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Research Article

Study on the Influence of Liquid Nitrogen Cold Soaking on the Temperature Variations and Seepage Characteristics of Coal Samples with Different Moisture Contents

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Due to its advantages such as environmental friendliness and remarkable permeability enhancement effect, the technology of liquid nitrogen cold soaking (LNCS) cracking coal has become a hot spot in the research on coal seam permeability enhancement in recent years. The frost heave force generated by water-ice phase transformation and the temperature stress are the main mechanisms of LNCS cracking water-containing coal. This paper focuses on the effect of LNCS on the temperature variations and seepage characteristics of coal. To further this purpose, the temperature measurement test and the permeability test were conducted on coal samples with different moisture contents under LNCS, respectively. In addition, by comparing the computer tomography test results of coal samples before and after LNCS, the internal pore structure changes of coal samples were further analyzed from a three-dimensional perspective. The test results show that the coal sample with a higher moisture content consumes a shorter time to reach internal temperature equilibrium and experiences faster temperature changes. LNCS can enhance coal permeability, and the growth rate of permeability increases exponentially with the increase of moisture content. After the LNCS treatment, the dried coal sample is mainly sprouting new pores on the basis of primary pores; in contrast, for water-containing coal samples, new pores are sprouted while primary pores will penetrate each other spatially to form a fracture network. In the process of LNCS, moisture has a significant effect on the seepage characteristics of coal, so appropriately increasing the moisture content of the coal seam conduces to achieving a better permeability enhancement effect.

1. Introduction

Coalbed methane (CBM), an associated energy source with coal, is a clean fuel and chemical raw material [1]. Coal is generally considered to be composed of a coal matrix and a natural crack network. The low permeability of coal seams directly restricts the efficient extraction of CBM [2–4]. Therefore, auxiliary methods are needed to enhance coal seam permeability. The liquid nitrogen cracking technology is a green and efficient waterless cracking technology which enhances coal seam permeability by injecting large amounts of low-temperature liquid nitrogen into the coal seam through surface drilling or downhole drilling. When used as the cracking fluid, liquid nitrogen boasts the advantages of no pollution, low cost, and easy preparation. It can not only effectively solve the problems of waste of massive water resources, water lock, water sensitivity, etc., but it can also improve the cracking effect [5–7].

n the low-temperature environment, the temperature change of coal rock is a very important research point, and some have conducted related studies on the temperature variation of rock-soil mass under the effect of low-temperature environment. Liu et al. [8] established a microscopic structural model of frozen rock using the digital image processing technique and converted the model into a finite element physical model by means of geometric vector conversion. Based on the converted model, they analyzed the temperature field distribution law during freeze-thaw cycles of inhomogeneous frozen rock under negative temperature conditions. By adopting the research methods of numerical simulation and experimental research, Xiong et al. [9] analyzed the basic law of heat transfer in frozen soil under freeze-thaw cycles based on the influence mechanism of heat conduction and latent heat of phase transformation on temperature, and studied the characteristics of temperature propagation in frozen soil after construction. Tan et al. [10] investigated the temperature field variation of tunnel-surrounding rock mass in a cold region by using a combination of numerical simulations and engineering field measurements, and they found that the latent heat released by the water-ice phase transformation process can change the temperature field of rock and soil porous media by changing the rate of ice formation. Park et al. [11] investigated the variation of thermophysical parameters of rocks with ambient temperature by physical parameter testing experiments. The results showed that when the temperature was reduced from 40°C to -160°C, the coefficient of thermal expansion and specific heat of rocks decreased with decreasing temperature, while the thermal conductivity changed less significantly with decreasing temperature. The above researches gave an insight into the variation pattern of the internal temperature of coal rock under the effect of a low-temperature environment. However, previous researches on coal rock temperature field variations under low-temperature freezing conditions were mostly conducted by means of numerical simulation, and few experimental studies in this field have been reported.

During liquid nitrogen cold soaking (LNCS) on coal rock, the temperature stress generated by the temperature gradient, the expansion stress generated by liquid nitrogen vaporization, and the freezing and swelling force generated by the volume expansion of water-ice phase transformation will all promote fracture generation, development, and connection, thus enhancing coal rock permeability. Cai et al. [12] tested permeability changes of a coal sample before and after liquid nitrogen treatment under the conditions of 0.25 MPa air pressure and 2.25 MPa surrounding pressure. In addition, considering the effect of moisture content, a series of liquid nitrogen permeability enhancement techniques have been proposed. Li et al. [13, 14] investigated the effect of LNCS on the permeability and pore structure of coal rock, and they found increases in the permeability, porosity, and pore radius of coal rock treated with LNCS. Yang et al. [15] treated saturated and dried coal samples under -10°C, -25°C, -40°C, and LNCS conditions, respectively, and tested their permeabilities. The results revealed that the coal sample experienced more significant permeability variations under LNCS conditions than under -10°C, -25°C, and -40°C conditions. Cai et al. [16, 17] conducted NMR analysis on rock samples that had been frozen with liquid nitrogen and found that the freezing effect of liquid nitrogen caused fracture expansion and porosity and permeability enhancement of the samples. By conducting NMR tests on coal samples before and after LNCS, Wei et al. [18] found that the porosity and pore radius of coal increased with the increase of water content saturation. This finding indicated that pore water played a crucial role in coal; besides, the higher the water saturation, the more obvious the damage characteristics of coal after LNCS. The above studies show that LNCS freezing promotes the pore development and seepage characteristics of coal rocks, but there is a lack of studies on the effects of LNCS on the seepage characteristics of coal with different moisture contents.

At present, the influence of moisture content on the coal cracking effect of LNCS has been extensively researched on. However, most of the researches are focused on the effect of moisture content on pore structure variations before and after LNCS cracking, and crack variations are often characterized by parameters such as sound velocity and crack expansion [19–23]. The influences on temperature changes and seepage characteristics in coal are rarely considered. In this paper, the influences of LNSC on the temperature changes and seepage characteristics of coal samples with different moisture contents were explored. First, coal samples with different moisture contents were prepared. Next, a series of tests related to LNCS were carried out on the coal samples. Furthermore, the temperature variations and permeability characteristics of coal are analyzed and discussed on the basis of the experimental results. The research results are beneficial for the improvement of coal permeability and the enhancement of CBM extraction efficiency.

2. Experimental Methods and Sample Preparation

2.1. Experimental Facilities. The temperature measurement test was conducted with the aid of a temperature measurement device independently developed by the laboratory, as shown in Figure 1. The permeability test was conducted with the aid of a laboratory coal rock triaxial creep-seepage test system (Figure 2) which consisted of a high-pressure gas cylinder, a clamping device, a flow rate acquisition device, an axial pressure and confining pressure loading device, and a constant-temperature device. The observation experiment of the development and expansion of the internal cracks in the coal samples used a laboratory coal and rock industrial CT scanning system (Figure 2).

2.2. Preparation of Test Samples. The test coal samples were anthracite taken from the Zhujiao Coal Mine of Henan Energy and Chemical Industry Group Co., Ltd., Anyang, China. The size of the coal samples used for the metrology measurement test was $\Phi 50$ mm × 100 mm. The basic parameter information of the coal samples is shown in Table 1. Coal samples for temperature measurement tests require internal temperature collection through temperature measurement boreholes. Please refer to the previous work for detailed processing information of experimental sample preparation for temperature measurement [24]. The prepared coal samples are shown in Figure 3. The coal samples with different

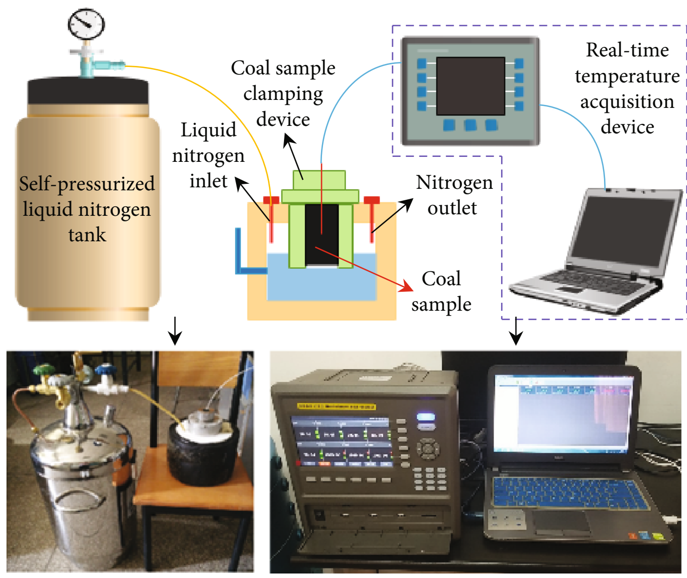


Figure 1: Temperature measurement test device.

Figure 2: Experimental overall flowchart.

Table 1: Basic parameters of coal samples.

Sample	TRD (g·cm^{-3})	ARD (g·cm^{-3})	M_{ad} (wt%)	A_{ad} (wt%)	V_{daf} (wt%)	FC_{ad} (wt%)
Coking coal	1.45	1.33	0.74	8.25	20.21	70.09

Note: TRD represents true density; ARD represents apparent density; M_{ad} represents moisture, air-drying base; A_{ad} represents ash yield, air-drying base; V_{daf} represents volatile matter, dry ash-free basis; FC_{ad} represents fixed carbon content.

moisture contents used in the test were prepared in accordance with the following procedure.

(1) The prepared coal sample was placed in the drying oven whose temperature was set to 70°C to be dried for over 48 h. The mass of the coal sample after drying was recorded as m_d

(2) The coal sample was subjected to vacuum water saturation treatment through a water saturation device, during which it was taken out to be weighed every 12 h until its mass no longer increased. In this case, the coal sample was considered to be saturated. At this time, the mass of the water-saturated coal sample was recorded as m_s

FIGURE 3: Test coal samples.

TABLE 2: Permeability test conditions.

Axial pressure (MPa)	Confining pressure (MPa)	Pressure at the high-pressure seepage end (MPa)	Pressure at the low-pressure seepage end (MPa)	Seepage gas
1	2	0.45	0.1	N_2

(3) The saturated coal sample was put into the drying oven, during which it was constantly taken out to be weighed. The time for weighing was adjusted according to actual needs. When reaching the target drying mass, the coal sample was taken out and immediately put in a sealing bag to cool to room temperature for later use. The preset water saturation degrees were 0%, 20%, 40%, 60%, 80%, and 100%, respectively. The target drying mass was calculated by $m = s_t(m_s - m_d) + m_d$, where m is the target drying mass, s_t is the preset water content saturation, and m_s is the mass of the saturated coal sample

(4) The above steps were repeated to prepare coal samples with different water saturation degrees. The drying oven and the vacuum water saturation device are shown in Figure 2

2.3. Experimental Process

2.3.1. Temperature Measurement Test. First, liquid nitrogen was injected into the heat preservation tank through the self-pressurized liquid nitrogen tank, and the air outlet port was kept open. The liquid nitrogen interface was kept constant by controlling the amount of liquid nitrogen injected in line with the amount of liquid nitrogen vaporized. Meanwhile, the internal measurement point temperature of the coal sample was acquired in real time at an interval of 1 min. During this period, liquid nitrogen was intermittently injected into the sample container, and the liquid nitrogen interface should always just submerge to the bottom of the coal sample. When the temperature change at the measuring point became less than 0.05°C/min, the internal temperature of the coal sample was considered to have reached equilibrium stability, and the data acquisition could stop. Finally, the coal sample must be taken out. During the coal sample temperature measurement, silicone rubber was applied to the bottom part of the sample holder in contact with the coal sample in order to prevent the low-temperature nitrogen gas generated by liquid nitrogen vaporization from penetrating the wall of the sample and interfering with the experiment

2.3.2. Permeability Test. The permeability tests of coal samples with different moisture contents before and after LNCS were carried out through the laboratory coal rock triaxial creep-seepage test system (sample size: Φ50 mm × 100 mm). In order to exclude the influence of the temperature measurement borehole on the native pore structure of the coal samples, the permeability test coal samples were left untreated. In addition, the experimental conditions during the liquid nitrogen cold soaking were the same as the temperature measurement tests. The permeability test conditions are listed in Table 2

The flow rate data acquired in the seepage experiment are used to calculate the permeabilities of samples:

$$k = \frac{2p_0 \mu L Q}{A\left(p_1^2 - p_2^2\right)}, \tag{1}$$

where k is the permeability of the coal sample (mD); p_0 is the atmospheric pressure (0.1 MPa); μ is the nitrogen dynamic viscosity (10^{-6} Pa·s); L is the length of the coal sample (cm); Q is the average flow rate of gas permeating the coal sample (cm^3/s); A is the size of the bottom and top area of the coal sample (cm^2); p_1 and p_2 are the pressures at the inlet and the outlet, respectively (MPa).

The specific test steps are as follows: (1) The permeabilities of all coal samples were measured by the triaxial seepage test system. Each coal sample was measured 3 times, and the calculated average value was taken as its permeability before LNCS. (2) After the initial permeability test was completed,

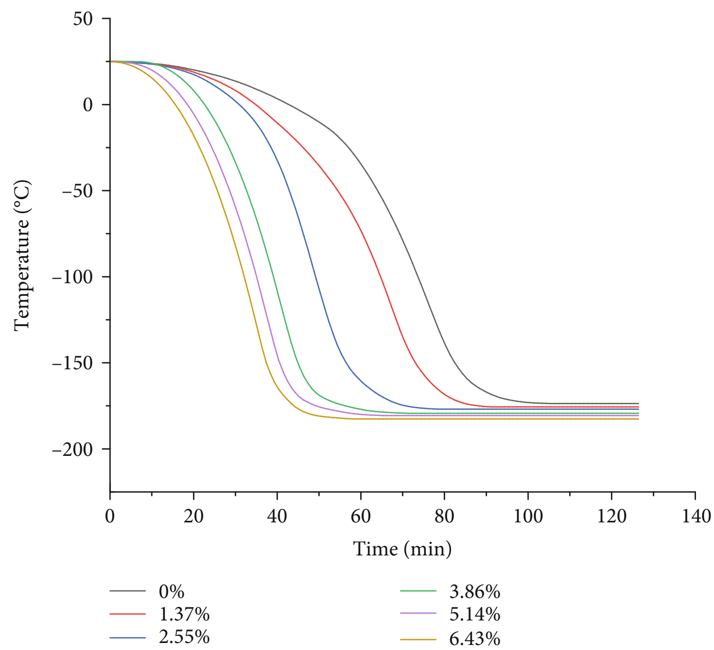


Figure 4: Temperature change curve of internal measurement points of coal samples with time.

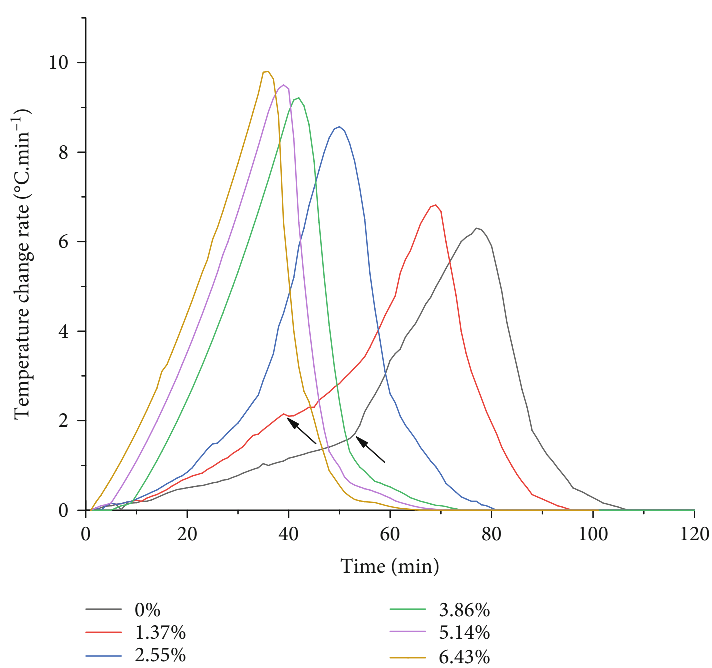


Figure 5: Temperature change rate curve of internal measurement points of coal samples with time.

the LNCS temperature measurement system was employed to perform LNCS treatment on coal samples, and the treatment time was the same the temperature measurement time. (3) After completion of LNCS temperature measurement, the permeability of the coal sample was measured after it returned to room temperature.

2.3.3. Micro-CT Scanning Test. LNCS treatment was carried out on coal samples through the liquid nitrogen temperature measurement system (the test conditions were the same as the temperature measurement test), and the samples were scanned before and after LNCS, respectively. The resolution of industrial CT scanning is related to the size of the sample, and the smaller the size, the more accurate the CT scanning. To improve the scanning accuracy, small size coal samples ($\Phi 50\,\text{mm} \times 100\,\text{mm}$) were used to observe the changes of pore structure. The CT image reconstruction and processing method is as follows: CT data reconstruction software was used to define the sample CT reconstruction area, the reconstruction parameters are set, beam hardening correction is

Table 3: Contrast of permeability of coal samples before and after treatment.

Sample	Moisture content (%)	Water saturation (%)	Average permeability (mD) Before LNCS	Average permeability (mD) After LNCS	Permeability increase rate (%)
J_1	0	0	0.1365	0.2406	76.26
J_2	1.37	20	0.1432	0.3214	124.44
J_3	2.55	40	0.1392	0.4403	216.31
J_4	3.86	60	0.0949	0.3869	307.70
J_5	5.12	80	0.1288	0.6979	441.85
J_6	6.43	100	0.0828	0.6492	684.06

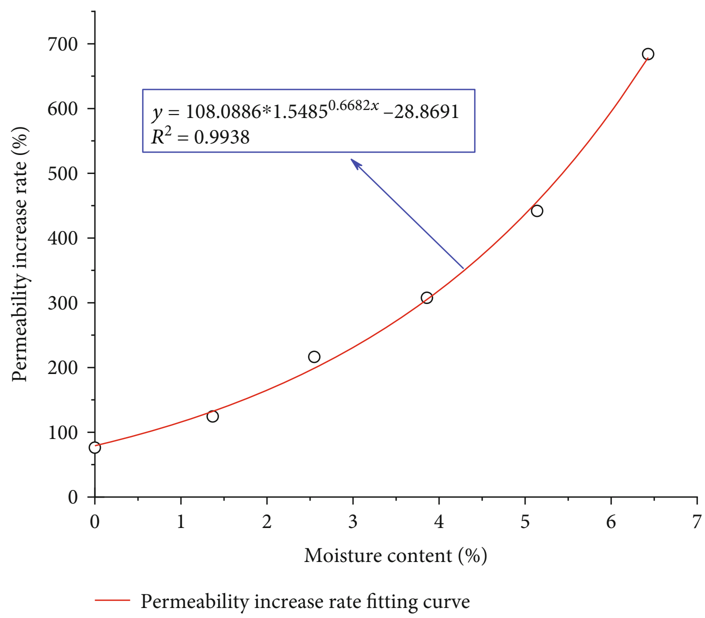


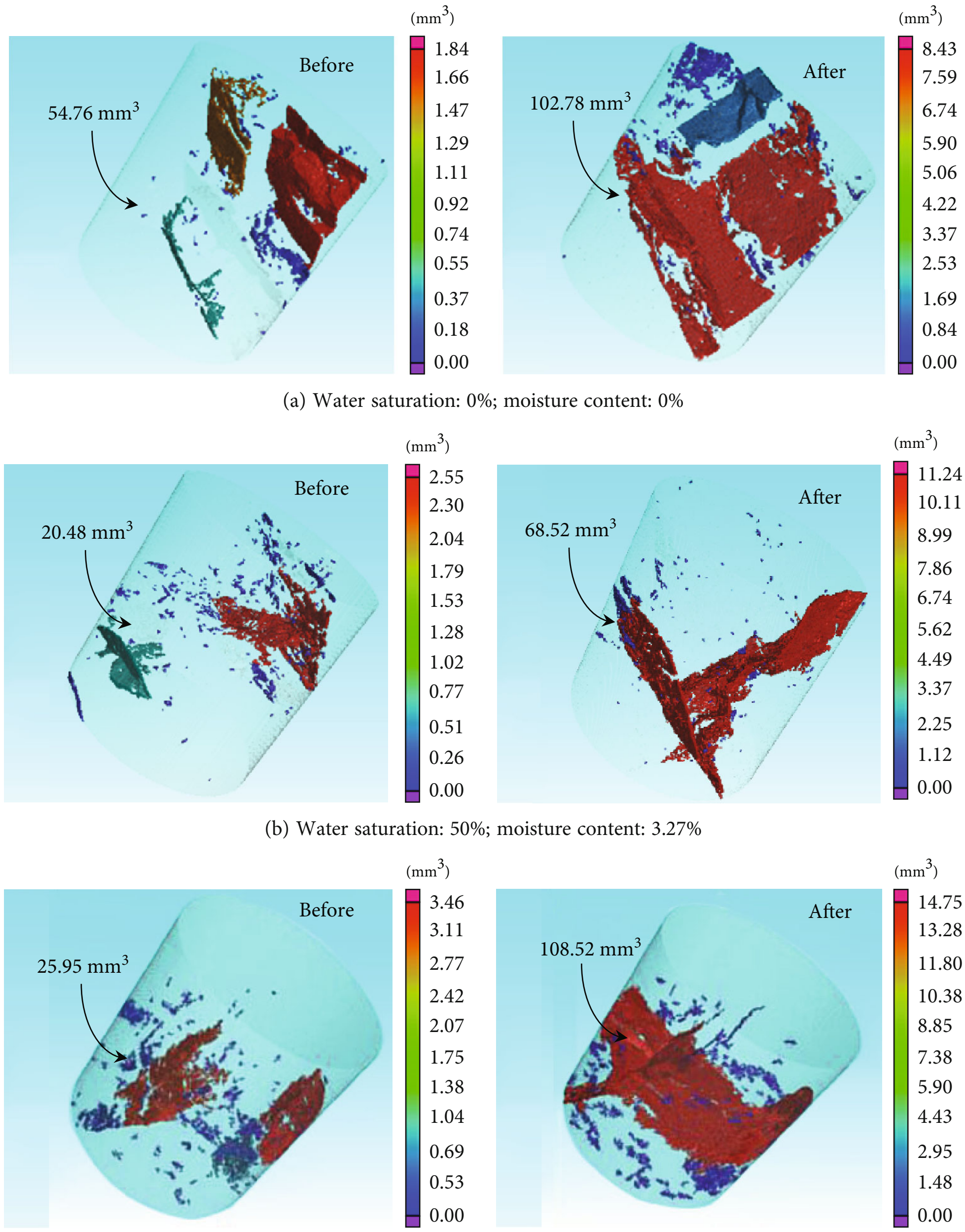
Figure 6: Fitting curve of permeability increase rates of coal samples after LNCS.

performed, automatic geometry correction is performed, and artifact compensation on the acquired raw images is performed to obtain better imaging quality. After the image reconstruction was completed, the image analysis was performed using VGStudio MAX image processing software to extract the internal fracture information of the coal samples at different scanning points and analyze them to obtain the pore structure changes inside the coal samples. The experimental flowchart is given in Figure 2

3. Results and Discussion

3.1. Effect of LNCS on the Temperature Variation of Coal. To disclose the effect of LNCS on the temperature variations within coal under different moisture content conditions and further explore the effect of moisture on the temperature variation of coal under LNCS, the experiment of temperature measurement of a coal sample under LNCS was carried out. Based on the results of the temperature measurement test, the temperature change curves of coal samples with different moisture contents under LNCS conditions were obtained (Figure 4).

As can be seen in Figure 4, the dried coal sample consumes about 110 min to reach temperature equilibrium at the internal measurement point, while the saturated coal sample only consumes 65 min. Besides, the larger the moisture content of a coal sample, the shorter the time it consumes to reach temperature equilibrium at the internal measurement point. As shown in Figure 5, for the coal sample with a higher moisture content, its temperature change rate requires a shorter time to reach the peak value and the peak value is larger. Considering the water-ice phase transformation aspect, when the coal sample is exposed to liquid nitrogen, an ultra-low-temperature medium, the pore water inside it will quickly freeze into ice. Since the thermal conductivity changes suddenly after water turns into ice, it affects the variation of the internal temperature of coal. As early as 1958, Hobbs obtained the following conclusion about the heat conduction between ice and water by summarizing previous experiments: when the temperature drops to 0°C, the thermal conductivities of ice and water are 2.2 W/m°C and 0.58 W/m°C, respectively. In addition, the thermal conductivity of ice will continues to increase continuously as the ambient temperature decreases [25].



(a) Water saturation: 0%; moisture content: 0%

(b) Water saturation: 50%; moisture content: 3.27%

(c) Water saturation: 100%; moisture content: 6.59%

Figure 7: 3D views of coal samples.

Since liquid nitrogen is an ultra-low-temperature medium, it has a greater influence on the thermal conductivity of the ice inside coal. Therefore, the coal sample with 6.43% water content corresponds to the highest temperature change rate. In Figure 5, sudden change points (marked by the arrows) occur on the temperature change curves of coal samples with 0% and 1.37% moisture contents, which agrees with the results of Li et al. The reason for such a phenomenon is that the infiltration of low-temperature nitrogen into the internal temperature measurement points of the coal samples accelerates temperature variation [24].

3.2. Effect of LNCS on the Permeability Changes of Coal. Coal is a porous medium containing pores and fractures instead of an idealized continuous medium [26, 27]. When coal is frozen by liquid nitrogen, the difference in thermal conductivities will produce uneven temperature stress, and the phase transformation effect of pore water will cause volume expansion, which will induce the expansion and connectivity of pores in coal. To investigate the effect of LNCS on the change of permeability of coal under different moisture content conditions, the changes of permeability of coal samples before and after LNCS were comparatively analyzed, as shown in Table 3 and Figure 6.

As presented in Table 3 and Figure 6, the permeabilities of all coal samples exhibit a rising trend after LNCS. Among them, the dried coal sample corresponds to the lowest permeability increase rate, and the saturated coal sample corresponds to the highest rate which is 8.97 times higher than that of dried coal samples. The relationship between the moisture content and the permeability increase rate is not linear but exponential (Figure 6). The mechanism and degree of influence of LNCS on the permeability changes of dried and water-containing coal samples are different.

The pore structure change of the dried coal sample results from the temperature stress caused by the temperature difference effect. When the coal sample is exposed to the low-temperature medium, the temperature field of the coal at the contact surface of the cold source will change rapidly, and part of the coal matrix grains will undergo volume contraction, so a large local temperature stress will be formed between the internal grains. The increase of temperature stress to a certain degree will promote the pore structure of coal to expand and connect, and then the coal permeability will increase. For water-containing coal samples, the pore structure change mainly results from the frost swelling force generated by the water-ice phase transformation and the temperature stress generated by the temperature difference effect, and the frost swelling force gradually becomes dominant as the water saturation increases. When the water-containing coal samples come into contact with the low-temperature medium, the water-ice phase transformation of the pore water can produce a volume expansion of about 9%, and the frost heave force generated by the phase transformation action is theoretically capable of reaching 207 MPa [28–30]. When the combined effect of frost heave force and temperature stress exceeds the strength limit of coal, their synergistic effect causes a large number of new pores to be generated as well as the connectivity of the primary crack network.

The following conclusion can be drawn based on the above analysis. The damage mechanism of dried coal is relatively single, dominated by the pore and fracture damage caused by temperature stress. With the introduction of moisture, the damage effect caused by moisture occurs in addition to the internal structure damage caused by temperature stress. In the case of a low moisture content, some water inside coal freezes and generates pressure. When coal gets saturated, the water inside pores and fractures is completely frozen, and volume expansion causes the contraction and destruction of the coal matrix. In short, the larger the moisture content of a coal sample, the better the permeability enhancement effect.

3.3. Effect of LNCS on the Pore Structures of Coal. The permeability test results suggest that LNCS has a remarkable promotion effect on the permeabilities of coal samples under different moisture content conditions, especially for the saturated coal sample. To investigate the permeability enhancement effect of LNCS on coal samples under different moisture content conditions, the pore structure of coal samples were analyzed by CT scanning test from a 3D perspective.

Figure 7 shows the 3D views of the CT scanning test results of coal samples with different water saturation degrees before and after LNCS. By comparing the spatial distribution of coal pores before and after LNCS, the effect of LNCS on the development and expansion of the pore structure of coal samples with different water saturation degrees is analyzed. Before LNCS, the total pore volumes of coal samples with 0%, 50%, and 100% water saturation are 54.76 mm^3, 20.48 mm^3, and 25.95 mm^3, respectively. After LNCS, they increase to 102.78 mm^3, 68.52 mm^3, and 108.52 mm^3 by 87.69%, 234.57%, and 318.19%, respectively. Compared with an unsaturated water coal sample, the total pore volume of a saturated coal sample increased more significantly. The total pore volume increase of the saturated coal water sample is 3.63 times that of the dried coal sample. A comparison among the 3D micro-CT pore models of coal samples with different water saturation degrees reveals that the dried coal sample is mainly sprouting new pores on the basis of primary pores after LNCS; in contrast, after the coal samples with 50% and 100% water saturation degrees are treated with liquid nitrogen, new pores sprouted while primary pores will penetrate each other spatially to form a crack network.

From the above analysis, it can be seen that after LNCS treatment, the coal sample with a higher moisture content experiences more vigorous development and expansion of its pore structure and a greater change in its seepage characteristics. Therefore, when applied to a water-containing reservoir, LNCS can significantly improve the permeability of the reservoir, thereby promoting the rate of CBM drainage.

4. Conclusions

In this study, the effect of LNCS on the temperature variations within coal samples with different moisture contents were studied. Moreover, the pore structure of coal samples was quantitatively and qualitatively characterized by permeability and CT scanning tests. The study draws the following conclusions

(1) The experimental results of coal temperature changes during LNCS show that the coal sample with a higher moisture content consumes a shorter time to reach internal temperature equilibrium and experiences faster temperature changes. The continuous increase of thermal conductivity after phase transformation of water-ice under low-temperature freezing conditions can accelerate the coal temperature change rate

(2) The permeability test results of coal samples reveal that the permeability increase grows exponentially with the increase of moisture content under the effect of LNCS, and the permeabilities of coal samples are enhanced by 76.26%-684.06%

(3) The 3D CT scanning results of coal samples disclose that after the LNCS treatment, the dried coal sample is mainly sprouting new pores on the basis of primary pores; in contrast, for water-containing coal samples, new pores sprouted while primary pores will penetrate each other spatially to form a crack network

(4) Moisture has a remarkable effect on the seepage characteristics of coal during LNCS, and the freezing and swelling force generated by volume expansion resulting from the water-ice phase transformation promote the development and connection of coal fractures and enhance coal permeability. The joint action of temperature difference effect and water-ice phase transformation exerts a stronger promotion effect on coal permeability. In the process of LNCS cracking coal, the integrated consideration of moisture content conduces to achieving a better permeability enhancement effect

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare no competing interests.

Acknowledgments

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