Methodology for Lost Gas Determination from Exploratory Coal Cores and Comparative Evaluation of the Accuracy of the Direct Method

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ABSTRACT: In the coal exploration of China, the commonly used direct method within 120 min has potential errors in lost gas calculation of deep coal seam for its complex geological conditions. The exploration of deep coal resources by drilling holes in Huainan of Eastern China offered an opportunity to starting research into developing a new method. A developed method with error analysis was constructed to estimate the lost gas using the total desorption process obtained from exploratory coal cores. The accuracy of the direct method was also evaluated comparatively. The result shows that the desorption curve of tested coal samples matches the fitted curve equation. Desorption temperature and the tectonic coal with associated pore characteristics significantly affect the variation of the adsorption characteristics and the estimation of lost gas. The direct method obviously underestimates the lost gas, and methodology using a new lost gas estimation procedure with additional residual gas allows for achieving relatively accurate results of the determination of gas content in coal seams. The calculated result of the new method is about 1.00–1.41 times that of the direct method. The error analysis of desorption results allowed us to determine the dependence between the time (retrieval time and desorption time) and determination method. The time used for desorption in the tank is allowed to extend to less than 400 min or more than 1000 min, which is very potentially important to accurately get the coalbed gas content for coring samples, especially deep exploratory cores for field application.

1. INTRODUCTION

Coalbed methane (CBM) refers to the emitted methane in the underground mining process, either from coal seam being mined or from other methane-containing formations near the mined coal seam.1 The coalbed methane represents one of the alternative sources of energy to reduce greenhouse gas emissions and has a wide range of applications and a high rate of commercial value. The commercial extraction of coalbed methane has now been well established in many countries around the world, including the United States, Australia, China, India, and Canada.3 Except as clean energy, methane hazard is one of the most dangerous threats in Chinese coal mines during underground mining practices. Methane present in coal seams has a bad influence on safety in underground mines as it is emitted during mining works.5 There are some parameters that are used to describe the gas hazard. One of the parameters used to describe the level of gas hazard is coal gas content. The field test data of coal seam gas content from exploratory coal cores are used both in the mining industry, to predict and control the level of gas emissions in underground coal mines, and in the gas industry, to calculate in-place gas resources in subsurface coal beds.5

There are many methods to calculate the coal seam gas content,1–6 which can be obtained by the empirical relationship between the gas content and the coal reservoir properties or by the isothermal adsorption curve, hydrostatic pressure, and other indirect methods. But the most credible and reliable method to obtain gas content is still obtained by the direct method. After the direct method was put forward in 1970,7 the decline curve method and the Smith and Williams method were proposed and developed.8–12 In addition, the GRI method,13 polynomial method,14 and the Amoco method15,16 have been applied to different degrees. It is worth noting that the direct method using
early desorption data to estimate lost gas will lead to the decline of calculation accuracy, and the Amoco method using full desorption data for fitting calculation may get more accurate results.\textsuperscript{1,17,18} At present, the most widely applied mean to predict coal gas content is the direct method, especially in China. However, the coal gas content usually varies with different geological environments, especially under high temperature and pressure conditions with the increasing burial coal depth. Compared with the conventional shallow coal seam, when collecting the deep coal seam samples, the gas escape time in the process of drilling is ahead of time, while the conventional CBM content test method does not consider the amount of free gas escape in the calculation of lost gas, which leads to the low test results. The higher the gas content of coal seam, the more broken the coal sample is, the longer the sampling time is, the greater the proportion of gas loss is.\textsuperscript{6,7,19} There is still a big uncertainty when the direct method is applied to the calculation of deep coalbed methane content. The application of the traditional gas content calculation method restricts the objective evaluation effect of deep CBM content and CBM geological conditions. To improve the accuracy of coal seam gas content calculation and enrich the basis of deep coal seam gas content calculation, this paper takes deep borehole coal core samples in Huainan mining area as an example to carry out the calculation research of CBM content. After core coal samples were taken, field gas content tests were carried out to get the lost gas, desorption gas, and residual gas. An analysis of the field test and evaluated results was conducted to develop a new method for determining the accurate gas content in coal seams, which can avoid the calculation error of gas content and accurately provide parameters for the safety of coal mine.

### 2. RESULTS

#### 2.1. Field Gas Desorption Characteristics

It can be seen from the desorption curves (Figure 1a,b) that the desorption amount and desorption speed of different coal samples are different at the same desorption time. The desorption capacity of coal sample increases rapidly in the initial stage of desorption, especially in 0–4 min; then, the desorption capacity curve tends to be flat in about 4–50 min, but the desorption capacity still increases rapidly; after about 50 min, the slope of desorption capacity curve decays slowly, the desorption capacity per unit time decreases, and the desorption capacity increases steadily. The desorption rate decreased with time. In the initial period of less than 4 min, except for 1-8 coal samples, the desorption rate decreased slowly, the rest of the coal samples showed significant attenuation and obvious turning point. When the desorption time is more than 10 min, the desorption rate tends to be flat, generally at 0.02 mL/(g·min). The desorption rate is related to the gas content of coal itself, coal structure, pore characteristics, temperature and pressure conditions of coal seam, etc. Coalbed methane desorption amount and desorption time show a typical curve (Figures 1, 4, and 7) rather than a linear relationship. It is generally believed that the more seriously the coal body is damaged, the larger the exposed area is, and the smaller the particle is, the faster the desorption rate is.\textsuperscript{11,20} The change of gas from adsorption state to free state is fast and continuous. With decreasing pressure difference, the gas is released in a law of deceleration (not uniform deceleration) all the time until it is in equilibrium with the ambient pressure.

#### 2.2. Potential Error Analysis of Direct Method

With the increase of coal seam depth, the extension of drilling time will advance the escape time of coalbed methane, and there will be a large error in linear regression fitting of initial desorption data to calculate the lost gas. For deep coal seam, due to the limitation of the traditional drilling technology and coring technology, retrieval time (from the time core retrieval began to the time core reached surface) is long. Using the linear regression diagram, based on the data points at the initial stage of desorption, the lost gas cannot reflect the actual gas content of coal seam. Different initial desorption time is selected to fit the initial desorption data points of the typical coal samples (1-8) in the study area (Figure 2). Although the fitting effect is good ($R^2 > 0.99$), with the delay of initial desorption point, the slope of the fitting line is decreasing, which is reflected in the decrease of the intercept of the fitting line on the vertical axis in the figure, and the calculated lost gas is also decreasing (Figure 2).

To analyze the desorption data error of direct method, the following method is adopted to analyze the lost gas of coal seam obtained by setting the retrieval time, and the formula is as follows

$$RE = 100\% \times \left[ \frac{V_0(t_1')} {V_0(t_1)} \right]$$

where $RE$ is the relative error, $\%$; $V_0(t_1)$ is the lost gas of the minimum simulated retrieval time $t_1'$, i.e., the $V_0$ value obtained by the direct method under the condition of the actual field desorption and downhole time $t_1$; $V_0(T_1')$ is the lost gas of the retrieval time $t_1$ which can be obtained at the simulated retrieval time of $t_1'$, where

$$V_0(t_1') = V_0'(t_1') - V_0(t_1')$$

where $V_0'(t_1')$ is the actual desorbed gas volume when the simulated retrieval time is $t_1'$ and $V_0'(t_1)$ is the lost gas volume.
when the simulated retrieval time is $t_1'$, which is solved according to the simulated retrieval time $t_1'$. The total desorption time $T_0'$ is the sum of the simulated lost gas time $t_0'$ and the observed desorption time $t$ of the coal core sample in the desorption tank.

$$T_0' = t_0' + t$$

(3)

where

$$t_0' = 1/2t_1' + t_2$$

(4)

where $t_0'$ is the lost gas time of simulated design, $t_2$ is the surface time, and $t_1'$ is the downhole time (i.e., the total simulated retrieval time of the core sample in the borehole, $t_1' \geq t_1$), which is the sum of the real retrieval time $t_1$ and 2 times the desorption time of the coal core in the desorption tank, i.e.

$$t_1' = t_1 + 2 \times T_0$$

(5)

where $T_0$ is the desorption time of coal core in desorption tank under an assumed time of $t_1'$.

The lost gas analysis of the above four samples by linear regression calculation is carried out, and the results are shown in Figure 3. Samples 2-7, 3-7, and 4-13 show an increasing trend with an increase in simulated time. It is revealed that there will be a large error when using the direct method to calculate the gas content of deep coal seam. A few simulated lost gas content did not increase with time, such as simulated times of 153 min (sample 2-7) and 125 min (sample 4-13). As the increase of simulated retrieval time will gradually deviate from the real retrieval time, the cumulative desorbed gas content will deviate from the actual law of initial gas desorption. If the simulation time is longer, then the simulation results are more unreliable and it is difficult to express the conclusion. The law revealed by sample 1-8 is contrary to the above, which shows that the direct method is not suitable for the calculation of lost gas of the sample. However, a better explanation is in short supply. The desorption curve in Figure 1 and pore distribution (Figure 13) of the 1-8 sample all confirm that they have unique characteristics which are different from other samples.

Taking the 2-7 sample as an example, the lost gas contents of each simulated lost gas time were 306.64, 330.73, 366.55, 332.96, and 335.29 mL when the retrieval times of the simulated coal sample were 73, 93, 113, 133, and 153 min, respectively. In these simulated drilling times, the actual desorbed gas volumes are 0, 49.66, 78.29, 107.94, and 133.03 mL, respectively. According to formula 2, the lost gas contents when the lost gas time is 73 min are 306.64, 281.07, 288.26, 225.02, and 202.26 mL, respectively. With the increase in exposure time, the lost gas at 118 min calculated by simulation decreased. A simulated retrieval time of 153 min is equivalent to the actual retrieval time in the hole of 73 min, and the surface desorption time is 40 min. The gas of the coal in the tank has desorbed 133.03 mL, and the fitting calculation of lost gas is 202.26 mL, which is less than the calculation of lost gas when the retrieval time is 73 min, and also less than the desorbed gas in the tank for 90 min. This is quite different from reality. The simulated retrieval time is 73–153 min, and the absolute error increases from 8.34 to 34.04%. With the increase of coal seam depth and the extension of retrieval time, the error of direct method becomes larger and larger.

### 2.3. Variant Langmuir Equation Fitting and Point Correction Method

According to the characteristics of coalbed methane desorption curve (Figure 1a), it is found that the cumulative desorption capacity increases with the increase of square root of desorption time and that the increase amplitude of cumulative desorption capacity gradually decreases with the increase of desorption time. Using the simulation software 1stOpt, more than 3500 curve models were used to fit the relationship between the unit gas desorption amount and the square root of desorption time. The comparative analysis of fitting results of various curve models showed that the desorption data points fit the following equation

$$\frac{1}{Y} = \frac{1}{\alpha X + d} + C$$

(6)

If $\sqrt{T} = 1/X$ and $V = 1/Y$, then the above formula can be changed to...
If $A = 1/d$ and $B = d/c$, then

$$V = \frac{T}{c + d\sqrt{T}} + C$$

where $V$ is the desorbed gas content of coal, $A$ is the maximum desorbed gas, $B$ is the fitting parameter related to desorption speed and adsorption heat, and $C$ is the lost gas.

The equation can be used to describe the desorption curve of coalbed methane and calculate the lost gas. However, due to the short time of field desorption data (about 120 min) with a few data points, when formula 8 is used for fitting calculation, the curve will be distorted, resulting in a great difference between the estimated and the actual results. If the lost gas and residual gas are desorbed in the desorption tank, the release law of desorption gas in the tank can be extended to the retrieval time before desorption and the released time of residual gas after desorption process in the tank. In other words, if CBM is not lost in the process of coal core retrieval, the lost gas should also be included in the cumulative desorbed gas of coal. Similarly, the residual gas in the later stage can also be included in the cumulative desorbed gas content. Therefore, the degassing

### Table 1. Comparison of Lost Gas Obtained by Direct Method and Suggested Correction Method

| sample | lost gas by direct method (mL) | estimated value (mL) | ratio to the direct method$^a$ | estimated value (mL) | ratio to the direct method$^b$ | estimated value (mL) | ratio to the direct method$^c$ |
|--------|-------------------------------|----------------------|-----------------------------|----------------------|-----------------------------|----------------------|-----------------------------|
| 1-8    | 898.48                        | 643.99               | 0.72                        | 987.46               | 1.10                        | 953.4                | 1.06                        |
| 2-7    | 306.64                        | 295.25               | 0.96                        | 305.48               | 1.00                        | 310.33               | 1.01                        |
| 3-7    | 341.74                        | 1405.28              | 4.11                        | 480.62               | 1.41                        | 479.75               | 1.40                        |
| 4-13   | 164.08                        | 307.95               | 1.88                        | 165.42               | 1.01                        | 166.61               | 1.02                        |

$^a$The ratio of the lost gas obtained by the proposed method to that obtained by the linear method. $^b$The ratio of the lost gas obtained by the proposed method with double-point correction to that obtained by the linear method. $^c$The ratio of the lost gas obtained by the proposed method with single-point correction to that obtained by the linear method.

Figure 4. Desorption fitting curve and error of four coal core samples.
amount of vacuum heating before comminution and degassing amount after comminution can be regarded as a part of the amount of cumulative gas and corrected to the corresponding time point of the above fitting curve in the direct method lost gas estimation graph. The corrected fitting curve is obtained by plotting the accumulated desorption points, and the lost gas is estimated from the curve fitting equation. For the single-point correction method, we use the sum of the vacuum heating degassing amount before crushing and the degassing amount after crushing to correct the desorption data. First, the coordinates \((\sqrt{t_0 + t_1}, V_2 + V_1)\) are established and mapped on the coordinate paper (the corresponding desorption time is \(t_0\)), and the coordinate points are repeatedly fitted and optimized by formula 8 so that the coordinates can meet the extension trend of formula 8 as accurately as possible. Finally, the desorption time coordinates of the optimal fitting results and the degassing volume after fitting the equation were obtained. For the double-point correction method, the coordinates \((\sqrt{t_0 + t_{-1}}, V_2 + V_1), (\sqrt{t_0 + t_1}, V_2 + V_3 + V_4)\) are set up and projected on the coordinate paper first. The desorption times of the two coordinate points are \(t_{-1}\) and \(t_1\), respectively. Formula 8 was used to optimize the two coordinate points, and the desorption time coordinate of the optimal fitting result was obtained. Both methods can obtain the values of parameters \(A\) and \(B\) of eq 8 and the values of \(C\).

Applying the above analysis process and method, the desorption data, single-point correction, and double-point correction fitting analysis of the four coal samples are carried out, and the results are shown in Table 1 and Figure 4. The additional corrected data (just one data point or two data points) shows that it takes a long time to reach the cumulative desorption amount, resulting in a fitting curve that appears to approximate a straight line. The results show that the proposed method for different samples only has a large fitting error at the initial desorption point, which shows that the initial gas desorption data is unstable, but the overall fitting effect is good. When formula 8 is used to fit the original desorption data directly, the estimated lost gas is 0.72–4.11 times higher than that of the direct method, and the estimated lost gas is 1.01–1.40 times and 1.00–1.41 times higher than that of the direct method after single-point correction and double-point correction, respectively.

3. DISCUSSION

The field desorption time of all of the coal samples is only about 120 min. To further explore the desorption characteristics of coalbed methane in the study area and the adaptability of formula 8, two core samples (5-5 and 6-1) were taken for more than 2000 min in situ desorption observation. Within 60 min after desorption observation started, the first three data points are separated by 1–3 min, and the later data points are separated by 5 min; within 60–200 min, the data points are separated by 10 min; within 200–1000 min, the data points are separated by 30 min; after 1000 min, the data points are separated by 60 min. About 73 and 68 valid data points were obtained from the two samples. Among them, the time interval of a small number of data points does not fully comply with the above rules, but does not affect the specific analysis. The desorption rate curves of the two samples are shown in Figure 5. The desorption rate has similar variation characteristics to that of 1-8 coal samples and has obvious power function variation relationship. The desorption rate is less than 0.10 mL/(g·min).

![Desorption characteristic curve of the validation sample.](image)

3.1. Potential Error of Retrieval Time. According to the above error analysis method, error analyses of the direct method and proposed method are carried out for the two samples, as shown in Figures 6 and 7, respectively. Figure 6 shows that the simulated retrieval time is from short to long, and the simulated lost gas is from small to large. The simulated retrieval time of the two samples increased from 22 and 31 min to 182 and 191 min, respectively, and the simulated lost gas increased from 223.66 and 270.50 mL to 457.88 and 306.64 mL, respectively. Among them, the actual retrieval time of the two samples corresponding to 22 and 31 min is the lost gas volume estimated at this time, which is the lost gas obtained by direct method through the initial desorption points. Figure 7 shows that the lost gas in different simulated retrieval times increases with the extension of retrieval time. However, the amount of lost gas corresponding to different simulated retrieval time is significantly higher than that obtained by the direct method. The results showed that the lost gas increased with the increase of retrieval time.

The simulated lost gas of real retrieval time can be further calculated by the obtained lost gas of different simulation time. Based on the estimation of lost gas obtained by the direct method and variant Langmuir method, simulated amounts of lost gas of two coal samples (5-5 in Figure 8a and 6-1 in Figure 8b) under the two estimation methods at real drilling times of 22 and 31 min are obtained. The results are shown in Figure 8. For the two verification samples, the simulated lost gas of the real retrieval time obtained by simulating different retrieval time increases with the extension of the simulated retrieval time. The results show that the lost gas obtained by the variant Langmuir method is larger than that obtained by the direct method and has a good fitting relationship; for the 6-1 sample, the lost gas was obtained by the direct method when the retrieval time is 31 min is lower at the last two simulation points. From the perspective of relative error, the simulated lost gas of two samples obtained by the direct method is larger than that obtained by real retrieval time (the minimum absolute error is 36.09%), and the lost gas is lower than that obtained by real retrieval time. But the deviation range of lost gas obtained by variant Langmuir method is smaller (the maximum absolute error is 21.72%). In particular, with the extension of the simulated retrieval time, the error of the variant Langmuir method is larger than that of the direct method. The results show that under the premise of similar geological conditions and desorption characteristics, the proposed method has better fitting effect, can more accurately estimate the lost gas of deep coal seam, and the calculated value is closer to the real lost gas content.
Figure 6. Diagram of lost gas estimated by direct method with variable retrieval time.

Figure 7. Variation diagram of lost gas estimated by the proposed method with retrieval time.

Figure 8. Variation of lost gas with simulated retrieval time and relative errors obtained by the two methods.

Figure 9. Deviation of lost gas before (a) and after (b) correction compared with the longest time using the variant Langmuir method (take the 5-5 sample as an example).
3.2. Potential Error of Desorbed Time. The length of coal sample desorption time and the number of data points will cause the curve distortion of the proposed method and affect the estimation of lost gas. Taking the 5-5 sample as an example, the desorption time (measuring data point) was changed, and the lost gas was estimated and analyzed. It is assumed that the longest gas desorption time (i.e., the largest number of gas desorption data points) is the best data point and the lost gas calculated using the group of data points and the proposed method is the best fitting value. Then, the relative error of lost gas caused by different desorption time and data points can be calculated

$$\text{RE}_V = 100\% \times \frac{(V^n_k - V^n_n)}{V^n_1}$$

(9)

where $V^n_k$ corresponds to the amount of gas lost at the desorption time point $t_k$ obtained from formula 8; $k$ is the observation time point, $k = 1, 2, 3, 4, ..., n$; and $V^n_n$ is the amount of lost gas at the longest observation point, i.e., the amount of lost gas when $k$ is the maximum.

The lost gas of two samples is estimated by the proposed method, and the variation trend of deviation ratio of lost gas is obtained by formula 9, as shown in Figure 9. Similarly, the $V^n_n$ value in the variation formula 9 is set as the lost gas volume obtained by the direct method. The corrected lost gas volume can be obtained by single-point correction combined with the changed gas desorption time. The deviation curve of the lost gas can be obtained by comparing it with the uncorrected lost gas by the proposed method and lost gas by the direct method, as shown in Figure 10.

Figure 9 shows that as the measured points decrease, the error deviation calculated by formula 9 before and after correction is not consistent. The overall positive deviation is less than 7%, and the negative deviation is more than −10%. The former shows a state of positive and negative deviation fluctuation, and the negative deviation is relatively large when the desorption time is about 400–1000 min; the latter shows a positive deviation: when the desorption time is more than 400 min, the deviation of lost gas basically decreases with time and the deviation ratio increases, while when the desorption time is less than 400 min, the deviation ratio has a decreased trend with time. Figure 9 indicates that the lost gas is more accurate with the increase of desorption time. When the desorption time is only 120 min (traditional gas desorption time), the deviation of the former is 3.13%, and the latter is 6.61%. When the desorption time is 400–1000 min, there is a large error of lost gas; when the desorption time is longer than 1000 min, the deviation ratio of lost gas reaches the minimum and its absolute value is less than 2%.

For Figure 10, the lost gas calculated by different desorption time is larger than that calculated by direct method, and the deviation ratio is between 20 and 40%. It shows that the lost gas calculated by direct method is probably lower than the real gas content. With the extension of desorption time, the deviation ratio of lost gas does not have a linear trend. According to Figure 9, if it is considered that the lost gas obtained with desorption time of 1000 min or longer is closer to the actual lost gas, then the lost gas estimated by direct method is at least 20% lower than the real gas content. If the variant Langmuir method does not carry out fitting correction by additional data points (Figure 10a), the lost gas estimated is high, and the high value even reaches about 35% for 120 min gas desorption data. For these data after single-point correction (Figure 10b), the estimated...
lost gas of different desorption time is lower than that before correction. However, with the change of time, the decreasing trend is not consistent. When the gas desorption time is 120 min according to the GB/T 23249-2009, the lost gas calculated by direct method is about 26% lower than that by the proposed method. The above rules show that if the optimal desorption time is about 300 min or more than 1000 min, the estimated content of lost gas is closer to the real gas content.

3.3. Effect of Temperature on Gas Desorption. The changed temperature of coring coal before tanking increases the error of lost gas estimation. To reflect the influence of temperature on lost gas determination, the field desorption experiments of core samples (1-5, 1-6, and 1-8) from the same coal seam were set up under the same environmental conditions and different water bath temperatures (reservoir temperature and atmospheric environment temperature). As shown in Figure 11, the initial desorption amount of samples with the same mass under reservoir temperature and ambient temperature is basically the same, but with the increase of desorption time, the gap between them gradually widens. That is to say, the desorption curve and desorption rate line at reservoir temperature are higher than those at ambient temperature. It indicates that the desorption amount at reservoir temperature is greater than that at ambient temperature in unit time, and the desorption rate at reservoir temperature is greater than that at ambient temperature. The desorption law at ambient temperature is similar to that at reservoir temperature. The increasing trend of desorption quantity decreases with time, and the desorption rate decreases slowly with time. It is revealed that different temperatures acting on gas desorption in unit time will lead to gas content error. At the same time, it also shows that with the increase of coal seam depth, the reservoir temperature increases. The increase of temperature will accelerate the coal seam gas desorption, and the lost gas will increase when sampling deep coal seam.

It takes a certain time for the water bath to reach the reservoir temperature and balance after the coal sample taken out from the borehole and put into the desorption tank. In the process of coring and desorption in the tank, the sample temperature will change, and it has experienced the process of gradually decreasing from the reservoir temperature to the ambient temperature, and then increasing the equilibrium to the reservoir temperature. Therefore, the first few measuring points may not accurately reflect the desorption law of coal sample when estimating the lost gas amount. Therefore, if the measurement points at the beginning of gas desorption are used, the estimation result of lost gas will be smaller.

Taking the 1-6 sample (reservoir temperature and ambient temperature) from conventional core as an example (Figure 12), the lost gas estimated under variable temperatures will be different (520.88 and 903.60 mL) using the early measurement points, which are unstable and do not reach the reservoir temperature balance. However, using the stable data points eliminating early unstable points, the lost gas estimated is 460.85 and 731.59 mL. The desorption rate of coal sample is obviously higher at the beginning of the gas desorption. This may have a great relationship with the friable coal in the study area. So, after the coal sample is sealed into a desorption tank, the gas released from the coal begins to desorb rapidly. For the 7-13 wireline coring sample with desorption under constant reservoir temperature (Figure 12), the initial desorption point is unstable and the desorption rate is small, the desorption starts to be stable from the fifth measuring point, and the measuring point basically shows a linear increasing trend. Using the first few unstable measuring points to estimate the lost gas will make the gas content smaller (about 42.85 mL), while using the stable measuring point indicates a normal estimated value of lost gas (about 104.41 mL).

3.4. Influence of Pore Distribution on Gas Desorption. The pore fracture system in coal reservoir plays a key role in gas desorption characteristics. Three samples 3-7, 1-8, and 4-13 in Figure 1 were tested by the mercury injection method. The advance and retreat mercury curves and pore size distribution are shown in Figure 13. The advance and retreat mercury curves show that the 3-7 sample has a certain volume difference of mercury advance and retreat. The hysteresis ring is narrow and small, which illustrates that most of the pores in coal are open. But the mercury retreat curve is concave, which indicates that there are a considerable number of semiclosed pores. The 1-8 sample has a large volume difference of mercury advance and retreat, the hysteresis ring in low-pressure section is wide, and the mercury retreat curve is slightly convex, which indicates that there are more open pores and the pore connectivity is the best. The 4-13 sample has a certain amount of initial mercury removal, but there is almost no mercury removal afterward, indicating that macropores are developed and the connectivity of pores is poor.

In terms of pore size distribution, the macropores and fractures of the 3-7 and 4-13 samples above 10 000 nm are relatively developed in varying degrees, but the former still has a large number of pores when the pore size is less than 10 nm (micropores), while the latter relatively lacks this part of pores. Figure 1b shows that the desorption rate of the 3-7 and 4-13 samples is greatly reduced within 10 min after the desorption.
started. There is an obvious inflection point after the desorption rate of 3-7 and 4-13 samples is reduced. This is consistent with the pore distribution of the samples. Because the macropores and fractures provide a good channel for gas releases, the gas in the coal can be released rapidly at a large speed at the beginning of desorption, and then the desorption rate decreases rapidly. The main source of gas in the later desorption time may mainly be the adsorbed gas in the micropores. The lack of micropores (<10 nm) and transition pores (10−100 nm) of the 3-7 and 4-13 coal samples is one of the possible reasons why the desorption rate is still lower than that of the 1-8 coal sample. The peak pore size of the 1-8 sample appears at about 1000 nm and disperses to both sides, showing the lack of macropores above 10 000 nm and pores below 100 nm. The desorption rate of this sample decreases gently, and there is no obvious inflection point in the process of decreasing to a low value, which is consistent with the pore distribution curve. The results show that the lack of macropores limits the rapid release of gas, and the characteristics of opening and connectivity revealed by the advance and retreat mercury curve (Figure 13a) show that the gas migration channel is more smooth than the other two samples, which makes the adsorbed gas continuously release, so the desorption rate is relatively high.

4. CONCLUSIONS

(1) The gas desorption characteristics of deep coal seam in Pansie, Huainan, are in line with the variant Langmuir curve. The error analysis of linear regression shows that with the increase of retrieval time, the error of lost gas increases gradually. The lost gas can be calculated by the variation of the Langmuir equation with single-point or double-point correction method based on the addition of vacuum heating degassing amount before comminution or the sum of vacuum heating degassing amount before comminution and degassing amount after comminution, respectively.

(2) The test results show that the error of the variant Langmuir equation is smaller than that of the direct method to estimate the lost gas with the increase of retrieval time. The decrease of desorption time results in fluctuating deviation of the calculation results of lost gas. Taking the most data points as the best value, the calculated lost gas is positive deviation when the desorption time is 120 min, and the deviation is minimum when the desorption time is less than 400 min and more than 1000 min, which indicates that this time is the best time for gas desorption on site.

(3) For the deep coal seam, appropriate desorption time in the canister and proposed method may be a wider choice to improve the accuracy of coal gas estimation compared with the direct method. Thus, the method can satisfy the needs of gas content estimation of deep coal seam within the allowable deviation range of the field application.

5. EXPERIMENTS AND METHODS SECTION

Coal seam samples with different depths in the peripheral coal survey area of Panji coal mine in Huinan of Eastern China were selected for field gas content test. The relevant geological background has been detailed in previous studies.22−26 The research group published relevant experimental data and results27,28 in the early stage. The test samples collected from coal seams are Carboniferous and Permian; the depth of the test sample ranges from 1000 to 1500 m; the test coal seams are 13-1, 11-2, 8, and other coal seams in the area; and the average maximum reflectance of the vitrinite of the test sample is between 0.64 and 0.75%, which indicates that the coal is medium−low metamorphic bituminous coal. About 76 samples were tested on the site and 56 samples were tested effectively. For the labeling of samples, the first and second numbers represent boreholes and coal seams, respectively. All of the tests were conducted at the drilling site. Most of the collected coal samples are in the form of fragments with a mass of 200−300 g. After the coal core is put out of the wellhead, it is loaded into the desorption tank for sealing as soon as possible and the gas desorption is carried out by the drainage gas gathering method. The temperature and water temperature of the site vary from −2.5 to 34 °C and from 2 to 48 °C, respectively, and the temperature of gas desorption water bath is from 26 to 44 °C. According to the process of core retrieval and desorption, the downhole time (the time from the core retrieval began to the surface), the surface time (the time a sample spends on the surface prior to being sealed in a gas-tight container), and the desorption time in the desorption canister and the cumulative desorption amount with time were all recorded. The determination process and method refer to GB/T 23249-2009,29 and the continuous observation time of desorption is about 120 min. The used volume data of the gas desorption amount are the volume data under the corrected standard conditions. The porosity was measured by an Autopore IV 9500 mercury porosimeter with a maximum pressure of 414 MPa.

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Notes
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