Influence of water soaking on swelling and microcharacteristics of coal

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Abstract
Improving the coal seam permeability is an important measure for increasing the coal bed methane (CBM) production and preventing gas disasters. Hydraulic technologies are effective ways of improving coal seam permeability. However, hydraulic technologies can also cause water to enter the coal seam, allowing the coal seam to soak for a long time. In this study, to obtain the influence of water soaking on the microscopic characteristics of coal, X-ray diffraction (XRD), scanning electron microscopy (SEM), free swelling ratio tests, and low-temperature nitrogen adsorption tests (LT-NATs) were conducted. The mineral compositions of raw coal samples, the variation regularities of micromorphologies, and pore characteristics of the samples with different soaking times were obtained. The results showed that the coal samples contained about 8.5% clay minerals, of which 71% were illite/smectite mixed-layer. Expansions of different sizes in the areas where the surface of the soaked coal samples contained clay minerals were observed, and the swelling was also observed macroscopically. The swelling not only led to an increase in the coal sample volume but also led to a decrease in pore volume. This change was magnified with the increase in soaking time (within 30 days). The cumulative pore volume of the samples soaked for 30 days was 0.00681 cm³/g. This was a reduction of 29.9% compared to the unsoaked samples. Moreover, the pore volumes show a logarithmic dependence on soaking time. This study provides evidence that the coal containing clay minerals will swell obviously when soaked in water, and the hydration swelling of clay minerals has a great influence on the swelling of coal. This swelling would lead to a decrease in the pore volume and the efficiency of CBM transport, thus affecting the effect of hydraulic measures.

KEYWORDS
coal, coal bed methane, microcharacteristics, pore structure, water soaking

1 | INTRODUCTION

Gas disasters severely threaten the safety of coal mines, especially coal and gas outbursts.¹ ² ³ Coal bed methane (CBM) extraction is an effective way to prevent gas disasters, and the extracted CBM can be used as a source of clean energy.⁴ ⁵ However, most CBM reservoirs in China are characterized by high crustal stresses and low permeabilities,
creating difficulties in extracting CBM in these coal seams.\textsuperscript{5,7,8} Increasing the permeability of the coal seam is an effective means to improve the recovery of CBM.\textsuperscript{9-11} Hydraulic technologies, including hydraulic fracturing\textsuperscript{12,13} and hydraulic slotting,\textsuperscript{14-16} are often used to prevent the gas disasters and enhance CBM recovery. These methods use high-pressure fluids to create fractures in the coal seams, thereby increasing permeability to cause more gas to desorb, create more space for gas diffusion, and improve the CBM production.

Water is widely used in underground hydraulic technologies in Chinese coal mines due to the low cost and ready availability.\textsuperscript{17} These hydraulic methods not only promote the development of fractures but also cause water to enter the fractures. Water present in coal seams is of profound importance in relation to CBM production.\textsuperscript{18,19} Its effects are manifold. The presence of water in coal reduces gas sorption capacity and gas diffusivity, and the sorption of water vapor by coal leads to several percent of swelling.\textsuperscript{19-21} Generally, these effects are usually caused by changes in the external morphology and internal structure.\textsuperscript{22} Many studies have been performed to identify the variation of coal in response to absorption of water or water vapor.\textsuperscript{23-25} Zhang et al\textsuperscript{25} compared dry and soaked coal samples using X-ray microcomputed tomography, and the results showed that the cleats in the coal matrix closed upon water absorption, while the cleats in the mineral phase were not affected. Yang et al\textsuperscript{17,26} compared the influence of water and viscoelastic surfactant fracturing fluid on coal samples, and the results showed that the permeability of the coal samples saturated with the viscoelastic surfactant fracturing fluid was higher than that of the samples saturated with water. Zhang et al\textsuperscript{27} measured nanoscale rock mechanical properties via nanoindentation tests for dry and wet heterogeneous coal. The indentation moduli by nanoindentation decreased by 60%-66%, but a 16.6% increase was measured in the dynamic bulk measurement. Liu et al\textsuperscript{19} reports dilatometry experiments conducted on 1 and 4 mm sized cubic high-volatile bituminous coal samples, the results show that the volumetric swelling strains attained at equilibrium show a near-linear dependence on relative humidity, reaching 1.37%-1.43% at around 95% relative humidity.

Hydraulic technologies, especially hydraulic fracturing, require a high pressure to be maintained for a period of time to expand the fractures in coal seams.\textsuperscript{13} This also pressed water into a deeper area. Meanwhile, after the water flow-back, due to fluid leak off and water blocking damage, some water still exists in the coal seam causing the coal seam to soak for a long time.\textsuperscript{22,23} This water soaking may have an influence on the coal, thereby affect the permeability and gas seepage. However, few studies have focused on the influence of water on the swelling and microcharacteristics of coal after long soaking times, and the variation of coal samples in different soaking times is not clear.

In this study, the coal samples were taken from the No. 8 coal seam in the Songzao mining area. Coal samples with different soaking times were analyzed by X-ray diffraction (XRD), scanning electron microscopy (SEM), low-temperature nitrogen adsorption tests (LT-NATs), and free swelling ratio tests. The mineral composition of the coal samples was obtained by XRD. SEM was used to study the variations in the surface morphologies of the coal samples with different soaking time. Through the LT-NATs, the variations in the pores of coal samples with different soaking time were obtained. Through the free swelling ratio tests, the swelling characteristics of the coal samples were obtained. These results demonstrate the analysis of the swelling and microscopic characteristics of coal when exposed to water and provide support for further research on the swelling mechanism of coal.

2 | MATERIALS AND METHODS

2.1 | Materials

A high-rank anthracite coal block was obtained from the No. 8 coal seam of Songzao mining area in Chongqing City (China) (Figure 1). The major coal-bearing rock series is the Permian Longtan Formation. The average seam thickness of the No. 8 coal seam is 2.5 m, as shown in Figure 2. The maximum gas content and pressure measured in the coal seam were 18.17 m$^3$/t and 2.56 Mpa, respectively.\textsuperscript{28} The proximate analysis of coal samples is summarized in Table 1, measured by Chinese Standards DL/T 1030-2006 and GB/T 212-2008.

2.2 | X-ray diffraction

The mineral compositions of the coal samples were identified by XRD (X’Pert3 Powder, Th Netherlands). The coal samples cut from the coal block were pulverized to below 320 mesh, after which they were treated by low-temperature ashing to remove organics to ensure the integrity of the mineral compositions in the coal samples.\textsuperscript{29} The ashed coal samples were dried for 24 hours at 100°C. The coal samples were

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{songzao.png}
\caption{Location of the Songzao mining area in Chongqing City, China}
\end{figure}
divided into three parts, and XRD was conducted. Each part was >3 g. The mineral composition and content were measured by the Chinese Standard SY/T 5163-2010.

### 2.3 | Scanning electron microscopy

The surface topographies of the coal samples were observed using field emission scanning electron microscope (FESEM; FEI Quanta FEG-250, USA). Coal samples with fresh cross-sections and areas of 0.5-1 cm² were selected for this study. The samples were soaked in deionized water. Three coal samples each soaking time of 0, 1, 5, 15, and 30 days (0 days samples were not soaked). The samples were dried for 48 hours by vacuum freeze-drying (vacuum freeze-drying has the advantage of retaining the sample's original physical properties and chemical composition). The surface attachments of the coal samples were blown away. The samples were sputter coated with gold for 60 s, after which they were observed by FESEM.

### 2.4 | Free swelling ratio test

The free swell ratio is the ratio of the sample's volume increase after soaking in water to the original volume without a confining pressure. The coal samples were pulverized to 60-80 mesh and dried for 48 hours by vacuum freeze-drying. A 40-g sample was divided into two equal parts. One part was soaked and stirred in 100 mL deionized water in a measuring cylinder, followed by static settling for 96 hours. The other part was untreated.

### 2.5 | Low-temperature nitrogen adsorption

The porosities of coal samples were determined by LT-NATs (Micrometrics ASAP2020 analyzer, USA). The coal samples that were separated from the fresh coal block were pulverized to 60-80 mesh and soaked in deionized water. Next, 5 g samples were taken after soaking times of 0, 1, 5, 15, and 30 days. The samples were dried for 48 hours by vacuum freeze-drying. Prior to the tests, the coal samples were degassed under a vacuum for at least 12 hours, after which the LT-NATs conducted.
3 | RESULTS AND DISCUSSION

3.1 | Mineral compositions of coal samples

XRD spectra of the ashed coal samples are presented in Figure 3. According to the Chinese Standard SY/T 5163-2010, the XRD spectra were analyzed. The compositions and contents of minerals and clay minerals are shown in Tables 2 and 3, respectively.

The main mineral components of the coal sample were clay minerals, calcite, dolomite, quartz, and anhydrite. Quartz, calcite, dolomite, and hematite have no chemical reaction with water, and they are hardly soluble in water, so these minerals have less influence on coal after water soaking. Illite, smectite, and kaolinite are all aluminosilicate clay minerals. The clay mineral content of the ashed coal samples was 47%, of which 71% was an illite/smectite mixed-layer, and the ratio of illite to smectite was 20%. Table 1 shows that the coal ash accounted for 18% of the coal sample. Thus, the clay mineral and illite/smectite mixed-layer accounted for about 8.5% and 6.0%, respectively. Smectite is a category of clay minerals with a three-layer crystalline structure that exhibits hydration swelling. When exposed to water, the adsorbed exchangeable cations dissociate to form a diffusive double layer, which produces electronegativity. The crystal layers repel each other, and the spacing increases, causing expansion. Although the content of smectite was relatively low, the hydration swelling ratio of the smectite was large, greater than 30 times its original volume. After the use of hydraulic methods, the coal mass within range becomes saturated with water. This may cause swelling of the smectite and influence the effect of the hydraulic methods. Kaolinite and illite have strong water absorption ability which is same as smectite, but they only have weak expansibility, so the effect on the coal is small.

3.2 | Influence of water soaking on surface topography

Figure 4 shows the FESEM results under 1500× magnification after soaking times of 0, 1, 5, 15, and 30 days. As shown in Figure 4A, the surface of the unsoaked coal sample was relatively flat, with a small number of coal particles attached. The structure was compact and exhibited good continuity and a regular shape. There were a few pores and fractures on the surface, the natural existed fractures tend to be irregular in shape and size. After 1 day of soaking, the surface of the samples became eroded, flaky particles were exfoliated, and small pores had formed at the exfoliated place, as shown in Figure 4B. The exfoliated particles attached to surfaces or filled the pores and fractures. As shown in Figure 4C–E, with the increase in soaking time, the edges of the flaky structure became blurred, the surface structure was gradually destroyed, and became loose and fragmented, and peeled particles with various sizes and shapes gradually increased.

After soaking, considerable clay swelling occurred. As shown in Figure 5A, part of the coal sample swelled significantly and bulged over the surface after soaking (1500×). A portion of the image is further magnified 6000× in Figure 5B. The expansion was composed of spherical particles of different sizes, which were closely packed and irregular. There were a large number of reticulated fractures around the expansion. The fractures connected with each other, and the widths were generally <5 μm. These fractures divided the surface of the coal sample into layered fragments of different sizes, and there was also a large number of smaller, irregularly accumulated, round particles on the fragments. This multifissure structure resulted in more water entering the coal mass. In addition to the large volume expansion, there was also some relatively small expansion. As shown in Figure 6A, a number of expansions were present (1500×). The expansions were 5-10 μm in diameter and surrounded by dark areas. The dark area indicated that it contained hydrated clay minerals. After the water soaking, interlayer water existed in the clay minerals, resulting in a decrease in its total atomic number (the backscattered electron quantity with the decrease of atomic number). The electronic signal decreased accordingly, forming dark areas. It was observed in the dark area that there are semicircular fractures with a width between 0.1 and 0.5 μm around the expansions (Figure 6B and 6), and there are also cases where clay minerals swelled but no fractures occurred (Figure 6D). The results indicate that the water soaking will erode the surface of the coal and cause the clay mineral to swell.

3.3 | Influence of water soaking on sample volume

Figure 7 shows the volumes of the unsoaked and soaked coal samples. The volume of unsoaked coal sample is 23 mL (Figure 7A). After soaking for 96 hours, the coal samples were divided into two parts: one part was precipitated, with a volume of 32 mL, and the other part was

![Figure 3 XRD spectra of coal samples](attachment:image.png)
suspended, with a volume of 2 mL, corresponding to a total volume of 34 mL (Figure 7B). The free swelling ratio was calculated as follows:

\[ d_{ef} = \frac{V - V_0}{V_0} \times 100\% \]

where \( d_{ef} \) is the free swelling ratio, \( V_0 \) is the volume of the unsoaked coal sample, and \( V \) is the volume of the soaked coal sample. The calculated free swelling rate of the coal sample treated with water was 47.8%.

To determine the influence of clay minerals on the swelling of the coal samples, a KCl solution with a concentration of 0.5% was prepared, which has a good effect on inhibiting the clay hydration swelling. The coal samples were treated with the solution, and the results are shown in Figure 8. The coal samples were also divided into two parts: the precipitated part was 27 mL, and the suspended
After being treated with KCl solution, the free swelling ratio of the coal sample decreased significantly. This indicates that clay minerals play an important role in the swelling of coal when exposed to water. The swelling effect of clay minerals in coal must be taken into account, and corresponding measures should be taken.

3.4 | Influence of water soaking on pore structure

Figure 9 shows the low-temperature nitrogen adsorption isotherms of the test samples processed with deionized water after soaking times of 0, 1, 5, 15, and 30 days. With the increase in the relative pressure, the adsorbed nitrogen volumes of all the samples increased. In the low relative pressure ranges, the growth rate was maintained at a low level. After the relative
pressure reached 0.80, the adsorbed volume increased rapidly until it reached a maximum value. The isotherms of the test samples were type II adsorption isotherms, which means that the sample mainly contained both macropores (≥50 nm) and mesopores (2-50 nm). Comparing the adsorption isotherms after different soaking times, the coal samples with longer soaking times had lower adsorption capacities. This indicates that the pore volume decreased as the soaking time increased. Figure 9A and 9 show the fitting curves of quantity adsorbed at inflection point \( p/p_0 = 0.035 \) and \( p/p_0 = 0.8 \), respectively. It obtained that the quantity adsorbed of coal samples showed a logarithmic dependence on soaking time.

With the increase of soaking time, the quantity adsorbed in the low relative pressure stage \( (p/p_0 = 0.035) \) did not exhibit significant variation, while in the high relative pressure stage \( (p/p_0 = 0.8) \), the quantity adsorbed gradually decreased until it reached relative stability after soaking for 30 days. This indicated that water soaking will significantly reduce the pore volume of the coal sample, and the volume of micropore did not change much with the increase of soaking time, while the volume of macropores and mesopores changed greatly, and remained stable after soaking for 30 days. According to the International Union of Pure and Applied Chemistry (IUPAC) classification, the hysteresis loops between the adsorption and desorption isotherms of the samples after a soaking time of 0 and 30 days belong to type H3, as shown in Figure 10, corresponding to slit-shaped pores. The pore shapes of the sample did not change after soaking.

A nonlocal density functional theory (NLDFT) model was used to analyze the pore size distribution (PSD) using the SAIEUS software. The NLDFT model was suitable for the PSD analysis of micropores, mesopores, and macropores with high accuracy. The calculation of the PSD was based on the integral adsorption equation:

\[
N(p/p_0) = \int_{D_{\text{min}}}^{D_{\text{max}}} N(p/p_0, D) f(D) dD, \tag{2}
\]

where \( N(p/p_0) \) is the adsorption isotherm data, \( D \) is the pore width, \( N(p/p_0, D) \) is the kernel of theoretical isotherms, and \( f(D) \) is the PSD.

Figures 11 and 12 show the PSDs and pore volumes of the samples after soaking times of 0 and 30 days with pore widths from 1 to 100 nm. Due to the limitation of the LT-NATs, the micropores only contained pores of 1-2 nm in this study. Mesopores and macropores accounted for a large proportion of the pore volume, which was consistent with the characteristics of type II adsorption isotherms.
incremental and cumulative pore volumes of the soaked samples decreased significantly compared with those of the unsoaked samples. The cumulative pore volume of the soaked samples was 0.00681 cm$^3$/g, which was a reduction of 29.9% compared to the unsoaked samples whose cumulative pore volume was 0.00972 cm$^3$/g. The decrease of pore volume was driven mainly by a decrease in pore width between 2-6 nm, 35-45 nm, and 65-75 nm. This is mainly attributed to the hydration swelling of smectite after water soaking. After the coal samples are soaked in water, the water enters into the coal along with the pores and fractures, and the smectite near the pores and fractures hydrates and gradually swells. Due to the limitation of coal structure, expansions will invade into pores and fractures, resulting in the decrease of pore volume. It seems to contradict the results of SEM, which shows the new fractures on the surface of the coal sample due to expansion. This is mainly because only a small amount of clay mineral is exposed to the surface of the coal samples; after water soaking, the pore volume reduced by internal expansion is larger than the pore volume generated by surface expansion, and the peeled particles can fill the pores and fractures. It should be noted that compared with bigger mesopores and macropores, the pores with width between 2 and 6 nm have a greater impact on CBM transport. As shown in Figure 13, after water soaking, the decrease in pore volume of these smaller mesopores may be accompanied by the closure or narrowing of the pore throats, resulting in an increase in closed pores or dead-end pores.

The results are different from the findings of some scholars. Zhai and Song claimed that the water soaking will dissolve the organic and inorganic substances in the coal, thus increasing the pore volume and pore size. This difference is probably due to the differences in composition of the coal samples. Since the minerals in the coal samples collected from Songzao have a low solubility in water, the pore volume increased by dissolution is small. Meanwhile, the smectite in the coal samples hydrates and swells after water soaking, resulting in a decrease in pore volume.

### 3.5 Discussion

Coal is a typical porous medium. The pore structure plays an important role for the CBM adsorption and transport in coal. The smectite in the coal samples swells, causing changes in pore structure.

Nanopore (<100 nm) is an important channel for CBM transport. After a coal seam is invaded by water, due to the water blocking damage, if the formation driving force cannot overcome the capillary pressure, it will cause the pores to be blocked by water in the invaded zone, the CBM in the fractures cannot be extracted (Figure 13). The Young-Laplace equation is used to calculate the capillary pressure:

$$P_c = \frac{2y \cos \theta}{r},$$

where $y$ is the surface tension, $\theta$ is the contact angle, and $r$ is the radius of the pore.
where $P_c$ is the capillary pressure, $\gamma$ is the interfacial tension between the water and the air, $\theta$ is the contact angle between the water and coal, and $r$ is the radius of the capillary. Due to water soaking, the pore volume and size decreased. According to Equation 3, interfacial tension and the contact angle are constant and with a decrease in the pore width, the capillary pressure will increase correspondingly. Therefore, the decrease in pore size caused by water soaking will aggravate the damage of water blocking effect, resulting in a decrease in the permeability of coal.

The nanopores have greater adsorption capacity, and the pores smaller than 100 nm accounts for more than 80% of the total specific surface area, which indicates that most of the CBM is adsorbed in the nanopores. In general, 100 nm is the threshold of diffusion and seepage, and the CBM transport in pores below 100 nm is dominated by diffusion. As a key flow property for CBM extraction, diffusion is considered as the first step of CBM transport in coal. The Knudsen diffusion coefficient is shown in the following equation:

$$D_K = \frac{d_p}{3} \sqrt{\frac{8RT}{\pi M_A}}$$  (4)

where $D_K$ is the Knudsen diffusion coefficient, $d_p$ is the pore diameter, $R$ is the ideal gas constant, $T$ is the temperature, and $M_A$ is the molecular mass of gas. After the water soaking, the pore size and pore volume decreased, according to Equation 4, Knudsen diffusion coefficient decreased correspondingly. After the implementation of the hydraulic measures, the CBM will be desorbed, and most of the CBM diffused into the fractures through the nanopores. However, as the soaking time increased, the pore volume and pore size decrease, and the CBM diffusion rate will decrease accordingly which greatly reduced the efficiency of CBM transportation. Therefore, it is necessary to focus on the coal swelling caused by water soaking to reduce its impact on hydraulic technologies.

4 | CONCLUSION

This study aimed to obtain the influence of water soaking on swelling and microscopic characteristics of coal. Based on the experiments conducted, it was found that the coal samples obtained from the No. 8 coal seam of the Songzao mining area contained small amounts of clay minerals dominated by illite/smectite mixed-layer, whose content was about 6%. With the increase in soaking time, the surface of the coal gradually eroded, resulting in a large number of flaky particles of different sizes. In the areas where the surfaces of soaked coal samples contained clay minerals, expansions composed of spherical particles of different sizes were observed, and fractures formed on the surface. Through the free swelling ratio test, the soaked coal samples also showed significant swelling, and the hydration swelling of the clay was the main reason for the expansion of the coal sample. Meanwhile, the pore volumes of the soaked coal samples decreased with the increase of soaking time until soaking for 30 days, and it shows
a logarithmic dependence on soaking time. The pore volume of the coal sample soaked for 30 days was 0.00681 cm$^3$/g, which was a reduction of 29.9% compared to the unsoaked samples. The reduction in pore volume was mainly due to a reduction in the number of macropores and mesopores.

We concluded that the coal samples that contained clay minerals, especially smectite, which were prone to hydration, exhibited significant swelling after water soaking. The swelling effects resulted in the increase in the coal sample volume and the decrease in the pore volume, and this change was magnified with the increase in the soaking time (within 30 days). This decrease of pore volume will lead to a decrease in pore width, an increase in closed pores, and a decrease in pore connectivity, which will exacerbate the water blocking damage and affect CBM transport and the efficiency of hydraulic measures.

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