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LAB STUDIES OF GAS COMPOSITIONS ON COAL OUTBURST

Ming Qiao¹, Zhongbei Li¹, Patrick Booth¹, Ting Ren¹ and Dennis Black²

ABSTRACT: Coal and gas outburst remains one of the most severe dynamic hazards to many underground coal mining operations worldwide, posing great threats to mine safety and productivity. To understand the influence of gas composition on coal and gas outburst propensity, bulk coal samples were collected from underground coal mine sites in NSW and QLD, and subjected to experimental studies. Isotherm adsorption experiments were carried out using the gravimetric isotherm testing method to investigate the impact of coal seam gas composition on gas adsorption characteristics with a range of coal sample particle sizes, to a maximum gas pressure of 4 MPa, at 35°C. The seam gas composition employed in the tests included 100% CH₄, a gas mixture of 50% CH₄ and 50% CO₂, and 100% CO₂. For all test coal samples of different particle sizes, the adsorption capacity of CO₂ was observed to be the highest, followed by the CO₂/CH₄ mixture and CH₄. For a given gas content, the equilibrium gas pressure of a CH₄ rich coal sample is significantly greater than the equivalent CO₂ rich coal sample. Given that gas pressure provides energy to induce outbursts, it is reasonable to suggest that CH₄ rich coal contains greater outburst initiating energy. Hysteresis occurs during the CH₄ and CO₂ sorption and is calculated by an improved hysteresis index (IHI) method. CO₂ sorption hysteresis is more significant than CH₄ sorption hysteresis, with the ratio of IHI_CO₂/IHI_CH₄ ranging between 1.50 and 2.25. At equivalent adsorption gas content, the amount of CO₂ desorption is less than that of CH₄, making it difficult to provide sufficient supply of desorption gas, resulting in low gas desorption energy, which is less conducive to the development of outburst. The research results can provide useful theoretical support for mine site gas management in underground coal mines, particularly those operating in areas with moderate to high composition of CO₂ seam gas.

INTRODUCTION

Coal and gas outburst is a complex dynamic phenomenon, which is stimulated by excavation disturbance under the combined effect of geo-stress, gas, coal, and surrounding rock (He and Zhou, 1991, Paterson, 1986). It can suddenly eject a large amount of gas and broken coal from a coal seam roadway or stope in a short time, forming a pear-shaped cavity in the coal seam, and causing powerful dynamic damage (Ren et al., 2017, Xue et al., 2011, Tan et al., 2021). With the further increase in mining depth, the severity and frequency of coal and gas outbursts will further increase. It not only threatens the safety of coal miners but also severely hinders the production of coal mines, causing substantial economic losses (Lei et al., 2021).

When coal and gas outburst occurs, the ejected gas is mostly CH₄ or CO₂, and sometimes a mixture of CH₄ and CO₂. At present, most studies on coal and gas outbursts are related to CH₄, and there are few studies on CO₂ outbursts. However, CO₂-related outburst accidents have occurred many times in Australia, the Czech Republic, China, Poland and other countries (Li et al., 2011, Beamish and Crosdale, 1998). Outburst threshold limit values (TLVs) in Australian underground mines assume that coal containing CO₂ seam gas has a significantly higher risk of outburst compared to coal containing CH₄ seam gas. Some field observations have casted doubt on the current outburst LTVs in CO₂ rich coal seams. However, there has been limited research in this area to challenge the claims made by the past researchers.

Reported laboratory testing of outburst propensity using coal briquettes in small-scale outburst simulation apparatus measuring burst response of samples saturated with CO₂ and CH₄ at equivalent gas pressures (Skoczylas, 2012, Zhao et al., 2016, Sobczyk, 2011), failed to sufficiently explain the fact that the effective gas content of the CO₂ test sample will be approximately twice that of the CH₄ test sample due to inherent sorption capacity of the coal material being tested. Therefore, the higher gas emission response from the CO₂ sample is to be expected due to the sample containing approximately

¹ School of Civil, Mining and Environmental Engineering, University of Wollongong

² South32 – Illawarra Metallurgical Coal

twice the gas volume relative to the CH₄ test sample. This fact was also acknowledged by Beamish and O'Donnell, who reported, for a given coal seam gas pressure, coal samples will contain a larger volume of CO₂ and the gas emission problems will therefore appear more acute (Beamish and Donnell, 1992). Several published works suggest that outbursts associated with CO₂ are more violent, more difficult to control and more dangerous (Hargraves, 1980, Hanes, 2001, Lama and Bodziony, 1998, Xu and Jiang, 2017). However, at equivalent gas content, there is little evidence to support the claim that an outburst of CO₂ will be more violent than CH₄.

This study aims to demonstrate, through laboratory testing, that CO₂ rich coal does not necessarily represent a significantly greater outburst risk than methane rich coal, in equivalent coal seam/sample conditions. A series of representative bulk coal samples, obtained from underground coal mine sites in NSW and QLD, have been subjected to isotherm adsorption experiments to better understand the impact of coal seam gas composition on coal and gas outburst propensity.

EXPERIMENTAL WORK

Sample preparation

In this study, a series of representative bulk coal samples obtained from host sites in NSW and QLD have been subjected to various characterization experiments. The collected coal samples were prepared into particles with particle sizes of 212 µm, <0.85 mm, 0.85~2.4 mm, 2.4~5.0 mm and intact coal. The basic information of the samples is shown in **Table 1**. Proximate analysis undertaken during this study was performed as prescribed in relevant Standards AS1038.3 and ASTM D7582-15.

Table 1: Basic information of the tested coal samples

Sample ID	Seam	Proximate analysis (wt%)				Classification of coal ash
		Moisture	Ash	Volatile matter	Fixed carbon	
A	Hoskisson	3.82	14.03	25.81	56.34	Medium ash
B	Bulli	0.88	17.36	30.75	51.01	Medium ash
C	Goonyella Middle	1.44	10.26	19.77	68.53	Medium ash

Isotherm adsorption experiment

For all isotherm testing in this study, coal samples were dried at a constant 60°C under vacuum until zero moisture content was achieved. All isotherms were completed at a constant temperature of 35 °C to allow direct comparison between sample locations, and sample intra-seam horizons at the same location. Coal samples with particle sizes of 212 µm, <0.85 mm, 0.85~2.4 mm, 2.4~5.0 mm and intact coal were tested sequentially with 100% CH₄, 50/50% CH₄/CO₂ mixture, and finally 100% CO₂. The maximum pressure of the sorption tests was set at 4 MPa. The single-component gas Langmuir volume and pressure was calculated by non-linear fitting the adsorption data using the Langmuir equation (Langmuir, 1918).

$$V = \frac{PV_L}{P + P_L} \quad (1)$$

Here V is the amount of gas adsorption under the equilibrium pressure condition, cc/g. P is the equilibrium pressure, MPa. V_L is the Langmuir volume, representative of the maximum adsorption capacity of gas, cc/g. P_L is the Langmuir pressure, corresponding to the pressure at which the adsorbed amount V reaches 50% of the Langmuir volume, MPa.

EXPERIMENTAL RESULTS

Proximate analysis result

Table 1 lists the coal samples analyzed, including the proximate analysis results, coal density, the maximum reflectance of the vitrinite and the firmness coefficient. The table shows that the coals are mainly of medium ash, with the moisture ranging from 0.88% to 3.82%, volatile matter varying from 19.77% to 30.75%, fixed carbon ranging from 51.01% to 68.53%.

Gas sorption isotherms

Gas sorption measurements at 35°C provide a common basis to analyze the correlation between gas adsorption properties and pore characteristics of coal samples. The adsorption isotherms of gas (CH₄, CO₂, and 50/50% CH₄/CO₂ mixture) fitted by the Langmuir model are shown in **Figure 1**. The Langmuir fitting parameters are summarized in **Table 2**. The results show that the Langmuir model fits the gas adsorption isotherms quite well. The fitting coefficient R² is greater than 0.99, except for the sample B coal of CO₂ adsorption isotherm (**Figure 1(b)**), for which R² is 0.98682. The combined effects of Langmuir volume V_L and Langmuir pressure P_L can reflect the essential difference in gas adsorption characteristics of different components (Liu and He, 2017). From **Table 2**, it is shown that the different composition gas adsorption characteristics exhibit significant differences among coal samples. The Langmuir volume for CH₄ (V_LCH₄) ranges from 20.25 to 21.46 m³/t and the Langmuir pressure for CH₄ (P_LCH₄) ranges from 2199.74 to 2655.52 kPa. The Langmuir volume for CO₂ (V_LCO₂) ranges from 35.65 to 43.60 m³/t and the Langmuir pressure for CO₂ (P_LCO₂) ranges from 1359.77 to 1801.89 kPa. The Langmuir volume for CH₄/CO₂ mixture (V_LCH₄/CO₂) ranges from 20.51 to 28.20 m³/t and the Langmuir pressure for CH₄/CO₂ (P_LCH₄/CO₂) ranges from 1524.71 to 2943.51 kPa.

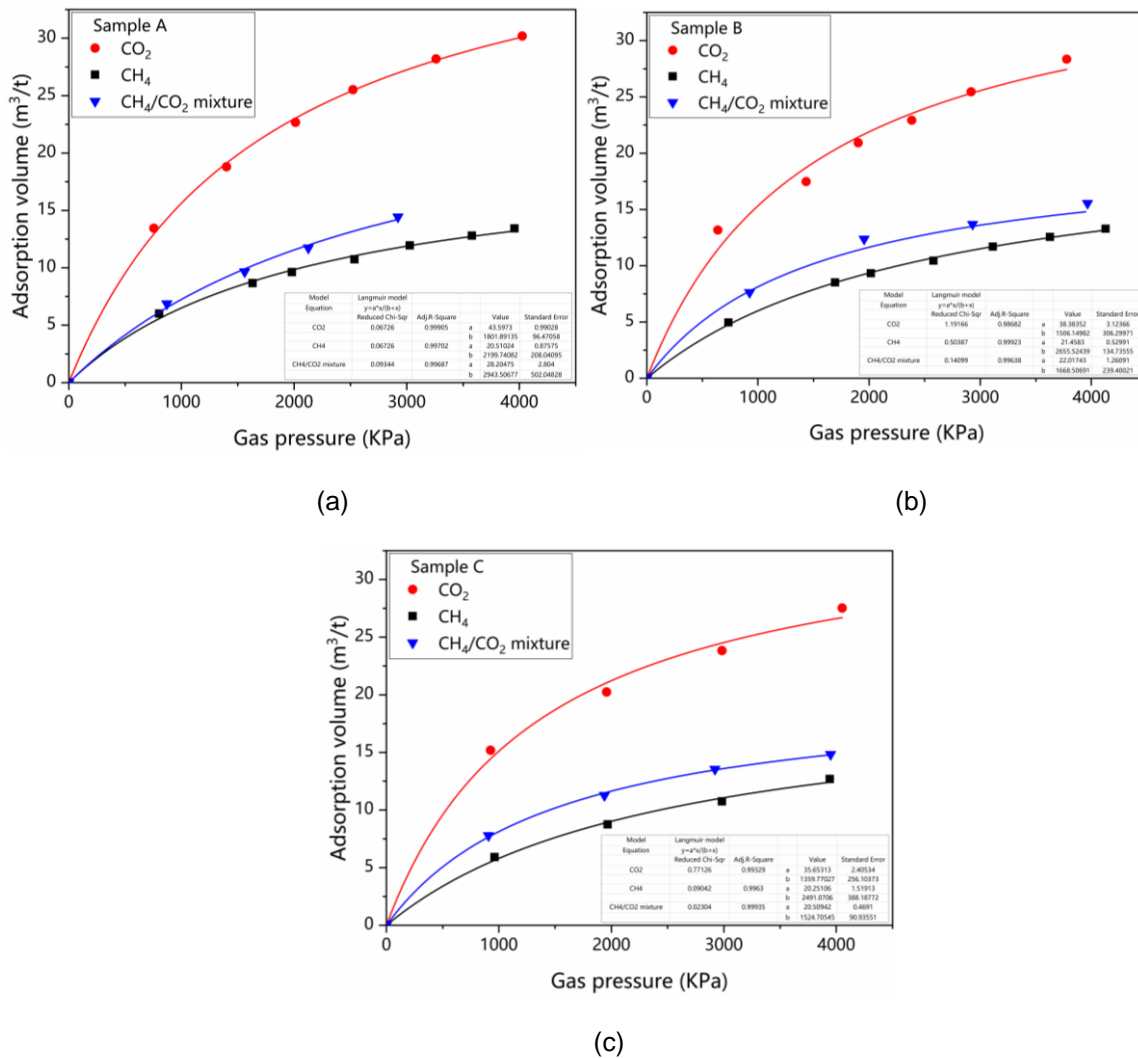


Figure 1: Different Gases (CH₄, CO₂, and CH₄/CO₂ mixture) adsorption isotherms of samples and the corresponding Langmuir fitting curves

Table 2: Langmuir model parameters of different gases adsorption

Sample	CO ₂ adsorption			CH ₄ adsorption			CH ₄ /CO ₂ mixture adsorption		
	V _L (m ³ /t)	P _L (kPa)	R ²	V _L (m ³ /t)	P _L (kPa)	R ²	V _L (m ³ /t)	P _L (kPa)	R ²
A	43.60	1801.89	0.9991	20.51	2199.74	0.9970	28.20	2943.51	0.9969
B	38.38	1506.15	0.9868	21.46	2655.52	0.9992	22.02	1668.51	0.9964
C	35.65	1359.77	0.9933	20.25	2491.07	0.9963	20.51	1524.71	0.9994

For all test coal samples of different particle sizes, the adsorption capacity of CO₂ was observed to be the highest, followed by the CO₂/CH₄ mixture and CH₄.

ANALYSIS AND DISCUSSION

Gas type and storage

Physical and chemical properties of gas molecules and their constituent atoms have a profound effect on gas behavior during adsorption, desorption, diffusion, and bulk flow. The quantity and species of gas measured in the mining environment during various stages of the mining cycle may indicate one or more of the gas processes are occurring at any particular stage (Booth et al., 2016).

The species of gas which may enter the mining environment at any stage of the mining process also determines the specific type of risk that gas presents to mine personnel (Booth et al., 2020). Given the toxic physiological effect that CO₂ has on the human body, it is agreed that CO₂ outbursts are more dangerous. However, there is little evidence to support the suggestion that an outburst of CO₂ will be more violent than CH₄ at equivalent gas content. In fact, with reference to the CO₂ and CH₄ isotherms presented in **Figure 1**, for a given gas content, the equilibrium gas pressure of a CH₄ rich coal sample is significantly greater than the equivalent CO₂ rich coal sample. Therefore, given gas pressure provides energy to induce outbursts, it is reasonable to suggest that CH₄ rich coal contains greater outburst initiating energy.

Prior research suggests gas may be stored in coal through as many as five separate mechanisms including; adsorption upon carbon surfaces, absorption within the carbon molecular structure, as a solute in water within coal and other carbonaceous material, as free gas within voids including cleats and fractures, and condensed as solid or liquid or stored in carbonate precipitates (Faiz et al., 2007, Moore, 2012, Black, 2011, Crosdale et al., 1998). For gas stored in coal, the adsorbed quantity may account for up to 90% of the total gas stored through all mechanisms (Hu et al., 2020), or be an order of magnitude greater in volume than simple open pore volume (Saghafi et al., 2007). Hence adsorption processes relevant to coal seam gas storage, transport and emission require detailed discussion. Up to 15 models for these adsorption and desorption principles are described in the literature, the most common being the Langmuir single layer model (Langmuir, 1918), the BET multi-layer model (Brunauer et al., 1938), and the Dubinin pore-filling model presented schematically in **Figure 2**.

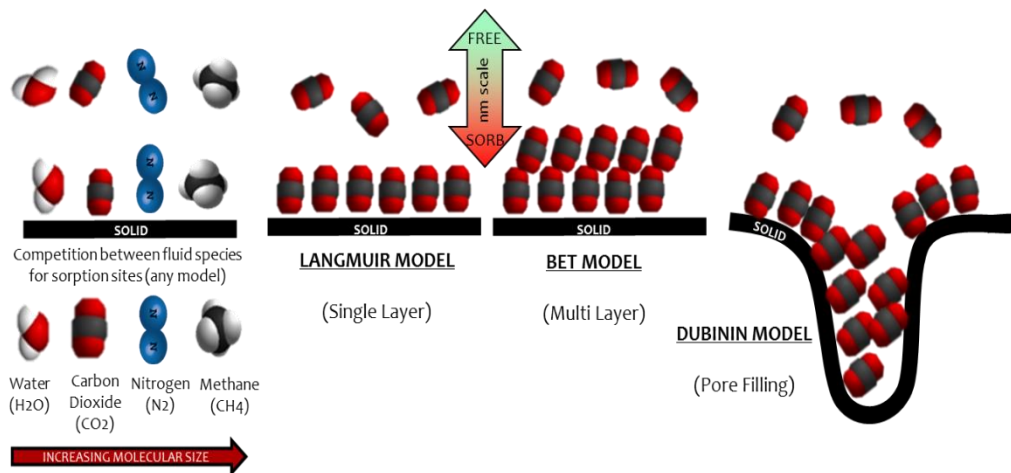


Figure 2: Typical models for fluid to solid sorption process at Nanometre (nm) scale (Booth et al., 2020)

Gas composition is a fundamental controlling variable in determining total possible sorption capacity. This is due to the relative size, structure, and energy levels of relevant gas molecules, particularly to CO₂. The precise nature of the adsorption bond depends on the properties of the species involved, but the adsorption process is generally classified as physisorption, characterised by Van der Waals forces, or chemisorption characterised by covalent bonding (Booth et al., 2016). Adsorption may also occur due to electrostatic attraction. Hamaker approximated the interaction energy of the Van der Waals force (F_{vw}) between two spheres of constant radii (R_1 and R_2) as a function of the size of the spheres and the separation distance (r), using a material dependent constant (A) measured in joules shown in equation

(2) (Mosher et al., 2013). This approximation was derived using London's dispersion energy equation, and the knowledge that the force on an object is proportional to the negative derivative of the potential energy function.

$$F_{vw}(r) = -\frac{AR_1R_2}{(R_1 + R_2)6r^2} \quad (2)$$

According to this equation, the interaction potential between CO₂, CO₂-C, CH₄, CH₄-C molecules are plotted, as shown in **Figure 3**. It can be seen that the maximum attractive force between CO₂ molecules is more significant than that between CH₄ molecules, and the maximum attractive force between CO₂-C molecules is more significant than that between CH₄-C molecules. Van der Waals forces are greater when the distance (r) between involved atoms is much smaller than the radii of those involved atoms, but Pauli's exclusion principle asserts that (r) can never be zero. When applied to fluid to solid interactions, Van der Waal's forces are also dependent on the topography of the surface involved. Surface asperities or pore protuberances may result in greater total contact area, and large increases in the total number of molecules per unit of volume may create a tendency for mechanical interlocking, particularly between carbon dioxide molecules.

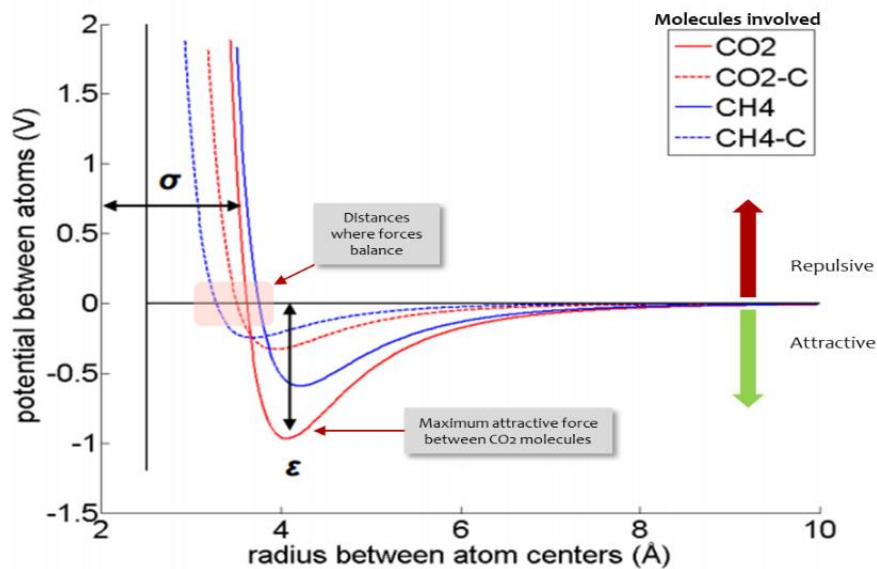


Figure 3: Relevant molecular interaction potential by distance (Mosher et al., 2013)

Relationship between gas composition and gas adsorption

Coal has different adsorption capacities for CO₂ and CH₄, and the adsorption capacity for different gases under the same conditions is CO₂>CH₄/CO₂ mixture>CH₄. The correlation between gas composition and adsorption capacity was studied by comparing CO₂, CH₄, and CH₄/CO₂ mixture Langmuir adsorption parameters (V_L and P_L), which are shown in **Figure 4 (a)** and **Figure 4 (b)**. The Langmuir volume for CO₂ (V_{LCO_2}) ranges between 35.65 to 43.60 m³/t. The ratio of V_{LCO_2}/V_{LCH_4} ranged between 1.76-2.13, and the ratio of P_{LCO_2}/P_{LCH_4} ranged between 0.54-0.81. CO₂ adsorption contains the largest V_L for the same coal sample, indicating that CO₂ has the strongest adsorption capacity. This is because the micropores in coal are highly developed, and gas is mainly stored in coal in the form of micropore filling. Compared with CH₄, CO₂ has a smaller molecular dynamic diameter, enabling it to enter smaller pores. Thus more adsorption sites are available for CO₂ adsorption. CO₂ adsorption contains smaller P_L than CH₄ adsorption, which means it takes less time for CO₂ to reach 50% of V_L . It is easier to adsorb CO₂ at the low-pressure stage compared with CH₄. Similarly, during the desorption process, when the amount of CO₂ desorption reaches 50% of V_L , the corresponding P_L is less than P_{LCH_4} , indicating that compared to desorbing a large amount of CH₄ in a short time, desorbing a large amount of CO₂ requires a higher pressure drop and more time. It is harder to desorb CO₂ at the high-pressure stage compared with CH₄. From this perspective, the outburst propensity of CO₂ is lower than that of CH₄.

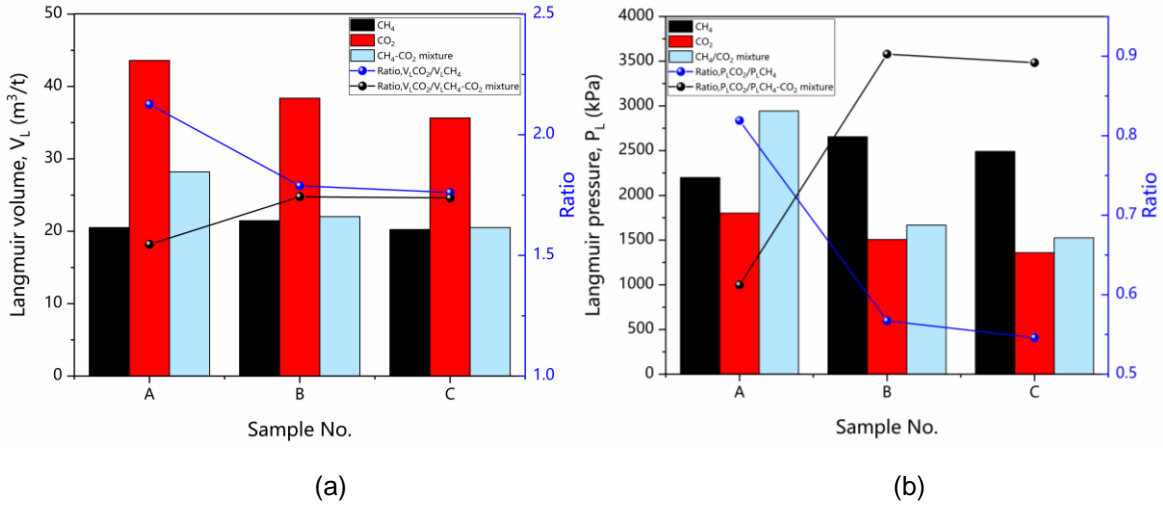


Figure 4: Comparison of CH₄, CO₂ and CH₄/CO₂ mixture adsorption capacity

(a) Langmuir volume, (b) Langmuir pressure

Effect of gas composition on sorption hysteresis

The difference or hysteresis between adsorption and desorption isotherms are referred to as “sorption hysteresis”. Usually, the adsorption process and desorption process are regarded as fully reversible, and the sorption hysteresis is ignored. In the laboratory experiment process, coal desorbs less than the adsorption volume at the same pressure, and sometimes this difference is significant. Hence sorption hysteresis should not be ignored. Sorption hysteresis is very useful for evaluating the coal seam gas drainage and predicting gas emissions during the coal and gas outburst process (Zhang and Liu, 2017). To quantify the sorption hysteresis, an improved hysteresis index (IHI) is used to calculate the sorption hysteresis gas volume (Wang et al., 2014), as illustrated in Figure 5. When IHI is 0, it means no hysteresis is observed and it is the utterly reversible state. When IHI is 100%, it means it is an utterly irreversible state. In this section, the sorption hysteresis is evaluated through this index and comparisons are made. All the analyses are based on this calculation.

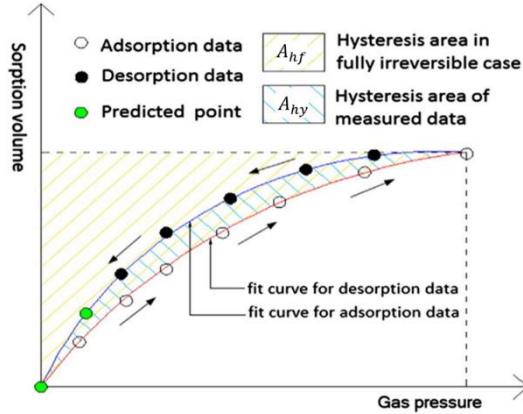


Figure 5: Explanation of the improved hysteresis index (Wang et al., 2014)

$$IHI = \frac{A_{hy}}{A_{hf}} = \frac{A_{de} - A_{ad}}{A_{sf} - A_{ad}} \times 100\% \quad (3)$$

Here A_{hy} is the measured hysteresis area, A_{hf} is the hysteresis area in the fully irreversible case, A_{hy} and A_{hf} can be calculated by the following equations:

$$A_{hy} = \int_0^{P_{max}} f_{de}(P) dP - \int_0^{P_{max}} f_{ad}(P) dP \quad (4)$$

$$A_{hf} = P_{\max} f_{de}(P_{\max}) - \int_0^{P_{\max}} f_{ad}(P) dP \quad (5)$$

Here P_{\max} is the maximum adsorption pressure, $f_{ad}(P)$ is the best fitting model of the isotherm adsorption curve, $f_{de}(P)$ is the best fitting model of the isotherm desorption curve.

In the previous studies, it is regarded that CO₂ sorption hysteresis is more significant than CH₄ sorption hysteresis, but most of the studies are qualitative. In this section, the CH₄ and CO₂ sorption hysteresis was evaluated through the IHI index. 100% CH₄ and 100% CO₂ sorption experiments on 212 um powdered coal were used for sorption hysteresis studies. Three samples in total were collected from Mine B from different locations. **Figure 6** shows the adsorption and desorption results. The IHI for CH₄ and CO₂ were calculated shown in **Figure 7**. It can be seen that CO₂ sorption hysteresis is stronger than CH₄ sorption hysteresis for all coal samples. The Ratio of IHI_CO₂/ IHI_CH₄ ranges between 1.50 and 2.25, as illustrated in **Figure 7**. Compared with CH₄, CO₂ molecules have a smaller kinetic diameter (Charrière et al., 2010) and a larger quadrupole moment, resulting in higher interaction energy with the coal matrix (Wang et al., 2014). Thus more CO₂ molecules can enter the constricted pores at the same gas pressure.

In the process of coal and gas outburst, it is necessary to provide enough desorption gas in a very short time to meet the transportation work (Lei et al., 2021). The desorption gas energy occupies a large proportion of the total outburst energy. Therefore, sufficient desorption gas supply is the key to the development of the outburst. At equivalent adsorption gas content, the amount of CO₂ desorption is less than that of CH₄ because of stronger sorption hysteresis, making it difficult to provide a sufficient supply of desorption gas, resulting in low gas desorption energy, which is less conducive to the development of outburst.

Gas molecules are dissolved into the coal microstructures. During the desorption process, only the adsorbed gas on the internal surface comes out of the pores, leaving behind the dissolved molecules in the coal structure. Recently, a novel explanation of gas adsorption on coals is that more than 90% of the gas molecules are adsorbed in coal in the micropores molecules filling (pore diameter < 1.5 nm) rather than monolayer covering (Hu et al., 2020). This theory can explain that CO₂ molecules are more likely to be trapped in the micropores.

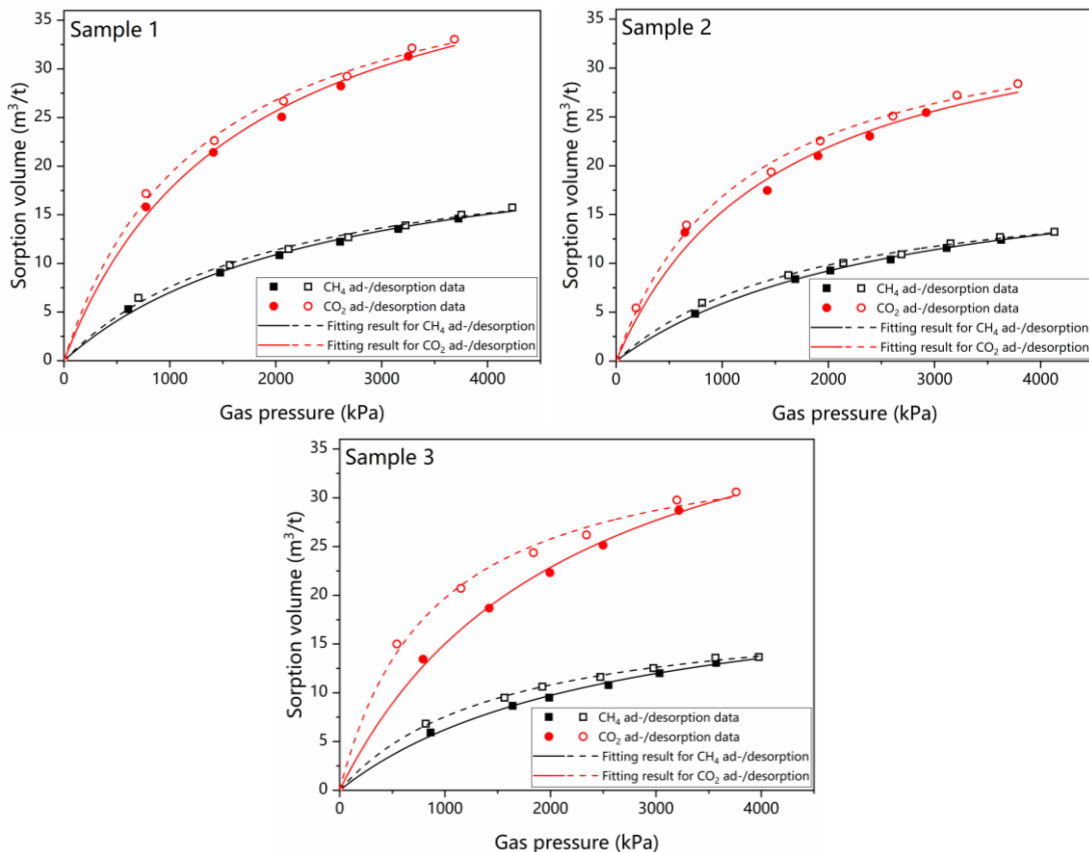
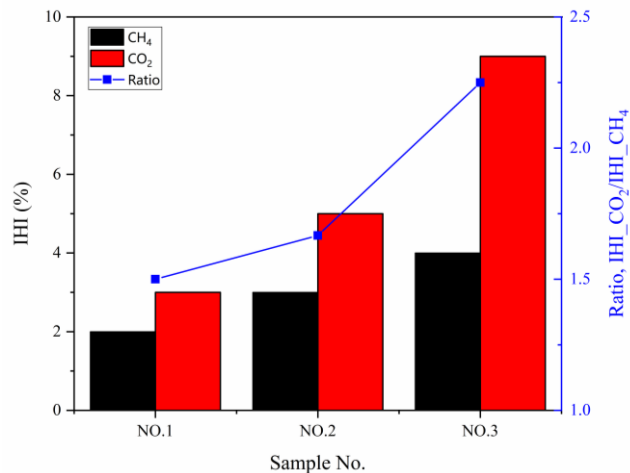


Figure 6: Sorption hysteresis results of Mine C, all with 212um coal

Figure 7: Comparison of IHI between CH₄ and CO₂ sorption

Gas adsorption-induced matrix strain changes the gas diffusion paths (Wang et al., 2014). The adsorption process would induce matrix swelling. As a result, the swelling effect would reduce the aperture of the gas diffusion channels. Coal contains constricted micropores with narrow pore throats, which are smaller than the kinetic diameters of CH₄ and CO₂. The gas molecules can enter the constricted pores with increasing gas pressure, but this leads to swelling of the coal matrix, which further narrows the pore throats. During depressurization, the gas molecules which have entered can escape from the constricted pores through the further narrowed pore throats, but this requires more energy than that which enables them to enter the pores. In other words, fewer gas molecules can escape from the constricted pores of coal during the desorption process than the sorption volume in the adsorption process at the same gas pressure. This accounts for the methane and CO₂ sorption hysteresis. The constricted micropores with narrow pore throats, which is 0.38 nm for CH₄ and 0.33 nm for CO₂ play a significant role in sorption hysteresis.

5. CONCLUSION

(1) Coal has different adsorption capacities for CO₂ and CH₄, and the adsorption capacity for different gases under the same conditions is CO₂>CH₄/CO₂ mixture>CH₄. Compared with CH₄, the molecular dynamic diameter of CO₂ is smaller, and the maximum attractive forces between CO₂ molecules and between CO₂-C molecules are greater, enabling it to enter smaller pores, thus more adsorption sites are available for CO₂ adsorption.

(2) With reference to the CO₂ and CH₄ isotherms, for a given gas content, the equilibrium gas pressure of a CH₄ rich coal sample is significantly greater than the equivalent CO₂ rich coal sample. Therefore, given that gas pressure provides energy to induce outbursts, it is reasonable to suggest that CH₄ rich coal contains greater outburst initiating energy.

(3) CO₂ adsorption/desorption contains smaller P_L than CH₄ adsorption/desorption, indicating that compared with CH₄, CO₂ is easier to adsorb at the low-pressure stage, and but more difficult to desorb at the high-pressure stage. For a given gas pressure, desorbing a large amount of CO₂ requires a higher pressure drop and more time. From this perspective, the outburst propensity of CO₂ rich coal is lower than that of CH₄.

(4) CH₄ and CO₂ sorption hysteresis may have significant impacts on coal and gas outbursts. CO₂ sorption hysteresis is more significant than CH₄ sorption hysteresis, at equivalent adsorption gas content, the amount of CO₂ desorption is less than that of CH₄, making it difficult to provide sufficient supply of desorption gas, resulting in low gas desorption energy, which is less conducive to the development of outburst.

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