

Article

Heterogeneity of Pore and Fracture Structure in Coal Reservoirs by Using High-Pressure Mercury Intrusion and Removal Curve

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Abstract: The pore structure determines the desorption, diffusion and migration of coalbed methane, and the heterogeneity of the pore structure seriously restricts the diffusion and seepage process and productivity of coalbed methane. Therefore, this paper takes eight coal samples in the Linxing area as the research target and uses the high-pressure mercury injection test to describe the pore structure distribution. On this basis, three kinds of single and multifractal models are used to calculate the progressive mercury removal curve, and the correlation analysis is carried out to determine the physical significance of the mercury removal fractal dimension. Finally, the relationship between the fractal dimension of the mercury curve and the pore structure parameters is defined, and the applicability of fractal models in characterizing pore structure heterogeneity is discussed. The conclusions of this paper are as follows. (1) Samples can be divided into two categories according to porosity and mercury removal efficiency. Among them, the mercury removal efficiency of sample 1–3 is higher than 35%, and porosity is less than 9.5%, while those of sample 4–8 are the opposite. The seepage pore volume percentage of sample 1–3 is 35–60%, which is higher than that in sample 4–8. (2) The difference of the samples' fractal dimension calculated with the Menger and Sierpinski models is small, indicating that the pore structure distribution heterogeneity of the two types is similar. The multifractal model shows that the adsorption pore and macro-pore heterogeneity of sample 4–8 are stronger than those of sample 1–3, and the pore distribution heterogeneity is controlled by the low value of pore volume. (3) The results of the two single fractal calculations show that the pore structure distribution heterogeneity of sample 4–8 is stronger than that of sample 1–3. The multifractal model calculation shows that the adsorption pore distribution heterogeneity of sample 4–8 is stronger, and the low value of pore volume controls the pore distribution heterogeneity. (4) The mercury fractals based on the Menger model can reflect the adsorption pore distribution and macro-pore distribution heterogeneity, while the Sierpinski model can only reflect the adsorption pore distribution heterogeneity at the mercury inlet stage.

Keywords: coal reservoir; pore structure; mercury injection curve; mercury removal curve; the fractal dimension



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1. Introduction

Relevant literature and engineering practice show that China's coal reservoirs have the characteristics of low permeability, low porosity and low saturation, which have become the key to restricting China's coalbed gas drainage and production [1–3]. Abundant literature shows that the pore structure has an important influence on the desorption, diffusion and migration of coalbed methane. Pore and fracture distribution heterogeneity (PFDH) restricts the seepage process of coalbed methane, leading to decreased CBM production and ultimate recovery. The application of mercury intrusive porosimetry (MIP) into the estimation of the

soil water characteristic curve (SWRC) of soft clayey soil under wetting and drying cycling conditions should be reviewed as well because the MIP greatly contributes to the soil water retention behavior of deformable soil (Hu et al., 2013) [4]. Therefore, pore structure has become important in determining coalbed methane productivity [5–9].

For this reason, the low-temperature liquid nitrogen test (LPN₂GA), the carbon dioxide adsorption test (LPCO₂GA), low-field nuclear magnetic resonance technology (LF-NMR), the high-pressure mercury injection test (HPMI), scanning electron microscopy (FE-SEM) and other experiments are used to study the pore and fracture of coal reservoirs [10–14]. Among them, the high-pressure mercury injection test has the advantages of being fast, convenient and low cost and is widely used in characterizing the pore structure parameters of coal reservoirs. On this basis, fractal theory is used to characterize pore and fracture distribution heterogeneity [15–17]. Xiao [18] used a high-pressure mercury injection test to conduct the fractal calculation of pore structure based on the mercury injection curve (MIC) and found that D_M can reflect pore structure distributions. Wu [19] found that the fractal dimension of coal reservoir pores is affected by porosity, specific surface area and other factors based on the mercury injection curve.

To sum up, many studies have been conducted on pore and fracture distribution heterogeneity based on the mercury injection curve and fractal calculation, but relevant literature shows that the mercury removal curve (MRC) can be used to characterize pore connectivity and has a better effect on the pore structure distributions of coal reservoirs [20–22]. However, there are few studies on the fractal characteristics of the mercury removal curve, and the fractal relationship between mercury intake and mercury removal remains to be clarified [23]. On this basis, this paper takes a block coal reservoir in Ordos as the research target and uses the high-pressure mercury injection test to clarify coal pore distribution. Two kinds of single and multiple fractal models are used to calculate the mercury curve, and pore and fracture distribution heterogeneity is characterized. The applicability of different fractal models to characterize pore and fracture distribution heterogeneity is discussed. Finally, a correlation analysis of the mercury inlet and mercury removal fractal dimensions is carried out, and the relationship between the fractal dimension of the mercury curve and pore structure parameters is clarified. It should be noted that the Hodot pore classification scheme is adopted in this paper, and pores are divided into adsorption pores (<100 nm), seepage pores (100–1000 nm) and large pores (>1000 nm) [24,25].

Due to the complex deformation and evolution history of China's coal basins, the original shape and structural style of the basins are changeable, there are unique geological characteristics and reservoir structures, and there is no systematic theory of coalbed methane mining at home and abroad. Therefore, it is of great practical significance to strengthen the study of the pore structure characteristics of coal reservoirs and their influence on adsorption and desorption and to clarify the relationship between them to guide the exploration and development of coalbed methane in this region and, also, to study the basic theory of coalbed methane.

On this basis, this paper takes the Jurassic coal reservoir in Ningxia as the research target and describes pore distribution with HPMI. Four kinds of single and multiple fractal models are used to calculate the MIC and MRC, and the applicability of different fractal models in characterizing PFDH is discussed. At the same time, the physical significance of the mercury removal fractal dimension is defined by comparing the correlation between the mercury inlet fractal dimension and mercury removal fractal dimension. On this basis, the correlation between the fractal dimension of the mercury curve and pore structure parameters is discovered, and the characterization of PFDH based on MRC and fractal theory is realized.

2. Experimental Testing and Fractal Theory

2.1. Sample Preparation and Experimental Test

The samples are taken from the Linxing area in the eastern Ordos Basin. The main coal-bearing strata are the Shanxi Formation and Taiyuan Formation. Among them, the

Taiyuan Formation is an epicontinental sea deposit, and the lower, middle and upper parts are a barrier sedimentary environment, tidal flat sedimentary environment, lagoon-facies sedimentary dark mudstone and carbonaceous mudstone. The Shanxi Formation consists of a delta plain deposit, marsh deposit, distributary channel sandstone deposit and sand mudstone interlayer, respectively [26,27]. The basic information of the samples is shown in Table 1.

Table 1. Percentage of pore volume with different pore sizes.

Sample Number	Pore Volume Percentage		
	1000–10,000 nm	100–1000 nm	<100 nm
Sample 1	0.306338	0.60915493	0.084507
Sample 2	0.518116	0.35144928	0.130435
Sample 3	0.483871	0.35483871	0.16129
Sample 4	0.320833	0.35	0.329167
Sample 5	0.339902	0.300492	0.359606
Sample 6	0.608924	0.26509126	0.125985
Sample 7	0.728643	0.2361807	0.035176
Sample 8	0.639567	0.30894289	0.05149

First, the experimental sample is ground into a powder, and its industrial and microscopic composition is tested. Second, the polished sample is dried at 60 °C for 48 h. Finally, the AUTOPORE 9505 mercury injection instrument is used to perform the pore rupture experiment. The instrument has 2 high-pressure points and 4 low-pressure points; the pressure range is 0–100 mp, the aperture measurement range is 0.019–280.310 µm, and the test temperature is 20 °C ± 1 °C. The mercury injection method of this instrument adopts continuous mercury injection, and the pore distribution and specific surface area are measured by changing the pressure [28,29].

2.2. Fractal Theory

The Menger model (M model) is shown in Equation (1) [30]:

$$\lg(dv/dp) \propto (D - 4)\lg(p) \quad (1)$$

where D_M is the fractal dimension without dimension; P is the injection pressure, Mpa; and V is the total injected volume, $\text{cm}^3 \cdot \text{g}^{-1}$.

The Sierpinski model (S model) is shown in Equation (2) [31]:

$$\ln(v) = (3 - D)\ln(p - p_t) + \ln a \quad (2)$$

where V is the volume of mercury injected, mL; P is the injection pressure, Mpa; P_t is the threshold pressure, MPa; D_S is the fractal dimension; and a is a constant.

$q \sim D(q)$ is a basic language to describe the local features of multifractal. The calculation formula of $D(q)$ is as follows:

$$D(q) = \frac{\tau(q)}{q - 1} \quad (3)$$

where $\tau(q)$ is the mass index function, and q is the statistical moment order. For details on the process, see literature [11]. The box-counting method is adopted to study the multifractal of adsorption data using LPN_2 and CO_2 GA technologies. When analyzing the volume probability of PSD in the interval $[A, B]$, the scale and measure value need to be determined. The scale and resolution are expressed as

$$\varepsilon = 2^{-k}L \quad (4)$$

$$\overline{\Delta n_i} = \frac{\Delta n_i - \Delta n_{\min}}{\sum_{i=1}^N (\Delta n_i - \Delta n_{\min})} \quad (5)$$

where ε is the scale size, k is a positive integer, and L is the interval length.

For adsorption data, relative pressure is divided into several boxes of equal length for gas adsorption, and the box size is represented by the scale size. The analysis interval of HPMP ranges from 0 to 199 MPa. Therefore, the corresponding scale size is different.

In the case of different scales, the distribution probability of pore volume in the interval $[A, B]$ satisfies

$$p_i(\varepsilon) \sim \varepsilon^{-a_i} \quad (6)$$

where $p_i(\varepsilon)$ is the mass probability function of the i th box, which is used to analyze the distribution of total gas adsorption in each box, and a_i is the singularity index.

The value of a_i characterizes the singularity strength in the i th box, and a higher value indicates higher smoothness or regularity. Conversely, a smaller value indicates a greater changing degree or stronger irregularity. a_i is related to its location, reflecting the probability of the area.

The data of the fractal model are MIC, and PFDH is discussed by calculating the fractal dimension. Whether MRC has fractal and its limits on the changes in porosity and permeability are to be discussed. Based on the MRC of the same sample, the fractal dimension of the two single fractal models and multifractal is calculated. The difference in the fractal between MIC and MRC of the same sample is discussed.

3. Results and Discussion

3.1. Pore Distribution Based on High-Pressure Mercury Injection Test

Based on the basic sample information and experimental parameters of HPMP, all samples are divided into types A and B according to porosity and mercury removal efficiency. Mercury removal efficiency (MRE) reflects the uniformity of pore throat distribution, while porosity reflects the reservoir capacity, permeability, pore water pressure and lithology identification of rocks, which are important indicators of rock pore properties. Among them, type A has high MRC and small porosity, which are more than 35% MRC and less than 9.5% porosity. Type B has low MRC and large porosity, which are less than 35% MRC and more than 9.5% porosity (Figure 1).

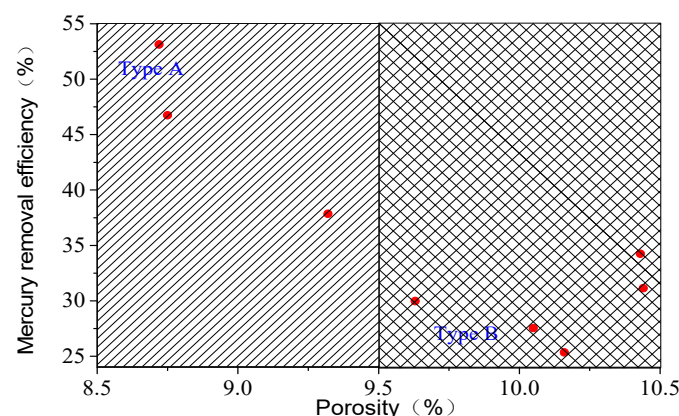


Figure 1. Sample division results of high-pressure mercury injection test.

Figure 2a shows that, in type A when the pressure is less than 1 MPa, the MIC is slow. When the pressure is 1~100 MPa, the MIC increases vertically, indicating that the samples develop micro-pores. In mercury removal, the MRC is steep, and the MRE is high, indicating that pore connectivity is good. Figure 2b shows that, in type B when the pressure is less than 0.3 MPa, the MIC is slow. When the pressure is 0.3~100 MPa, the MIC

increases. In mercury removal, the MRC is smooth, and the MRE is low, which indicates that porosity and fracture connectivity are poor. According to different research objectives, relevant literature classifies the sample types according to pore structure parameters, mineral composition and other factors. Figure 2c,d show that all samples can be divided into two categories according to pore structure parameters (total pore volume, pore volume percentage from 10 to 100 nm and >100 nm) and porosity. All samples can be divided into two categories. In order to achieve fine characterization of pore and fracture structure, pore and fracture are divided into macro-pore (1000–10,000 nm), seepage pore (100–1000 nm) and adsorption pore (<100 nm).

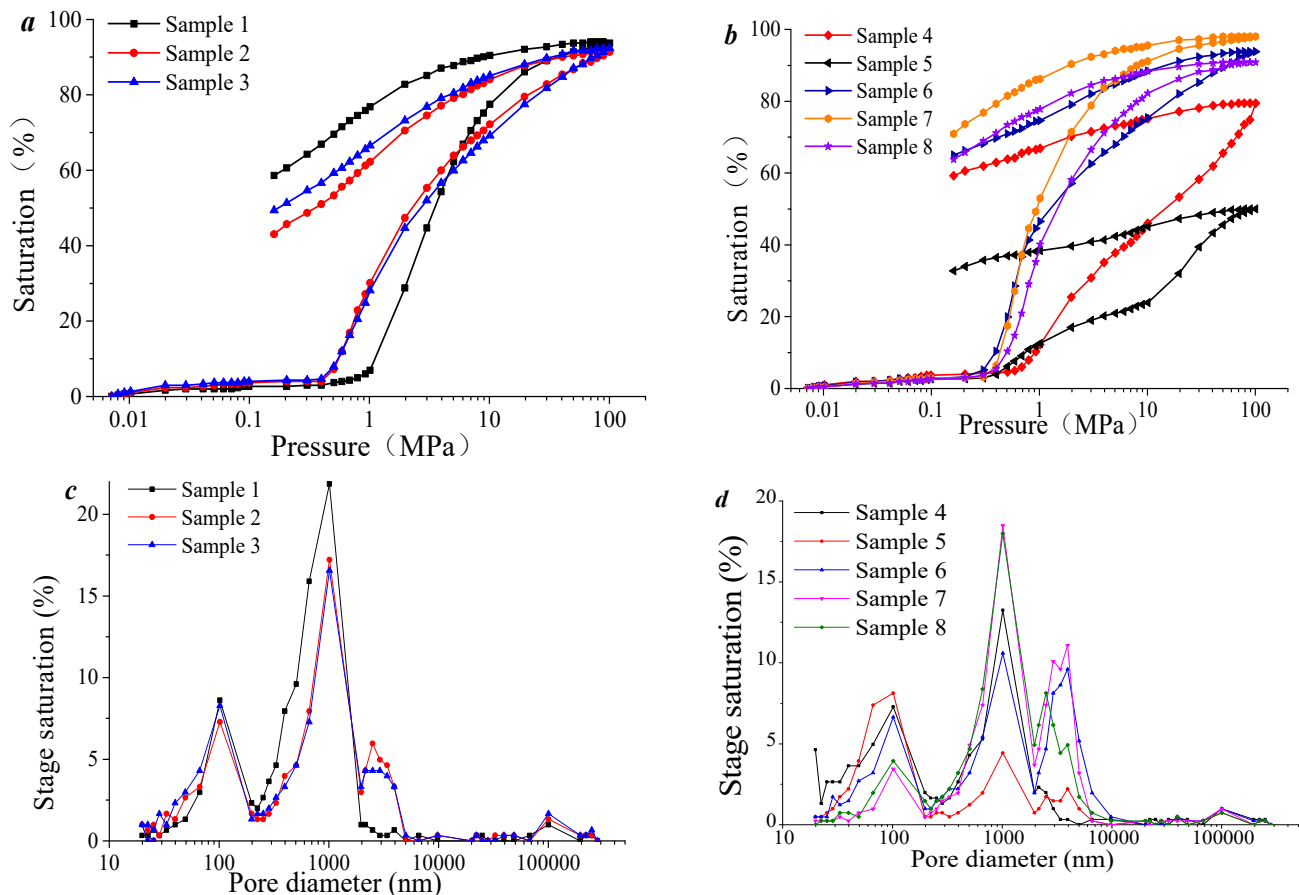


Figure 2. Mercury injection curves and pore distribution of different samples (a–d).

The macro-pore volume percentage of type B is 30–70%, which is higher than that of type A. The seepage pore volume percentage of type A is 35–60%, which is higher than that of type B, but the adsorption pore volume percentage of type B is higher than that of type A (Figure 3). In general, the seepage pores of type A are developed, and the macro-pores and adsorption pores of type B are more developed.

3.2. Refined Description of Pore Structure Distribution Heterogeneity Based on the Mercury Inlet Curve

D_M can be calculated with the M model. The fractal curve can reflect the linear negative correlation between $\lg p$ and $\lg (dv/dp)$, which indicates that the mercury inlet fractal can be well-reflected by this model. The linear fit of type A ranged from 0.83 to 0.86, and D_M ranged from 3.1 to 3.2. The linear fit of type B ranged from 0.7 to 0.95, and D_M ranged from 3 to 3.2 (Figure 4). By comparison, D_M of type A is similar to that of type B, indicating that the PFDH of type A and B are similar.

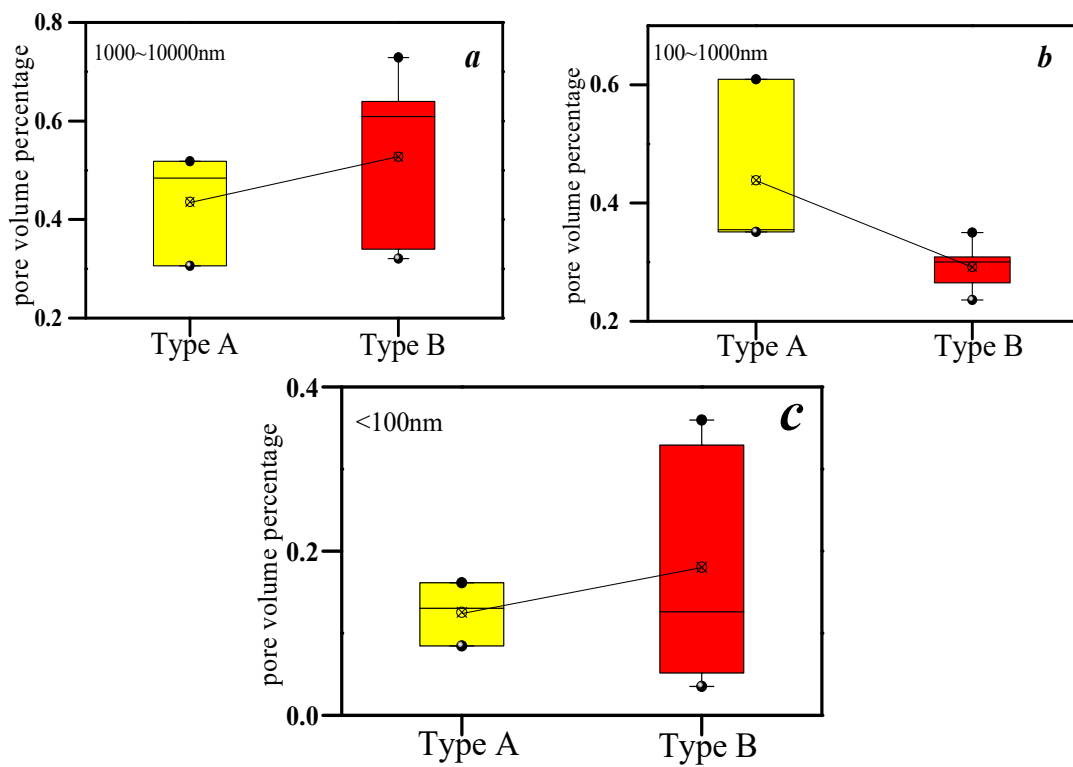


Figure 3. Pore distribution of different samples is compared (a–c).

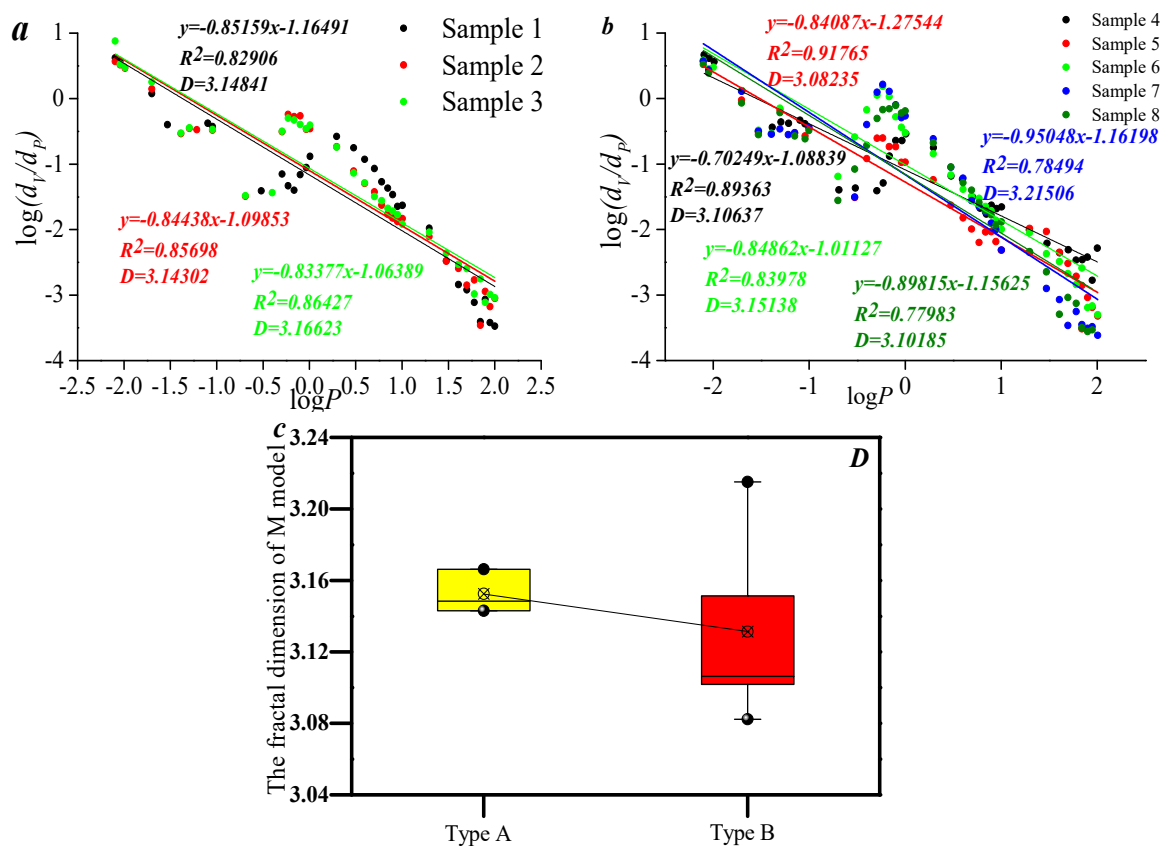


Figure 4. Fractal dimension of different samples based on the M model is compared (Based on mercury injection curve) (a–c).

D_S can be calculated with the S model. The fractal curve can reflect the linear positive correlation between $\ln p$ and $\ln v$, which indicates that the fractal of the samples can be reflected by this model. Among them, the linear fitting degree of type A is 0.50~0.60, and D_S is 2.4~2.5. The linear fit of type B ranged from 0.50 to 0.60, and D_S ranged from 2.4 to 2.5 (Figure 5). By comparison, D_M of type A is similar to that of type B, indicating that the PFDH of type A and B are similar. The calculated results of the S model are consistent with those of the M model.

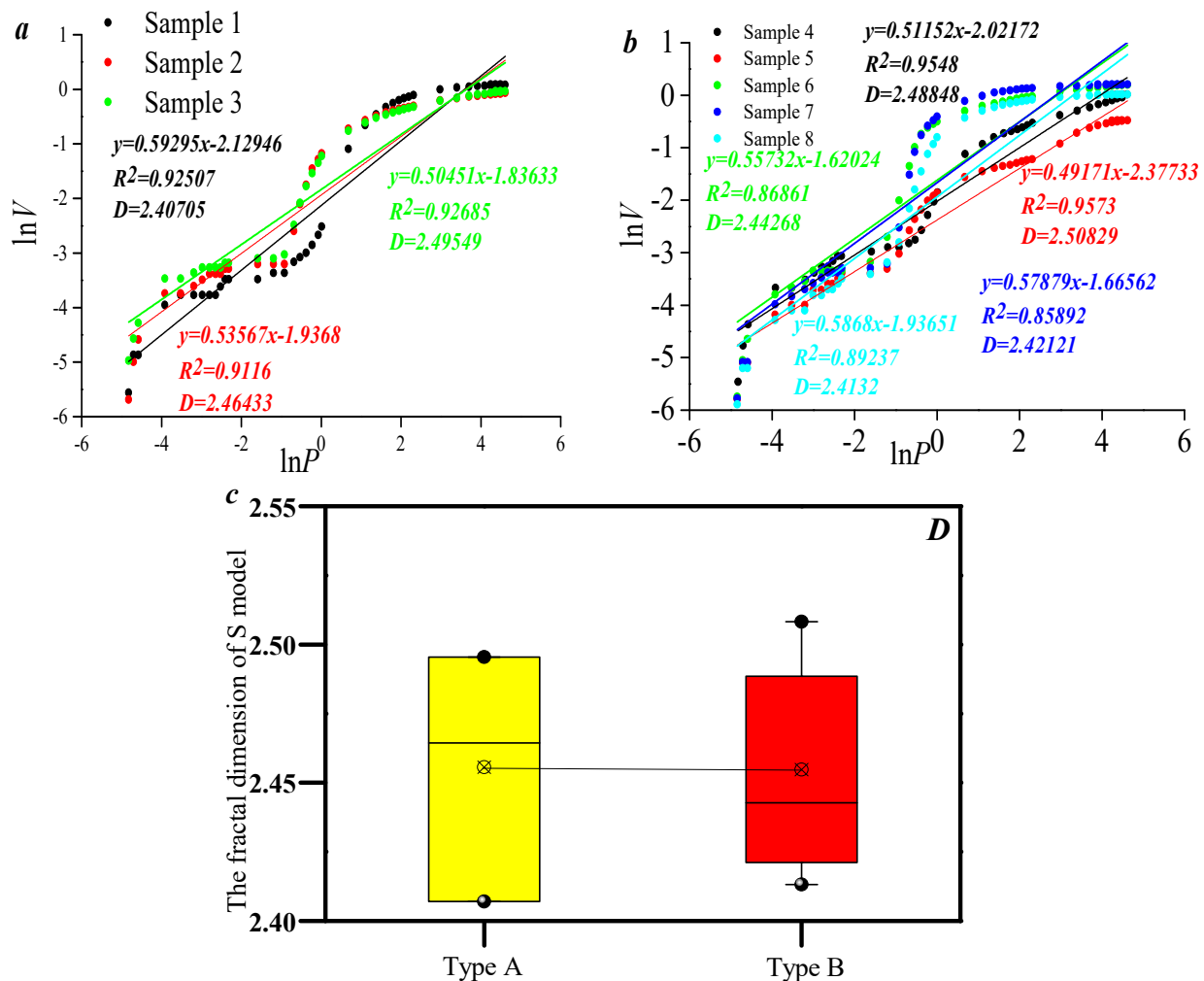


Figure 5. Fractal dimension of different samples based on the S model is compared (Based on mercury injection curve) (a–c).

Figure 6 shows that the $q \sim D(q)$ spectra show an inverse S, indicating that the pore distribution is characterized by multifractal characteristics and the pore structure is heterogeneous. $D_{-10} - D_0$ of type A ranged from 0.40 to 1.42, and $D_0 - D_{10}$ ranged from 0.40 to 0.71. $D_{-10} - D_0$ of type B ranged from 0.4 to 2.12, and $D_0 - D_{10}$ ranged from 0.13 to 0.87. The literature shows that the left spectral width represents heterogeneity in the low-value pore volume region, and the right spectral width represents heterogeneity in the high-value pore volume region. The curve on the left side of type B is larger than that of type A, indicating that type B has a strong heterogeneity in the adsorption pore volume distribution. The curve on right side of type B is larger than that of type A, indicating that type B has a strong heterogeneity in the large pore volume distribution.

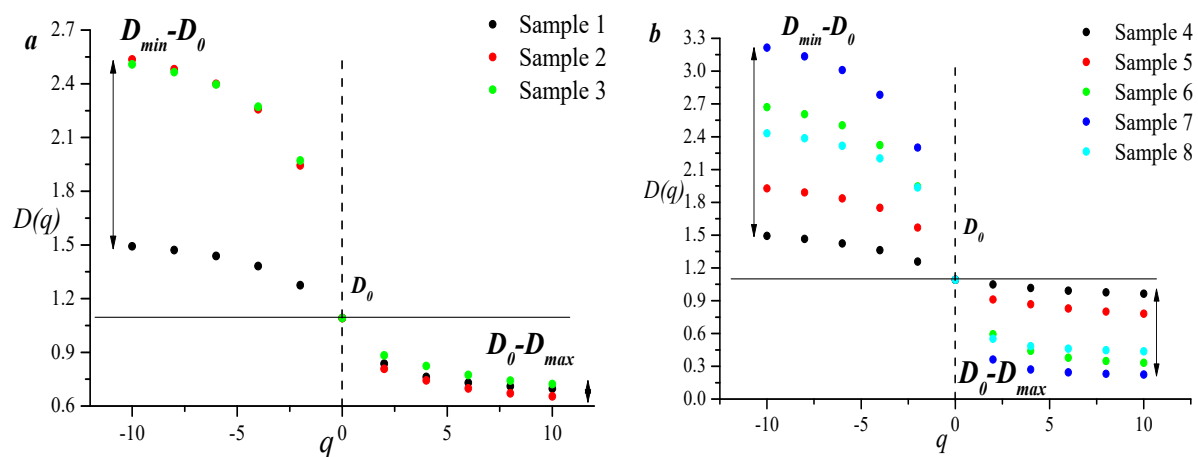


Figure 6. Multifractal of different samples based on mercury injection stage is compared (a,b).

Figure 7 shows that type B is higher than type A in the variation range of $D_{-10}-D_0$, D_0-D_{10} and D , which indicates that the adsorption pore and large pore heterogeneity of type B is stronger than that of type A, and this result is consistent with that of Figure 6.

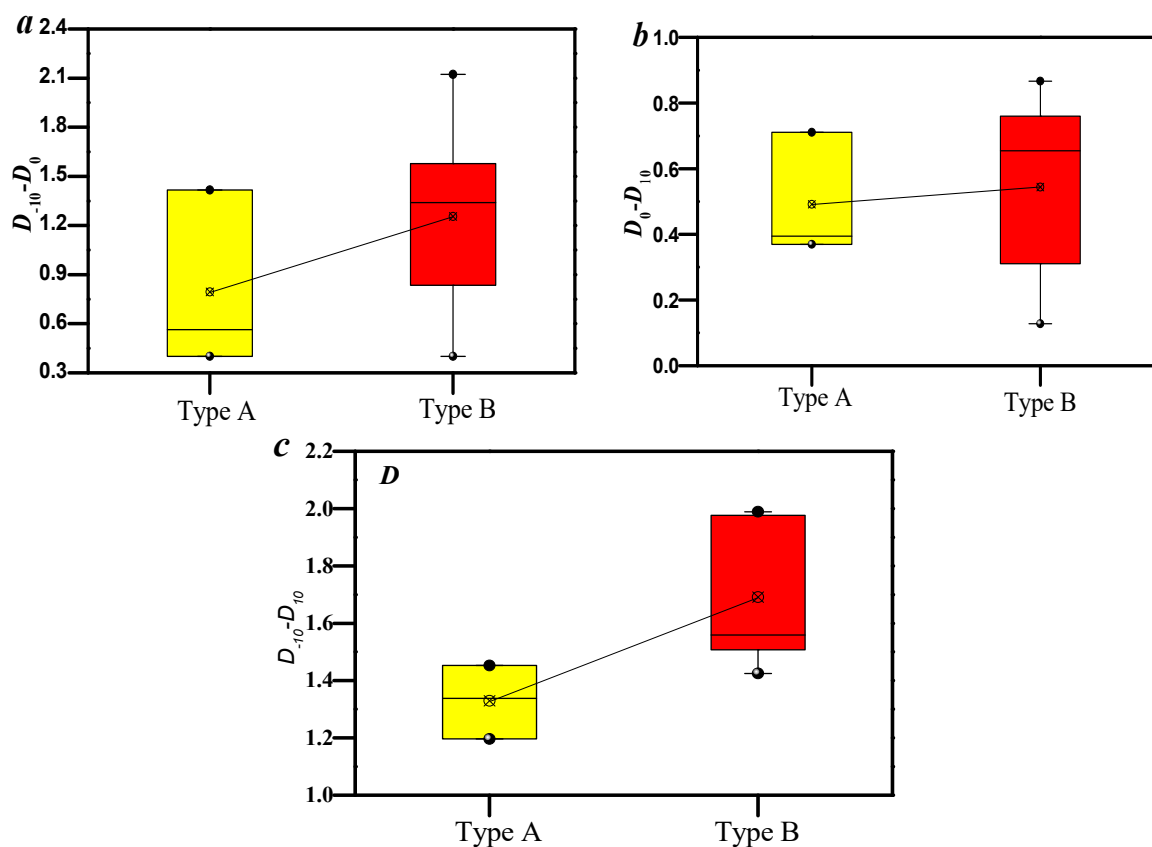


Figure 7. Multifractal of different samples in the mercury injection stage is compared (a–c).

The results of the multifractal calculation show that the relationship between $D_{-10}-D_0$ and $D_{-10}-D_{10}$, D_0-D_{10} and $D_{-10}-D_{10}$, and $D_{-10}-D_0$ and D_0-D_{10} is positive, and the relationship between $D_{-10}-D_0$ and $D_{-10}-D_{10}$ is more significant. This indicates that the low pore volume region controls the PFDH (Figure 8).

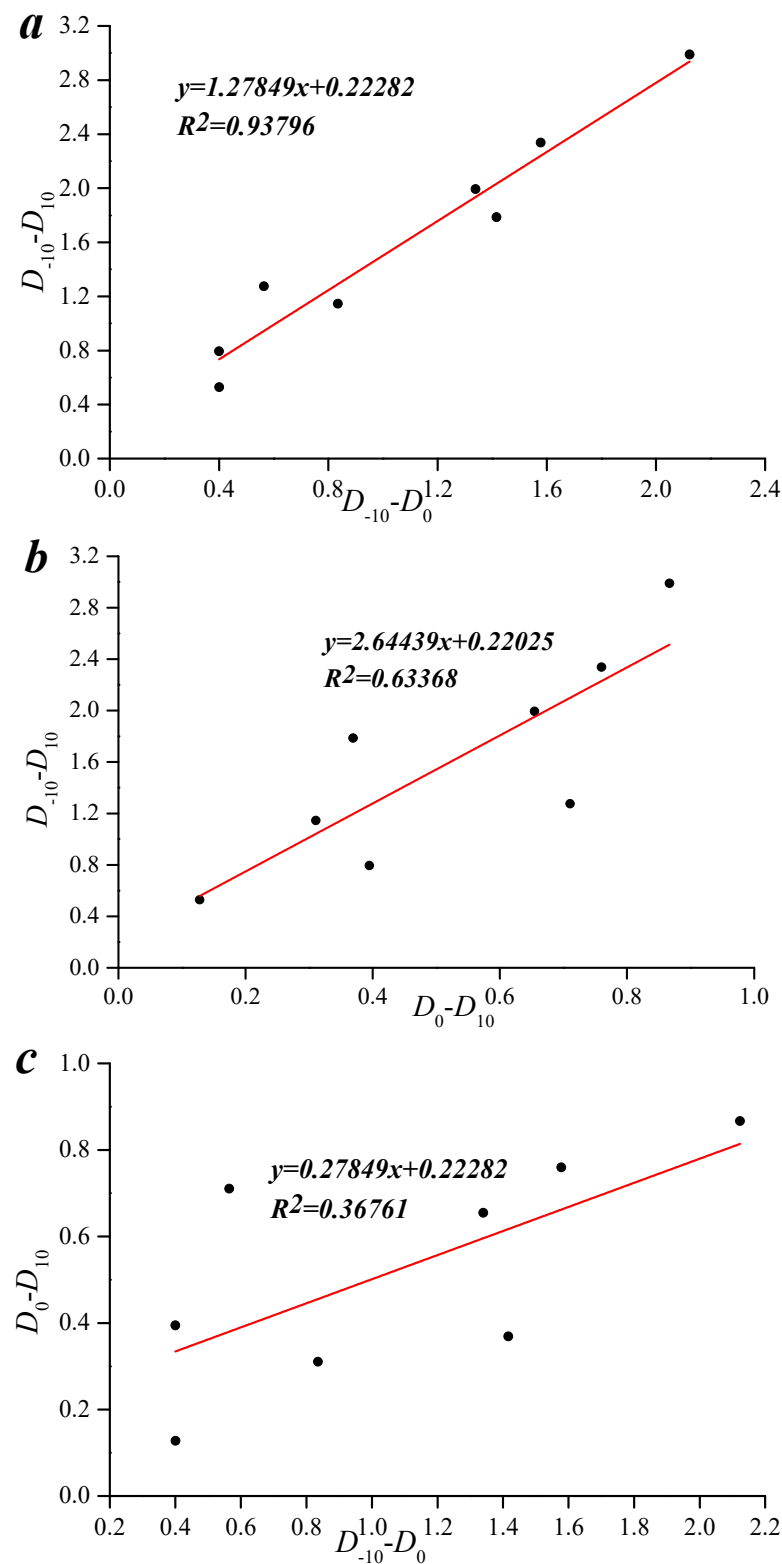


Figure 8. The relationship between different multifractal dimensions based on mercury injection curves (a–c).

3.3. Quantitative Characterization of Pore Structure Distribution Heterogeneity Based on the Mercury Removal Curve

D_M can be calculated with the M model. The fractal curve can reflect the linear negative correlation between $\lg p$ and $\lg (dv \setminus dp)$, indicating that the MRC has fractal characteristics.

The linear fit of type A ranged from 1.15 to 1.37, and D_M ranged from 2.6 to 2.8. The linear fit of type B ranged from 1.0 to 1.4, and D_M ranged from 2.6 to 3.0 (Figure 9). By comparison, D_M of type B is higher than that of type A, indicating that the PFDH of type B is stronger than that of type A.

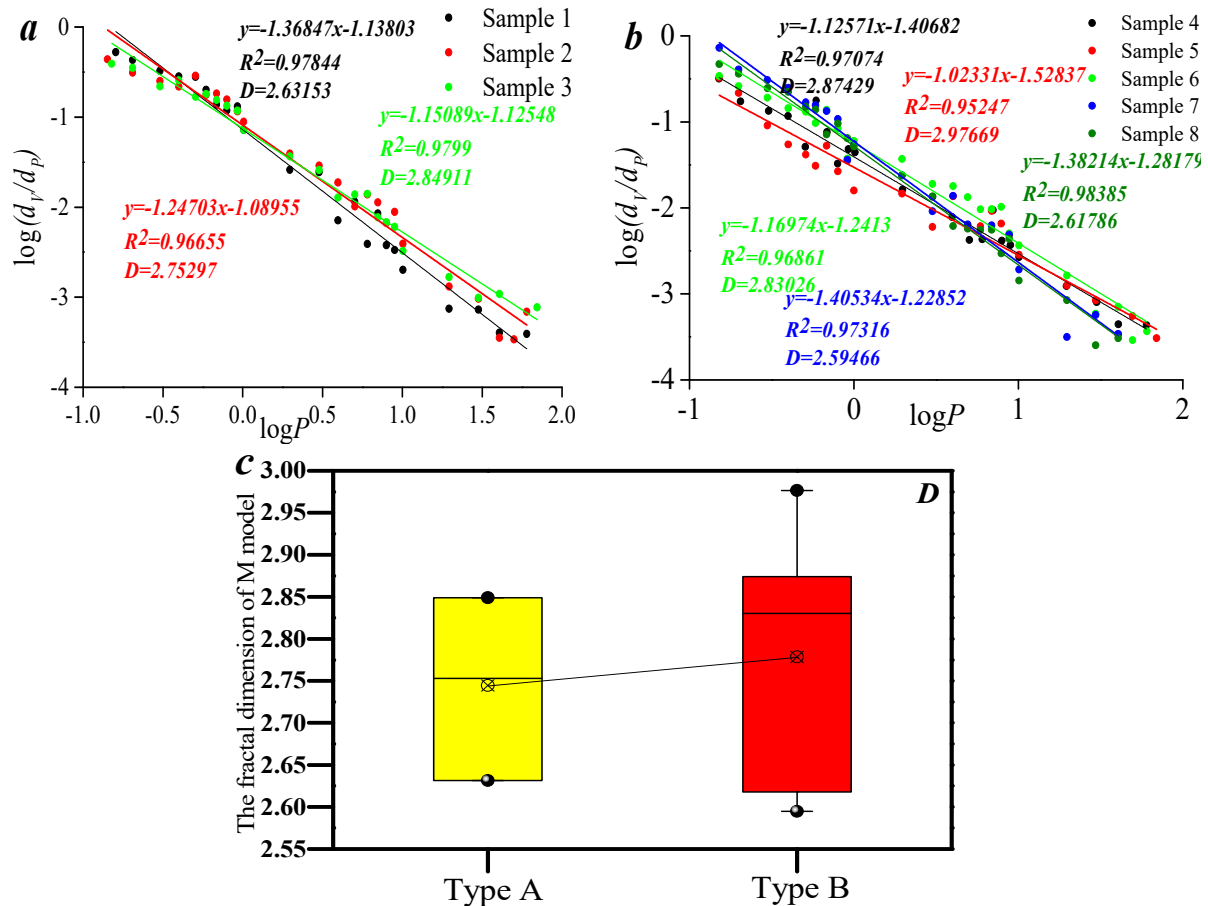


Figure 9. Fractal dimension of different samples based on the M model is compared (Based on the mercury removal curve) (a–c).

D_S can be calculated with the S model. The fractal curve can reflect the linear positive correlation between $\ln p$ and $\ln v$, which indicates that the fractal of the samples can be reflected by this model. Among them, the linear fitting degree of type A is 0.07–0.12, and D_S is 2.88–2.93. The linear fit of type B ranged from 0.04 to 0.06, and D_S ranged from 2.94 to 2.96 (Figure 10). By comparison, D_S of type B is higher than that of type A, which is the same as Figure 9 but different from Figure 5; this indicates that the calculation results of the fractal model based on the MRC have a good consistency.

Figure 11a,b show that the $q \sim D(q)$ spectra show an inverse S, indicating that the pore distribution based on the MRC has multifractal characteristics. $D_{-10} - D_0$ of type A ranged from 0.40 to 0.60, and $D_0 - D_{10}$ ranged from 0.73 to 0.86. $D_{-10} - D_0$ of type B ranged from 0.5 to 1.10, and $D_0 - D_{10}$ ranged from 0.90 to 0.97. The literature shows that the left spectral width represents heterogeneity in the low-value pore volume region, and the right spectral width represents heterogeneity in the high-value pore volume region. The curve on the left side of type B is larger than that of type A, indicating that type B has a strong heterogeneity in the adsorption pore volume distribution. The curve on the right side of type A is larger than that of type B, indicating that type A has a strong heterogeneity in the large pore volume distribution. This understanding is inconsistent with that of MIC.

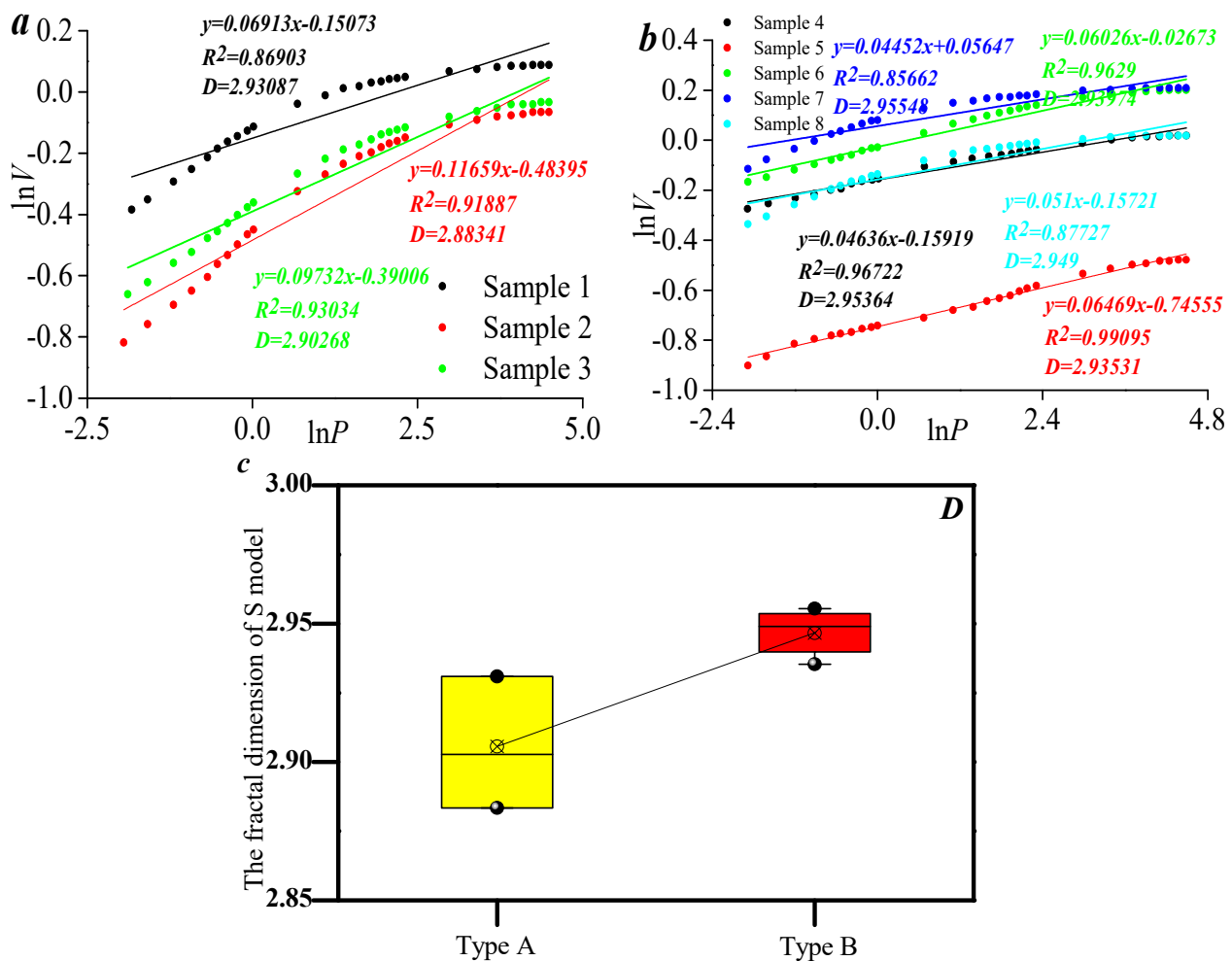


Figure 10. Fractal dimension of different samples based on the S model is compared (Based on the mercury removal curve) (a–c).

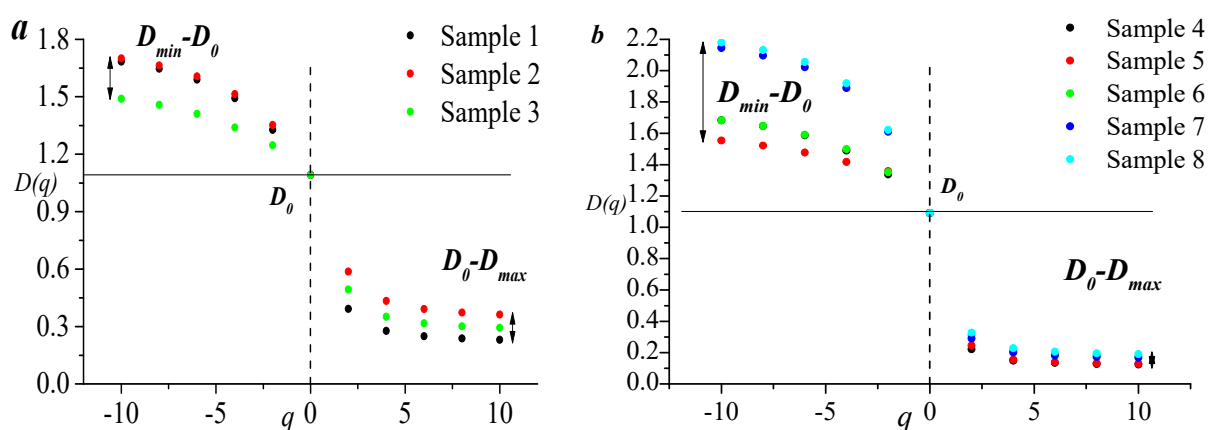


Figure 11. Multifractal of different samples based on the mercury removal stage is compared (a,b).

Figure 12 shows that type B is larger than that of type A in the $D_{-10}-D_0$ variation range, which indicates that the adsorption pore heterogeneity of type B is stronger than that of type A. This result is consistent with Figure 7. Type A is larger than that of type B in the D_0-D_{10} variation range, which indicates that the large pore heterogeneity of type A is stronger than that of type B. This result is inconsistent with the result in Figure 7.

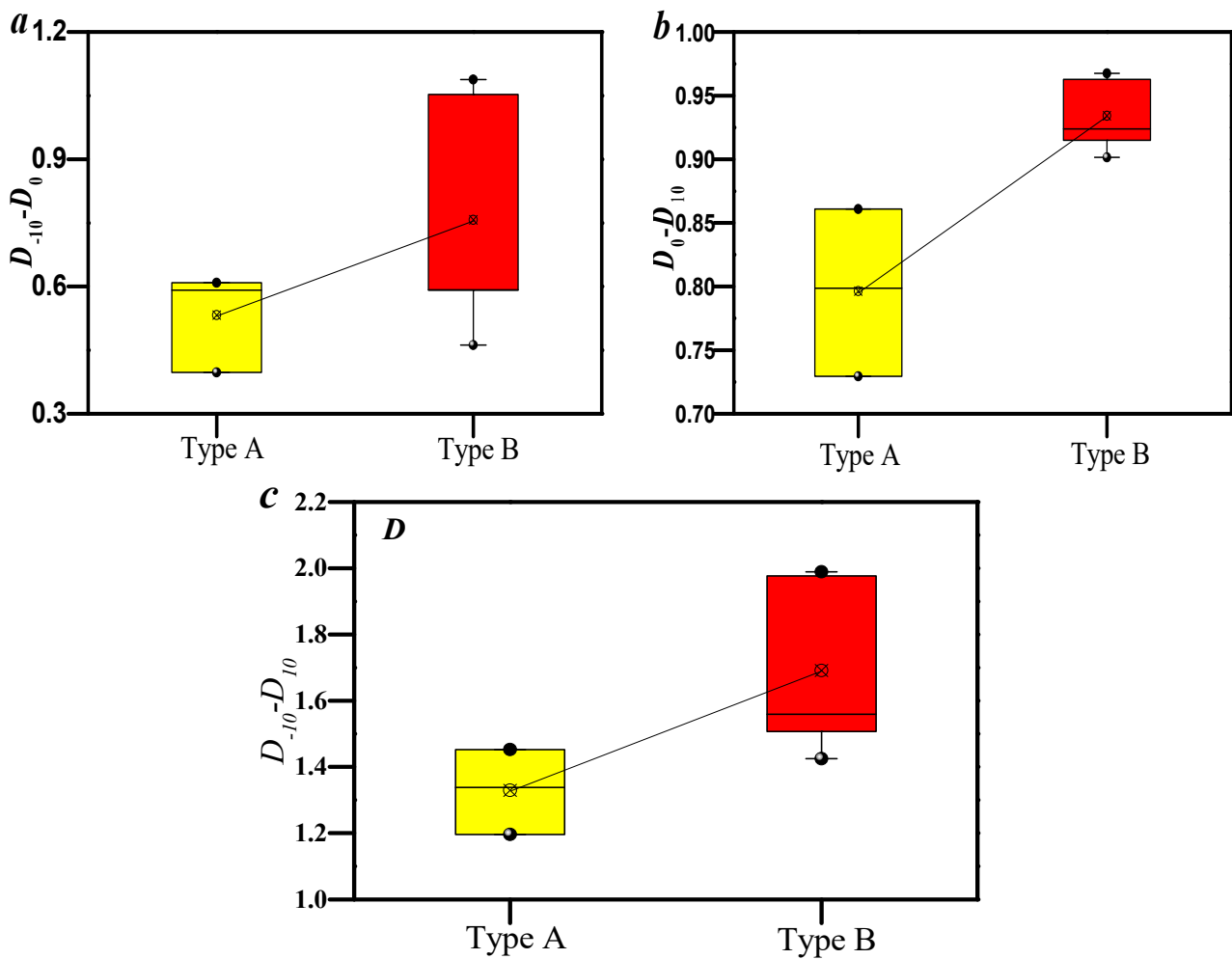


Figure 12. Multifractal of different samples in the mercury removal stage is compared (a–c).

Figure 13 shows that there is no correlation between $D_{-10}-D_0$ and D_0-D_{10} , $D_{-10}-D_0$ is positively correlated with $D_{-10}-D_{10}$, and D_0-D_{10} is positively correlated with $D_{-10}-D_{10}$. The correlation between $D_{-10}-D_0$ and $D_{-10}-D_{10}$ is stronger. This indicates that the low pore volume area controls the PFDH of the samples, which is consistent with the result of the mercury injection.

3.4. Influencing Factors Restricting Pore Fractal Characteristics and Applicability Analysis

Figure 14a shows that there is a significant negative correlation between the adsorption pore volume percentage and D_M , and a significant positive correlation between the adsorption pore volume percentage and D_T , indicating that both M and T models can reflect adsorption PFDH. Figure 14b shows that there is no correlation between the seepage pore volume percentage and both D_M and D_T , indicating that the M and T models cannot reflect seepage PFDH. Figure 14c shows that the large pore volume percentage has a linear positive correlation with D_M , but no linear relationship with D_S , indicating that, compared with the S model, the M model can better characterize macropore PFDH. In summary, in the mercury injection stage, the M model can reflect adsorption pore and macropore PFDH, while the S model can only reflect adsorption PFDH.

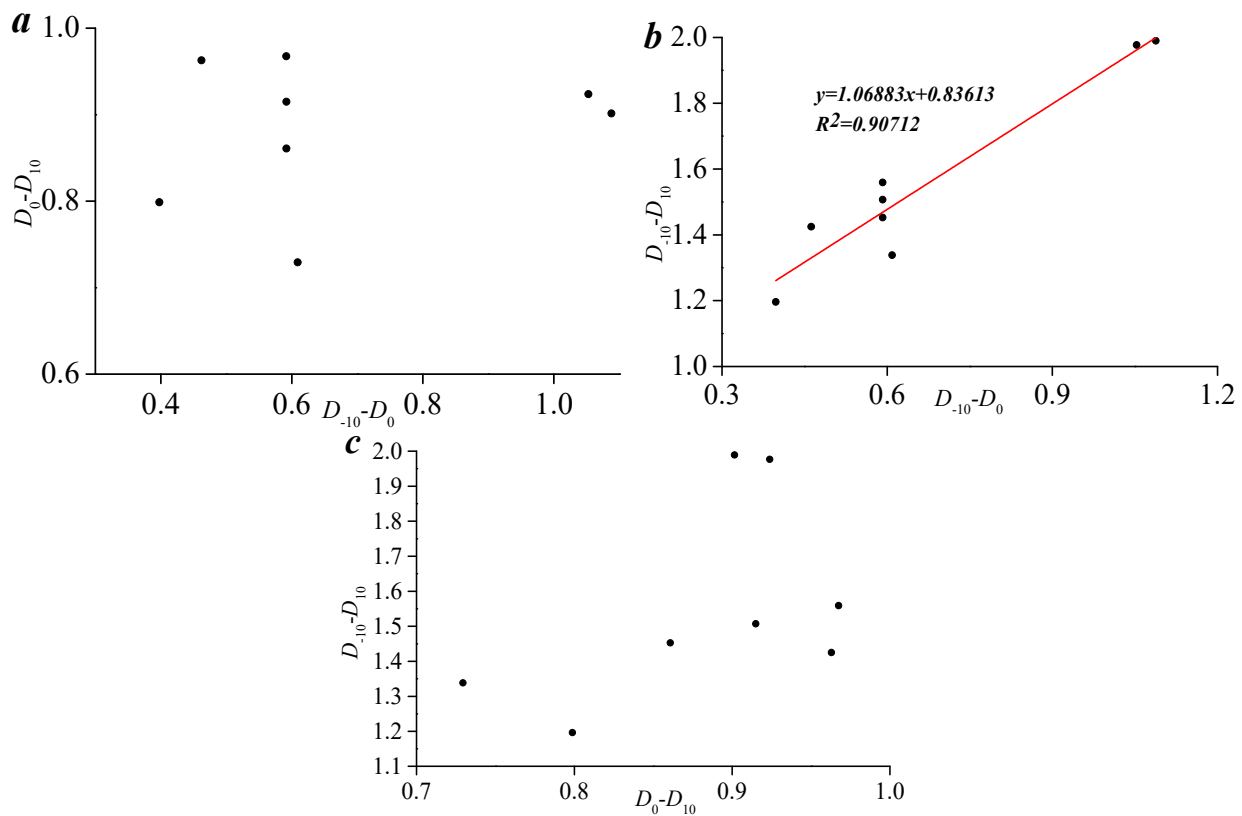


Figure 13. The relationship between different multifractal dimensions based on mercury removal curves (a–c).

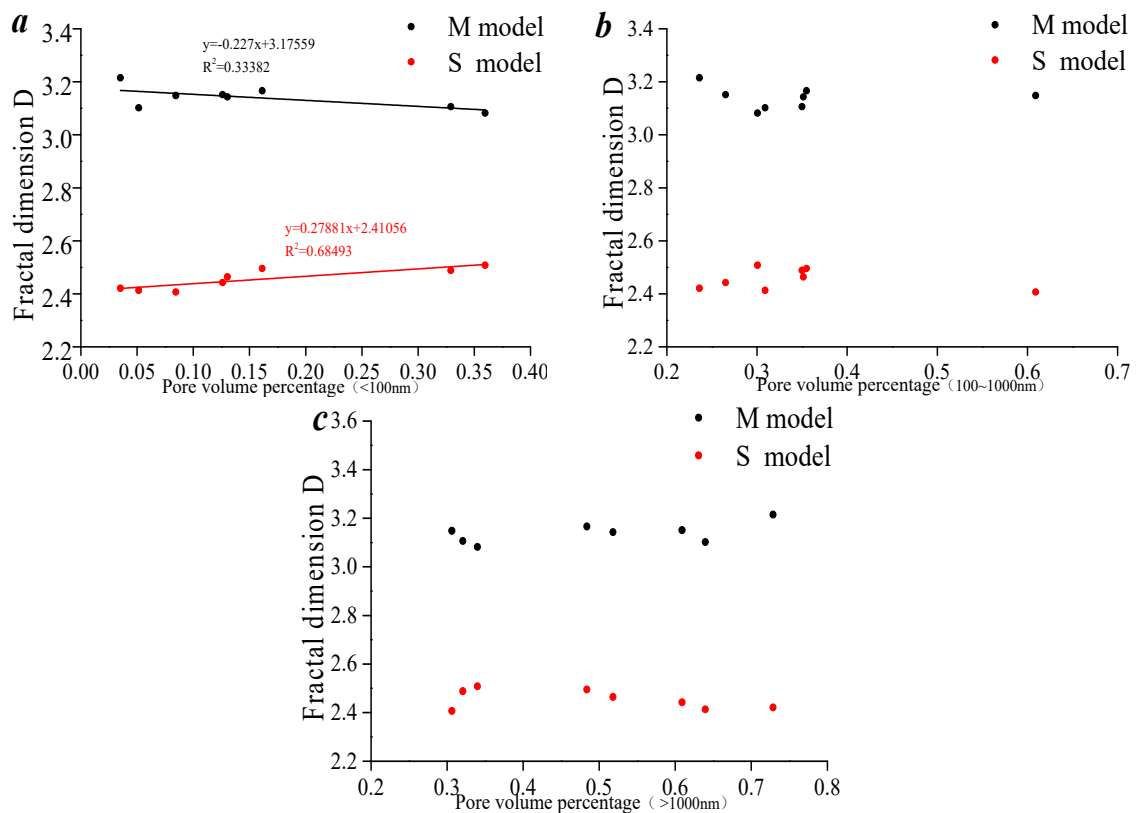


Figure 14. Correlation analysis between pore volume parameters and fractal dimension based on the mercury removal curve (a–c).

Figure 15a shows that there is a linear positive correlation between the adsorption pore volume percentage and D_M , but no relationship between the adsorption pore volume percentage and D_S , indicating that the M model can reflect adsorption PFDH. Figure 15b shows that there is no linear relationship between the seepage pore volume percentage and both D_M and D_S , indicating that neither the M nor S model can characterize seepage PFDH. Figure 15c shows that the macropore volume percentage has a linear negative correlation with D_M but no linear relationship with D_S , indicating that the M model should also be used to characterize macropore PFDH. In summary, the M model can characterize the PFDH of adsorption and macropores in the mercury removal stage, and the results are consistent with the mercury injection stage. The S model could not characterize the PFDH at each stage, and the result is different from that at the mercury inlet stage.

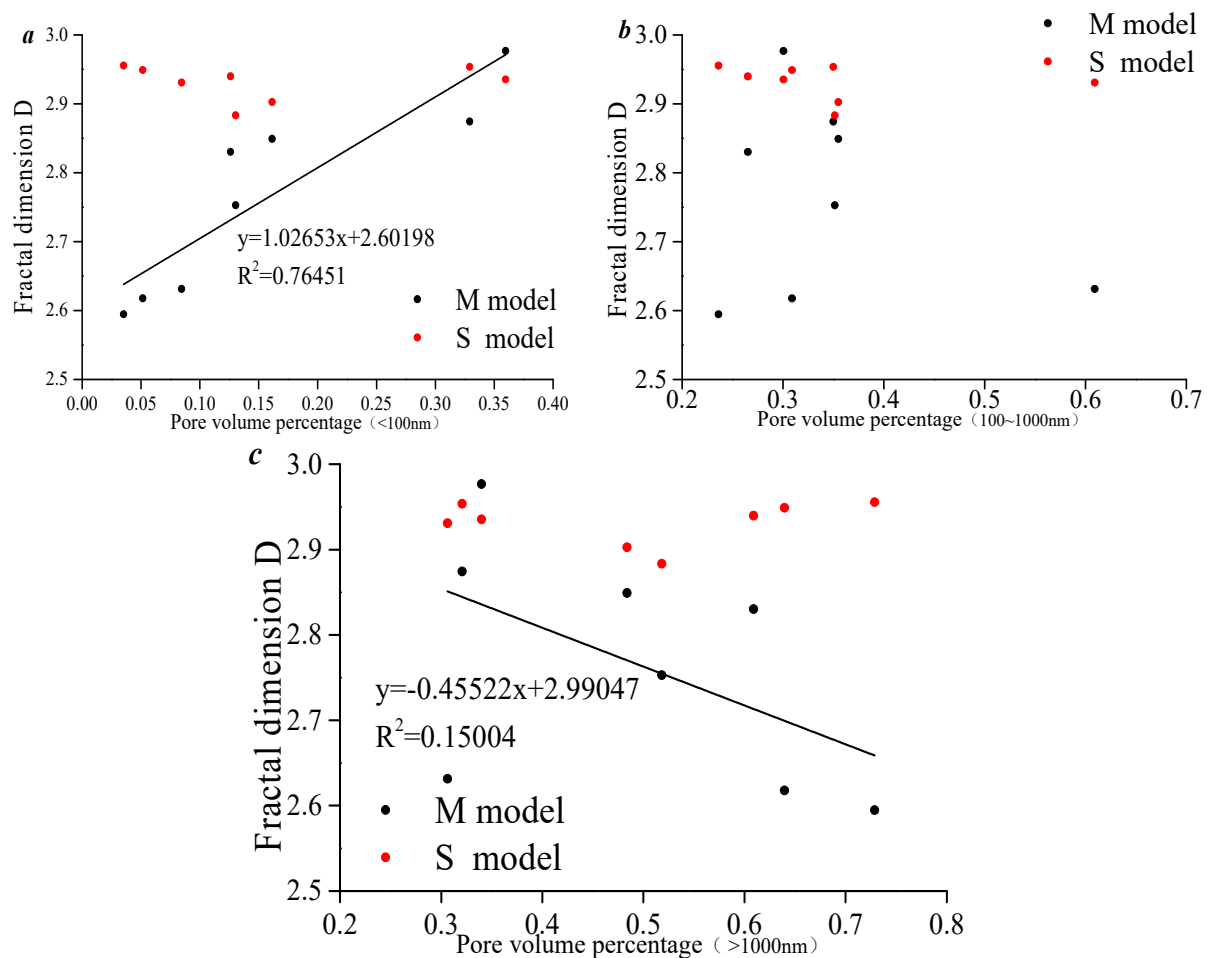


Figure 15. Correlation analysis between pore volume and fractal dimension based on the mercury removal curve (a–c).

Figure 16 shows a linear negative correlation between D_{M1} based on the mercury inlet stage and D_{M2} based on the mercury removal stage. However, there is no linear correlation between D_{S1} based on the mercury inlet stage and D_{S2} based on the mercury removal stage, which indicates that the fractal of the M model in the mercury stage is similar, while the physical significance of the S model is different in characterizing the fractal characteristics of the mercury curve.

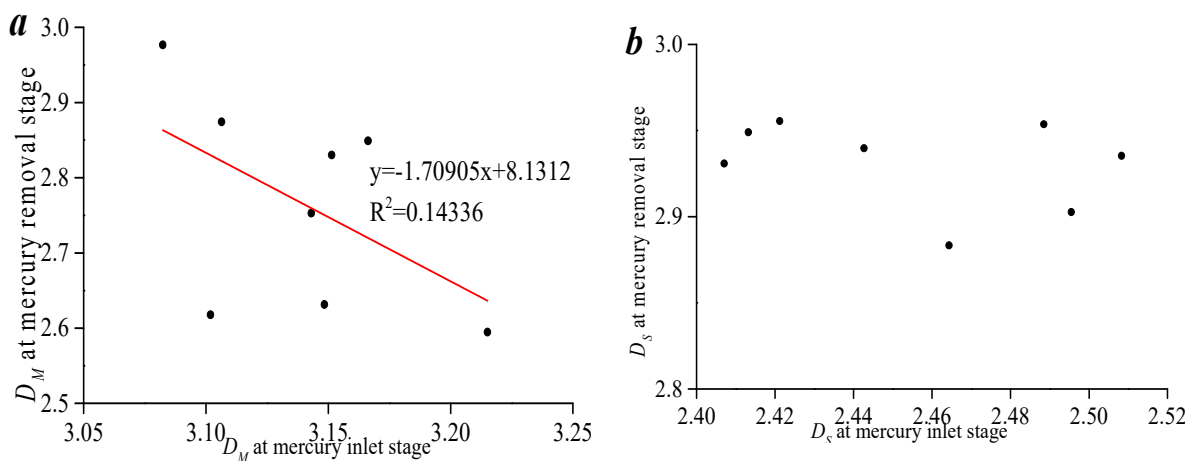


Figure 16. Relation of mercury fractal dimension (a,b).

Figure 17 shows that D_{10} , D_{-10} , $D_{-10}-D_0$ and D_{-10}/D_{10} of the multiple models based on the mercury stage show a linear relationship, while there is no correlation between D_0-D_{10} and D_{-10}/D_{10} , indicating that the correlation of multifractal parameters under mercury curves is weak. This result is similar to the single multiple fractal parameter, which indicates that the physical significance revealed by the single multiple fractal parameter based on the regression mercury curve is slightly similar, but correlation is not strong.

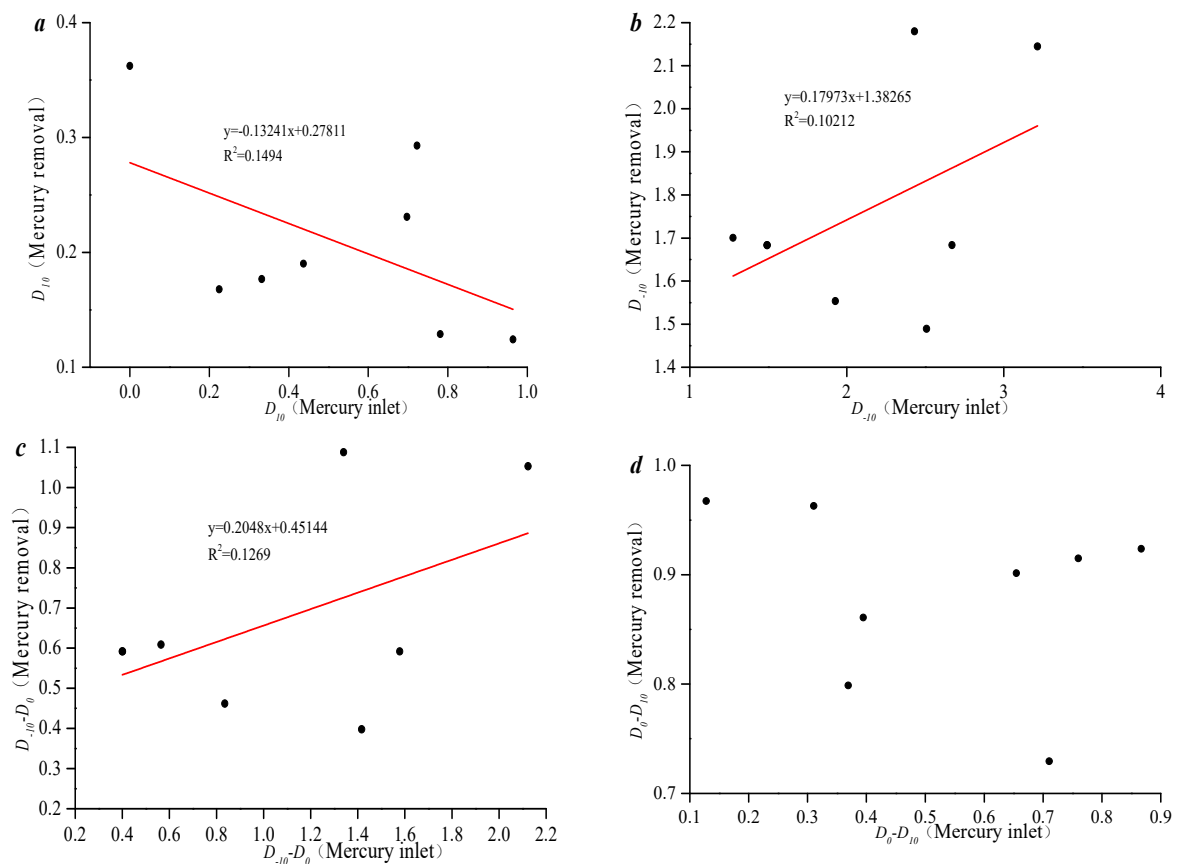


Figure 17. Cont.

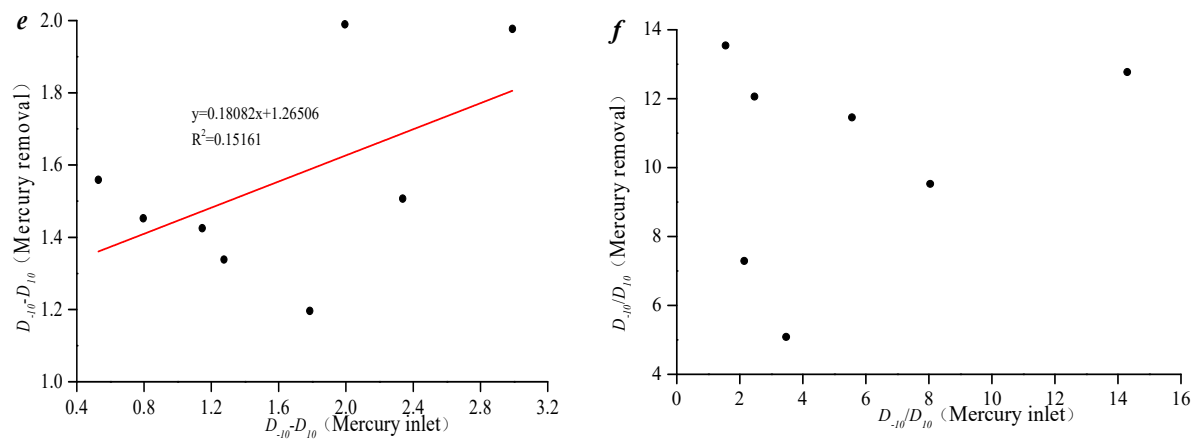


Figure 17. Multifractal parameter relationships based on the regression mercury curve (a–f).

4. Conclusions

In this paper, pore distribution characteristics are described by the high-pressure mercury injection test. On this basis, two kinds of single and multiple fractal models are used to calculate the mercury curve, and the applicability of various fractal models in characterizing pore and fracture distribution heterogeneity is discussed. The conclusions reached are as follows. The pore structure has an important effect on the desorption, diffusion and migration of coalbed methane. The pore and fracture distribution heterogeneity restricts the seepage process of coalbed methane, leading to the decrease in coalbed methane production and the ultimate recovery efficiency. Therefore, pore structure has become an important factor in determining coalbed methane productivity, which is of great significance to engineering practice.

- (1) Samples can be divided into two categories according to porosity and mercury removal efficiency. Among them, the mercury removal efficiency of type A (see Figure 1 for details) is higher than 35%, and the porosity is less than 9.5%, while those of type B (see Figure 1 for details) are the opposite. The seepage pore volume percentage of type A is 35–60%, which is higher than that of type B. In general, type A developed seepage pores, while type B developed adsorption pores.
- (2) Fractal characteristics of the mercury inlet curve. The difference of the samples' fractal dimension calculated with the *Menger* and *Sierpinski* models is small, indicating that the pore structure distribution heterogeneity of the two types is similar. The multifractal model shows that the adsorption pore and macro-pore heterogeneity of type B is stronger than that of type A, and the pore distribution heterogeneity is controlled by the low value of the pore volume.
- (3) Fractal characteristics of the mercury removal curve. The results of the two single fractal calculations show that the pore structure distribution heterogeneity of type B is stronger than that of type A. The multifractal model calculation shows that the adsorption pore distribution heterogeneity of type B is stronger, and the low value of the pore volume controls the pore distribution heterogeneity, which is consistent with the results of the mercury inlet fractal.
- (4) The mercury fractals based on the *Menger* model can reflect the adsorption pore distribution and macro-pore distribution heterogeneity, while the *Sierpinski* model can only reflect the adsorption pore distribution heterogeneity at the mercury inlet stage. The physical meanings revealed by the single–multiple fractal parameters based on the mercury curve are different, and the physical meanings are different.

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Abbreviations

LPCO ₂ GA	Carbon dioxide adsorption test
HPMI	High-pressure mercury injection test
LPN ₂ GA	Low-temperature liquid nitrogen test
LF-NMR	Low-field nuclear magnetic resonance technology
M model	Menger models
MIC	Mercury injection curve
MRC	Mercury removal curve
PFDH	Pore and fracture distribution heterogeneity
S model	Sierpinski models
FE-SEM	Scanning electron microscopy

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