Particle Size Effect and Temperature Effect on the Pore Structure of Low-Rank Coal
Teng Li,* Jian-jun Wu, Xing-gang Wang, and Hai Huang

ABSTRACT: High moisture content and high volatile content are typical characteristics of low-rank coal. To acquire the pore structure characteristics of low-rank coal accurately, the particle sizes and the pretreatment temperatures are two key parameters that should be considered when the low-pressure liquid-nitrogen adsorption is used. In this study, a low-rank coal sample was collected from Ordos Basin, and it was polished into four different particle sizes, 40–80 mesh, 80–120 mesh, 120–160 mesh, and 160–200 mesh, respectively. Besides, the low-rank coal samples are handled under seven various pretreatment temperatures (ranging from 120 to 300 °C); then, the pore structure characteristics of low-rank coal under various particle sizes and pretreatment temperatures are acquired. The dynamic change of pore volume and pore-specific surface area for low-rank coal is coincident. Under the same pretreatment temperatures, the mesopores’ volume continuously decreases. When the pretreatment temperature reaches 300 °C, a faint increase in their volume is observed. These results mean the mesopores are damaged during the progressive pulverization and heating procedures. When it comes to the same particle sizes, the mesopores’ volume also decreased with the increased pretreatment temperatures. Contrarily, the macropore volume is stable. This is mainly due to the decomposition of volatile matters and collapse of mesopores under the high pretreatment temperatures. However, the enrichment of ash in the mesopores could maintain the coal skeleton. The particle size effect and temperature effect mainly relate to the mesopores in low-rank coal, and the pores with the aperture below 5 nm contribute predominantly, followed by the pores with the aperture ranging from 5 to 10 nm.

1. INTRODUCTION
Pore structure is an essential parameter to evaluate the unconventional reservoir. The nanometer pores are developed in the unconventional reservoir, and with the low-temperature liquid-nitrogen adsorption measurements, the pore structure parameters of the unconventional reservoir could be acquired. The pore structure of the porous mediums with various particle sizes has been previously studied.1−6 Besides, the porous medium samples would be dried before the measurement to discharge the impurities in the samples. For the same sample, the various particle sizes and pretreatment temperatures may lead to different results of pore structures, which would finally influence the evaluation of the unconventional reservoir.

The particle size of the coal would be changed when being heated, and the pore structure would be sequentially different. During the rapid heating, the evaporation of moisture and decomposition of volatiles in the coal would produce heat, which would lead to the breaking of the coal particles,7−9 and then, the pore structure of the coal may be changed. The maturity, particle sizes, content of moisture, volatility, and porosity are the key factors that lead to the change of coal particle sizes when the temperature is increased.10 For the low-rank coal, the particle size would be reduced with the continuous heating because of its loss of moisture.11 The content of volatiles in the coal can lead to various results of particle sizes during heating. The higher or lower content of volatiles commonly features a faint influence on the particle sizes, while the moderate content of volatiles always leads to the decrease of the particle sizes.11,12 When the coal with a certain particle size is heated, the temperature for the surface and internal of the coal is different; the thermodynamic activity would lead to the development of fractures.13,14 When the temperature is lower than 200 °C, the microfractures would be born; once the temperature is increased to 300 °C, the microfractures would be enlarged to connected fractures; and with persistent increase of temperature to approximately 400 °C, the newborn fractures would decrease, but the porosity of
the coal would increase because of the decomposition of the volatiles.\textsuperscript{13–15}

The coal particle sizes have an influence on the pore structure\textsuperscript{17,19,21} and will ultimately change the adsorption isothermal curves.\textsuperscript{17} The smaller the coal particle size is, the bigger the specific surface area will be. Then, the fine-particle coal will present a higher adsorption capacity and adsorption rate.\textsuperscript{17,19} For primary coal, the adsorption capacity is mainly determined by the pores with apertures below 10 nm.\textsuperscript{19} However, the adsorption capacity of deformed coal is mainly contributed by pores with diameters less than 8 nm.\textsuperscript{20} A study of three high-volatility bituminous coal samples ($R_{\text{max}} = 0.6406$, 0.8134, and 1.0754\%) with various particle sizes ($<0.074$, 0.074–0.2, 0.2–0.25, 0.25–0.5, 0.5–1, and 1–3 mm) indicated that coal properties present faint changes with various particle sizes, whereas there are significant changes in pore structure during pulverization procedures.\textsuperscript{21} The meso- and macropore pore volume and pore specific surface area values in high-volatility bituminous coal will continuously increase during pulverization,\textsuperscript{19,22} whereas the values of micropores are complicated\textsuperscript{19} and may even present no changes.\textsuperscript{23} For deformed coal, the volume of micropores may even decrease with reduced coal particle sizes,\textsuperscript{23} owing to damage to the micropore.\textsuperscript{3} There is a massive closed pore volume in the high-rank coal;\textsuperscript{6,24–26} these closed pores will be opened with decreased coal particle sizes,\textsuperscript{26} and then, the meso- and macropore volume and specific surface area will continuously increase.\textsuperscript{27} In addition, pyrite and clay minerals were commonly blocked into the pores during the formation of the coal, and these minerals may drop during pulverization,\textsuperscript{27} leading to a further increase of the pore volume. With decreasing coal particle sizes, the content of the minerals and ash yield will increase,\textsuperscript{17,22,27,28} which will reduce the total pore volume. In that regard, the relationship between the content of minerals and the micropore volume is complicated,\textsuperscript{2,22,28} and the minerals contribute significantly to the pore volume and pore-specific surface area.\textsuperscript{25} With decreasing coal particle sizes, long pores transform to short pores,\textsuperscript{27} complicated pores become simple, and the pore structure is much simpler, favoring the storage and migration of gas.\textsuperscript{21}

It can be founded that both the pretreatment temperatures and particle sizes could influence the pore structure of the coal. However, almost all of the previous studies only focused on temperatures or particle sizes. The temperatures or the particle sizes may have an influence on the pore structure of coal with a certain range, while less attention is paid on it. With the detailed study of the temperature effect and particle size effect on coal would help us to enhance our knowledge on the dynamic change of pore structure of the coal. In this study, the collected low-rank coal sample from Ordos Basin was pulverized to four different particle sizes, and seven pretreatment temperatures were designed to investigate the dynamic change of pore structure with the low-pressure liquid-nitrogen adsorption (LP-N$_2$A) measurements.

2. RESULTS AND DISCUSSION

2.1. Maturity and Proximate Analysis of the Coal. The vitrinite reflectance of the Dafosi coal sample is 0.62%, which features as a low-rank coal. The proximate analysis results of sub-coal samples with various particle sizes show that the Dafosi coal samples contain high contents of volatile matters, ash yield, and moisture, while the contents of fixed carbon is low. Besides, with the decreased particle sizes, the contents of ash yield and volatile matters increase continuously, while that for moisture and fixed carbon feature contrary (Table 1).

| sub-coal sample | $R_{\text{max}}$/% | $M_d$ | $A_d$ | $V_{ad}$ | $F_{Cad}$ |
|-----------------|-------------------|-------|-------|---------|---------|
| DFS-4           | 0.62              | 5.44  | 14.74 | 44.23   | 44.96   |
| DFS-8           | 0.62              | 5.20  | 16.67 | 45.58   | 43.00   |
| DFS-12          | 0.62              | 4.98  | 17.18 | 46.00   | 42.50   |
| DFS-16          | 0.62              | 4.70  | 19.24 | 47.08   | 40.73   |

2.2. Characteristics of the Adsorption and Desorption Curves of Coal. The adsorption isotherm curves of the porous medium are commonly of six types, and the hysteresis loops feature four types.\textsuperscript{29} The adsorption isotherm curves of the sub-coal samples with various particle sizes and pretreatment temperatures present the IV type, and there are various types of hysteresis loops. With the same particle size, the maximum adsorbed quantity of the sub-coal samples tend to decrease with the increased pretreatment temperature (Figure 1). The adsorbed quantity is mainly determined by the total specific surface area of the porous medium, especially the internal surface area. The decreased maximum adsorbed quantity indicates that the pore structure of the sub-coal samples has been changed with the increased pretreatment temperatures. Besides, it can be found that the hysteresis loop is larger under the lower pretreatment temperatures, and the hysteresis loop becomes smaller with the increased pretreatment temperatures, meaning the connectivity of the pores in the sub-coal samples was enhanced, and the pore structure in the sub-coal samples varies from complexity to simple. With the lower pretreatment temperatures, the desorption curve is not coincided with the adsorption curve under the lower relative pressure range, but it tends to coincide under the higher pretreatment temperatures, and the type of hysteresis loop changes from $H_2$ to $H_4$. The change of the hysteresis loop indicates that the pore shape changes from cylindrical and spherical pores to parallel plate pores. It can be concluded that not only the pore structure but also the pore shape of the sub-coal samples were modified with the increased pretreatment temperatures. Under the same pretreatment temperature, the influence of the particle sizes on the pore structure is faint. The LP-N$_2$A results show that the hysteresis loop type is stable, but the maximum adsorbed quantity decreases, indicating that the dynamic particle sizes mainly change the pore structure of the sub-coal samples.

2.3. Influence of Particle Sizes and Pretreatment Temperatures on Pore Structure. 2.3.1. Characteristics of Pore Volume. For the sub-coal samples with the same particle size, the pore volumes of the sub-coal sample tend to decrease with the increased pretreatment temperatures; especially when the pretreatment temperature exceeds 210 °C, the pore volume decreases sharply (Figure 2). When the pretreatment temperature is lower than 210 °C, the pore volumes feature a linear decrease with the increased pretreatment temperature for the sub-coal samples with various particle sizes. The decrease of the pore volume for the sub-coal samples with larger particle sizes is more significant, and pore volume differences between the sub-coal samples tend to decrease under the same pretreatment temperatures, indicating that the particle size
effects play an essential role in the change of pore volume under the lower pretreatment temperatures. When the pretreatment temperatures exceed 210 °C, the pore volume differences of the sub-coal samples with various particle sizes further decrease, and the temperature effect contributes predominantly to the sharp decrease of the pore volume.

The study on the dynamic change of pore volume with high-rank coal shows that the pore volume increases with the decreased particle sizes, and this is mainly due to the continuous opening of the closed pores in the high-rank coal,\(^6,^{26}\) but this is contrary for that of the Dafosi coal samples in this study. The Dafosi coal is low-rank coal, and with the low maturity, the quantity of the closed pores is less. During the progressive pulverization, part of the pores in the coal may be damaged by the mechanical forces, which finally leads to the decrease of the pore volume. Besides, the thermodynamic activity may also contribute to the decrease of pore volume in the coal sample.

For the sub-coal samples with the same particle size, the characteristics of the incremental pore volume are similar under various pretreatment temperatures; the pore size distribution features as bimodal distribution (Figure 3). The pore volumes for the pores with various pore apertures decreased with the increased pretreatment temperatures. The pore volume of pores with the apertures below 20 nm tends to decrease significantly. However, the pore volume of pores with

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**Figure 1.** Adsorption and desorption curves of the coal samples with various particle sizes and pretreatment temperatures.

**Figure 2.** Dynamic change of pore volume for the sub-coal samples with various particle sizes and pretreatment temperatures.
pore diameters larger than 20 nm is almost unchanged, meaning that the increased pretreatment temperature has a significant influence on the pores with aperture below 20 nm, and this is obviously for the sub-coal samples with larger particle sizes (40−80 mesh and 80−120 mesh).

Under the same pretreatment temperature, the incremental pore volume of the sub-coal samples also decreases with the decreased particle sizes (Figure 3). When the pretreatment temperature reaches 270 °C, the pore size distribution differences among various pretreatment temperatures decrease. For the sub-coal samples with particle sizes below 120 mesh, the pore size distribution is almost the same as under the high pretreatment temperature (>270 °C), indicating that the thermodynamic activity has a faint influence on the pore structure.

Figure 3 shows that the bimodal distribution of pore structure is significant with the increased pretreatment temperatures, and pore size distribution tends to be uniform. The right peak of the pore size distribution continuously right shifts, indicating that the pore aperture of the sub-coal samples may be increased. The results of the average pore apertures under various particle sizes increase with the increased pretreatment temperatures (Figure 4), which supports this view, and the average pore aperture increases sharply when the pretreatment temperature rises to 180 °C.

2.3.2. Characteristics of Pore-Specific Surface Area. The pore-specific surface area of the sub-coal samples decreases with the increased pretreatment temperatures. When the pretreatment temperatures exceed 210 °C, the decrease of the pore-specific surface area increases (Figure 5). The pore-specific surface area is mainly provided by the micro- and mesopores in the coal, leading to the similar dynamic characteristics with pore volume. However, it should be noted that the dynamic change of the pore-specific surface area of the sub-coal samples with particle sizes of 40−80 mesh and 80−120 mesh features two stages with the knee point at the...
pretreatment temperature of 180 °C. For the sub-coal samples with particle sizes of 120−160 mesh and 160−200 mesh, the pore-specific surface area decreases linearly.

For the sub-coal samples with the same particle sizes, the pores with aperture below 10 nm contribute predominantly to the pore-specific surface area, and the pore-specific surface area decreases significantly with the increased pretreatment temperatures. When the pretreatment temperature reaches 270 °C, the pore-specific surface area of the sub-coal samples with particle sizes below 120 mesh is almost stable (Figure 6).

2.3.3. Influence of Particle Sizes on Pore Structure of Mesopores. The IUPAC’s pore aperture classification method was utilized in this study, and the pores are divided into micropores (<2 nm), mesopores (2−50 nm), and macropores (>50 nm). With the same pretreatment temperature, the pore structures of the sub-coal samples with various particle sizes are different, and this difference is mainly focused on the pores with aperture below 20 nm (dominantly mesopores), then the pore structure characteristics of these pores are studied in detail. In order to distinguish the change of pores with various diameters, the pores are divided into four parts, < 5, 5−10, 10−15, and 15−20 nm, respectively.

The dynamic changes of pore volume and pore-specific surface area feature a certain synchronism. Under the same pretreatment temperature, the pore volume of pores with aperture below 20 nm decreases with the decreased particle sizes. The decrease of pore volume for pores with aperture below 10 nm features the obvious decrease, and the differences for pores with various diameters tend to decrease with the increased particle sizes. The smaller the particle sizes of the sub-coal sample, the better connectivity of pores in the coal. When the pretreatment temperatures increased, the differences

Figure 5. Dynamic change of pore-specific surface area of the sub-coal samples with various particle sizes and pretreatment temperatures.

Figure 6. Dynamic change of incremental pore-specific surface area of sub-coal samples with various particle sizes and pretreatment temperatures.
Figure 7. continued
of the pore structure with various particle sizes decreased (Figure 7).

For the low-rank coal in Dafosi coal mine, the decrease of the pore volume mainly comes from the sharp decrease of the pore volume with pore aperture below 10 nm. During the progressive pulverized procedures, parts of the mesopores with aperture below 10 nm are easily damaged by the mechanical force. When the pretreatment temperature is lower, the mechanical disruption contributes predominantly to the decrease of the pore volume. The temperature would play an important role in the decrease of pore volume under higher pretreatment temperatures.

Figure 7. Curves of dynamic change of pore volume and pore-specific surface area for the pores with aperture below 20 nm.
2.3.4. Influence of Pretreatment Temperatures on Pore Structure of Mesopores. For the sub-coal samples with the same particle sizes, the pore volume decreases significantly when the pretreatment temperature is lower than 210 °C, and

![Figure 8. Histogram of dynamic change of pore volume and pore-specific surface area for the pores with aperture below 20 nm.](image-url)
the decrease of pore volume for pores with aperture range from 10 to 20 nm is faint. Once the pretreatment temperature exceeds 210 °C, there is a significant decrease of pore volume for the pores, and the decrease of pore volume is much more significant for the pores with aperture below 5 nm. The decrease of the pore volume for the sub-coal samples with larger particle sizes features obvious decrease. With the continuous decrease of the particle sizes, the decrease of pore volume for the pores is faint when the pretreatment temperature exceeds 210 °C. It indicates that the temperature has a significant influence on the pore structure of coal with larger particle sizes (Figure 8).

The content of volatile matter in the Dafosi coal is high. When the pretreatment temperature is increased, some volatiles would be decomposed (Figure 9), which may lead to the decrease of pore volume.

Due to the decomposition of the volatiles, the mass of the coal sample would be decreased. In this study, the sample mass decrease ratio was introduced to present the dynamic change of sample mass with various particle sizes and pretreatment temperatures.

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d_m = 100\% \times \left( \frac{m_i - m_0}{m_0} \right)
\]

where \(d_m\) is the mass decrease ratio, %; \(m_i\) is the sample mass when it is pretreated at temperature of \(i\); \(m_0\) is the sample mass before it is treated; \(i\) is the various pretreatment temperatures, 120, 150, 180, 210, 240, 270, and 300 °C, respectively.

With the increased pretreatment temperatures, the mass of the sample decreases (Figure 10). When the pretreatment temperature is lower than 180 °C, the mass decrease ratio of the sub-coal samples with larger particle sizes features superior than that of smaller particle sizes. With the continuous increase of the pretreatment temperature, the mass decreases sharply for the sub-coal samples. When the pretreatment temperature reaches 300 °C, the maximum mass decrease ratio is approximately 1.2%, which is quite lower than the content of volatiles in the coal. This indicates that the decomposition of volatiles in the low-rank coal is not the dominant factor that leads to the damage of pore structure.

The decomposition of small quantity of volatiles in the Dafosi coal cannot lead to the massive decrease of the pore volume. The probable explanation for the decrease of pore volume under higher pretreatment temperatures would be the collapse of the pores, which results in the continuous decrease of pore volume. The content of ash yield in the Dafosi coal sample is high, but the scanning electron microscopy (SEM) and energy spectrometer results show that the Dafosi coal is compact (Figure 11), and there are less inorganic minerals distributed on the surface of the coal matrix, then the inorganic minerals may be enriched in the pores of the coal, and the apertures of these pores may be less than 20 nm. Although the pores collapse under higher pretreatment temperatures, the inorganic minerals in the pores could sustain the shape of the pores. The content of ash yield in the sub-coal samples with smaller particle sizes is higher than that with larger particle sizes, then the decrease of the pore volume is significant. Besides, the supporting function of ash yield in the pores leads to the faint decrease of pore volume under high pretreatment temperatures.

2.4. Uncertainty. In order to analyze the particle size effect and temperature effect on the dynamic change of pore structure, the same sub-coal samples were repeatedly used.
for seven times. The sub-coal samples not only experienced repeatedly high temperature and low temperature but also high pressure and low pressure. The fatigue effect may also influence the pore structure of sub-coal samples, but this

Figure 11. SEM and EDS of Dafosi coal samples.
cannot be represented currently. In fact, when the pretreatment temperature exceeds 270 °C, the dynamic change of the pore structure with pore aperture below 10 nm is fluctuant, and this may be related to the fatigue effect of sub-coal samples.

3. CONCLUSIONS

(1) The pretreatment temperatures and particle sizes could both influence the pore structure of the low-rank coal. With increased pretreatment temperatures and decreased particle sizes, the pore volume and pore-specific surface area of the low-rank coal would decrease, and this mainly focuses on the pores with aperture below 20 nm, especially that for the pores with diameters less than 5 nm.

(2) The particle size effect predominantly influences the pore structure of low-rank coal with larger particle size. The pretreatment temperatures feature a complete influence on the pore structure of low-rank coal, especially the high pretreatment temperature. Under high pretreatment temperatures, the decomposition of the volatiles in the low-rank coal is less, which contributes to parts of the decrease of the pore volume. The rapid collapse of mesopores (pore aperture < 20 nm) is the dominant factor to the sharp decrease of pore volume for the low-rank coal with larger particle size. The low-rank coal only experiences a certain degree of thermal evolution; some amounts of the produced liquid hydrocarbon may not be migrated. Due to the high adsorption capacity of mesopores, the residual liquid hydrocarbon would condensate and accumulate into the mesopores, especially that for the pores with aperture below 20 nm. When the low-rank coal is heated again, some of the hydrocarbon would be decomposed and evaporated from the mesopores, which leads to the collapse of the mesopores.

4. METHODS

4.1. Samples. The coal sample was collected from the work face of no. 4 coal seam in Dafosi coal mine, Binchang mining area, Ordos Basin, China. The collected coal sample features a size of 20 cm × 10 cm × 10 cm, and the coal sample was packaged with the vacuum bag and sent to launch the relative measurements rapidly.

4.2. Measurement Procedures. The maturity of the coal sample was first measured based on the method of determining microscopically the reflectance of vitrinite in coal (GB/T 6948-2008, Chinese national standard). Following that, the coal sample was pulverized, and the laboratory sieving method was utilized to select four different coal sample particle sizes, 40–80 mesh (DFS-4), 80–120 mesh (DFS-8), 120–160 mesh (DFS-12), and 160–200 mesh (DFS-16), respectively. The proximate analyses of these four sub-coal samples were performed according to the proximate analysis of coal (GB/T 212-2008, Chinese national standard). The sub-coal samples are sprayed with gold, and the SEM of these coal samples is viewed with the general specification of energy-dispersive X-ray spectroscopy quantitative analysis with the probe microanalyzer and scanning electron microscope (GB/T 17359-1998, Chinese national standard). Finally, the LP-N₂A measurements with these sub-coal samples were launched to investigate the characteristics of the pore structure under various particle sizes and pretreatment temperatures, and the determination of the specific surface area of solids by gas adsorption using the BET method (GB/T 19587-2017, Chinese national standard) was taken as a reference to start the LP-N₂A measurements.
120 °C under vacuum. When the vacuity reaches approximately 0.5 mbar, the sample would be vacuumed with another 3 h to achieve the measurement standard. The pretreated sub-coal samples would launch the LP-N2A measurements under −196 °C with the relative pressures range from 0 to 1. The high-purity nitrogen was used as the adsorbate. To avoid the heterogeneity, the sub-coal samples after the LP-N2A measurements would be repeatedly used to launch LP-N2A measurements under various pretreatment temperatures. In this study, seven various pretreatment temperatures were set from 120 to 300 °C with a step length of 30 °C. Figure 12 shows the procedures of LP-N2A measurements.

After the LP-N2A measurement, the pore volume and pore size distribution of the sub-coal samples were calculated with the Barrett–Joyner–Halenda (BJH) model, and the pore-specific area of the sub-coal samples was acquired with the Brunauer–Emmett–Teller (BET) model. The BET model was deduced from the classical statistical theory to present the adsorption of gases in multimolecular layers. The BET model is the theory base for the adsorption of gases on the particle surface, and it was widely used to study the adsorption capacity of particles. An inference of the BJH model is that the shape of pores in the porous medium is cylindrical, and the pore apertures could be calculated with the Kelvin equation, as shown in eq 3.

$$P/V(P_0 - P) = [1/V_m \times C] + [(C - 1/V_m \times C) \times (P/P_0)]$$

where $P$ is the nitrogen partial pressure; $P_0$ is the saturated vapor pressure of nitrogen; $V$ is the adsorption quantity of nitrogen, cm$^3$/g; $V_m$ is the single layer saturated adsorption quantity of nitrogen, cm$^3$/g; and $C$ is the adsorption constant, which is related with the porous medium itself.

The calculation of the pore apertures and pore size distribution of porous medium is mainly based on the capillary condensation theory and volume equivalent substitution theory. An inference of the BJH model is that the shape of pores in the porous medium is cylindrical, and the pore apertures could be calculated with the Kelvin equation, as shown in eq 3.

$$R_k = -0.414/\log(P/P_0)$$

where $R_k$ is the Kelvin radius; $P$ is the nitrogen partial pressure; and $P_0$ is the saturated vapor pressure of nitrogen.

**AUTHOR INFORMATION**

**Corresponding Author**

Teng Li – College of Petroleum Engineering, Xi’an Shiyou University, Xi’an 710065, China; Shandong Key Laboratory of Depositional Mineralization & Sedimentary Mineral, Shandong University of Science and Technology, Qingdao 266590, China; orcid.org/0000-0001-9379-8733; Email: tli@xsyu.edu.cn

**Authors**

Jianjun Wu – Institute of Engineering Technology, PetroChina Coalbed Methane Company Limited, Xi’an 710082, China
Xinggang Wang – Exploration & Development Research Institute, Tuha Oilfield Company, Hami 839009, China
Hai Huang – College of Petroleum Engineering, Xi’an Shiyou University, Xi’an 710065, China

Complete contact information is available at:
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**Notes**

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