Determination principle of residual Gas and Analysis of influencing factors on the results of Laboratory Measurement

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Abstract: In view of the current situation that the concept of residual gas content in coal seam in our country is relatively chaotic, the definition of residual gas content and the measurement principle of degassing and desorption methods in “The direct method of determining coalbed gas content in the mine” are analyzed. The standard unified method and the explicit concept are proposed. Through the laboratory desorption experiments in some mining areas, it is found that the residual content of gas and the degree of coal metamorphism are positively correlated as a whole. There is a negative correlation between the content of ash and internal moisture in coal and the residual content of gas. The order of the residual gas content of different sampling methods is as follows: core sampler ≈ hydraulic slag sampler ≈ wind slag sampler, coal structure is complete, the change range of measured data is the smallest with core sampler, which can truly reflect the law of coal desorption and status.

1. Introduction
Coal has a strong adsorption capacity. After desorption at normal temperature and pressure, some gas is still adsorbed in the coal body. This part of gas is called residual gas content [1-3] which is composed of desorption and loss. The coal's original gas content is an important basic data for mine gas emission prediction, gas drainage design, gas drainage compliance evaluation, and coal seam gas outburst. To clarify the concept of residual gas content, and to determine the true, accurate and effective residual gas content is of great significance to the implementation of coal mine gas standards and disaster management measures [4-5].

At present, the entire industry has little research on coal gas residues, and it is not in-depth. The definition of gas residue content is ambiguous, and there are many interpretations, which can easily cause misunderstandings in understanding. It is necessary to unify the definition of residual gas content. There are two domestic methods for measuring the residual gas content in the country. One is to directly measure the residual gas content in the coal through the laboratory, and the other is based on the coal gas adsorption characteristics experiment. It is assumed that the residual gas content of coal is the approximate calculation of coal gas absorption under standard pressure. By comparing a large number of experimental data, the author found a more common phenomenon: that is, there is a large difference in the residual gas content measured in the same coal seam in the same mining area under the same geological conditions. To this end, this paper analyzes the data of laboratory gas residual content determination in some domestic mining areas, and analyzes the main influencing factors of laboratory gas residual content by analyzing the changes of gas residual quantity under different sampling methods and different coal quality backgrounds.
2. Expression of residual gas in coal seam

At present, there are many expressions in the current industry codes and standards on the residual gas content of coal seams. On the one hand, it is believed that the residual gas content is approximately equal to the coal methane adsorption capacity under standard pressure. On the other hand, it is believed that the residual gas content is related to the coal exposure time. According to different production needs and the length of the coal body exposure time, the residual gas content is divided into several categories [6]:

① The residual gas content of the coal after being transported out of the mine; ② Residual gas content in adjacent coal seam; ③ Residual gas content in coal seam after mining impact; ④ Residual gas content in coal seam after extraction; ⑤ Residual coal gas content in goaf; ⑥ Residual volume after natural desorption in desorption method; ⑦ Degassing method The residual gas content and so on.

It can be seen from the above description that there are many descriptions of residual gas content, and different meanings of residual gas content are required for different working stages. In actual work, the phenomenon of mixed residual gas content often occurs, sometimes with large errors, especially in mines. During the prediction of gas emission [7], in the review report, the residual gas content of the laboratory measured by the degassing method is often substituted for the residual gas content of the coal sample after the mine is transported out. If there is an error in the selection of the residual gas content, the calculation result will be magnified several times, seriously affecting mine safety production planning and design. At the same time, in many jobs where the gas content cannot be measured, theoretical calculations are often used instead of actual measurements. Practice has shown that the theoretically calculated gas content is a fixed value, and the actual data is a wide range. It is not scientific to use a fixed value to represent a wide range. Sex and effectiveness.

This article mainly discusses the definition of residual amount in the process of measuring gas content in coal mine desorption method. The meaning of this residual gas content is that after the coal sample is desorbed 60 to 120 minutes downhole, the gas content that remains in the coal sample without desorption remains. GB / T23250-2009 "Method for Direct Measurement of Gas Content in Coal Seam Underground" drafted by Shenyang Coal Science Institute and Chongqing Coal Science Institute [8], although there is such a definition for the amount of residual gas: The amount of gas remaining in the coal sample after desorption", but no specific measurement or calculation process of residual gas content is given. The standard adopts two methods for measuring gas content, natural desorption and degassing.
The characteristic of the degassing method proposed by Shenyang Coal Science Institute is that coal samples are degassed under negative pressure. The gas content measured in the laboratory is divided into vacuum degassing amount before crushing and vacuum degassing amount after crushing. It is not desorbed under normal pressure. According to the definition of terms in the standard, the residual gas content can be understood as consisting of vacuum degassing amount before crushing and vacuum degassing amount after crushing, that is, the coal sample is naturally desorbed in the coal mine after 60 to 120 minutes. The gas content in the sample.

The natural desorption rules proposed by Chongqing Branch are all carried out under normal pressure. The gas content measured in the laboratory includes the natural desorption amount before crushing and the natural desorption amount after crushing. Under normal pressure, the content of undesorbed gas remaining in the coal sample after crushing and desorption is called the normal pressure non-desorbable amount. The atmospheric non-desorbable amount is not measured in the laboratory, but based on the adsorption characteristics of coal to gas. For formula calculation, see Equation 1.

\[
X_b = \frac{0.1a}{1+0.1b} \times \frac{100-A_d-M_{ad}}{100} \times \frac{1}{1+0.31M_{ad}} + \pi
\]

In the formula: \(X_b\) is the amount of gas that cannot be desorbed under standard atmospheric pressure; \(a\) and \(b\) are the adsorption constants of coal to gas; \(A_d\) is ash; \(M_{ad}\) is moisture; \(\pi\) is porosity; \(\gamma\) is bulk density.

According to the definition of terms in the standard, the non-desorption amount under normal pressure can be understood as the residual gas content. At this time, the definition of residual gas content in the standard is contradictory (see Figure 1). The range of residual gas content in the degassing method (the amount of degassed before crushing and the amount of degassed after crushing) is larger than the range of residual gas in the natural desorption method (No desorption amount under normal pressure). And one is the measured value and the other is the theoretical value, which is not comparable. The author uses two laboratory desorption methods to measure in the same coal seam in the same mine area, and finds that the amount of laboratory desorption measured by natural desorption method (natural desorption amount before crushing + natural desorption amount after crushing + normal pressure non-desorbable amount) is often slightly larger than degassing The measured laboratory desorption amount (degassing amount before crushing + degassing amount after crushing). This is because the normal pressure non-desorption amount calculated by the natural desorption method is often greater than the actual gas content remaining in the coal sample. This results in the same standard, different methods, and different results. Therefore, it is necessary to systematically analyze and integrate the two methods in the standard, or use only one standard to eliminate this systematic error.

3. Degassing gas residual measurement method
Because the residual gas content in the natural desorption method is theoretically calculated, and the residual gas content determined by the degassing method is all measured in the laboratory, which is more authentic and comparable, so this paper uses the degassing method for related research.

3.1 Degassing device and preparation work before degassing
The degassing device in the degassing method consists of a thermostat, a sealed tank, a water collection bottle, a cooling tube, a mercury vacuum gauge, a water isolation bottle, a suction tube, a drainage bottle, a suction bottle, a vacuum bottle, a measuring tube, a leveling bottle, a drying tube, Vacuum pump and other components (see Figure 2). Before degassing, put the coal sample tank into the water for 5 minutes to check whether there is a gas leak, and those who are leaking will be treated as waste samples. The degasser is placed at its maximum vacuum for 240 min, and the mercury meter liquid level rise is not more than 5 m.
3.2 Degassing coal samples before crushing
Coal samples are first degassed under vacuum at 30 °C, and then heated to a constant temperature of 95 °C to 100 °C for degassing until the amount of degassing is less than 10 cm³ every 30 minutes. After degassing, turn off the vacuum gauge, remove the sealed tank, and immediately put the coal sample into the ball mill tank for sealing. Using SQ-205 gas chromatograph to analyze gas composition, calculate the volume percentage concentration of each gas component.

3.3 Degassing after crushing
First, check the airtightness of the ball mill tank, crush the large coal sample to below 25mm, tighten the lid of the tank to seal, start the ball mill, and crush the coal sample to a weight of less than 0.25mm and the weight exceeds 80%. After the coal sample is crushed, degassing is carried out according to the steps before crushing. After the degassing is completed, after the tank is cooled, the weight of the coal sample is weighed, and the coal sample is reduced according to GB474-1996 "Preparation Method of Coal Sample", according to the analysis of GB / T212-2001 Mad, Aad, Vdaf method.

4. Research on Influencing Factors of Gas Residual Measurement
The author is at the Coal Mine Safety Basic Laboratory of Shenyang Research Institute of Coal Science and Technology Group, from the Xingfeng Coal Mine, Pingyuan Coal Mine, Shunfeng Coal Mine, Qingshan Coal Mine in Guizhou Province, Walnut Chong Coal Mine, Tangpo Coal Mine, Ji Ke Coal Mine in Yunnan Province, Jin Coal Group Temple in Shanxi 211 samples of different coal quality and different sampling methods in the mining areas of He Coal Mine, Chengzhuang Coal Mine, Yuecheng Coal Mine and other mining areas were used to determine the residual gas. There are many factors affecting the measurement results, such as human error, instrument system error, sampling method, test method, coal quality, etc. This paper studies the coal quality and sampling methods, analyzes the relationship between coal sample moisture, ash, volatiles and gas residuals and the impact of different sampling methods on the measurement results of gas residuals.

4.1 Correlation analysis of volatile matter and gas residual
The scatterplot (Figure 1) is generated by the statistical analysis of raw coal gas residual data and volatile content data in the laboratory. It can be seen from the figure that the gas residual content data is between 0.41 m³ / t ~ 10.58 m³ / t. The sub-data is between 4.41% and 29.44%. Overall, the residual content of raw coal gas gradually decreases with the increase of volatile matter. When the volatile content is less than 20%, the residual coal gas content is between 0.53 m³ / t ~ 10.58 m³ / t, which is more discrete. The data between 3 m³ / t ~ 7 m³ / t accounts for 80% of the total; When the volatile content is greater than 20%, the residual coal gas content is 0.41 m³ / t ~ 4.73 m³ / t, the data is relatively concentrated.
Lu Shouqing\textsuperscript{[10-]12} and others believed that the amount of gas (methane) adsorbed on coal decreased with increasing volatile matter. Wu Jun\textsuperscript{[13]} and Chen Changguo\textsuperscript{[14]} believed that the amount of coal gas (methane) adsorption is related to the change of the specific surface area of coal, and the specific surface area of coal is the smallest between $V_{daf} = 20\% - 30\%$, and the gas adsorption capacity is the weakest, and the gas adsorption capacity is the smallest. The coal gas adsorption capacity changes in a "U" shape with the increase of volatile matter. This is similar to the relationship between gas residues and volatiles tested in our experiment. Therefore, it can be considered that the overall trend of gas residues is limited by the adsorption of coal to gas, but the specific data points are more dispersed, as shown in Figure 4, When the volatile content $V_{daf} = 8\%$, the residual gas content varies from 1.02 to 10.03 m$^3$/t. Therefore, from the perspective of coal metamorphism (volatile matter), especially high-metamorphic coal, it is not possible to directly determine the amount of gas remaining, but only to define a broader range.

4.2 Correlation analysis of ash and gas residual

By comparing and analyzing the coal sample ash and residual gas content in each mining area, a scatterplot is generated (Figure 5). It can be seen from the figure that there is no obvious correlation between ash content and residual gas content, except that the ash content is greater than 40\% A few data points have small gas content, and the other data points are basically evenly distributed, with no obvious change trend.

In order to remove the influence of other factors such as coal seam metamorphism, the coal samples of Chengzhuang Coal Mine with the same metamorphism are separately formed into a scatter plot (see Figure 6). It can be seen from the graph that with the increase of ash content, the residual gas is obvious There is a decreasing trend. The increase in ash content leads to a decrease in organic matter, and the coal's gas generation capacity is reduced. At the same time, the coal's pore structure changes and the
porosity decreases, resulting in a reduction in coal's adsorption capacity, resulting in a low residual gas content. The ash content in coal is inversely related to the residual gas content.

\[ y = -0.0589x + 5.7851 \]
\[ R^2 = 0.198 \]

Fig.5 Relationship between ash content and residual gas content in Chengzhuang mine

4.3 Correlation analysis of moisture and gas residual

Related research shows that when water occupies the pore space of coal, it will inevitably lead to a decline in coal gas adsorption performance \(^{[15]}\). The moisture in coal can be divided into external moisture and internal moisture according to the cause. The external water is related to the external conditions and has nothing to do with the coal quality itself. It is mainly adsorbed on the surface of the coal and the larger capillary when the coal body is exposed to the air, The external water easily evaporates. The internal water is the water absorbed by the small capillary inside the coal. This part of the water is related to the coal quality and is an inherent component of the coal body. It can only be lost when heated. It is generally considered that the internal water is the main factor affecting the coal gas adsorption.

In this paper, under the background of large samples, a statistical analysis of coal sample moisture and gas residual content in the three mining areas was conducted to generate a scatter plot (Figure 7). As can be seen from the figure, there is no obvious correlation between moisture and gas residual content relevance.

In order to remove the influence of coal metamorphism and other factors such as external moisture, the metamorphism of the Guizhou mining area was selected to be approximately the same, and the dry-sampling samples of the spiral drill pipe were used for analysis. The measurement data of residual gas content is between 0.31 ~ 2.87m³ / t. As shown in Figure 8, we can see that with the increase of moisture, the residual gas volume has a tendency to decrease, but it is not very obvious. The residual gas volume has a weak negative correlation with the moisture inside the coal seam. Relationship, but due to the limited number of samples, it is not possible to calculate an accurate formula for the relationship between residual gas content and natural moisture in coal.
4.4 Analysis of the influence of coal sample sampling method on gas residual

At present, the underground sampling methods of coal mines mainly include core sampling method, spiral drill pipe cuttings method, wind slag sampling method, hydraulic slag sampling method, etc. The core point of the core extractor is accurate, the core is complete, and it can retain most of the original coal structure. Most of the sampling is columnar (> 25mm), part is massive (5mm ~ 25mm), a small amount is particulate (1mm ~ 5mm), pole less powdery (<1mm) gas desorption speed is slow, more gas remains in the coal sample, the operation is relatively complicated, and the coal sample is exposed for a long time; the spiral drill pipe cuttings method, the equipment is light, the construction is more convenient, the drilling cuttings There is no sortability, and it is easy to mix the coal samples in the deep part of the hole with the front part, the sampling accuracy is not high, and the construction labor intensity is large. The coal sample of wind slag discharge method has short exposure time, rapid sampling, and accurate sampling position. The disadvantage is that the original structure of the coal body is severely damaged. The sampling part is particulate and part is powdery. The sampling working environment is poor, and the wind has a certain sortability. The coal sample is poorly represented; the hydraulic slag sampling method is easy to operate, the coal sample exposure time is short, the sampling working environment is good, and the sampling position is more accurate. The disadvantage is that the original structure of the coal body is seriously damaged, and the sampling part is particulate and part is powder The hydraulic power has a large sorting property, the coal sample is poorly represented, and additional water is added, causing secondary pollution to the coal sample.

Table 1 Characteristic of different sampling method

| Sampling method       | Core tube method | Wind slagging      | Hydraulic slagging |
|-----------------------|------------------|--------------------|--------------------|
| Coal sample status    | Columnar, blocky | Coal powder, fine  | particle           |
|                       |                  | particles          |                    |
| Coal sample exposure time | <10min         | <5min              | <5min              |
| Sortability           | none             | Weak sorting       | Strong sorting     |
| Coal sample purity    | high             | low                | low                |
| Additional moisture   | none             | none               | Have               |

In this study, three sampling methods including core sampling, wind slag discharge and hydraulic slag discharge were selected to sample the No. 3 coal seam mining face in Chengzhuang Coal Mine, and the effects of different sampling methods on the measurement results of coal seam gas residuals were analyzed. The sampling borehole diameter is 94mm, the sampling depth is 20m ~ 30m, a total of 32 samples are taken, of which 12 are taken by the core extractor method, 12 are air-drilled cuttings samples, 8 are water-drilled cuttings samples, and sent to Shenyang Research Institute The laboratory conducts a degassing experiment, and the residual gas content results and the corresponding sampling method are statistically formed into a scatter plot (see Figure 9). It can be seen from the figure that the residual gas content measured by the core sampling method is the largest, and the residual gas The content is between 3.8 m³ / t ~ 6.4m³ / t. The residual gas content measured by wind slag sampling method is between 2.1m³ / t ~ 5.9m³ / t. The residual gas content
measured by hydraulic slag sampling method is between $1.6m^3/t \sim 5.9m^3/t$.

Fig.8 Relationship between sampling method and residual gas content

From the experimental data, it can be seen that for the 3 # coal seam in Chengzhuang Coal Mine, the order of residual gas content measured by different sampling methods is: core sampling $>$ hydraulic slag sampling $\approx$ wind slag sampling. The author analyzes the reasons as follows:

(1) Factor of particle size of coal sample: Due to different sampling methods, the sample exists in four states: columnar ($>50mm$), massive ($5mm-50mm$), particulate ($1mm-5mm$), and powdery ($<5mm$). The desorption rate of coal has a negative correlation with the particle size of the coal sample, that is, the smaller the particle size of the coal sample, the greater the desorption rate, and the larger the particle size, the smaller the desorption rate. Li Deyang [16] analyzed the coal samples of Shenyang Mining Bureau and other regions, and believed that the residual gas content of coal increased with the increase of particle size. Among them, low-metamorphic coal had a smaller change range, and high-metamorphic coal had a larger change range. This is roughly consistent with the results of this study.

(2) Factors of coal sample purity: The purity of the sample directly affects the representativeness of the coal sample [17]. The core sampling method is convenient for personnel to identify and remove gangue and other debris from the coal, while the wind slag discharges. The two sampling methods with hydraulic slag discharge are not conducive to personnel identification and removal of debris due to the small sample size. The mixing of gangue will inevitably seriously affect the accuracy of the measurement value of coal seam gas content.

(3) External moisture factor: Related studies have shown that as the water content of coal increases, the coal's ability to adsorb methane decreases significantly. When the hydraulic slag discharge method is used for sampling, the coal sample is wetted by water, and the added water will occupy the coal surface and some pores, block the channel of gas diffusion and penetration, and reduce the gas desorption rate. Too small. This study found that the two sampling methods of wind slag discharge and hydraulic slag discharge are significantly different from the added moisture, but the results of the determination of residual gas content are similar, and the difference is not significant. This is inconsistent with the conclusion of the previous research on the adsorption capacity of water and coal. The author analyzes that the internal moisture of coal plays a more important role in the adsorption performance of coal seam gas. During the slagging process, the exposure time of the coal sample is short, and the action time of water on the coal seam is limited. Therefore, the results reflected in the measurement data of the residual gas content of the coal are not obvious.

5. Conclusion

5.1 At present, there is no unified expression of the residual gas content in coal seams in China. The results of the two methods of degassing and desorption have certain errors. Experiments show that the use of degassing can avoid the error of gas remaining due to calculation.

5.2 Under the background of large samples, the residual gas content has a good positive correlation with the coal metamorphism as a whole. The higher the coal metamorphism, the greater the residual gas
content. At the same time, the residual gas content of coal samples with the same metamorphic degree varies greatly. It is difficult to establish the relationship between the two using theoretical formulas, and only a broad range can be provided according to the specific conditions of the mining area.

5.3 Ash and internal moisture in coal have a negative correlation with the residual gas content. Ash and internal moisture change the pore structure inside the coal body, occupying the pore space, reducing the adsorption capacity of coal, and thus reducing the residual gas content.

5.4 According to the different sampling methods, the order of the measured residual gas content is: core sampling > hydraulic slag sampling ≈ wind slag sampling, core sampling can preserve the original structure of the coal body, and the residue in the coal. The gas volume is the largest, and the variation range of the measurement data of the same coal sample is the smallest, which can reflect the true gas occurrence status of the coal seam to the greatest extent. Therefore, it is recommended to use a core sampler when taking coal samples.

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