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Characteristics of Acid Leaching and Vibration Coupling Desorption of Gas-Saturated Coking Coal

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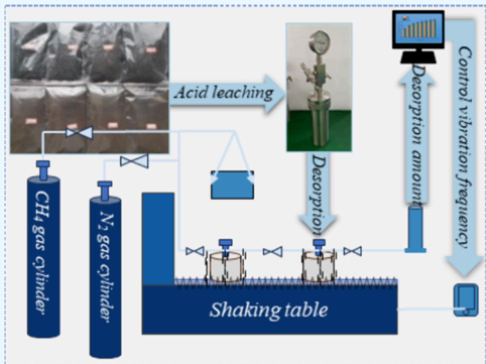
ABSTRACT: Permeability is a key factor affecting efficient gas drainage from coal seams, and acidification and vibration shock are effective means to increase permeability in original low-permeability coal seams. To study the gas desorption characteristics of coking coal under the coupling effect of mining disturbance and acidification permeability enhancement, taking the coal seam of Shoushan No. 1 coal as the research object, a self-built adsorption−desorption vibration test platform was used. Acid leaching vibration coupling desorption experiments at vibration frequencies of 0, 30, 60, and 100 Hz were conducted on selected particle coals with particle sizes of 0.18−0.25 and 1−3 mm. The experimental results show that the gas desorption amount of particle coal with the same particle size first increases and then decreases with the increase of vibration frequency, among which the desorption effect is the best under 60 Hz vibration condition. Under the condition of fixed vibration frequency, the desorption amount, initial desorption velocity, and velocity attenuation coefficient of particle coal increase as the particle size decreases. Under the same particle size and vibration frequency conditions, the acid leaching and vibration of coal samples have a synergistic effect on gas desorption, which is manifested in the promotion of gas desorption on the outer surface of the coal sample and the surface of open macropores. The research can provide theoretical reference for coal seam acidification and permeability enhancement under the influence of mining disturbance.



1. INTRODUCTION

The acidification modification of coal samples can affect the pore structure of coal, the structural parameters of coal surface functional groups, and dissolve inorganic minerals such as clay minerals in coal to promote gas desorption and diffusion, thereby effectively increasing the transparency of coal seams.[1,2] At present, scholars in the industry have done sufficient research on coal seam acidification, acid liquid ratio, and other fields and have been widely used in various fields such as coal mining and gas control.[3,4] The permeability and porosity of acidified coal in different bedding directions are increased,[5,6] and it has varying degrees of impact on the fatty structure, oxygen-containing compounds, and aromatic structure of coal.[7] Research on acidified coal from the perspective of fractal dimension shows that the pore structure characteristics of acidified modified coal have changed significantly.[8] Research on the gas adsorption properties and microscopic mechanism of acid-leached coal samples found that acid leaching can change the adsorption properties of coal samples.[9] Nowadays, acidification modification has become an effective method for underground gas control and coal seam transparency enhancement. However, during the deep mining process, the disturbance caused by mining machinery inevitably affects the desorption of coal seam gas,[10] and its impact cannot be ignored. Research on the desorption characteristics of gas under mechanical disturbance is crucial for coal mining.

Under vibration conditions, cracks in the coal body increase and expand, and the temperature of the coal itself increases to promote gas desorption. At the same time, the "throwing" effect caused by vibration promotes the gas molecules to break away from the surface of the coal body, and the coal body's ability to desorb gas is different under different vibration conditions. Significantly, currently, scholars in related fields have analyzed the gas desorption characteristics under mechanical vibration conditions from different angles.[11−14] Li et al.[15−17] analyzed the impact of low-frequency vibration on the porosity, permeability, and diffusion rate of coal based on self-designed experiments and further revealed the impact mechanism of low-frequency vibration on the volume strain and porosity of gas-containing coal. Ou et al.[18] used a self-developed coal and gas outburst simulation device to simulate underground mine drilling

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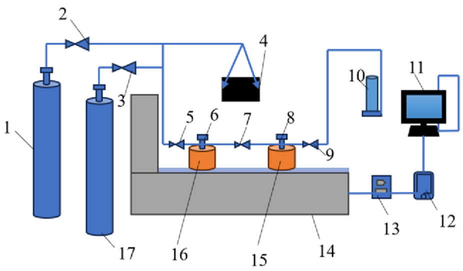

1. N_2 gas cylinder 2/3. Pressure reducing valve 4. Vacuum pump 5/7/9. Stop valve 6/8. Pressure sensor 10. Graduated cylinder 11. Computer 12. Signal generator 13. Power amplifier 14. Vibration table 15. Coal sample tank 16. Reference tank 17. CH_4 gas cylinder

Figure 1. Schematic diagram of the system device.

construction and proposed a coupling transition instability mechanism of coal and gas outbursts induced by drilling construction. Yong[19] believes that, under the condition of fixed vibration frequency, as the vibration time increases, the volume of the coal body gradually shrinks, the pores decrease, and the permeability weakens. Xuexi et al.[20] established a vibration-adsorption−desorption experimental system to explore the impact of mechanical vibration on the gas diffusion characteristics of granular coal. The results show that vibration can increase the gas diffusion amount and diffusion speed of granular coal and have a more obvious impact on small-particle-sized coal samples. Li et al.[21] analyzed the changing characteristics of the natural vibration frequency during the vibration damage process of coal, and rock through experiments, and believed that the acceleration and frequency of dynamic disturbance under the action of vibration are closely related to the decreased rate of the natural vibration frequency of the coal body. Relevant scholars have made certain achievements in studying the physical property changes and desorption characteristics of coal under the influence of vibration. However, experiments rarely provide an acidified environment. The gas desorption characteristics of acidified coal samples under the influence of vibration are bound to be different from those of conventional experimental conditions.

In summary, research on coal desorption under the unilateral influence of acidification or vibration has achieved a certain understanding, but there are few reports on the desorption characteristics of granular coal under the coupling effects of acid leaching and vibration. This paper takes the coal seam of Shoushan No. 1 mine as the research object and uses a self-built adsorption−desorption vibration test bench to carry out desorption experiments on acid-leached coal samples of different particle sizes at different vibration frequencies. This study hopes to reveal the desorption law of particulate coal under the coupling effect of acid leaching and vibration and provide a theoretical basis for improvement of underground gas control methods.

2. EXPERIMENTAL EQUIPMENT AND COAL SAMPLE PREPARATION

2.1. Desorption and Diffusion Experimental System under Acid Leaching-Vibration Coupling. The experimental device uses a self-built adsorption−desorption vibration test bench, which is mainly composed of an adsorption−desorption system, shock vibration system, and data acquisition system. The principle of the device is shown in Figure 1. To fix the coal sample tank and vibration table, in this experiment, a coal sample tank fixing frame by itself was designed, as shown in Figure 2.

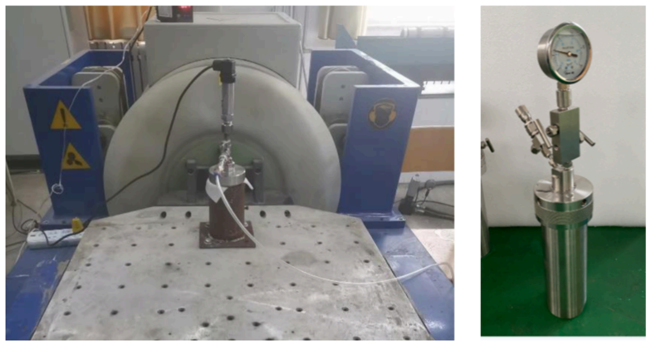

Figure 2. Coal sample tank holder (left) and a coal sample tank (right).

The adsorption−desorption system consists of a gas supply system, pressure regulation system, reference kettle and test kettle, pneumatic control system, pressurization system, temperature-increasing system, and data acquisition and control system.

The shock vibration system consists of three separate parts: the vibration table, power amplifier, and Vibstar controller.

The data acquisition system mainly consists of hardware (computer, printer, and data acquisition card), software (for instrument control and data acquisition and processing functions), and circuit control group layers.

2.2. Coal Sample Selection and Preparation. The coal samples used in this experiment were collected from the coal seams of Shoushan No. 1 mine in Pingdingshan. The coal type was coking coal. The primary structure of the coal was damaged and is granular. The organic components are mainly vitrinite and semivitrinite, and the inorganic components are mainly clay minerals, followed by carbonates. The coal samples used in the experiment were collected from freshly exposed excavation working faces. The full thickness of the coal seam was collected, the gangue was removed, and it was reduced to 1 kg using the quartering method. To prevent the oxidation reaction caused by direct contact between coal and air and the loss of external moisture in the coal sample, the coal sample was sealed and stored immediately after collection.

According to industrial analysis experiments, the coal sample analysis results are listed in Table 1. The maximum reflectance of coal vitrinite is $R_{0,\text{the max}}$ is 1.5%, and the apparent density is 1.58 t/m^3.

The following steps to process the collected coal samples were followed below.

2.2.1. Preparation of the Coal Sample for the Isothermal Desorption Experiment.

Table 1. Industrial Analysis Parameters of Coal

	adsorption parameters		analyze results		
sample	a (m^3/t)	b (m^3/t)	M_{ad} (%)	A_{ad} (%)	V_{ad} (%)
coking coal	22.818	0.972	0.87	13.29	20.8

1. All collected coal samples are crushed through a pulverizer, and coal samples with particle sizes of 1−3 and 0.18−0.25 mm are screened out and placed in drying trays, as shown in Figure 3.

Figure 3. 1−3 mm (top) and 0.18−0.25 mm (bottom).

2. The drying tray containing the coal sample was put into the drying box, adjusted the temperature to 100 °C, kept for 1 h, and then taken out, and it was put into the desiccator to cool to room temperature.

2.2.2. Granular Coal Acid Infiltration Process. As shown in Figure 4, the acid-infiltrated granular coal sample was prepared by using the MesoMR23-060H-I vacuum device of the Key Laboratory of Gas Geology and Gas Control of Henan Polytechnic University for negative pressure infiltration treatment.

Figure 4. MesoMR23-060H-I vacuum device.

1. The prepared coal sample was placed into a beaker containing 5% hydrochloric acid solution and then placed in a sealed glass cover of a vacuum saturation device.
2. The experimental temperature was set to 60 ± 1 °C, turned on the vacuum pump, waited until the vacuum gauge indication stabilizes, turned off the vacuum pump, and performed negative pressure infiltration treatment.
3. After 12 h, the airtight glass cover was opened; the soaked coal sample was washed, filtered, and dried until the pH test paper shows neutral.
4. The processed coal sample was put into a drying box, adjusted the temperature to 60 °C, kept for 4 h, taken out, put in a sample bag, and then cooled to room temperature for storage until use.

3. RESULTS AND DISCUSSION

3.1. Acid Leaching-Vibration Coupling Effect of the Coal Gas Desorption Experiment of Different Particle Sizes. Coal samples were selectedwith a particle size of 1−3 mm, divided into two groups of 200 g each, and then numbered. One group is randomly selected for infiltration treatment with 5% hydrochloric acid, while the other group is left untreated and stored after drying. The two groups of prepared coal samples was put into coal sample tanks, respectively, and high-pressure nitrogen was used to check the air tightness of the experimental system. After passing the inspection, the coal samples were degassed again to remove impurity gases in the coal sample tanks and coal samples. The temperature of the constant temperature water bath was adjusted to 300 °C. After degassing the coal sample, the coal sample tank was filled with 1.0 MPa gas for constant pressure adsorption. After 10 h of adsorption, the adsorption equilibrium is reached. Desorption experiments were conducted under vibration conditions of 0, 30, 60, and 100 Hz, and the changes in desorption amount during the experiment were recorded.

The gas desorption rules of untreated coal samples and treated coal samples under different vibration frequencies are listed in Figure 5.

As can be seen from Figure 5, the gas desorption amount of the coal sample after hydrochloric acid infiltration is significantly reduced. This is due to the decrease in the adsorption capacity of the acidified modified coal sample, and the desorption amount of the untreated coal sample is also significantly reduced in the same time period. At the same time, the gas desorption effect of the two coal samples is optimal under the vibration condition of 60 Hz. Therefore, the following article focuses on analyzing the desorption characteristics of particle coal with particle sizes of 1−3 and 0.18−0.26 mm under acid leaching vibration coupling under 0 and 60 Hz vibration conditions.

3.2. Analysis of Experimental Results at 0 and 60 Hz Vibration Frequencies. Figure 6 shows that, under the same vibration conditions of untreated coal and hydrochloric acid-infiltrated coal, coal samples with large particle sizes have a small total gas desorption amount. For untreated coal samples, the gas desorption amounts of 1−3 mm particle coal without vibration and with a vibration frequency of 60 Hz are 2.50 and 2.67 mL/g, respectively. The gas desorption amounts of 0.18−0.25 mm particle coal without vibration and with a vibration frequency of 60 Hz are 2.76 and 2.98 mL/g, respectively. For hydrochloric

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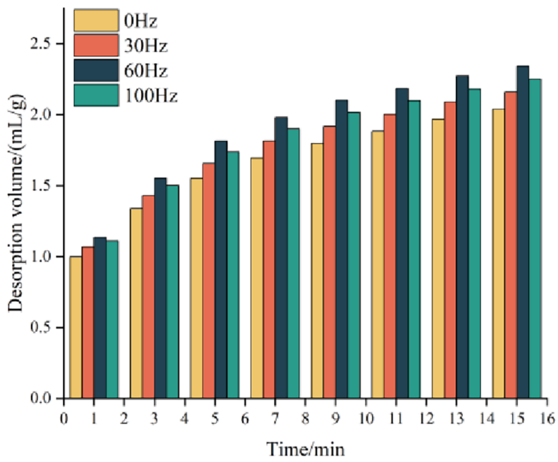
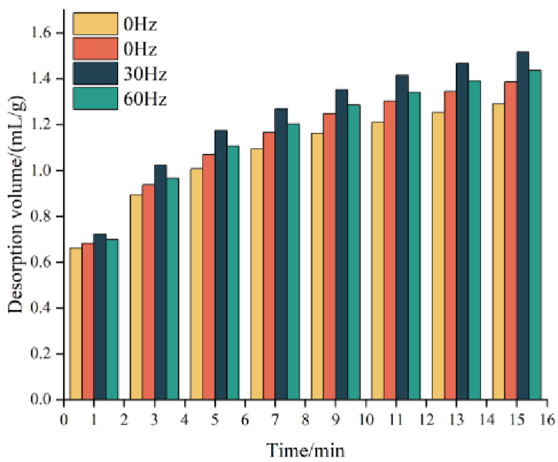


a. Changes in desorption amount of untreated coal gas



b. Changes in desorption amount of hydrochloric acid infiltrated coal gas

Figure 5. Histogram of gas desorption changes under different vibration frequencies.

acid-infiltrated coal, the gas desorption amounts of 1−3 mm particle coal without vibration and with a vibration frequency of 60 Hz are 1.63 and 1.75 mL/g, respectively. The gas desorption amounts of 0.18−0.25 mm particle coal without vibration and with a vibration frequency of 60 Hz are 1.82 and 2.01 mL/g, respectively. It can be seen that, when no vibration is added, the gas desorption amount changes slightly with the change in coal sample particle size. After vibration is applied, the desorption amount changes greatly as the particle size of the coal sample changes.

Figure 6 shows that the gas desorption rate gradually decreases with an increase of time. In the early stage of desorption, the gas desorption rate decreased rapidly. After 10 min, the gas desorption rate decreased and stabilized until it became stable. Under the same vibration conditions, the desorption speed of coal samples with large particle sizes in the early stage of desorption is significantly slower than that of coal samples with small particle sizes. The desorption rate and time models are listed in Tables 2 and 3.

It can be seen from Tables 2 and 3 that, under the same conditions, the initial gas desorption velocity and attenuation coefficient of coal samples with small particle sizes are large. When the particle size of the coal sample remains unchanged, vibration increases the initial velocity and attenuation coefficient of gas desorption in the coal sample. For untreated coal samples, when no vibration is added, when the particle size of the coal sample decreases from 1−3 to 0.18−0.25 mm, the initial gas desorption velocity increases by 11.10%, and the attenuation coefficient increases by 1.18%. When 60 Hz vibration is applied when the coal sample particle size decreases from 1−3 to 0.18−0.25 mm, the initial gas desorption velocity increases by 13.08%, and the attenuation coefficient increases by 1.89%. Similarly, for hydrochloric acid-infiltrated coal samples, without vibration, when the particle size of the coal sample decreases from 1−3 to 0.18−0.25 mm, the initial gas desorption velocity increases by 13.64%, and the attenuation coefficient increases by 1.54%. When 60 Hz vibration is applied when the coal sample particle size decreases from 1−3 to 0.18−0.25 mm, the initial gas desorption velocity increases by 17.65%, and the attenuation coefficient increases by 1.62%. Under vibration conditions, the initial gas desorption velocity and attenuation coefficient increase more significantly, the particle size is inversely proportional to the initial gas desorption velocity and attenuation coefficient, and the increase is more obvious.

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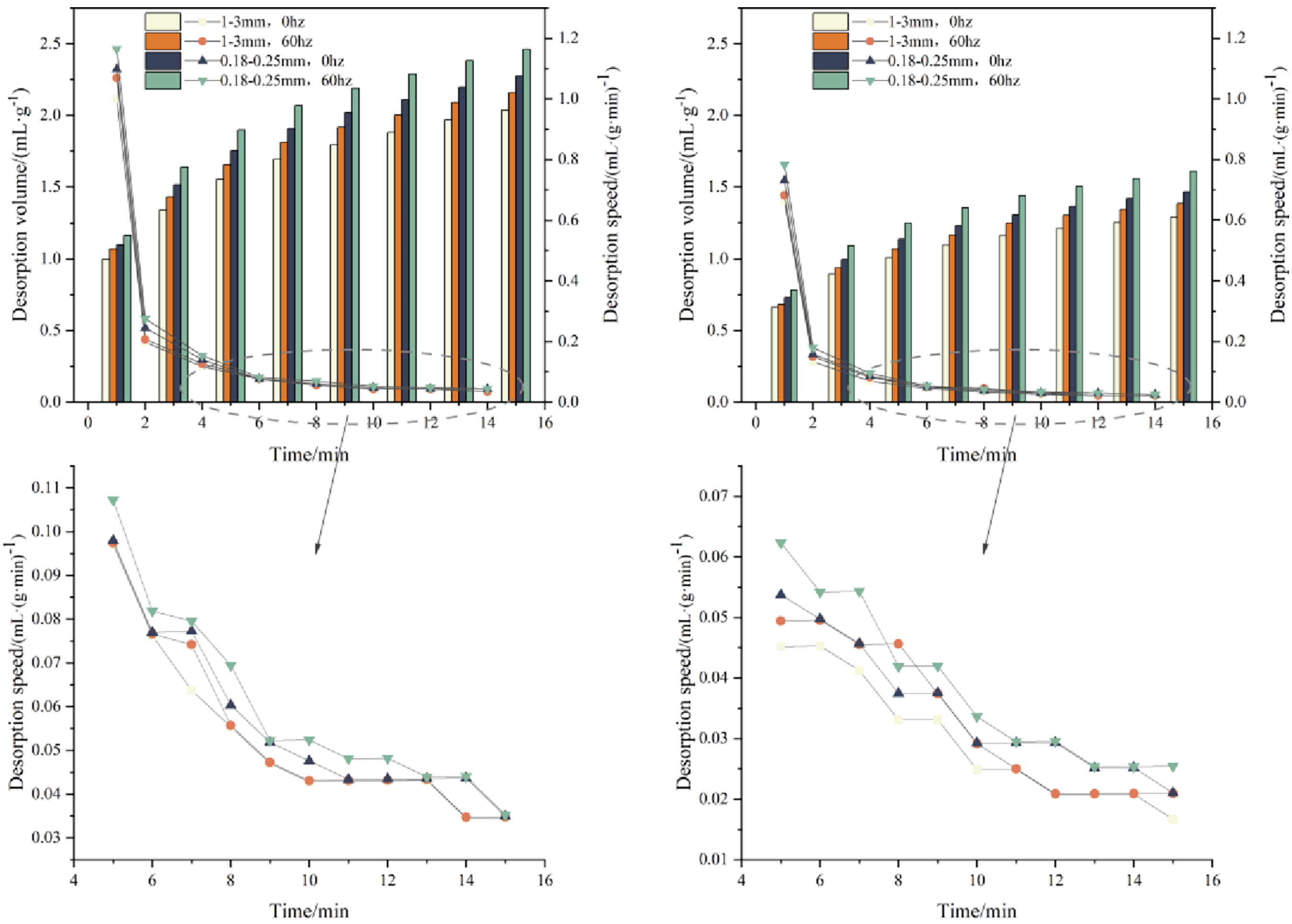

a. Desorption amount and rate of different particle sizes of untreated coal samples

b. Desorption amount and rate of different particle sizes after acid leaching treatment

Figure 6. Change curve of gas desorption amount of coal samples with different particle sizes.

Table 2. Fitting Equations for Gas Desorption Rates of Untreated Coal Samples with Different Particle Sizes

coal sample particle size/mm	vibration frequency/Hz	q_0/mL*(min.g)$^{-1}$	attenuation coefficient	fitting equation	R^2
1−3	0	0.99	1.052	$V = 0.99(1 + t)^{-1.052}$	0.8265
	60	1.07	1.056	$V = 1.07(1 + t)^{-1.056}$	0.8255
0.18−0.25	0	1.10	1.071	$V = 1.10(1 + t)^{-1.071}$	0.8382
	60	1.21	1.076	$V = 1.21(1 + t)^{-1.076}$	0.8459

Table 3. Fitting Equations for Gas Desorption Rates of Hydrochloric Acid-Infiltrated Coal Samples with Different Particle Sizes

coal sample particle size/mm	vibration frequency/Hz	q_0/mL*(min.g)$^{-1}$	attenuation coefficient	fitting equation	R^2
1−3	0	0.66	1.040	$V = 0.66(1 + t)^{-1.040}$	0.8214
	60	0.68	1.049	$V = 0.69(1 + t)^{-1.049}$	0.8351
0.18−0.25	0	0.75	1.056	$V = 0.75(1 + t)^{-1.056}$	0.8307
	60	0.80	1.066	$V = 0.80(1 + t)^{-1.066}$	0.8396

Treating granular coal as isotropic homogeneous spherical particles, the analytical solution of the "Fick diffusion" model is obtained:

$$\frac{Q_t}{Q_\infty} = 1 - \frac{6}{\pi^2}\sum_{i=1}^{\infty}\frac{1}{i^2}\exp\left(\frac{-D_{Fi}{}^2\pi^2 t}{r_0{}^2}\right) \quad (1)$$

In the formula, Q_t is the coal particle gas desorption amount at time t, mL; Q_∞ is the coal particle gas desorption amount at time $t \to \infty$, mL; r_0 is the coal particle radius, mm; and D_F is the diffusion coefficient, m^2/s.

Q_t/Q_∞ can be regarded as the relative desorption rate of gas within t time. When the t value is small, it can be simplified to

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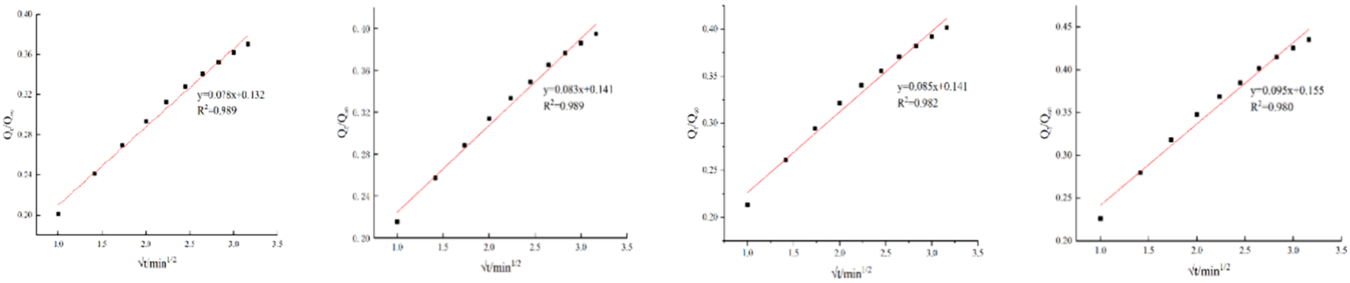

Figure 7. Fitting curve of desorption rate of untreated coal.

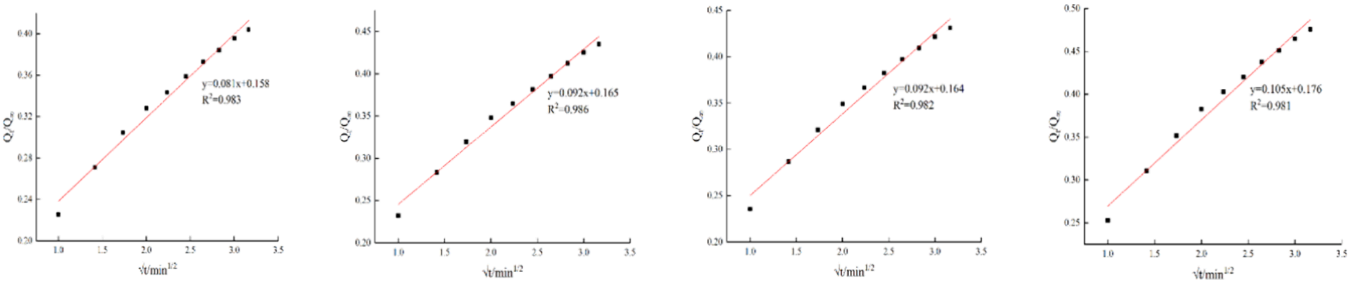

Figure 8. Fitting curve of the desorption rate of hydrochloric acid-infiltrated coal.

$$\frac{Q_t}{Q_\infty} = \frac{6}{r_0}(D_F t/\pi)^{0.5} \tag{2}$$

Let $k = \frac{6\sqrt{D}}{r_0\sqrt{\pi}}$, then $D = \frac{k^2 r_0^2 \pi}{36}$, the above formula can become:

$$\frac{Q_t}{Q_\infty} = k\sqrt{t} \tag{3}$$

In the desorption−diffusion model, the gas desorption amount includes the desorption amount Q_0 on the outer surface of the coal and the open macropore surface and the diffusion amount Q_t in the internal pores. However, eq 3 contains only the diffusion amount Q_t and cannot reflect the change of the desorption rate with time. The improved formula is as follows:

$$\frac{Q_t}{Q_\infty} = k\sqrt{t} + Q_0 \tag{4}$$

The relationship between Q_t/Q_∞ and $\sqrt{t}$ within 10 min before desorption is shown in Figures 7 and 8. The k and Q values of coal samples with different particle sizes under the influence of acid leaching vibration coupling are shown in Table 4.

Q_0 is the desorption amount on the outer surface of the coal sample and the open macropore surface. Figure 9a shows that the Q_0 values of coal samples with particle sizes of 0.18−0.25 and 1−3 mm increased by 25.53 and 19.95%, respectively, under the influence of acid leaching vibration coupling. This shows that acid leaching-vibration coupling can effectively increase the desorption amount on the outer surface of coal samples and open macropore surfaces and is inversely proportional to the particle size. The Q_0 values of raw coal and acid-leached-vibrated coal samples decreased by 8.24 and 12.38%, respectively, as the particle size increased, further proving that the particle size is inversely proportional to the desorption amount on the outer surface and open macropores, and the acid leaching-vibration effect is more obvious.

The k value is proportional to the initial effective diffusion coefficient, D_F, which reflects the initial desorption capacity of the coal sample. In Figure 9b, the k values of coal samples with particle sizes of 0.18−0.25 and 1−3 mm increased by 24.82 and 25.00%, respectively, under the influence of acid leaching vibration coupling, which confirms that acid leaching-vibration coupling conditions can improve the initial desorption capacity of coal, regardless of particle size.

Acid leaching-vibration coupling has a greater impact on the desorption amount on the outer surface of coal and open macropore surfaces but has a smaller impact on the initial desorption capacity.

Table 4. Initial Effective Diffusion Coefficient of Coal Samples under Different Conditions

coal sample	particle size/mm	vibration frequency/Hz	k	Q_0
untreated coal	1−3	0	0.078	0.132
		60	0.083	0.141
	0.18−0.25	0	0.085	0.141
		60	0.095	0.155
acid-leached coal	1−3	0	0.081	0.158
		60	0.092	0.165
	0.18−0.25	0	0.092	0.164
		60	0.105	0.176

4. CONCLUSIONS

1. Under shock and vibration conditions, acid-leached coal has a greater gas desorption amount and desorption speed than raw coal. At the same vibration frequency, as the particle size decreases, the gas desorption amount of acid-leached coal increases more than that of raw coal, confirming that acidification is beneficial to the diffusion and desorption of coal seam gas.
2. The vibration effect can increase the gas desorption rate of gas-containing raw coal and acid-leached coal, and the desorption efficiency is maximum at a vibration frequency of 60 Hz.

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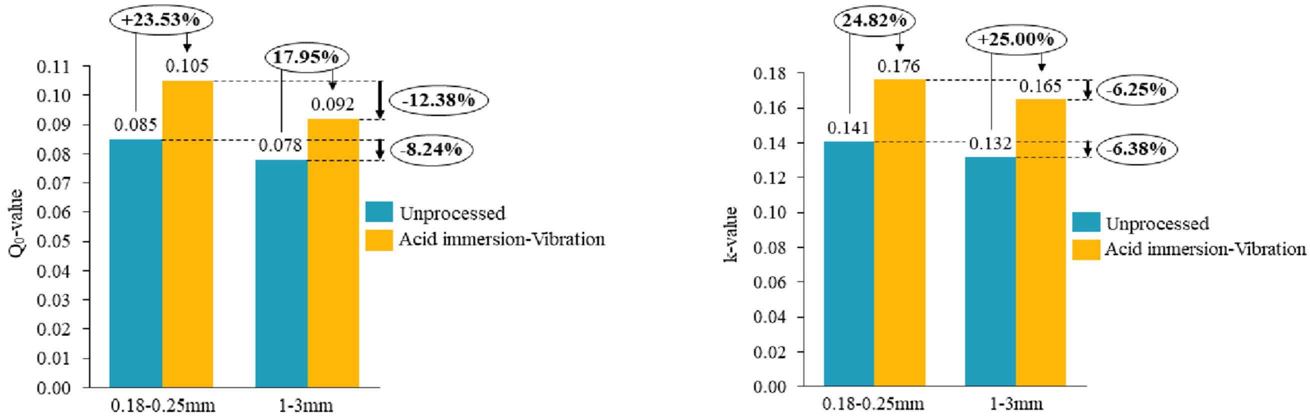

a. Q_0 values of different particle sizes under 60Hz vibration frequency

b. k values of different particle sizes at 60Hz vibration frequency

Figure 9. Q_0 value and k value of different particle sizes at 60 Hz vibration frequency.

3. The acidification-vibration coupling effect directly promotes the reaction of carbonate minerals in coal particles, which enhances pore connectivity and thereby increases the desorption rate of particle coal. The smaller the particle size, the larger the specific surface area of coal, and the faster the desorption rate of coal after acidification.

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Author Contributions

R.L.: conceptualization, methodology, visualization, formal analysis, funding acquisition, supervision, writing-original Draft, writing-review and editing. S.Z.: conceptualization, methodology, visualization, data curation, formal analysis, writing-original draft. L.Z.: methodology, data Curation, formal analysis. H.G.: methodology, data curation, formal analysis. L.J.: methodology, data curation, formal analysis. H.L.: methodology, data curation, formal analysis.

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