₃PO₄+HF, HCL+HF). Low-field nuclear magnetic resonance (LF-NMR) tests were conducted to measure parameters such as porosity, permeability, and pore size distribution of the coal samples before and after treatment with distilled water and the three mixed acid solutions. By analyzing the change patterns of these parameters, the effects of each mixed acid solution on the pore structure of high-rank coal were investigated to select the suitable acid type for high-rank coal; the orthogonal experimental method was used to optimize the acid fluid system and acidification parameters suitable for high-rank coal, providing experimental support and theoretical basis for promoting the application of acidification technology in coal seams.

2. Optimization of Acid Type Suitable for High-Rank Coal

2.1. Experimental Materials and Methods

The experimental coal samples used in this study were collected from the working face of the No. 15 mining area of Jiulishan Coal Mine, Henan Coking Coal Energy Co., Ltd. The maximum vitrinite reflectance, maceral composition, and proximate analysis results of the coal are shown in Table 1. As shown in Table 1, the maximum vitrinite reflectance of the coal is 2.59%, classifying it as anthracite. Coal samples were prepared strictly according to GB/T 474-2008 (China) 'Methods for Preparation of Coal Samples'. Coal cores with a diameter of ϕ 25 mm and length of 50 mm were drilled using a core drilling machine for low-field nuclear magnetic resonance (LF-NMR) testing to analyze the effect of mixed acid solutions on the coal pore structure, thereby optimizing the acid type suitable for high-rank coal.

Table 1: Vitrinite Reflectance and Maceral and Proximate Analyses of the Coal Sample(%).

coal sample	$R_{o,max}$	maceral content				Proximate analysis			
		V	I	E	MM	M _{ad}	A _{ad}	V _{ad}	F _{cad}
Jiulishan coal mine	2.59	78.3	14.1	0	7.6	3.15	8.24	6.3	82.31

Note: $R_{o,max}$ represents the maximum vitrinite reflectance; V represents vitrinite; I represents inertinite; E represents exinite; MM represents mineral; M_{ad} represents the moisture content of air dried basis; A_{ad} represents the ash content of air dried basis; V_{ad} represents volatiles; and F_{cad} represents the carbon content of air dried basis.

The mineral composition of the coal and the chemical reaction characteristics of each mineral with different acids form the material basis for acid selection and the implementation of acidification permeability enhancement technology. To determine the mineral composition and content of the Jiulishan Mine coal, the raw coal sample was tested using an X-ray diffractometer (XRD) with a testing angle range of 0-70° and a scanning speed of 5°/min. Jade 6.0 software was used to perform phase retrieval and quantitative analysis on the XRD test results, revealing that the main minerals in Jiulishan Mine coal include calcite, ankerite, quartz, kaolinite, chlorite, and barite, with proportions of the total mineral mass being 21.4%, 2%, 50.9%, 10.2%, 1.9%, and 13.6%, respectively. The X-ray diffraction pattern of the raw coal sample from Jiulishan Coal Mine is shown in Figure 1.

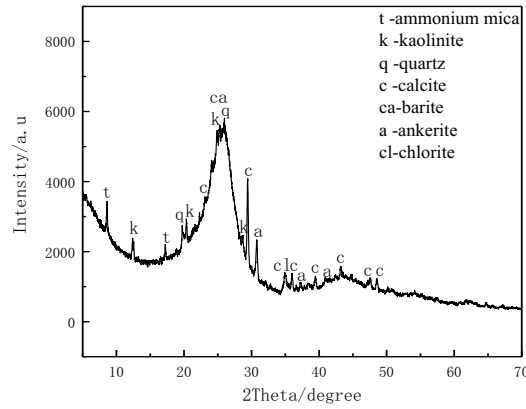


Fig. 1: XRD diffraction pattern.

The experimental procedure was as follows: ① Pre-treatment coal samples were placed in a constant temperature drying oven at 80 °C for 48 h. After cooling to room temperature, the mass of each dry coal sample was weighed using a balance. ② The dried coal samples were placed in a vacuum saturation device filled with distilled water and saturated for 24 h. ③ The mass, length, and diameter of each saturated coal sample were measured, and the volume was calculated. ④ LF-NMR experiments were conducted on each coal sample before acidification to measure the T_2 spectrum distribution. ⑤ Then, the samples were centrifuged for 1 h, and the mass of each centrifuged coal sample was weighed. ⑥ LF-NMR experiments were conducted on the centrifuged coal samples to measure the T_2 spectrum distribution. ⑦ 300 ml each of distilled water, Acid 1, Acid 2, and Acid 3 were prepared and stored in sealed inert plastic bottles. Four coal cores were dried and cooled, labeled J0, J1, J2, and J3, respectively, and then immersed in the corresponding solutions (distilled water, Acid 1, Acid 2, Acid 3) in sealed inert plastic bottles. These were placed in a constant temperature water bath at 30 °C to react for 24 h. ⑧ The treated coal samples were dried in a constant temperature oven at 80 °C for 48 h. After cooling to room temperature, the mass of each dry treated sample was weighed. Steps ②–⑥ were repeated to study the effects of the four solutions on the pore structure of the coal samples. The detailed experimental procedure is shown in Figure 2.

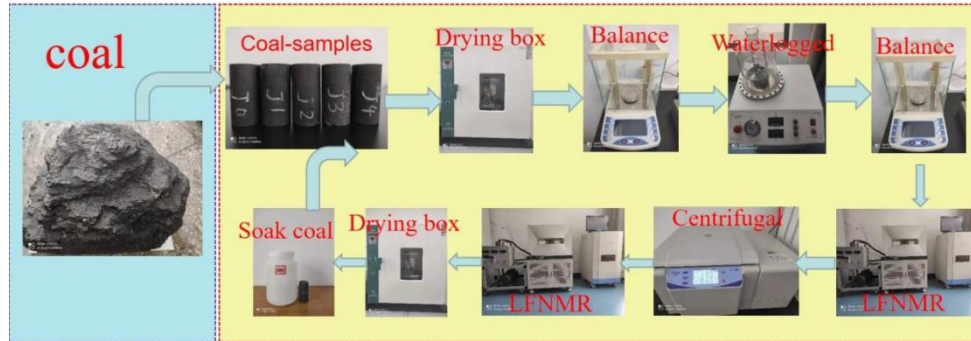


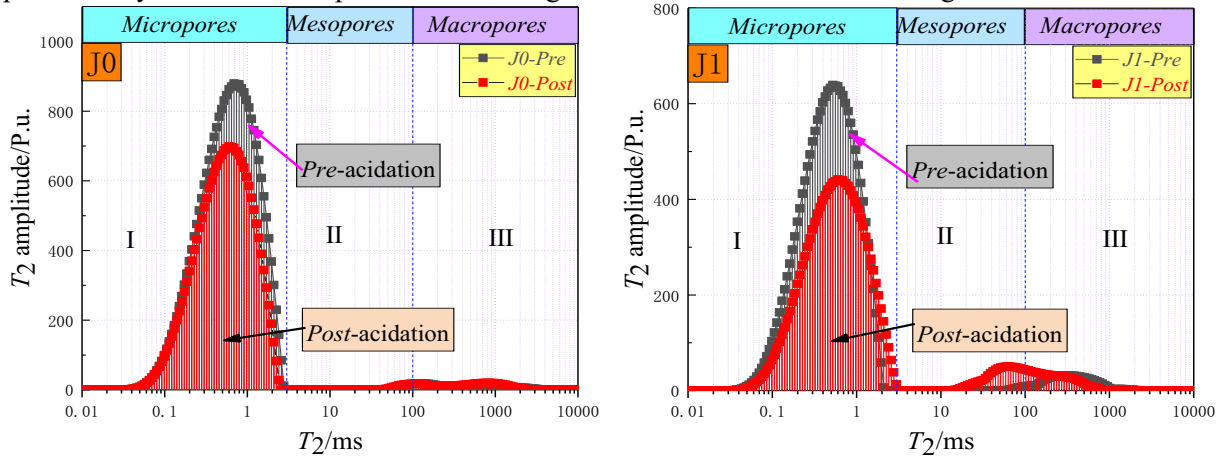
Fig. 2: Schematic diagram of the experimental process.

2.2. Analysis of T_2 Spectrum Results

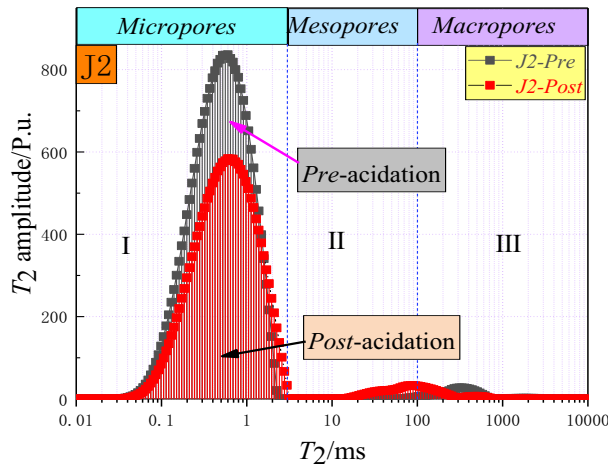
According to the principle of low-field nuclear magnetic resonance testing, the T_2 spectrum peak on the relaxation time axis is proportional to the pore size, the peak height is proportional to the number of pores at the corresponding size, and an increase in amplitude indicates an increase in the number of pores [10]. The T_2 spectrum curves of saturated coal samples before and after treatment with distilled water and the three acid solutions are shown in Figure 3. Before treatment, all four coal samples exhibited a three-peak characteristic. According to the Hodot pore classification method [11], the left peak I ($T_2 < 2.5\text{ms}$) corresponds to micropores; the middle peak II ($2.5\text{--}100\text{ms}$) corresponds to mesopores; and the right peak III ($T_2 > 100\text{ms}$) corresponds to macropores-microfractures. The areas enclosed by the T_2 spectra and the transverse relaxation time axis in regions I, II, and III represent the volumes of micropores, mesopores, and macropores-microfractures in the coal sample, respectively [12]. From Figure 3, the micropore peak I has the highest peak value, but

peak I exists independently of peaks II and III, indicating that the pore volume proportion of micropores is the largest and micropores are the most developed, but the connectivity between micropores and mesopores/macropores-microfractures is poor. The peak values of the mesopore peak and macropore-microfracture peak are relatively low, while the valley between the mesopore peak and the macropore-microfracture peak is relatively flat, indicating a lower pore volume proportion for mesopores and macropores-microfractures, although they are relatively developed, and the connectivity between mesopores and macropores-microfractures is good. After acidification, the T_2 spectrum distribution curves of coal samples J1, J2, and J3 treated with the three mixed acid solutions still show three peaks. However, the peak value of peak I, representing micropores, significantly decreased compared to before acidification, the right side of the peak shifted to the right, and the peak area decreased, indicating that pore sizes shifted towards mesopores and macropores, and the volume proportion of micropores significantly decreased. The peak values of peaks II and III, representing mesopores and macropores-microfractures, increased substantially compared to before acidification, the peak areas increased, and the peak ranges widened. The valley between the mesopore peak and the macropore-microfracture peak became smoother, indicating enhanced connectivity between mesopores and macropores-microfractures. This shows that after acidification, the number and pore volume proportion of mesopores and macropores-microfractures both increased significantly, with the increase in the number of mesopores and macropores being most prominent, and the connectivity between them was enhanced. This is consistent with the conclusions drawn in reference [13]. The dissolution effect of the mixed acid solutions dissolved minerals within the coal pores and fractures, cleared originally blocked pores and fractures, increased the connectivity between coal pores and fractures, causing some micropores to transform into mesopores and macropores. Therefore, after acidification, the volume proportion of micropores in the coal decreased, while the volume proportion of mesopores and macropores increased.

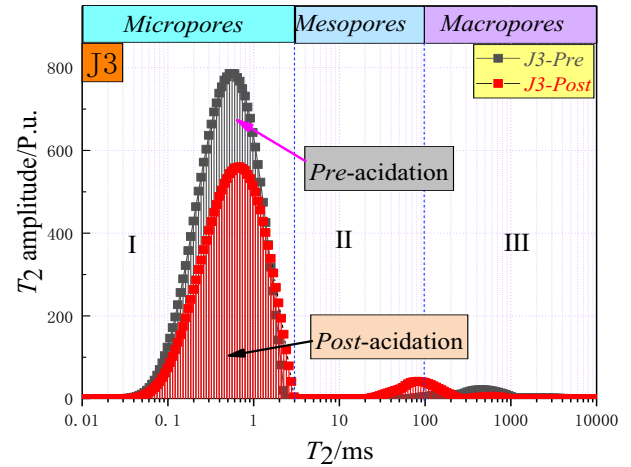
Comparing the T_2 spectrum distribution curves of the three coal samples after acidification, it is observed that compared to the J2 sample acidified with 9% H_3PO_4 +3% HF , the peak amplitudes and areas of peaks II and III, representing mesopores and macropores-microfractures, in the T_2 spectra of samples J1 and J3 acidified with 9% HCL +3% HBF_4 and 9% HCL +3% HF , respectively, increased significantly more. This indicates that, compared to the acidified J2 sample, the number and volume of mesopores and macropores-microfractures increased more in the acidified J1 and J3 samples, and the connectivity between pores was improved more effectively. This is because the dissolution effect of 9% HCL +3% HBF_4 and 9% HCL +3% HF on the minerals in coal pores and fractures is stronger than that of 9% H_3PO_4 +3% HF , indicating that the mixed acids 9% HCL +3% HBF_4 and 9% HCL +3% HF can better dissolve the minerals originally filling the coal pores and fractures compared to 9% H_3PO_4 +3% HF . This leads to a greater increase in the number and volume of coal pore fractures, allowing acidification to play a better role in clearing and expanding pores, enhancing the connectivity between coal pores and fractures to a greater extent, which is more conducive to gas migration and extraction from coal pores and fractures. Therefore, the mixed acids 9% HCL +3% HBF_4 and 9% HCL +3% HF are more suitable than 9% H_3PO_4 +3% HF for acidification permeability enhancement operations in the high-rank coal of the Jiaozuo mining area.



(a) Comparison of T_2 spectra before and after treatment of distilled water (b) Comparison of T_2 spectra before and after treatment of acid 1



(c) Comparison of T_2 spectra before and after treatment of acid 2



(d) Comparison of T_2 spectra before and after treatment of acid 3

Fig. 1: T_2 spectral curves of NMR tests before and after treatment with different solutions.

2.3. Analysis of Changes in Effective Porosity

Coal porosity is an important indicator characterizing the gas storage capacity and describing the structure of the coal body. By conducting LF-NMR tests on saturated and centrifuged coal samples before and after treatment with the four solutions, the saturated porosity and centrifuged porosity of the four samples before and after treatment were obtained. Saturated porosity is calibrated as total porosity, and centrifuged porosity is calibrated as irreducible water porosity; the difference between the two is the effective porosity [14]. Effective porosity refers to the portion of well-connected porosity in coal that facilitates the flow of movable fluids. It is an important indicator for evaluating the seepage capacity of the coal seam. The higher the effective porosity, the better the coal seam's seepage capacity.

To eliminate differences in the initial values of the coal samples, analyzing the change in effective porosity before and after acidification for each sample is more important. Comparative analysis of the change in effective porosity before and after acidification for each sample better describes the requirements for coal seam acidification permeability enhancement and improving gas migration efficiency, and is conducive to exploring the effects of different acid solutions on coal permeability enhancement, thereby enabling the optimization of the best acid type. The effective porosity and its changes calculated from the saturated and centrifuged porosities for coal samples before and after immersion treatment with the four solutions are shown in Table 2.

Table 2: Effective porosity measurement results (%).

No. of coal sample	Before treatment	After treatment	Absolute variation	Relative variation
J0	0.215	0.14	-0.075	-34.88
J1	0.259	0.386	0.127	49.03
J2	0.22	0.324	0.104	47.27
J3	0.12	0.342	0.222	185

Table 2 shows that after treatment with distilled water, the effective porosity of the coal sample decreased, with an absolute decrease of 0.075% and a relative reduction of 34.88%. This indicates that after distilled water treatment, the effective pore volume of the coal sample decreased, the connectivity between pores worsened, and the coal's seepage capacity declined. This phenomenon suggests that improper application of hydraulic fracturing or coal seam water injection technology might lead to a decrease in the effective porosity of the coal seam, thereby reducing coal permeability and hindering gas drainage.

After treatment with the three acid solutions, the effective porosity of samples J1, J2, and J3 all increased, indicating enhanced connectivity between pores after acidification. To optimize the most suitable acid type for acidification permeability enhancement in the high-rank coal of Jiulishan Mine, Jiaozuo mining area, the changes in effective porosity of the coal samples after treatment with the three acid solutions were compared and analyzed. As shown in Table 2, the effective porosity of the coal samples increased to varying degrees after treatment with the three mixed acid solutions. The effective porosity of samples J1, J2, and J3 after acidification increased by 0.127%, 0.104%, and 0.222%, respectively, corresponding to increase percentages of 49.03%, 47.27%, and 185%. Judging from the magnitude of the increase in effective porosity after acidification, the J3 sample treated with the 9%HCL+3%HF mixed acid solution showed the largest increase, indicating that for the high-rank coal of Jiulishan Coal Mine in the Jiaozuo mining area, 9%HCL+3%HF has the strongest ability to improve the coal pore structure and achieves the best acidification permeability enhancement effect.

2.4. Analysis of Changes in NMR Permeability

Changes in coal porosity manifest macroscopically as changes in permeability. Permeability reflects the ability of the coal seam to allow gas flow and production. Permeability can serve as the ultimate parameter for evaluating the effectiveness of acidification permeability enhancement. Based on parameters such as saturated porosity and centrifuged porosity measured by the LF-NMR instrument, the NMR permeability of the coal samples before and after acidification and its changes were calculated using an NMR permeability calculation model. The results are shown in Table 3.

Table 3: Permeability and its variation before and after acidification (%).

No. of coal sample	Permeability before treatment /mD	Permeability after treatment /mD	Absolute change in permeability /mD	Relative change in permeability /%
J0	0.043	0.011	-0.032	-74.42
J1	0.032	0.066	0.034	106.25
J2	0.036	0.060	0.024	66.66
J3	0.009	0.061	0.052	577.77

As seen in Table 3, after treatment with distilled water, the permeability of the coal sample decreased by 0.032 mD, a reduction percentage as high as 74.42%. After acidification treatment, the permeability of samples J1, J2, and J3 increased by 0.034 mD, 0.024 mD, and 0.052 mD, respectively, corresponding to increase percentages of 106.25%, 66.66%, and 577.77%. Thus, the J3 sample acidified with 9%HCL+3%HF exhibited the largest increase in permeability, indicating that for Jiulishan Coal Mine in the Jiaozuo mining area, the permeability enhancement effect of 9%HCL+3%HF is superior to that of 9%HCL+3% HBF_4 and 9% H_3PO_4 +3%HF. The change pattern of the coal sample's NMR permeability is consistent with the change pattern of effective porosity. Therefore, the authors believe that a mixed acid solution of HCL and HF can be considered the optimal acid type for implementing acidification permeability enhancement technology in Jiulishan Coal Mine, Jiaozuo mining area.

3. Optimization of Acid Fluid System Based on Orthogonal Experimental Method

3.1. Orthogonal Experimental Design and Results Analysis

Based on the low-field nuclear magnetic resonance experiments conducted on coal samples before and after acidification treatment, this study has preliminarily selected HCL and HF as the suitable acid types for high-rank coal. To achieve the optimal acidification effect for the high-rank coal of Jiulishan Mine in the Jiaozuo mining area, the orthogonal experimental method was employed to optimize the acid fluid system and acidification permeability enhancement parameters. Based on the selected acid types and analysis of factors affecting acidification effectiveness, four factors were chosen: hydrochloric acid concentration (A), hydrofluoric acid concentration (B), solid-liquid ratio (C), and reaction temperature (D), with three levels set for each factor. Nine portions of coal powder with particle sizes ranging from 0.18 mm to 0.25 mm, each weighing 3 g, were prepared. These coal samples were reacted with the acid solutions for 6 hours

under the designed orthogonal experimental conditions. The orthogonal experimental scheme for the four factors and their levels, along with the range analysis of the experimental results, is shown in Table 4.

Table 4: Orthogonal test scheme and range analysis results.

Test number	Influencing factor				Test result
	<i>A</i> /%	<i>B</i> /%	<i>C</i> /（g:mL）	<i>D</i> /℃	Dissolution rate/%
1	5	1	1:10	25	3.108
2	5	2	1:30	30	4.527
3	5	3	1:20	35	4.424
4	8	1	1:30	35	2.928
5	8	2	1:20	25	2.942
6	8	3	1:10	30	4.141
7	11	1	1:20	30	3.395
8	11	2	1:10	35	4.491
9	11	3	1:30	25	3.447
<i>K</i> ₁	12.059	9.431	11.740	9.497	<i>R</i> ₀ = <i>K</i> ₁ + <i>K</i> ₂ + <i>K</i> ₃ =33.403
<i>K</i> ₂	10.011	11.960	10.761	12.063	
<i>K</i> ₃	11.333	12.012	10.902	11.843	
<i>k</i> ₁	4.020	3.144	3.913	3.166	Total average 33.403/9=3.711
<i>k</i> ₂	3.337	3.987	3.587	4.021	
<i>k</i> ₃	3.778	4.004	3.634	3.948	
<i>R</i>	0.683	0.860	0.326	0.855	
Optimal level	<i>A</i> ₁	<i>B</i> ₃	<i>C</i> ₁	<i>D</i> ₂	
Primary and secondary factors	<i>B</i> > <i>D</i> > <i>A</i> > <i>C</i>				
Optimal combination	<i>A</i> ₁ <i>B</i> ₃ <i>C</i> ₁ <i>D</i> ₂				

Note: K_i is the sum of test indexes of all factors at the same level, $i=1,2,3$; k_i is the mean value of test indexes of all factors at the same level, $i=1,2,3$; R_0 is the sum of test indexes of n test schemes, $n=9$; R is the range, which is calculated by subtracting the minimum k_i value from the maximum k_i value of each factor.

From Table 4, based on the magnitude of the R values, the order of influence of the factors on the acidification effect is determined as $B>D>A>C$. This indicates that the hydrofluoric acid concentration has the greatest impact on the experimental index, while the solid-liquid ratio has the least impact. Based on the magnitude of the k_i values, the optimal parameter combination for the acidification effect is $A_1B_3C_1D_2$. This means that when the hydrochloric acid concentration is 5%, the hydrofluoric acid concentration is 3%, the solid-liquid ratio (g:mL) is 1:10, and the temperature is 30°C, the acidification effect on the high-rank coal of Jiulishan Mine in the Jiaozuo mining area is optimal.

3.2. Validation of the Optimal Scheme from Orthogonal Experiment

To verify the reliability of the optimal level combination selected by the orthogonal experimental method, a mixed acid solution with a concentration of 5% hydrochloric acid and 3% hydrofluoric acid was prepared at a solid-liquid ratio (g:mL) of 1:10. This acid solution was reacted with 3 g coal powder samples at 30°C for 6 hours. The dissolution rates from three parallel experiments were 5.53%, 4.677%, and 4.588%, respectively, with an average value of 4.932%. All these values are greater than the dissolution rates of the 9 experiments listed in the orthogonal test table, thus verifying the reliability of the optimized acid fluid system and acidification parameters selected in this study.

4. Conclusion

After acidification, both the effective porosity and NMR permeability of the coal samples increased. The increase in effective porosity and NMR permeability was most significant after acidification with 9%HCL+3%HF. For the high-rank coal of Jiulishan Mine in the Jiaozuo mining area, 9%HCL+3%HF demonstrated the strongest capability to improve the coal pore structure, resulting in the best acidification permeability enhancement effect. Therefore, the optimal acid type selected for implementing acidification permeability enhancement technology in Jiulishan Mine, Jiaozuo mining area, is a mixture of HCL and HF. The order of influence of factors on the high-rank coal acidification effect is: hydrofluoric acid concentration > reaction temperature > hydrochloric acid concentration > solid-liquid ratio. The acid concentrations and acidification permeability enhancement parameters optimized using the orthogonal experimental method are: hydrochloric acid concentration of 5%, hydrofluoric acid concentration of 3%, solid-liquid ratio (g:mL) of 1:10, and reaction temperature of 30°C. Finally, the reliability of the optimized acid fluid system and acidification parameters selected in this study was validated.

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