Experimental Study on Desorption Characteristics of Methane in the Soft and Hard Layer Coal of Stratified Structure in the Northwest of Guizhou, China

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Based on the importance of coal gas desorption characteristics to coal bed methane mining, extraction, and Abstract: prevention and control of coal and gas outburst, the coal samples from the soft and hard laver of stratified structure in the northwest of Guizhou were selected as research subjects, and a High Capacity Adsorption(HCA) device using high pressure capacity method was utilized to perform research on adsorption characteristics of the soft and hard layer coal under different temperatures and pressures. Seventy-two experiment groups have been carried out. The characteristics and rules of gas desorption of the soft and hard layer coal were analyzed comprehensively. The results showed that the gas desorption rate of the soft and hard layer coals was highest within the first minute after coal body was exposed or gas pressure was relieved, and gas desorption volumes increase almost linearly, then leveled off. The gas desorption of the soft and hard layer coals took place in the first 30 minutes after coal body was exposed or gas pressure was relieved, and the first minute gas desorption volumes accounted for 62.67-86.06% of the first two-hour gas desorption volumes. The initial gas desorption rate, the cumulative volume, and the desorption proportion of different time periods were affected by temperature and pressure, and increased slightly with the increase of temperature and pressure. Under the conditions of same temperature and pressure, the initial gas desorption rates of the soft layer coal were 1.282-1.892 times of the hard. The cumulative gas desorption of the hard layer coal would exceed that of the soft at the end of desorption. The shortest desorption process can happen in 10 minutes. The results provide a reference and guideline to coal gas mining and gas outburst control in the northwest of Guizhou.

1 INTRODUCTION

Although there are vast amounts of coal resources and tremendous coal mining projects in Guizhou province of China, the coal seams distribute in complex geological settings, and disasters due to coal and gas outburst accidents occur more frequently than other places (Li, 2013; Heng et al., 2015). A critical overview of a large number of coal and gas outburst cases shows that occurrences of these outbursts are closely related to complicated geological structure (Li and Shi, 2013; Li and Lin, 2010; Liu et al., 2015). Numerous investigations done by scholars around the world have shown that tectonically deformed coal has a strong gas diffusion ability, high gas desorption rate, high porosity, various strength, and favorable dynamic and mechanical conditions of gas accidents(Li et al., 2013; Li, 2011; Wei et al., 2008; Cao et al., 2013; Wang and Sun, 2015; Liang et al., 2014; Gao and Tan, 2015; Liu and Liu, 2015). Therefore, to prevent or control gas outburst in tectonically deformed coal, understanding the gas desorption characteristics of the coals is the key to gas control work (Li et al., 2010; Li et al., 2014; Xie

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and Chen, 2007; Li et al., 2011; Liu et al., 2010). There are many coal seams in the northwest coalfield of Guizhou, and most of them consist of layers of stratified structure. Many of these coal seams are thin and interlayed with layers of different lithology. Most coal seams have high gas contents and are close to coal seam group with coal and gas outburst dangers. These coal seams merge and bifurcate frequently, and their structure is complicated with low matrix permeability and interlayed by soft and hard intervals. In the mining process, the volume of coalbed gas emitted in Xiaotun coal mine in Dafang county, Qinglong coal mine in Qianxi county and Xinglong coal mine in Xishui county is large. Because the emission mechanism is complicated and the control of gas outburst is difficult, gas burst seriously threatens the safety of underground workers. It is noted that the gas emission from coal depends mainly upon gas desorption characteristics. In this paper, the experiments on gas desorption from the samples retrieved from Xiaotun, Qinglong, and Xinglong coal mines were conducted under different temperatures and pressures. By contrastive analysis on gas desorption characteristics of the samples from the soft and hard layer coals, the desorption mechanism and the factors that influence desorption characteristics were revealed. The results provide a reference and guideline to coal gas mining and gas burst control.

2 EXPERIMENTAL METHODS AND PROCESSES

2.1 Sample Collection and Preparation

The coal samples were collected from the soft and hard layers of the sixth coal seams of Xiaotun coal mine, the No. 16 coal seam of Qinglong coal mine, and the No. 18 coal seam of Xinglong coal mine. The coal samples were prepared according to the sample preparation standard, GB/T212-2008, issued by China. The coal samples were crushed, sieved, and then put into the bottle with ground stopper and sealed. Standard analysis, true density (*TD*), apparent density (*AD*), initial speed of gas emission (ΔP), speed of gas diffusion (ΔD), and firmness coefficient (*f*) of the samples are shown in Table 1. Notes: QLSC-The soft coal of Qinglong coal mine;

QLHC-The hard coal of Qinglong coal mine; XTSC-The soft coal of Xiaotun coal mine; XTHC-The hard coal of Xiaotun coal mine; XLSC-The soft coal of Xinglong coal mine; XLHC-The hard coal of Xinglong coal mine

2.2 Experimental Methods and Steps

The selected experimental equipment was the high pressure and capacity adsorption device HCA, which was manufactured by Chongqing Research Institute of China Coal Technology Engineering Group. According to the standard MT/T752-1997, experiments were conducted as follows:

- The fresh coal samples were crushed, sieved by the 0.2-0.25 mm standard sieve, then the particles of sizes between 0.2-0.25 mm were put into the bottles with ground stopper and sealed with a label.
- Fifty grams of samples, with an accuracy to 0.0001 grams, were weighed and put into dry containers and numbered, then dried for 8 hours under temperature of 85 °C and pressure of 13 Pa, and then were cooled down.
- One of the dried samples was loaded into a coal sample tank and vibrated, and the tank was sealed and filled with high pressure gas of 4MPa. Then the tank was put into a water bath and checked for air-tightness.
- The valve was slowly opened to release high-pressure gas in the tank, and the tank was connected to the degassing system, then put into the water bath.
- The temperature of water bath was increased to 60±0.1 °C, then the vacuum pump was started, and the vacuum degassing valve was slowly opened to remove the gas from the coal sample.
- After the vacuum gauge showed the pressure was below 4 Pa, the coal sample tank was continuously pumped for at least 4 hours, then the valve was closed, and the vacuum unit and vacuum gauge were turned off.

When gas desorption experiments of coal samples were carried out, experimental methods and steps were as follows:

Coal sample name	Water content (Mad)%	Ash content (Aad)%	Volatile content (Vdaf)%	TD (g/cm ³)	AD (g/cm ³)	△ P (mmHg)	△ D (ml)	f
QLSC	3.42	22.96	8.75	1.61	1.54	15.209	1.520	0.78
QLHC	2.18	10.06	7.14	1.51	1.43	10.053	0.850	1.223
XTSC	3.58	19.99	7.86	1.57	1.49	19.779	2.100	0.303
XTHC	1.66	12.86	6.59	1.57	1.48	12.533	1.018	0.707
XLSC	5.42	10.41	8.10	1.56	1.49	15.904	1.411	0.333
XLHC	3.15	7.58	7.50	1.43	1.37	4.961	0.470	0.673

Table 1: Basic parameters of coal samples.

- The temperature of water bath was adjusted to 25±1°C, and the coal sample tank was put into the constant temperature water bath.
- The inflatable tank was filled with a certain volume of high purity gas with concentration of 99.99%, then the valve of gas tank was closed.
- The coal sample tank was connected to the inflatable tank and the gas pressure data acquisition instrument. The valves of coal sample tank and the inflatable tank were opened, then the high purity gas flowed into the coal sample tank.
- When gas pressure of the coal sample tank reached a set value, the valve of the coal tank was closed, then the gas was fully adsorbed by the coal sample at a set temperature.
- The gas pressure inside the tank was read from the gas pressure data acquisition instrument, and the tank was filled with gas immediately when the pressure inside the tank was less than the set value, until the pressure reached the set value.

When the gas adsorption on the coal sample lasted more than 12 hours, the gas adsorption equilibrium experiment was finished. The temperature of water bath was adjusted to set values($20\pm1^{\circ}C$, $30\pm1^{\circ}C$, $40\pm1^{\circ}C$) before the gas desorption volume was measured. When the temperature of water bath reached a set value and became constant, experiment could begin.

The cylinder scale that was used to measure the gas desorption volume was read and recorded every 10 seconds within 1 minutes, and data were read and recorded every 1 minute from the beginning to 30 minutes, every 5 minutes from the 31 to 60 minutes, and every 10 minutes from the 61 to 120 minutes. The cumulative gas desorption volume per gram per minute measured from the beginning to 10 seconds is the initial gas desorption rate V1(ml/(g-min)), i.e. the gas desorption rate of coal after 10 seconds of exposure.

3 ANALYSIS OF EXPERIMENTAL DATA

3.1 Desorption Experiment Under Different Experimental Temperatures

The experiments of 6 coal samples (soft and hard layers) from 3 coal mines were carried out under 1.5MPa at 20 °C, 30 °C, 40 °C, respectively. The results were shown in Figures.1 to 6.

In order to study the relationship between the initial gas desorption rates of the soft and hard coals and the desorption temperature, the statistics analysis were performed on the desorption data of the soft and hard coals from Xinglong mine. The results were shown in Table 2.

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Figure 1: Desorption curves of QLSC at different temperatures.



Figure 2: Desorption curves of QLHC at different temperatures.



Figure 3: Desorption curves of XTSC at different temperatures.



Figure 4: Desorption curves of XTHC at different temperatures.



Figure 5: Desorption curves of XLSC at different temperatures.



Figure 6: Desorption curves of XLHC at different temperatures.

		0-1 mir	1.	2-5 min.			
Samples	Temperature	Cumulative gas desorption volume (ml/g)	Percentage (%)	Cumulative gas desorption volume (ml/g)	Percentage (%)		
XLSC	20±1 ℃	2.877	29.20	2.085	20.45		
	30±1 ℃	2.990	27.07	2.413	21.84		
	40±1 ℃	3.154	25.64	2.89	23.50		
XLHC	20±1 ℃	2.054	17.39	1.985	16.51		
	30±1 ℃	2.080	15.97	2.091	16.85		
	40±1 ℃	2.192	14.71	2.579	17.31		

Table 2: The cumulative gas desorption volumes and percentage of XLSC and XLHC within 5 minutes (at pressure of 1.5 MPa).

Figures.1 through 6 showed that the curves fitted the monotonically increasing function relationship, in which the cumulative gas desorption volume of the soft and hard interlayered coals increased with the time. However, gas was desorbed quickly within the first minute, and the desorption volume increased almost linearly with desorption time, then increased slightly. The gas desorption rates of the samples from different coal mine were different. The cumulative desorption volume increases when temperature increases for all coal samples.

Table 2 showed that when desorption pressure was kept constant, the cumulative gas desorption volume of soft coal was 11 percentage higher than that of hard coal. The gas desorption rate is the highest in the first minute, and then decreased later. Therefore, the increase of cumulative desorption volume slowed off gradually. The cumulative desorption volume from 2 to 5 minutes was obviously smaller than that of the first minute under the same temperature, but the decline rate decreased with the increase of temperature.

3.2 Desorption Experiments Under Different Desorption Pressures

The HCA experimental device was used, and desorption experiments were carried out under the temperature of 25 °C and the pressures of 0.74 MPa, 1.5 MPa, 3.0 MPa respectively. The experimental results were shown in Figures.7 through 12.

In order to study the relationships between the initial gas desorption rate (V1), cumulative gas desorption volume and gas adsorption equilibrium pressure, the data of desorption time from 0 to 2 hours at 25 ± 1 °C were analyzed by for the soft and

hard coals from Xinglong mine, which are shown in Table 3.



Figure 7: Desorption curves under different desorption pressures, QLSC.



Figure 8: Desorption curves under different desorption pressures, QLHC.

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Figure 9: Desorption curves under different desorption pressures, XTSC.





Figure 11: Desorption curves under different desorption pressures, XLSC.



Figure 12: Desorption curves under different desorption pressures, XLHC.

Figure 10: Desorption curves under different desorption pressures, XTHC.

Samples	Soft coal			Hard coal			
Gas equilibri	0.74 MPa	1.5 MPa	3 MPa	0.74 MPa	1.5 MPa	3 MPa	
The initial gas desorption rate(V1)(ml/(g·min))		9.066	12.074	12.918	7.542	8.196	8.478
0-1 min	Cumulative volume(ml/g)	2.541	2.997	3.264	1.963	2.152	2.298
	Percentage(%)	26.96	26.13	25.81	18.69	17.03	15.71
0-30 min	Cumulative volume(ml/g)	7.676	8.765	10.13	7.266	8.846	10.45
	Percentage(%)	78.42	82.28	83.21	69.18	70.00	71.48
0-2 h	Cumulative volume(ml/g)	8.792	9.909	11.398	8.970	10.90	12.85
	Percentage(%)	89.82	93.02	93.63	85.40	86.22	87.90

Table 3: The 0-2 hours cumulative gas desorption volumes and percentage of the soft and hard coals from Xinglong coal mine (25 $\pm 1~^\circ\text{C}$)

Figures.7 to 12 showed that the higher the gas equilibrium pressure, the higher the gas desorption rate was at constant temperature, and the gas content increased as the pressure increased. The gas desorption rate decreased with desorption time. The decline of desorption rate was the highest during the first minute. The initial gas desorption rates of the samples from the soft coals were larger than those from the hard coals. The attenuation of gas desorption rate of the soft coals was faster than that of the hard coals as well.

Table 3 showed that the percentage of 0-1 minute cumulative volume decreased when gas equilibrium pressure increased for both soft and hard coals from Xinglong coal mine. The cumulative gas desorption volume of the soft layer coal within the first 30 minutes accounted for 78.42-83.21 % of the cumulative desorption volume within 2 hours, and the cumulative gas desorption volume of the hard coal within the first 30 minutes accounted for 69.18-71.48% of the total cumulative desorption volume within 2 hours. Therefore, for both soft and hard coals, the amount of gas emission in the early stage of coal exposure or gas pressure relief changes dramatically, and the gas desorption rate decreased rapidly. Under the same gas equilibrium pressure, the initial gas desorption rates (V1) of the soft coals were 1.202-1.524 times of those of hard coals. The 0-1 minute cumulative volumes of the soft coals were 1.294-1.420 times of those of the hard coals. The percentages of 0-1 minute cumulative volumes in total desorption volumes of the soft coals were 1.442-1.643 times of those of the hard coals. For the same desorption time period, the cumulative amount of desorption increased with the increase of gas pressure. For example, the 0-2 hours cumulative volume of the soft coal at 3 MPa increased by 2.606 ml/g than the volume at 0.74 MPa. This explained that gas content in the coal mine increased as the pressure increased, and the probability of gas disaster increased.

3.3 Desorption Experiments Under Different Temperatures and Equilibrium Pressures

In order to study the desorption characteristics of soft and hard layer coal under different temperatures and pressures, the experiment on gas desorption characteristics of coal samples were carried out at different temperature and pressure combinations. Experiment pressures were set at 0.74MPa, 1.5MPa,

3MPa, and temperatures were set at 20° C, 25° C, 30° C, 35° C, 40° C.



Figure 13: Desorption curves of QLSC and QLHC under different temperatures and pressures.



Figure 14: Desorption curves of XTSC and XTHC under different temperatures and pressures.

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Figures.13 to15 showed that the 2-hour cumulative gas desorption volumes of both soft and hard coals from three different coal mines increased slightly with the increase of temperature and pressure. The 30-minute cumulative gas desorption volumes of most soft coals were larger than those of hard coals. However, the 2-hour cumulative gas desorption volumes of the soft coals were smaller than those of hard coals.

Table 4 showed that the 30-minute cumulative gas desorption volumes of most soft coals were larger than those of the hard ones from the same coal mine under same experimental conditions. The initial gas desorption rates (V1) of the soft coal were 7.116-12.94 ml/(g·min) while those of the hard coals were 3.762-10.09 ml/(g·min). This explained that there would be a large volume of gas emission of the soft coal at the beginning of sudden pressure relief, and the risk of coal and gas outburst in the soft coals would be higher than that in the hard coals.

The percentage of 1-minute desorption volume in total desorption volume of the soft coals were much higher than that of the hard coals. The 1minute desorption volumes of the soft coal samples from Qinglong coal mine were 1.367-1.652 times of the hard one. The percentage of 1-minute desorption volume in 2-hour desorption volume of soft coals were 1.592-1.865 times of those of the hard ones from same coal mine. For Xiaotun coal mine samples, the 1-minute desorption volumes of the soft coal samples from were 1.442-1.659 times of those of the hard ones, and the percentage of 1minute desorption volume in total desorption volume of the soft coals were 1.780-1.985 times of those of the hard coals. The 1-minute desorption volumes of the soft coal samples from Xinglong coal mine were 1.368-1.481 times of those of the hard ones, and the percentage of 1-minute desorption volume in the total desorption volume of the soft coals were 1.686-1.729 times of those of hard coals. The 30-minute cumulative gas desorption volume of the hard coals were larger than those of the soft ones under the same temperatures and pressures. These showed that the gas desorption rate decays quickly in the soft coals after the first minute. At the 30th minute, the cumulative gas desorption volumes of the hard coals were larger than those of the soft ones, and the difference in cumulative gas desorption volumes between the soft and the hard coals slowly increased afterwards.



Figure 15: Desorption curves of XLSC and XLHC under different temperatures and pressures.

The 30-minute cumulative gas desorption volumes of the soft layer coal accounted for 72.74-86.06 % of the 2-hour volumes. Meanwhile, during the same time period, it accounted for 62.67-72.79 % for the hard coals. The percentages of the soft coals were 1.161-1.182 times of those of hard coals. These indicated that the gas desorption of the soft and hard coals mainly occurred in the first 30-minute, and the gas desorption rates were decline significantly in the interval from the 30^{th} minutes to the 2^{nd} hour. The gas desorption rates of the soft coals were larger than those of the soft coals during this time interval, and the declines of the desorption curves of the hard coals were less than those of the soft ones.

With the increase of the equilibrium pressures and temperatures, the time for the desorption volumes of the hard coals to exceed those of the soft coals became shorter, and the shortest time observed in the experiment, which was 10 minutes, took place in samples from Xiaotun coal mine under the conditions of 40 ± 1 °C and 3MPa.

Samples	Gas equilibrium pressure(MPa)	The initial gas desorption rate(V1) (ml/(g·min))	0-1 min		2-30 min		30 min-1 h		1 h-2 h	
			Cumulative volume(ml/g)	Percentag (%)	Cumulative volume(ml/g)	Percentage (%)	Cumulative volume(ml/g)	Percentage (%)	Cumulative volume(ml/g)	Percentage (%)
QLSC	0.74	7.116	1.754	23.07	3.818	50.22	1.072	14.10	0.958	12.61
	1.5	8.316	2.003	21.59	4.842	52.20	1.304	14.06	1.157	12.47
	3	10.69	2.445	22.54	5.391	49.70	1.571	14.48	1.441	13.28
QLHC	0.74	3.762	1.062	12.37	4.320	50.30	1.567	18.25	1.638	19.08
	1.5	5.692	1.465	13.56	5.584	51.67	1.913	17.70	1.844	17.06
	3	5.962	1.623	12.27	6.909	52.24	2.461	18.61	2.233	16.88
XTSC	0.74	11.72	3.088	31.56	5.259	53.76	0.868	8.873	0.568	5.806
	1.5	12.19	3.266	29.83	6.131	56.01	0.934	8.532	0.616	5.627
	3	12.94	3.438	29.14	6.692	56.92	0.994	8.424	0.675	5.521
	0.74	6.950	1.988	17.73	6.166	54.99	1.784	15.91	1.274	11.36
ХТНС	1.5	7.500	1.969	15.03	7.501	57.26	2.004	15.30	1.626	12.41
	3	10.09	2.385	14.94	8.854	56.53	2.373	15.15	1.750	11.17
	0.74	10.81	2.737	28.54	5.316	55.44	0.936	9.761	0.600	6.257
XLSC	1.5	11.84	2.890	26.16	6.413	58.05	1.075	9.731	0.669	6.056
	3	12.92	3.328	25.76	7.457	57.71	1.370	10.60	0.766	5.928
XLHC	0.74	6.918	1.848	16.93	5.983	54.81	1.678	15.37	1.407	12.89
	1.5	7.812	1.970	15.13	7.179	55.13	2.091	16.06	1.781	13.68
	3	9.990	2.433	15.09	9.299	57.70	2.498	15.50	1.787	11.09

Table 4: The initial gas desorption rates and the cumulative gas desorption volumes of the soft and hard layer coal at different time periods(30±1°C).

4 CONCLUSIONS

The gas desorption characteristics of the coal samples that came from the soft and hard coals of 3 coal mines in the northwest of Guizhou were studied by the experiments under different temperatures and pressures. Based on the analyses of the experimental data, following conclusions can be drawn:

The curves fitted the monotonically increase of the cumulative gas desorption volumes of the soft and hard coals with the desorption time.

The gas desorption rates of the soft and hard coals were high at early stage, then slowed down. Gas was desorbed quickly within the first minute, and desorption volumes increased linearly, then leveled off. The gas desorption of the soft and hard coals mainly took place in the first 30-minute, and the gas desorption volumes within 30 minutes after coal exposure or gas pressure relief accounted for 62.67-86.06% of the 2-hour desorption volumes.

The initial gas desorption rate, the cumulative desorption volume and the desorption percentages of different time periods were affected by temperature and pressure. The desorption volume was proportional to both temperature and pressure.

Under the conditions of the same temperature and pressure, the initial gas desorption rates of the soft coals were 1.282-1.892 times of those of the hard coals.

Given enough desorption time, the cumulative gas desorption volumes of the hard coals would exceed those of the soft ones. The shortest time observed in the experiments is 10 minutes.

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