

The Response Characteristics of Pore Volume and Specific Surface Area of Different Phases of Carbon Dioxide Injection into Anthracite Coal Pores

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Abstract: After the coal sample reacts with CO₂, the porosity and pore volume change. Compared with dry raw coal sample, the porosity of coal after CO₂ adsorption is increased. With the increase of CO₂ injection pressure, the porosity of coal will gradually increase, especially when CO₂ enters the supercritical phase. In terms of pore capacity of coal samples, CO₂ adsorption time, CO₂ adsorption pressure and CO₂-H₂O treatment do not show obvious regularity in the changes of pore capacity of coal samples, but in general, the increase of CO₂ adsorption pressure and CO₂-H₂O treatment have a positive effect on the increase of pore capacity of coal samples, especially ScCO₂-H₂O. The mesoporous pore capacity increased by 137.1%. In terms of macro pore volume of coal sample, the change of macro pore volume after CO₂ adsorption is more regular. Extending CO₂ adsorption time, increasing CO₂ adsorption pressure and CO₂-H₂O treatment all have positive effects on the increase of macro pore volume of coal sample. Under 8MPaScCO₂-H₂O, macro pore volume reaches a maximum of 0.016462ml/g. The increase also reached 130.4 percent. On the mesoporous specific surface area of coal sample, increasing CO₂ adsorption pressure, prolongating CO₂ adsorption time and CO₂-H₂O treatment can increase the mesoporous specific surface area of the sample. The mesoporous specific surface area of the sample at 8 MPa ScCO₂-H₂O is 0.631 m²/g, which reaches the maximum in this experiment, with an increase of 334.27%.

Keywords: Porosity, Pore volume, Pore specific surface area, ScCO₂.

1. Introduction

Coal reservoirs, as a type of porous medium, are composed of interconnected pores and fractures. The development characteristics and connectivity of these pores and fractures directly impact the desorption, diffusion, permeation processes of methane, as well as the production characteristics, thereby determining the effectiveness of CO₂ displacement of CH₄ after injection into coal seams.

Currently, research on coal pore and fracture characteristics mainly focuses on: investigating the development characteristics of pore and fracture networks in coal reservoirs [1, 2]; studying the effects of pores and fractures on gas adsorption/desorption, fluid migration, gas content, permeability, coalbed methane production, and effective implementation of CO₂-ECBM based on various models. The genesis types and complex morphologies of coal pores vary widely in size. Scholars both domestically and abroad have conducted extensive research on the classification of coal pore structures for different research purposes. Among them, the most widely used in China's coalbed methane industry is the IUPAC classification system based on coal adsorption properties: micropores (<2 nm); mesopores or transitional pores (2~50 nm); macropores (>50 nm), and the decimal classification system proposed based on industrial adsorbents: micropores (<10 nm); transitional pores (10~100 nm); mesopores (100~1000 nm); macropores (>1000 nm). Researchers believe that the genesis, morphology, and development characteristics of coal pores are closely related

to coal maceral composition, metamorphism, and deformation [3].

2. Sample and Methods

2.1. Sample collection and preparation

The samples used in the experimental research of this paper were taken from the Datong Mine No. 3 coal mine in Gaoping City, Shanxi Province, China. These coal samples were all taken from adjacent positions of the same large block. To ensure the accuracy and reliability of the experiments, three small pieces of original samples were randomly placed in sealed bags. The reflectance of vitrinite and microcomponents were tested using the Axio Scope.A1 multifunctional microscope produced by ZEISS company. The experimental conditions were room temperature at 23°C and humidity at 60%. The tests were conducted according to the recommended standards of the petroleum industry SY/T 5124-2012 and SY/T 5124-1995. Table 1 shows the test results of this experiment. The results indicate that the random average reflectance of vitrinite is 2.34%, 2.29%, and 2.16% respectively, indicating high-rank coal. Microcomponent analysis shows that these samples are predominantly vitrinite, accounting for 97.5%, 96.0%, and 97.3% respectively, with some inertinite, accounting for 2.5%, 4.0%, and 2.7% respectively. The total organic matter accounts for 93.1%, 95.4%, and 96.3% respectively, pyrite accounts for 0.1% in all cases, and other minerals account for 6.8%, 4.5%, and 3.6% respectively.

Table 1. Basic information of the coal samples.

Sample source	Total rock (%)			maceral (%)			Vitrinite reflectance (%)		
	Total organic matter	Pyrite	Other minerals	Liptinite	Vitrinite	Inertinite	Min	Max	Average value
The 3 [#] coal of Datong Mine	93.1	0.1	6.8	/	97.5	2.5	2.11	2.51	2.34
	95.4	0.1	4.5	/	96.0	4.0	2.21	2.45	2.29
	96.3	0.1	3.6	/	97.3	2.7	1.99	2.35	2.16

2.2. Water injection and gas injection experiments

All samples in the experimental procedure were subjected to 24 hours of drying at 105°C in an oven to remove all moisture before conducting the triaxial experiments. Then, according to the experimental plan, the samples were prepared under the corresponding CO₂ pressure. After the

pressure gauge readings stabilized, continuous CO₂ injection was carried out for 3 days and 6 days at the given pressure and temperature [5, 6]. Studies have shown that the structure of coal still undergoes slow changes even after one year of CO₂ injection, but the main interaction between coal and CO₂ is completed within the first few days. Therefore, considering the timeliness of CO₂ adsorption, this study adopted continuous injection times of 3 days and 6 days.

Table 2. Experimental conditions for coal powder.

Coal sample identification number	Experimental temperature (°C)	Moisture condition	Gas injection type	Gas injection time (h)	Gas injection pressure (MPa)
1	40	Dry	/	/	/
2		Dry	CO ₂	72h	4MPa
3		Dry	CO ₂	72h	6MPa
4		Dry	CO ₂	72h	8MPa
5		Dry	CO ₂	144h	4MPa
6		Dry	CO ₂	144h	6MPa
7		Dry	CO ₂	144h	8MPa
8		Immersion water	CO ₂	144h	4MPa
9		Immersion water	CO ₂	144h	6MPa
10		Immersion water	CO ₂	144h	8MPa

2.3. Low-temperature N₂ adsorption experiment

The low-temperature nitrogen adsorption experiment was conducted using the ASAP 2460 multi-station automatic surface area and pore size analyzer manufactured by Micromeritics Instrument Corporation in the United States, following the international standards ISO 15901-2:2006 and ISO 15901-3:2007. The sample used in the experiment was coal powder with a particle size of 0.18-0.25 mm. Nitrogen was used as the adsorption medium, with the analysis bath temperature set at -195.85°C and the relative pressure range from 0.005 to 0.996. In the analysis of nitrogen adsorption data, the multi-layer adsorption model proposed by Brumauer-Emmett-Teller (BET) was used to calculate the specific surface area, and the Barrett-Joyner-Halenda (BJH) equation was used to determine the pore size. The BET model is a widely used theoretical tool for calculating adsorption isotherms, suitable for studying the adsorption behavior of adsorbent surfaces in gases or solutions.

**Figure 1.** Physical diagram of the low-temperature N₂ adsorption experimental instrument.

2.4. High-pressure mercury experiment.

The mercury intrusion porosimetry test gradually injects liquid mercury into the pores under external pressure. Smaller pore sizes require higher pressure to be filled. The experiment utilized the AutoPore IV 9520 fully automatic mercury porosimeter from Micromeritics Instrument Company, USA, following the international standard ISO 15901-1:2005. The coal sample used in the experiment was small coal blocks with a volume of about 3-4 cm³. During the experiment, the injection pressure of mercury ranged from 0.0099 MPa to

413.46 MPa. The measurement lower limit was 5 nm, employing a computer-program-controlled point-by-point measurement method, and the samples were dried for 24 hours before the experiment. The pore size calculation employed the Washburn equation, with a surface tension of mercury set at 0.48 N/m and a contact angle between mercury and coal set at 140°.



Fig. 2 High-pressure mercury intrusion porosimetry instrument physical diagram.

3. Results and Discussion

3.1. Changes in porosity and pore volume of coal after CO₂ injection.

Based on previous research findings, low-temperature N₂ adsorption experiments based on the BJH model are more accurate for characterizing pores smaller than 50nm, while there are significant errors in the high-pressure mercury intrusion method within the range of less than 30nm. Therefore, in order to obtain maximum experimental accuracy, 50nm is selected as the dividing point for the two aforementioned experimental methods for joint analysis (pores with diameters larger than 50nm are characterized using high-pressure mercury intrusion experimental data, while pores smaller than 50nm are characterized using N₂ adsorption experimental data).

The division of pore size is very important for studying the pore distribution characteristics of coal samples, and a targeted pore size division scheme can clearly reflect the distribution characteristics of different-sized pores. To ensure the accuracy of this experiment, the multi-scale pore structure characteristics of coal samples were studied based on low-temperature N₂ adsorption experiments and high-pressure mercury intrusion experiments, and the pores were divided into mesopores (2~50nm) and macropores (larger than 50nm).

Table 3. The porosity and pore volume of coal samples before and after reaction.

Number	Mercury Intrusion Porosity (%)	Porosity Growth (%)	Mesopores ($\times 10^{-4}$ ml/g)	Mesopore Growth (%)	Macropores ($\times 10^{-3}$ ml/g)	Macropore Growth (%)	Total Pore Volume ($\times 10^{-3}$ ml/g)	Total Pore Volume Growth (%)
1	3.50	-	2.48	-	6.897	-	7.145	-
2	3.69	5.43	3.17	27.82	7.903	14.59	8.22	15.05
3	4.09	16.87	1.43	-42.34	10.588	53.52	10.731	50.19
4	4.13	17.98	6.4	158.06	10.981	59.21	11.621	62.65
5	4.21	20.31	4.51	81.85	10.969	59.04	11.42	59.83
6	4.24	21.06	1.74	-29.84	11.28	63.55	11.454	60.31
7	4.38	25.13	4.23	70.56	98.43	42.17	10.266	43.68
8	4.52	29.02	3.57	43.95	13.299	92.82	13.656	91.13
9	4.64	32.58	1.4	-43.55	13.538	96.29	13.678	91.43
10	4.97	41.95	5.88	137.10	15.874	130.16	16.462	130.40

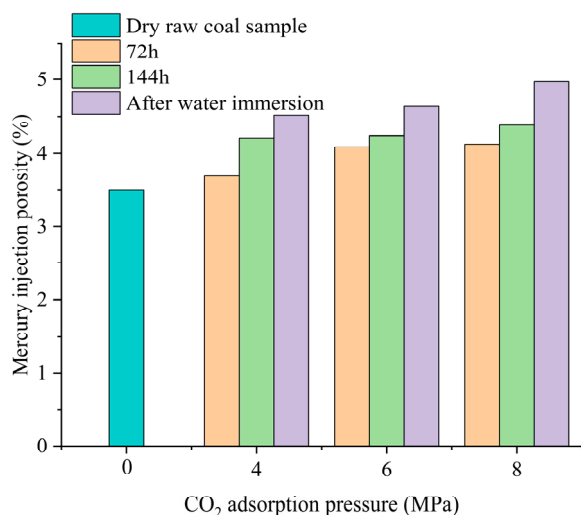


Figure 3. Change in Mercury Intrusion Porosity of Coal Samples Before and After Reaction.

The porosity of coal seams is an important parameter for evaluating permeability. In this experiment, the porosity was measured using the mercury intrusion method. With the CO₂ adsorption time remaining constant (72 hours), as the CO₂ adsorption pressure increases, the porosity of the coal sample gradually increases. When the adsorption pressure increases from 4 MPa to 6 MPa and then to 8 MPa, the porosity increases from the initial 3.5% to 4.13%, showing an increase of 17.98%. Similarly, with a CO₂ adsorption time of 144 hours, as the CO₂ adsorption pressure increases from 4 MPa to 6 MPa and then to 8 MPa, the porosity of the coal sample gradually increases from the initial 3.5% to 4.38%, showing an increase of 25.13%. It can be seen that the mercury intrusion porosity of the coal sample increases with the increase in CO₂ adsorption pressure. When the CO₂ adsorption pressure is held constant (4 MPa) and the adsorption time is extended from 72 hours to 144 hours, the porosity of the sample increases from 3.69% to 4.21%. Similarly, when the CO₂ adsorption pressure is 6 MPa, extending the CO₂ adsorption time increases the porosity of

the sample from 4.09% to 4.24%. When the CO₂ adsorption pressure is 8 MPa, extending the CO₂ adsorption time increases the porosity of the sample from 4.13% to 4.38%. Therefore, it can be observed that extending the adsorption time under constant CO₂ adsorption pressure also increases the mercury intrusion porosity of the coal sample. This experiment also considered the influence of the coupling factors of water and CO₂. After treatment with 4 MPa CO₂-H₂O, the porosity of the sample was 4.52%, after treatment with 6 MPa CO₂-H₂O, the porosity was 4.64%, and after treatment with 8 MPa CO₂-H₂O, the porosity was 4.97%. It can be seen that their porosity is greater than that of samples adsorbed with CO₂ alone, and the porosity also increases with the increase in CO₂ adsorption pressure. After treatment with supercritical CO₂ and water, sample 10 reached the maximum porosity (4.97%) in this experiment, with the maximum increase of 41.95%. Therefore, extending the CO₂ adsorption time, increasing the CO₂ adsorption pressure, and performing CO₂ adsorption after water immersion will increase the mercury intrusion porosity of the coal sample.

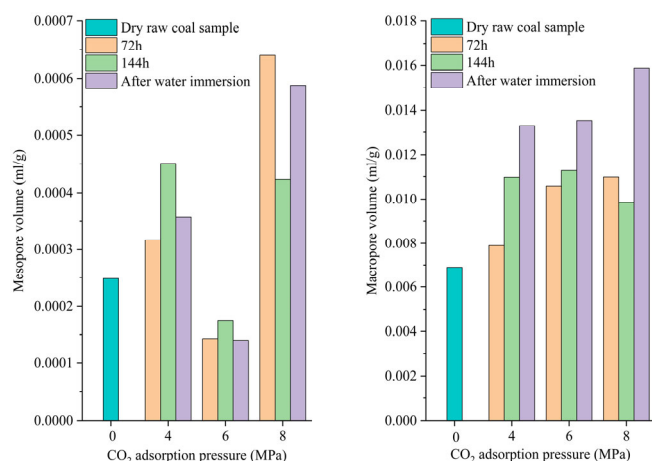


Figure 4. Changes in Porosity of Coal Samples Before and After Reaction.

The porosity in coal is mainly contributed by macropores, while mesopores have smaller porosity, and the porosity of some samples decreases after CO₂ adsorption. Mesopore porosity shows varying degrees of increase and decrease after CO₂ adsorption, while macropores only show different degrees of enlargement after CO₂ adsorption. From the perspective of CO₂ adsorption time, the mesopore porosity of coal samples adsorbed with 4 MPa CO₂ shows an increasing trend, specifically, the mesopore porosity increases from 0.000317 ml/g to 0.000451 ml/g after extending the adsorption time. The mesopore porosity of coal samples adsorbed with 6 MPa CO₂ shows a decreasing trend. The mesopore porosity of coal samples adsorbed with 8 MPa CO₂ increases, but with extended adsorption time, the increase in mesopore porosity decreases, from 158.06% to 70.56%. This data indicates that the change in mesopore porosity due to CO₂ adsorption time is not regular. From the perspective of CO₂ adsorption pressure, when adsorbed for 72 hours, 4 MPa CO₂ adsorption increases the mesopore porosity of the sample to 0.000317 ml/g, 6 MPa CO₂ adsorption decreases it to 0.000143 ml/g, and 8 MPa CO₂ adsorption increases it to a maximum of 0.00064 ml/g. When adsorbed for 144 hours, 4 MPa CO₂ adsorption increases the mesopore porosity of the

sample to a maximum of 0.00451 ml/g, 6 MPa CO₂ adsorption decreases it to 0.000174 ml/g, and 8 MPa CO₂ adsorption increases it to 0.000423 ml/g. Overall, increasing CO₂ adsorption pressure promotes an increase in mesopore porosity in coal samples. Additionally, this experiment considered the effect of CO₂ adsorption on mesopore porosity of coal samples after water immersion. Overall, CO₂-H₂O promotes an increase in mesopore porosity of coal samples, especially ScCO₂-H₂O, which increases mesopore porosity by 137.1%, reaching 0.000588 ml/g.

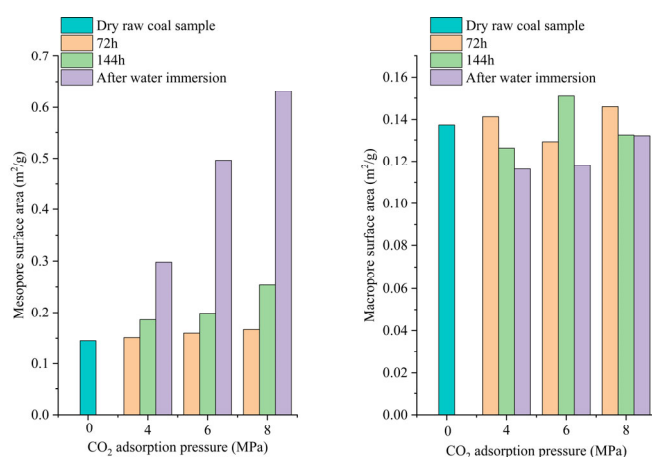
After CO₂ adsorption, the pattern of macropores in the samples becomes more pronounced. From the perspective of CO₂ adsorption time, the macropore porosity of coal samples adsorbed with 4 MPa CO₂ shows an increasing trend, with the macropore porosity increasing from 0.00822 ml/g to 0.01142 ml/g after extending the adsorption time. The macropore porosity of coal samples adsorbed with 6 MPa CO₂ also shows an increasing trend, with the macropore porosity increasing from 0.010731 ml/g to 0.011454 ml/g after extending the adsorption time. The macropore porosity of coal samples adsorbed with 8 MPa CO₂ also increases, but after extending the adsorption time, the increase in macropore porosity decreases slightly, from 62.65% to 43.68%. Therefore, extending CO₂ adsorption time under constant pressure has a positive effect on increasing macropore porosity in samples. From the perspective of CO₂ adsorption pressure, when adsorbed for 72 hours, the macropore porosity increases with increasing CO₂ adsorption pressure, specifically, the macropore porosity is 0.00822 ml/g at 4 MPa, 0.010731 ml/g at 6 MPa, and 0.011621 ml/g at 8 MPa. Similar trends are observed when adsorbed for 144 hours, with the exception of a smaller increase in macropore porosity at 8 MPa. This data indicates that increasing CO₂ adsorption pressure also has a positive effect on increasing macropore porosity in samples. After water immersion, followed by CO₂ adsorption, the data indicates that higher adsorption pressure results in larger macropore porosity, with the macropore porosity being 0.013656 ml/g at 4 MPa CO₂-H₂O, 0.013678 ml/g at 6 MPa CO₂-H₂O, and reaching a maximum of 0.016462 ml/g at 8 MPa ScCO₂-H₂O, with an increase of 130.4%. Additionally, the total porosity of coal samples is mainly contributed by macropores, so the variation in total porosity is similar to that of macropores. Therefore, extending CO₂ adsorption time, increasing CO₂ adsorption pressure, and performing CO₂ adsorption after water immersion all have a positive effect on increasing the total porosity of coal samples.

3.2. Change characteristics of pore specific surface area of coal after CO₂ injection

The gas adsorption capacity of coal is closely related to the pore volume and specific surface area of coal, especially the pore volume and specific surface area of nanopore. In this experiment, changes in specific surface area of different apertures were analyzed based on low-temperature N₂ adsorption experiment and high-pressure mercury injection experiment. The specific surface area of mesoporous pores was the BET specific surface area tested by liquid nitrogen method, and that of macroporous pores was the specific surface area tested by mercury injection method. The distribution of specific surface area of different apertures before and after CO₂ adsorption was shown in Table 4.

Table 4. Pore specific surface area before and after coal sample reaction.

Number	mesoporous (m ² /g)	Mesoporous increase (%)	macrohole (m ² /g)	Macrohole increment (%)	Total pore specific surface area (m ² /g)	Total pore specific surface area increase (%)
1	0.1453	-	0.137465	-	0.282765	-
2	0.1513	4.13	0.141311	2.8	0.292611	3.48
3	0.1598	9.98	0.129163	-6.04	0.288963	2.19
4	0.1669	14.87	0.146002	6.21	0.312902	10.66
5	0.1857	27.8	0.12622	-8.18	0.31192	10.31
6	0.1988	36.82	0.150999	9.85	0.349799	23.71
7	0.2539	74.74	0.132318	-3.74	0.386218	36.59
8	0.298	105.09	0.116302	-15.4	0.414302	46.52
9	0.4964	241.64	0.11801	-14.15	0.61441	117.29
10	0.631	334.27	0.13198	-3.99	0.76298	169.83

**Figure 5.** Changes of specific surface area before and after coal sample

The experimental data show that the total pore specific surface area of coal samples in this experiment is mainly contributed by mesoporous pores. The mesoporous specific surface area of the raw coal sample is 0.1453 m²/g, and the mesoporous specific surface area increases to 0.1513 m²/g under the adsorption condition of 4 MPa CO₂ for 72 hours, and 0.1598 m²/g under the adsorption condition of 6 MPa CO₂ for 72 hours. Under the condition of 8 MPa CO₂ adsorption for 72 hours, the specific surface area of the mesopore increased to 0.1669 m²/g. It can be seen that under the same CO₂ adsorption time, the specific surface area of the coal sample mesoporous will also increase with the increase of CO₂ adsorption pressure, which can also be clearly seen in Fig.3-3. This is also reflected in the case of 144 hours of CO₂ adsorption. The specific surface area of the mesopore increases to 0.1857 m²/g at 4 MPa CO₂ adsorption for 144 hours, and 0.1988 m²/g at 6 MPa CO₂ adsorption for 144 hours. Under the condition of 8 MPa CO₂ adsorption for 144 hours, the specific surface area of the mesopore increased to 0.2539 m²/g, and the increase rate reached 74.74%. When the CO₂ adsorption pressure is 4 MPa, the adsorption time is extended from 72 hours to 144 hours, and the specific surface area of the mesopore is also increased to 0.1857 m²/g. It can be seen that under the condition that the CO₂ adsorption pressure remains unchanged, the specific surface area of mesoporous pores will increase with the extension of CO₂ adsorption time. This is also reflected in the case of 6 MPa and 8 MPa, when the CO₂ adsorption pressure is both 6 MPa, the adsorption time is extended from 72 hours to 144 hours, and the specific surface area of the mesopore is increased

from 0.1598 m²/g to 0.1988 m²/g. When the CO₂ adsorption pressure is 8 MPa, the adsorption time is extended from 72 hours to 144 hours, and the specific surface area of the mesopore is increased from 0.1669 m²/g to 0.2539 m²/g. The increase of mesoporous specific surface area of the samples treated with CO₂-H₂O was significantly higher than that of the samples adsorbed CO₂ alone. In addition, when the samples were treated with CO₂-H₂O, the mesoporous specific surface area of the samples increased significantly with the increase of adsorption pressure. The mesoporous specific surface area of the sample at 4 MPa CO₂-H₂O is 0.298 m²/g, that of the sample at 6 MPa CO₂-H₂O is 0.4964 m²/g, and that of the sample at 8 MPa ScCO₂-H₂O is 0.631 m²/g, reaching the maximum in this experiment. The increase reached 334.27%.

Only the macropore specific surface area of 4 MPa CO₂ adsorbed for 72 hours, 8 MPa CO₂ adsorbed for 144 hours and 8 MPa CO₂ adsorbed for 72 hours was larger than the original macropore specific surface area, and the increasing trend was not regular from the three variables. The macropore specific surface area of the other samples is smaller than that of the original, but the decreasing trend is not consistent. Therefore, whether the CO₂ adsorption time is prolonged, the CO₂ adsorption pressure is increased, or the specific surface area of the sample is treated with CO₂-H₂O, the specific surface area of the sample shows inconsistent rules. Therefore, the adsorption of CO₂ has no regularity on the macro pore specific surface area of the coal sample. Since the total pore specific surface area of coal is contributed by mesoporous pores, its change trend is similar to that of mesoporous specific surface area, that is, increasing CO₂ adsorption pressure, prolonging CO₂ adsorption time and CO₂-H₂O treatment will increase the total pore specific surface area of coal samples.

4. Conclusions

(1) After the coal sample reacts with CO₂, the porosity and pore volume change. Compared with dry raw coal sample, the porosity of coal after CO₂ adsorption is increased. With the increase of CO₂ injection pressure, the porosity of coal will gradually increase, especially when CO₂ enters the supercritical phase. For example, when CO₂ is adsorbed for 4MPa72 hours, the porosity of the coal sample is 3.69%, and when CO₂ is adsorbed for 8MPa72 hours, the porosity of the coal sample is 4.13%. With the increase of CO₂ injection time, the porosity of coal will also increase. In addition, under the coupling effect of CO₂ and water, the porosity of coal will further increase. After the treatment of supercritical CO₂ and water, the porosity of sample 10 reaches the maximum in this

experiment (4.97%), and the increase rate also reaches the maximum (41.95%).

(2) In terms of the pore capacity of coal samples, CO₂ adsorption time, CO₂ adsorption pressure and CO₂-H₂O treatment do not show obvious regularity in the changes of pore capacity of coal samples, but in general, the increase of CO₂ adsorption pressure and CO₂-H₂O treatment have a positive effect on the increase of pore capacity of coal samples, especially ScCO₂-H₂O. The mesoporous pore capacity increased by 137.1%, reaching 0.000588ml/g. In terms of macro pore volume of coal sample, the change of macro pore volume after CO₂ adsorption is more regular. Extending CO₂ adsorption time, increasing CO₂ adsorption pressure and CO₂-H₂O treatment all have positive effects on the increase of macro pore volume of coal sample. Under 8MPaScCO₂-H₂O, macro pore volume reaches a maximum of 0.016462ml/g. The increase also reached 130.4 percent. In addition, the total pore volume of coal samples is mainly contributed by macro pores, so the change of total pore volume is basically the same as that of macro pores.

(3) For the mesoporous specific surface area of coal samples, increasing CO₂ adsorption pressure, prolongating CO₂ adsorption time and CO₂-H₂O treatment can increase the mesoporous specific surface area of the sample. The mesoporous specific surface area of the sample at 8 MPa ScCO₂-H₂O is 0.631 m²/g, reaching the maximum in this experiment, with an increase of 334.27%. On the macropore specific surface area of coal sample, the adsorption of CO₂

has no regularity. Since only the specific surface areas of mesoporous and macro pores are studied in this experiment, the total pore specific surface area of coal in this experiment is contributed by mesoporous pores, and the change of total pore specific surface area is basically consistent with that of mesoporous specific surface area.

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