



# The Influence of CO<sub>2</sub> Saturated Brine on Microstructure of Coal: Implications for Carbon Geo-Sequestration

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Carbon dioxide geological storage in deep underground oil and gas reservoirs or coal seam is considered a key technology to effectively mitigate carbon emissions. Deep coal seams are the potential host rock for CO<sub>2</sub> storage, with chemical reactions occurring when coal is exposed to CO<sub>2</sub>-saturated brine. However, limited studies have been conducted to reveal the effect of chemical reaction, particularly to unveil how this importantly affects the coal seam porosity at the micrometer scale. In this study, the CO<sub>2</sub> geological storage is simulated using a high-temperature and high-pressure reactor containing CO<sub>2</sub>-saturated brine and coal samples. The samples in the previous study on the effect of CO<sub>2</sub> on coal mostly contained high content of carbonate minerals and sulfide minerals. However, the tested Collie coal samples, which are characterized by high content of kaolinite and siderite, are rarely used. This coal was quantitatively analyzed using a scanning electron microscope before and after reaction. The results show that the microstructure of coal matrix was largely changed due to acid exposure. Kaolinite and siderite in the coal matrix were dissolved, the size and morphology of the cleats increased, and the absolute effective porosity also increased significantly. Most importantly, the connectivity of cleat network was improved, leading to an increase in CO<sub>2</sub> storage space and coal seam permeability. Therefore, it is concluded that the microstructure changes of coal can be measured on a microscopic scale, and it is better to be quantitatively evaluated to improve the accuracy and reliability of CO<sub>2</sub> storage in deep coal seams.

**Keywords:** CO<sub>2</sub> geological storage, coal seam, SEM, CO<sub>2</sub>-saturated brine, cleat structure

## 1 INTRODUCTION

CO<sub>2</sub> storage in deep geological formation is a vital technology that effectively resolves climate change caused by carbon emissions (White et al., 2005; Alemu et al., 2011; Zhou et al., 2017). The main storage places are underground geological reservoirs including deep undeveloped coal seams, deep brine aquifers, and oil and gas reservoirs (Bachu et al., 1994). Solubility trapping is one of the safest

and effective trapping mechanisms (Akhondzadeh et al., 2020; Al-Khdheawi et al., 2016; Iglauer et al., 2015; Iglauer and Al-Yaseri, 2021; Mitchell et al., 2010; Niu et al., 2015; Zhang et al., 2017) by dissolving CO<sub>2</sub> in the formation water. The density of CO<sub>2</sub>-saturated brine (also called live brine) is greater than that of the original formation water; thus, it sinks to the bottom of the reservoir. However, this CO<sub>2</sub>-saturated brine in the formation has a pH value of only 3–4, which is strongly acidic that will chemically react with coal seam (Deng et al., 2015; Liu et al., 2018; Menke et al., 2017).

The chemical reaction of coal and CO<sub>2</sub> has been extensively studied in the context of acid stimulation. Such reactions lead to the opening and interconnection of the cleats (Dawson et al., 2015; Hedges et al., 2007). Calcite, dolomite, and magnesite dissolved in the CO<sub>2</sub>-saturated brine, causing the opening of originally closed or semi-closed pores, and eventually improved porosity and permeability (Du et al., 2018; Liu et al., 2010). The dissolution of minerals filled in pores and cracks changed the mechanical properties of coal (Anggara et al., 2013; Faiz et al., 2007). However, quantitative micro-pore scale study is limited, which needs to be supplemented to improve the accuracy and safety of CO<sub>2</sub> storage in deep coal seams.

It is although significant to investigate the crucial influence of microstructure changes on the porosity and permeability of coal, current research mainly focuses on the qualitative description of geochemical changes (element migration and mineral changes) (Bertier et al., 2006; Creodoz et al., 2011; Farquhar et al., 2015; Gunter et al., 1993; Hedges et al., 2007). SEM has been widely used to study the evolution rules of coal cleats and to capture the cleat geometry in coal (Zhang et al., 2016; Zhang et al., 2017). Yu et al. (2018a) observed the coal cleat morphology through SEM and presented the effect on nanoscale mechanical heterogeneity. Liu et al. (2017) described pore connectivity and types of pores in high-rank coals.

Here, we thus used the reactor to simulate the reaction of CO<sub>2</sub> with the coal seam at high temperature and high pressure. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) are used to identify and image the changes of specific minerals and the microstructure before and after the reaction and then to evaluate the influence of CO<sub>2</sub>-saturated brine on coal microstructure.

## 2 MATERIALS AND METHODS

### 2.1 Geological Setting

The Collie Basin located 150 km southeast of Perth is composed of three sub-basins (Cardiff, Shotts, and Muja), covering an area of approximately 230 km<sup>2</sup>. It is one of the largest basins in Western Australia. The Permian strata were preserved in a northwest-trending graben (Hocking et al., 1994; Li et al., 2014). The Collie coal seam developed in the Permian throughout the basin (Backhouse, 1991; Yu et al., 2018a), which consisted of three sets of coal seams that were typical carbonaceous coal seams, whose rank was subbituminous and vitrinite reflectance was 0.41–0.61%. The total thickness of Collie coal seams is around 800 m.

## 2.2 Experimental Procedure

### 2.2.1 Experimental Materials

Three samples of the Collie formation in the Collie Basin named S1, S2, and S3 were collected to conduct a high-pressure and high-temperature (HPHT) geochemical simulation experiment. The samples are polished by p1200 silicon carbide (particle size is  $9.8 \pm 0.5 \mu\text{m}$ ) to ensure that the sample is sufficiently smooth for SEM experiments. We injected CO<sub>2</sub> into deionized water that dissolved 5 wt% NaCl to get the CO<sub>2</sub>-saturated brine (live brine).

### 2.2.2 Geochemical Simulation Experiment

We use the experimental instrument to carry out the HPHT geochemical simulation experiment, the volume of the experimental container is 1 L, and the experimental temperature and pressure are 50°C and 20 MPa, respectively. The experimental conditions are calculated from the geothermal gradient of the Collie Basin and the depth of the Collie coal seams. The geochemical simulation experiment was conducted as follows:

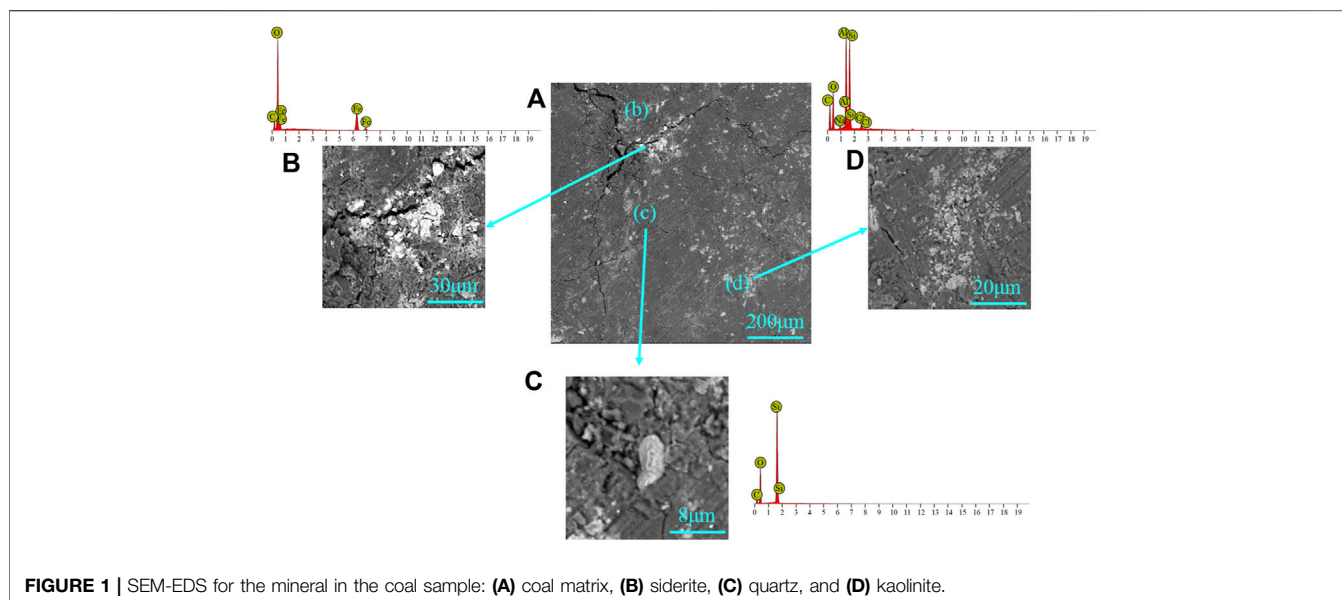
1. The samples were polished and imaged by SEM at room temperature and pressure.
2. The sample placed in a reactor and the system was vacuumed for 24 h to ensure no air remained.
3. CO<sub>2</sub> and brine were pressurized to 50°C and 20 MPa and then injected into the reactor. In order to ensure the stability of the reaction system, the temperature and pressure were continuously monitored during the experiment.
4. The sample was took out and put in a vacuum-drying oven after 24 h, and then the SEM experiment was performed.

### 2.3 SEM Image Processing

Scanning electron microscopy (SEM) was performed to scan the coal samples in different scales (200X–4000X) to investigate the morphological characteristics and mineral element composition of the coal sample. In addition, the energy dispersive spectroscopy (EDS) (Acquafredda et al., 1999) method was performed to get elemental composition, which thus applies to mineral identification and distribution. *Avizo* is used for SEM grayscale image processing. SEM grayscale images were first denoised with a *Gaussian filter* to obtain clearer and more detailed images (Blinchikoff and Zverev, 1976; Mayer et al., 2003; Yu et al., 2018b; Yu et al., 2019a; Yu et al., 2019b) and then segmented by *Watershed Segmentation* module, which can extract the microscopic cleats of the sample from the sample skeleton (Vincent and Soille, 1991; Szeliski, 2010; Zhang et al., 2020). Finally, we used the *Label Analysis* module to quantitatively obtain the sample porosity, equivalent diameter, perimeter, and area before and after solid–liquid reaction. The equivalent diameter refers to the diameter of a circle that is the same as the area occupied by the cleat and represents the size of the cleat. The area fraction refers to the ratio of the number of pixels occupied by cleats to the number of pixels in the overall picture and represents the cleat porosity of the sample.

### 2.4 X-Ray Diffraction Measurements

XRD experimental analysis was conducted with a Bruker-AXS D8 Advance diffractometer (Yuan et al., 2021). These cleated samples



were ground to 800-mesh and put into a container. Then, the element energy spectrum of the sample was acquired by XRD equipment, compared with the existing standard mineral energy spectrum to determine the mineral type and content.

## 3 RESULTS AND DISCUSSION

### 3.1 Mineral Composition and Microstructure

The mineral composition of the coal sample is mainly obtained from the XRD experiment (Yu et al., 2018a). There are three inorganic minerals (not including the coal matrix) in the Collie coal sample: kaolinite (49.1%), quartz (36.6%), and siderite (14.3%). Kaolinite  $[Al_4(Si_4O_{10})(OH)_8]$  is a common aluminosilicate mineral in sedimentary rocks, while siderite ( $FeCO_3$ ) is the carbonate mineral. These two minerals can dissolve in acidic solutions, which in total take more than 60% of the mineral composition.

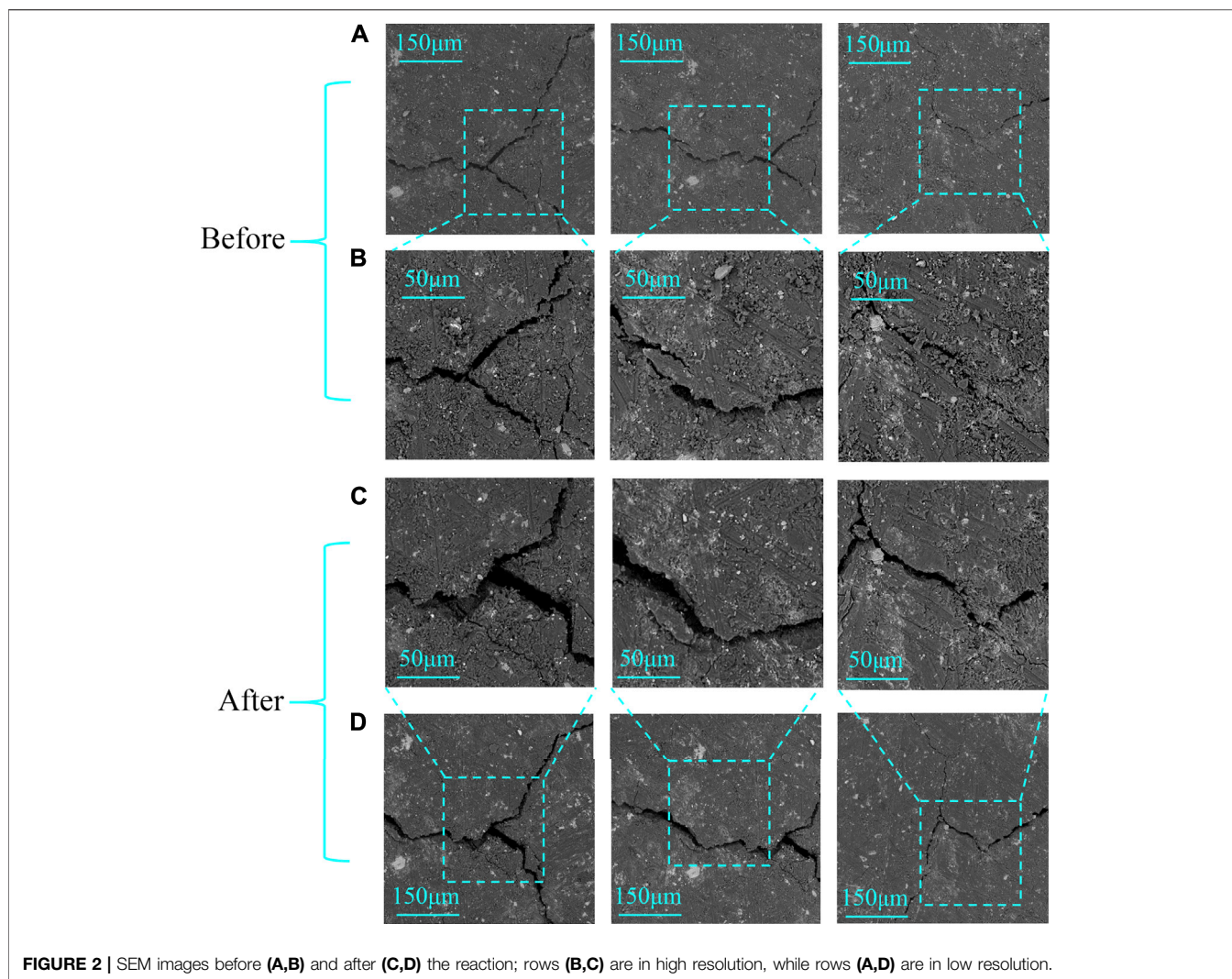
The microstructure of the coal sample was imaged by SEM. The SEM images showed the coal consisted of minerals and coal matrix. The coal matrix is very dense with almost no pores, and cleats can be clearly observed with width 0.1–1  $\mu m$ . The minerals in the SEM images show different brightness. The brighter area represented high-density minerals, and the dark parts were lower density minerals. EDS can obtain elemental composition, combining with SEM images to identify mineral types. SEM-EDS results indicated that panel (B) in **Figure 1** is siderite, as the EDS results showed the main elements in this mineral are iron, carbon, and oxygen, while the minerals are granular and gelatinous. Hence, panels (C) and (D) are quartz (silicon and oxygen) and kaolinite (aluminosilicate minerals and kaolinite booklets), respectively. These cleats provided a flow channel for live brine and increased the reaction area when the sample had contact with the acid system.

### 3.2 Microstructural Change

SEM images were obtained in different scale and resolution before and after the experiment (**Figure 2**). Rows (A) and (B) are the sample images before the experiment, whereas rows (C) and (D) are the results after the reaction. Among them, rows (A) and (D) are in low resolution for the large view of the sample, while rows (B) and (C) are in high resolution for accurate image processing.

It can be seen that there are rarely matrix pores in the sample; however, there are many cleats throughout the sample. These cleats provided a flow channel for live brine and increased the reaction area when the sample had contact with the acid system. The morphology and size of the cleats in the coal sample critically changed. Some cleats enlarged and some closed, and also new cleats were generated after the reaction (**Figure 3**). This phenomenon may be generated by mineral dissolution and small piece migration. The pH of  $CO_2$  saturated brine is very low at high temperature and high pressure, leading to mineral dissolution (Du et al., 2019).

We extracted the cleats from SEM images, shown in column (2) of **Figure 3**. The expansion, generation, and closure of the cleats are obviously shown. We found that most of the cleats especially primary cleats enlarged a lot in these three samples, while the disconnected narrow cleats expanded visibly after the reaction (points c and d, e and f, k and l), forming large cleats. Moreover, new cleats were generated (points g and h, i and j). However, the cleats (point a) in S1 significantly shrunk (point b) after the reaction. The dissolution of siderite and kaolinite is the main reason leading to cleat expansion and new cleat generation (He and Morse, 1993; O'Connor et al., 2000; Kanakiya et al., 2017). However, mineral dissolution resulted in the small pieces in the coal sample migration, in turn causing some cleats shrink or even disappear. These changes in the microstructure illustrate that the disappearance of small individual cleats makes the cleat network better connected.



**FIGURE 2** | SEM images before (A,B) and after (C,D) the reaction; rows (B,C) are in high resolution, while rows (A,D) are in low resolution.

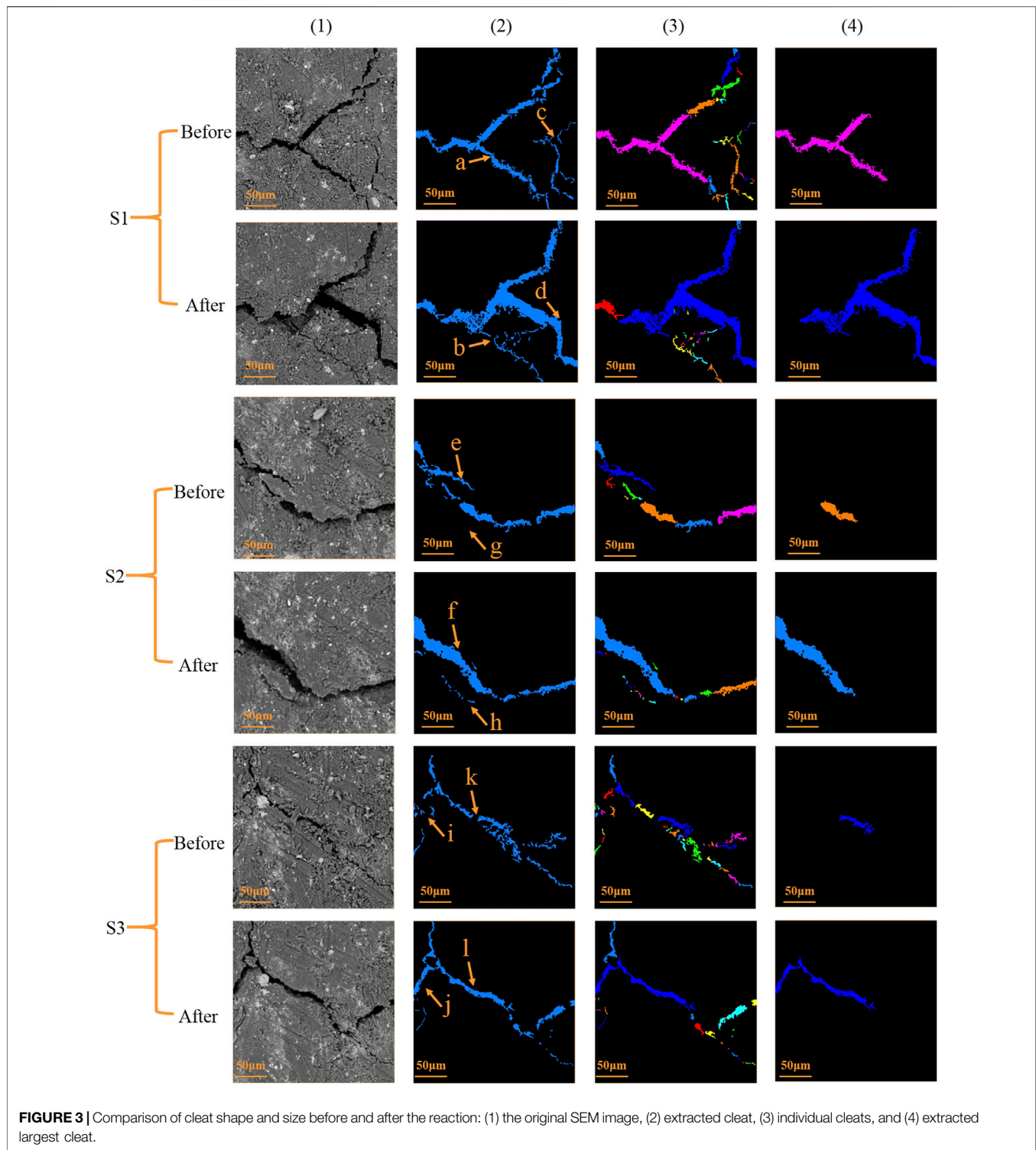
Both the dissolution and migration of minerals affect the microstructure of the coal sample. The SEM images showed that the cleat area has increased significantly, indicating that the porosity and pore volume of the coal sample have increased after the experiment, which is consistent with that reported in the previous research (Liu et al., 2015; Liu et al., 2017; Yu et al., 2018a; Zhang et al., 2016). It can also be observed that the original small cleats were connected forming a new large path, and such a change will greatly increase permeability (Cai et al., 2018).

### 3.3 Cleat Quantified Analysis

We processed the image and calculated the specific cleat change. All cleats were identified and marked with a different color, and the largest cleats (max-cleat) of each sample were also extracted (Figure 3). The tested samples were analyzed using the average cleat aspect ratio, cleat porosity, average cleat width, and cleat density. Meanwhile, the perimeter, area, aspect ratio, and fraction were applied for the maximum cleat. The results showed that the cleats in the coal sample after geochemical reaction dramatically changed, especially the largest micro-cleats (Figure 4).

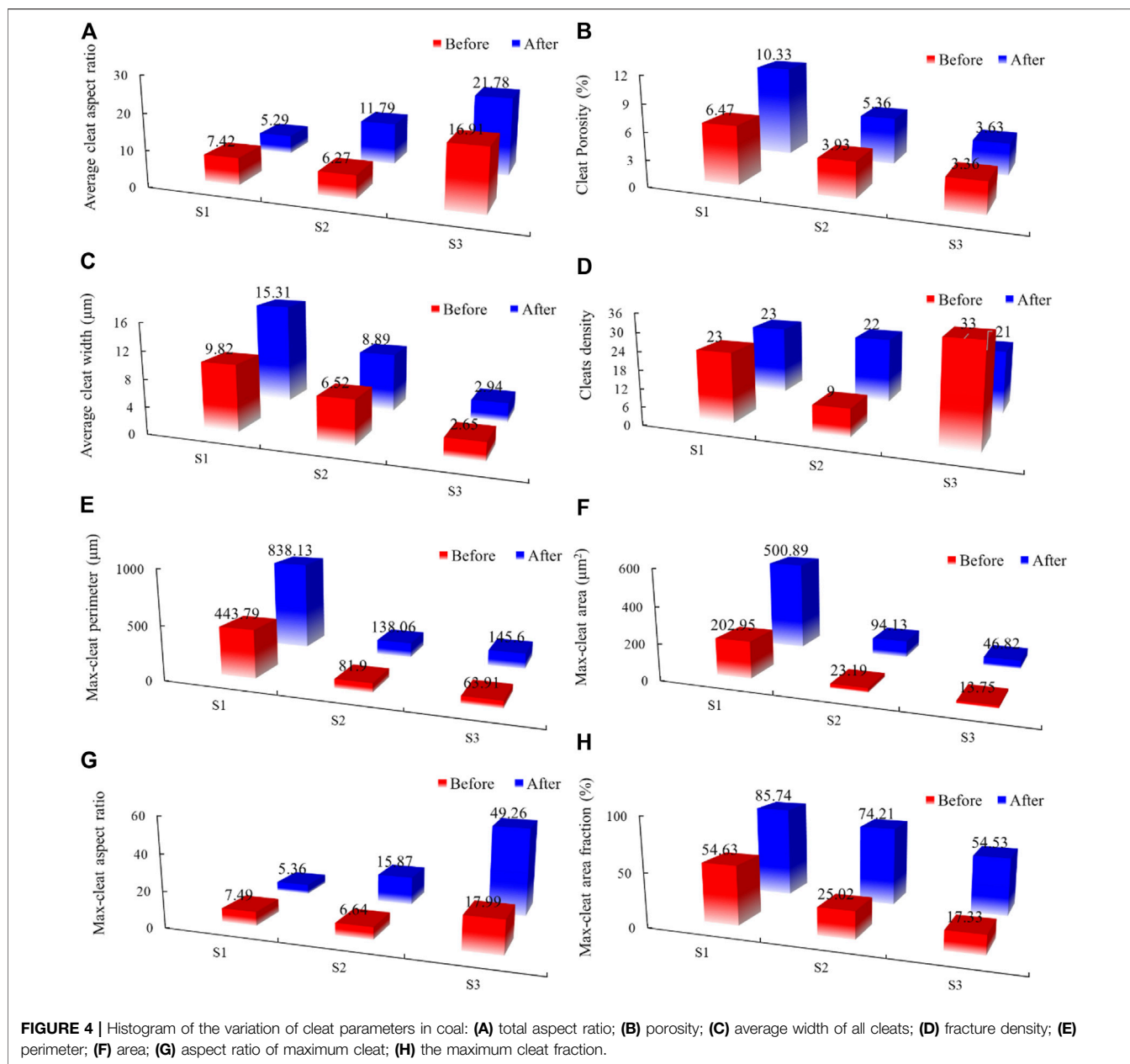
The cleat porosity of these three samples all rose before and after the reaction, changing from 3.36 to 10.33%. The average cleat width also increased after the reaction, and the variation was from 0.29 to 5.49. Such results indicated  $\text{CO}_2$ -saturated brine can indeed expand the cleats. The average aspect ratio varied in each cleat, from 6.27 to 21.78. The average aspect ratio of S1 decreased, while that of S2 and S3 increased after the reaction. The main reason that can explain this is many isolated cleats were connected after the reaction in S2 and S3, therefore leading to the rise in the average aspect ratio. The cleat density shows a distinct change, due to some smaller cleats merging into larger cleats and the formation of new cleats.

The max-cleat of each sample is significantly important for fluid and gas flooding; hence, we also analyzed the max-cleat (Figure 4). The size max-cleat also increased after the reaction: the largest increment value is 127.83% (max-cleat perimeter), 308.02% (max-cleat area), 31.27 (max-cleat aspect ratio), and 49.19% (max-cleat area fraction). It is concluded that the max-cleat changed mostly among all cleats, which would severely put an impact on the sample permeability.



The increment of cleat size after the reaction illustrated that the micro-cleat structure in the coal has greatly changed, leading to the increase of porosity and the connectivity improvement of the cleat network, thereby providing a better flooding path and storage space for CO<sub>2</sub> in the deep coal seam. It is worth mentioning that this result is consistent with that reported by

Zhang et al. (2018), who observed that the calcite-rich coal was partially dissolved during CO<sub>2</sub>-saturated brine flooding, resulting in a significant increase in absolute porosity and connectivity. Yu et al. (2018b) studied fractured shale exposed to CO<sub>2</sub>-saturated brine, and they also found that connectivity and apertures of micro-fractures in the shale were significantly improved after



CO<sub>2</sub>-saturated brine injection. However, CO<sub>2</sub>-saturated brine did not always increase rock permeability. The permeability of unconsolidated sandstone was greatly reduced after CO<sub>2</sub>-saturated brine injection. The decrease in permeability was induced by the blockage of pore throats, caused by the release, migration, and reattachment of fine particles at the initial stage of CO<sub>2</sub> injection (Yu et al., 2019a). Nevertheless, the storage of CO<sub>2</sub> in different formations still needs further research.

## 4 CONCLUSION

CO<sub>2</sub> injection into deep geological formations has been proven to be an effective way to mitigate climate change. However, the injected

CO<sub>2</sub> reacts with the formation water and forms acidic brine (Yu et al., 2018c; Yu et al., 2019a), which would react with the coal seam and change the microstructure of the coal seam. Therefore, it is very important to fully understand the interaction of CO<sub>2</sub>-saturated brine with coal seam and its impact on the microstructure. However, most of the work is focused on the qualitative geochemical changes (Du et al., 2019; Gunter et al., 1993; Hedges et al., 2007), and there is only limited work to better understand micron-scale dissolution, although microscale microstructure changes are critical to the CO<sub>2</sub> storage in deep coal seams.

Therefore, we conducted experiments to study microstructure changes before and after reaction between CO<sub>2</sub>-saturated brine and coal samples at the micrometer scale. The microstructure of coal partially dissolves after being exposed to live brine, which is

consistent with the change in cleat size and shape. The mineral dissolution at the edge of the cleat greatly increases the volume fraction of the cleat, resulting in a significant increase in the equivalent diameter and area of the cleat. In addition, it is important that the reaction between live brine and coal causes a better connectivity of the cleat network because the fraction of the maximum cleat has increased from 25 to 72.4%, which means that some isolated cleats connected increased the fraction of the maximum cleat, leading to an increase in CO<sub>2</sub> storage space and coal seam permeability.

Thus, overall, we have concluded that the changes in the microstructure of coal should be measured on a microscale, preferably combined with quantitative analysis, to improve the accuracy and reliability of CO<sub>2</sub> storage in deep coal seams.

## DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/Supplementary Material, and further inquiries can be directed to the corresponding author.

## AUTHOR CONTRIBUTIONS

XL was mainly responsible for analyzing and displaying research data, especially visualization data display, as well as performing research on published works and writing drafts. MAL conducted experiments and data collection, monitored the overall experimental

process, and proofread the experimental data. MIL and JZ provided the experimental samples and materials needed for the research. ZY conducted a preliminary review of the draft, proposed some suggestions for revision, and participated in subsequent revisions. WC and SC were responsible for making some histograms and looking up references cited in the text. HY proposed the idea and the design of the overall draft, checked the experimental results, and conducted a final critical review and comment on the draft with. During the second revision, YY and LUJ made changes to the grammar and vocabulary of the text, SD made changes to the figures.

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