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# Study on methane adsorption characteristics of different rank coals in presence of moisture

*Experiments on methane sorption of four coals from north China ranging from high-volatile bituminous to anthracite with different moisture have been carried out. The results show that the coal rank has a significant impact on the adsorption characteristic of the moist coal. The moisture content of coals varies with volatile matter shows a U-shape function and reaches the minimum at about 30% volatile matter under the same humidity and temperature condition. The methane adsorption isotherms of four different rank coals in the presence of moisture can be well fitted by the Langmuir model. The experimental results show that there is a critical moisture content above which the moisture has no inhibition effect on methane sorption. Furthermore, coal rank influences the critical moisture content. High rank coal usually has higher critical moisture than low rank coal. Moreover, the adsorption capacity of low rank coal reduces more by the moisture than high rank coal at the moisture content. A new empirical formula was proposed and it can well fit the data of the gas adsorption capacity of four coals with different moisture. This empirical formula explains the effect of coal rank on the adsorption capacity of moist coal from the perspective of mathematical theory.*

*Keywords: Moisture, methane, isothermal adsorption, coal rank*

## 1. Introduction

Coalbed methane recovery projects have been currently developed commercially all over the world [1]. Methane isothermal adsorption is an important experimental method to evaluate the yield of coalbed methane [2]. The coal in seam conditions is always saturated by water, so it is necessary to identify the effect of moisture on methane adsorption. Some studies have examined the methane adsorption characteristics of coals with different moisture contents [3-7]. It is generally agreed that the presence of moisture reduces the adsorption

capacity of methane on coal. There is a critical moisture content which is coal-dependent and above which further moisture has no effect on the methane sorption. Level et al [8] found that methane capacity decreased linearly with increasing moisture when the coal is at low moisture and this is similar to the relationship described by Ettinger et al [9] and Joubert et al [5,6]. Previous studies mainly focused on the methane adsorption regularity of the same coal with different moisture contents but the methane adsorption measurements of different rank coals with different moisture content levels are rarely mentioned.

Siemons and Busch measured CO<sub>2</sub> sorption isotherms on both dry and moist coals of various ranks, the study showed there was no obvious correlation between the sorption capacity and the coal rank in dry samples but the CO<sub>2</sub> sorption capacity increased with the increasing rank in moist samples. Prinz and Littke [10] also found that the adsorption capacity of moist coals was rank dependent. However, the law of coal rank affecting methane adsorption of coal in the presence of moisture is still not clear at present.

In this work, Methane isotherms of four coals from north China with different moisture content were measured. The effect of moisture content on the adsorption capacity of different rank coals was compared. After that, the authors analyzed the characteristics of coal rank affecting methane adsorption of moist coal. The study is expected to make some contributions to the coal seam gas occurrence theory and the evaluation work of coalbed methane production.

## 2. Coal sample

### 2.1 SAMPLES AND SAMPLE CHARACTERIZATION

Four coal samples of different ranks were collected from such sites in the north of China as Gencun Coal mine (GC) and Hong ling Coal mine (HL) in Henan Province, Tangshan Coal mine (TS) in HeBei Province and Changcun Coal mine (CC) in Shanxi Province. These samples were immediately sent to the laboratory for proximate analysis after being separated from the working face [11]. The coal proximate analysis followed the GB/T 30732-2014 and was conducted by the GF-A2000 auto proximate analyzer. The coals were

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divided into four ranks: low-volatile bituminous, medium-volatile bituminous, high-volatile bituminous and anthracite following the GBT/T 5751-2009. Details of the four samples are shown in Table 1.

## 2.2 SAMPLES WITH DIFFERENT MOISTURE

The four coals were all crushed and screened to obtain the desired sample size of 0.18-0.25mm. Then the samples were dried at 50°C in a drying oven for a week under nitrogen atmosphere to avoid coal oxidation. The samples were weight periodically until weight did not change anymore, then the sample was assured to be absent with moisture. The drying process of the coal samples took more than a week [12]. Then, we divided the dried coal samples into two groups and each group contains the four kinds of coal samples, one group coal samples were sealed and stored in a dry jar respectively, the other group coal samples were used to prepare moisturized samples.

The coal samples with different moisture were prepared in the HWS-70B humidity chamber, Three working condition which were 303 K and 50% humidity, 303 K and 80% humidity and 303K and 96% humidity were set orderly. The moist samples were weighed periodically and the equilibrium time of samples in each working condition needs about a week. After water balance, small aliquot was taken to measure moisture content by drying. The moisture content of the coal samples under different equilibrium condition were listed in Table 2. The coal samples with different moisture were prepared in the HWS-70B humidity chamber, Three working condition were set orderly which were 303 K and 50% humidity, 303 K and 80% humidity and 303K and 96% humidity. The moist samples were weighed periodically and the equilibrium time of samples in each working condition needs about a week. After the water balance, small aliquot was

taken to measure moisture content by drying. The moisture content of the coal samples under different equilibrium conditions were listed in Table 2.

## 3. Experiments

### 3.1 EXPERIMENTAL DEVICE

Fig.1 shows schematically the experimental device which consists of four systems: (1) A vacuum degassing system, which includes a vacuum pump, a vacuum gauge, a vacuum gauge tube, some valves and some hoses. This system is able to remove the gas existing in the pipeline network, the coal sample tank and the coal pore. A vacuum pump was used for degassing and the limiting vacuum of the pump is  $6.0 \times 10^{-2}$  Pa. The measuring range of the vacuum gauge is  $1.0 \times 10^{-8} \sim 1.0 \times 10^5$  Pa. (2) A gas supply system, which contains a high pressure CH<sub>4</sub> cylinder, a high pressure helium cylinder, a pressure reducing valve, a reference cell, a precise pressure gauge and some pipes. The measurement range of the pressure gauge is 0~10MPa and measurement accuracy is 0.001MPa. High-pressure helium gas is used to calibrate the volume of the reference cell and sample cell. Prior to the sorption experiment the void volume of the sample is also determined by the expansion of helium gas. (3) A thermostatic system, which includes a thermostatic water bath, an electric heater and a temperature sensor. This system is primarily used to ensure a constant temperature in the reference cell and coal sample cell in the course of the experiment. The adjustable temperature range is from the room temperature to 100°C. (4) An adsorption equilibrium system, which contains a coal sample cell and a pressure gauge. This system can ensure methane adsorption equilibrium in the coal sample cell.

### 3.2 EXPERIMENTAL METHODOLOGY

The methane adsorption isotherm was measured by the manometric method [13]. Six pressure steps were used from 1.6MPa to 5.8MPa. Before the conduction of an adsorption experiment, the volumes of reference cell and the volume of sample cell were calibrated already by a standard steel specimen with known volume and helium gas according to the calibration method

TABLE 1: PROPERTIES OF THE FOUR COALS

Sample	Coal mine	Coal rank	Proximate analysis (%)		
			M <sub>ad</sub>	A <sub>ad</sub>	V <sub>daf</sub>
GC	Changcun	High-volatile bituminous	4.14	8.38	39.34
TS	Tangshan	Medium-volatile bituminous	1.01	4.38	36.18
HL	Hongling	Low-volatile bituminous	0.58	9.48	18.57
CC	Changcun	Anthracite	0.70	10.62	9.79

TABLE 2: SUMMARY OF ISOTHERM ADSORPTION PARAMETERS OF THE FOUR COAL SAMPLES

Sample	Humidity (%)	Moisture (%)	V <sub>L</sub> (cm <sup>3</sup> /g, daf)	P <sub>L</sub> (MPa)	Sample	Humidity (%)	Moisture (%)	V <sub>L</sub> (cm <sup>3</sup> /g, daf)	P <sub>L</sub> (MPa)
GC	50	1.815	19.35	2.38	HL	50	1.600	34.87	2.62
GC	80	3.481	13.11	2.01	HL	80	3.349	31.95	4.13
GC	96	4.286	12.92	2.23	HL	96	4.031	31.12	4.35
TS	50	1.348	24.31	2.99	CC	50	2.884	39.62	1.77
TS	80	2.906	19.61	4.10	CC	80	4.956	25.59	2.14
TS	96	3.640	16.89	3.44	CC	96	5.713	23.19	2.02

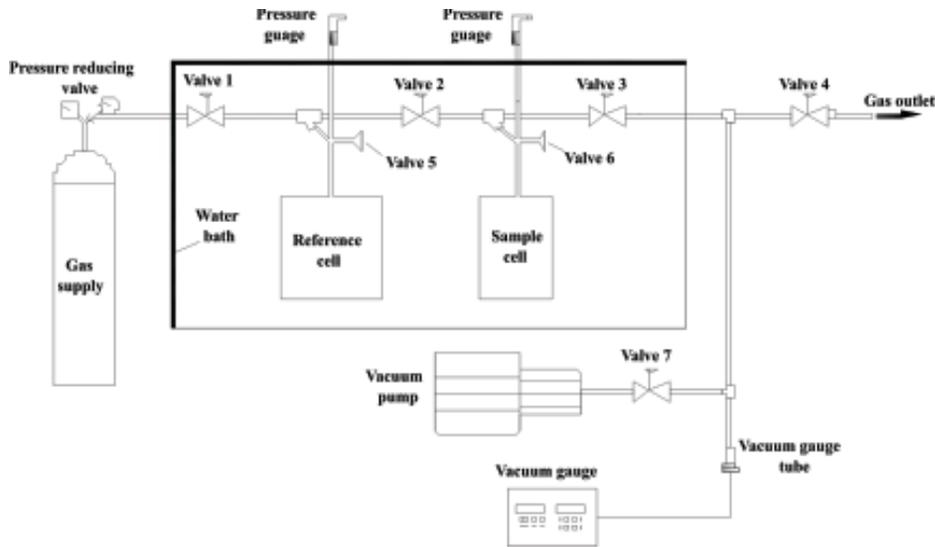


Fig.1 The schematic diagram of the adsorption device

of porosity instrument mentioned in SY/T 5336-2006. Then the methane adsorption isotherm was measured following the method mentioned in GB/T 19560-2008. The procedure is detailed as follows:

1. About 130g dry or moist sample weighted by a digital balance with a precision of 0.1 mg was put into sample cell.
2. Valve 1 ~ valve 6 were closed. A certain amount of helium gas was admitted to the reference cell by opening the valve 1 and valve 5, then valve 1 was closed and 45-60 minutes were allowed for pressure and thermal equilibration. The equilibration pressure  $P_r$  and atmospheric pressure  $P_a$  were measured.
3. Valve 2 and valve 6 were then opened and the helium gas was admitted to the sample cell. The pressure dropped and it was accurately monitored by the pressure gauge. 45-60 minutes were allowed for pressure and thermal equilibration. The equilibration pressure was recorded as  $P_e$ . The void volume of sample cell could be calculated by the following equation:
 
$$\frac{P_r V_r}{Z_r} + \frac{P_a V_{void}}{Z_a} = \frac{P_e (V_r + V_{void})}{Z_e} \quad \dots (1)$$
 where  $P_r$  is equilibration pressure after gas inflated into the reference cell,  $P_a$  is initial atmospheric pressure in sample cell,  $P_e$  is the equilibration pressure after gas inflated into the sample cell,  $V_r$  and  $V_{void}$  are volumes of reference cell and void volume of sample cell, respectively.  $Z_r$ ,  $Z_a$  and  $Z_e$  are the compressibility factors.
4. Valve 3 and valve 4 were opened, both the helium gas in reference and sample cells were evacuated. When the moist coal was used for experiment, the valve 6 was closed rapidly after the exhaustion to prevent the loss of moisture.
5. The temperature of the thermostatic water bath was

adjusted to  $30 \pm 0.1^\circ\text{C}$ .

6. The reference cell and sample cell were degassed, valve 1 and valve 4 were closed but other valves were opened. The system was vacuumed until the reading of vacuum gauge was lower than 4 Pa. It should be noted that special care should be taken for the system vacuuming before the experiments on moisturized samples. When vacuuming the apparatus, the sample cell valve 6 was closed and the pipelines were vacuumed first. Then the sample cell was vacuumed only 1min to avoid sample moisture loss to the

most extent. After degasing, all the valves were closed and the vacuum pump was turned off.

7. CH<sub>4</sub> gas, with purity over 99.99%, was expanded into the reference cell to 1.6MPa by opening valve 1 and adjusting the pressure reducing valve.
8. Valve 1 was closed, then, valve 2 and valve 6 between the reference cell and the sample cell were opened to expand CH<sub>4</sub> gas into the sample cell. When the pressure readings of both cells kept constant for at least 8 h, the equilibration pressure was recorded and valve 2 between the reference cell and the sample cell was closed.
9. Repeat Steps 6 to 8. The initial pressure in the reference cell was adjusted to 2.2MPa, 3.0MPa, 3.6MPa, 4.2MPa, 5MPa and 5.8MPa. The methane adsorption capacity can be calculated by the following equation:

$$Q_n = Q_{n-1} + \frac{V_m (P_{n0} V_r / Z_{n0} + P_{n-1} V_{void} / Z_{n-1} - P_n (V_r + V_{void}) / Z_n)}{mRT} \quad \dots (2)$$

where,  $n$  is the number of times of adsorption equilibrium processes,  $Q_n$  is the accumulative adsorption capacity after the CH<sub>4</sub> gas was expanded into the sample cell for the  $n$ th time ( $Q_0 = 0$ ),  $Q_{n-1}$  is the accumulative adsorption capacity after the CH<sub>4</sub> gas was expanded into the sample cell for the  $n$ th time,  $P_{n0}$  is the initial pressure of reference cell before the CH<sub>4</sub> gas was expanded into the sample cell at  $n$  time.  $P_n$  is the equilibrium pressure after the CH<sub>4</sub> gas was expanded into the sample cell for the  $n$ th time.  $P_{n-1}$  is the equilibrium pressure after the CH<sub>4</sub> gas was expanded into the sample cell for the  $(n-1)$ th time.  $Z_{i0}$ ,  $Z_{i-1}$  and  $Z_i$  are the compression factors.  $V_m$  is the molar volume of gas (22.4L/mol),  $m$  is the quality of coal sample,  $R$  is the gas constant ( $R = 8.735, \text{J/mol} \cdot \text{K}$ ),  $T$  is temperature of the water bath.

## 4. Results and discussions

### 4.1 MOISTURE CONTENT OF DIFFERENT RANK COALS

It is difficult to define the relationship between moisture and relative humidity prior to the analysis. Moisture and humidity data of four coals are summarized in Table 1. A cubic polynomial has been used to fit the moisture content and relative humidity.

According to the experimental data, a cubic polynomial curve can well reflect the relationship between relative humidity and moisture content (Fig.2). This is similar to the detailed moisture adsorption isotherm studies of Allardice and Evans on lower rank coals [14]. The results show that the moisture content of coal sample increases quickly under a low relative humidity, but it increase slowly when the relative humidity is closed to 100% relative humidity.

Fig.3 shows the moisture content distribution with volatile matter under the relative humidity of 50%, 80% and 96% at 30°C. Furthermore, Fig.3 shows the moisture content

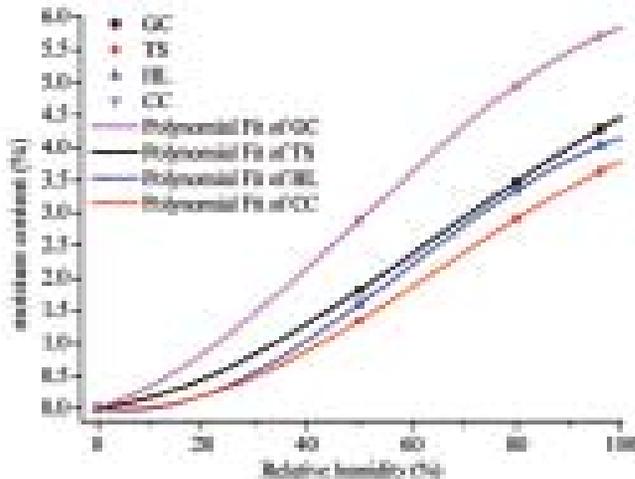


Fig.2 Relationship between relative humidity and moisture content

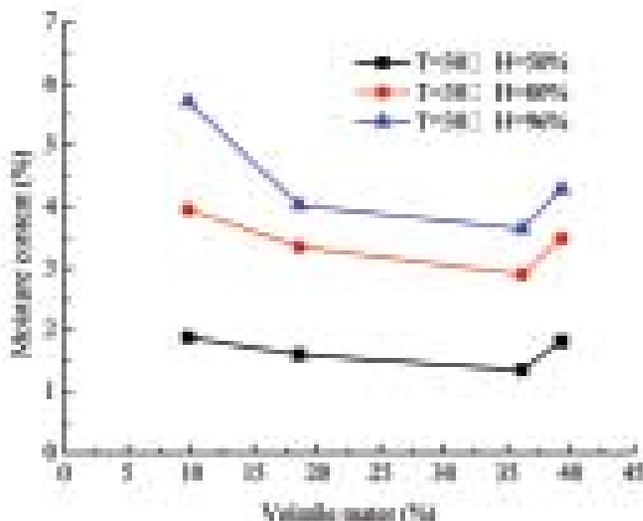


Fig.3 Moisture content distribution with the volatile matter

decreased with the relative humidity at first, reaching the minimum at about 36% volatile matter, and then increased with the relative humidity. It is similar to the result of the study of D. Prinz and R. Littke [10]. According to their study, the moisture content of coals varies with volatile matter ranking from lignite to anthracite shows a U-shape function and reaches the minimum at about 30% volatile matter and this parabolic behavior may be explained by the micro-pore volume distribution with coal rank.

### 4.2 MODELLING OF ADSORPTION ISOTHERM

Three types of isothermal adsorption models - Langmuir, Dubinin-Radushkevich (D-R) mode and Double Porosity model are widely used to analyze the adsorption experiment data [15]. This study shows that the Langmuir mode can well fit the methane adsorption isotherm of the four different rank coals (Fig.5). The Langmuir mode equation is as follows:

$$V = \frac{V_L P}{P_L + P} \quad \dots (3)$$

where,  $V$  is the volume of gas (standard state) adsorbed at constant temperature per gram of coal,  $P$  is the absolute pressure of methane,  $V_L$  is the Langmuir volume, and  $P_L$  is the Langmuir pressure.

Methane isotherm adsorption data on coal samples with different moisture contents are fitted by the Langmuir mode and the fitting parameters are summarized in Table 2. The Langmuir volume  $V_L$  indicates the maximum monolayer adsorption capacity. It clearly shows that  $V_L$  decreases with moisture content as shown in Figure 4(a). The Langmuir pressure  $P_L$  is pressure when the adsorption amount reached the half of the Langmuir volume.  $P_L$  represents methane adsorption rate of the coal. As Fig.4(b) shows, there is no specific relationship between the  $P_L$  and moisture content.

### 4.3 ADSORPTION ISOTHERMS OF DIFFERENT RANK COAL

The methane adsorption isotherms for each coal of four moisture contents are shown in Fig.5. The results show that the adsorption capacity of  $\text{CH}_4$  decreases with the increasing moisture, but there is a critical value, when the moisture is over the value, additional moisture does not reduce methane adsorption capacity of the coal. The same conclusion was also drawn in the study of Crosdale [3], Joubert et al [5,6].

According to the study of Day et al [16], the critical moisture content depends on the rank of the coal. The critical moisture content increased with the coal rank and was equivalent to the moisture content which would be attained if the coal was equilibrated at about 60 to 80% relative humidity. Figure 5(a) and 5(d) show that the critical moisture of low rank GC coal is about 3.481%, while the critical moisture of high rank CC coal is larger than 4.956%. It is obvious that high rank coal has higher critical moisture than low rank coal. What's more, the degree of capacity reduced by moisture depends on coal rank. Low rank coal is more affected by the

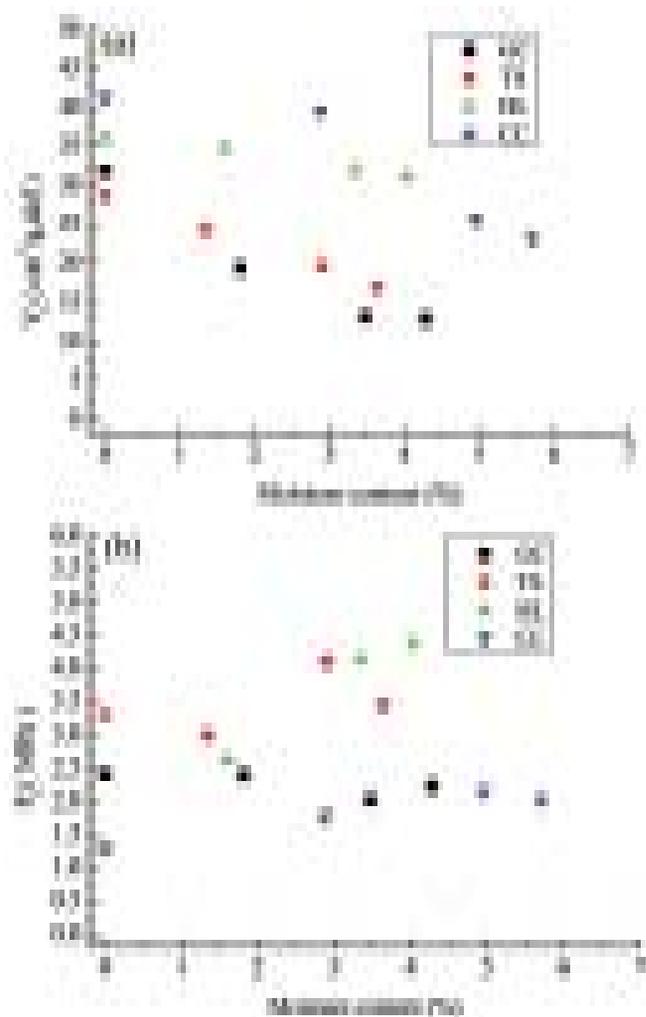


Fig.4 Moisture content distribution with the volatile matter

moisture than high rank coal. When the moisture content of the GC coal sample rises from 0 (dry) to 1.815%, the methane adsorption capacity falls to approximately 1/2 at the same pressure. While for TS, HL and CC coal sample, when moisture content from 0 to 2.906%, 3.349% and 4.956%, the methane adsorption capacity falls to 1/2 of the dry coal.

The mechanism which leads to a reduction in methane adsorption is that water molecules preferentially occupied the polar sites which are available for methane molecules. What's

more, when the moisture content is beyond the monolayer capacity, clusters of water molecules form around the polar sites [16]. Clusters at or across the opening of the pores can effectively block the entire pore space, cause the decrease of diffusivity and induce swelling thus further reduce the sorption capacity [17]. The methane is unlikely to attach to water, so it will be restricted to the hydrophobic sites not occupied by water. The primary polar sites where water adsorbed are the oxygen containing functional groups such as carboxylic groups and hydrophilic groups. Since low rank coal contains a greater proportion of oxygen containing functional groups [18], the reduction of methane capacity in the presence of water is greater than high rank coal. The adsorption surface area occupied by water increased with the increasing of moisture content. When the moisture content exceeds the critical level, the superfluous moisture only occupies large pores or inter-particle voids which is not accessible to methane adsorption [16]. For this reason, the moisture beyond critical level has no effect on the methane adsorption capacity of coal.

#### 4.4 RELATIONSHIP BETWEEN GAS CONTENT AND MOISTURE CONTENT

Many studies covered the relationship between the adsorption capacity of coal and the moisture content. Some empirical formulas were proposed to fit the relationship between moisture content and adsorption capacity. Among these formulas, one proposed by a scholar from former Soviet Union is most widely used [19, 20]. The formula is as follows:

$$V_w/V_d = 1/(1 + Am) \quad \dots \quad (4)$$

where  $V_w$  and  $V_d$  are the amounts of the methane adsorbed in moist and dry coal, respectively,  $m$  is the coal moisture content in weight percent; and  $A$  is a fitting coefficient.

According to the Langmuir volume and the Langmuir pressure in different moisture as shown in Table 2, the adsorption capacity at different pressure could be calculated by the Langmuir mode. Fig.6 shows the variation of the adsorption capacity with the moisture content at different pressure fitted by the formula (4). The fitting coefficient  $A$  is related to the volatile matter and pressure [5, 19]. Since the effect of pressure on the coefficient  $A$  is small, the average

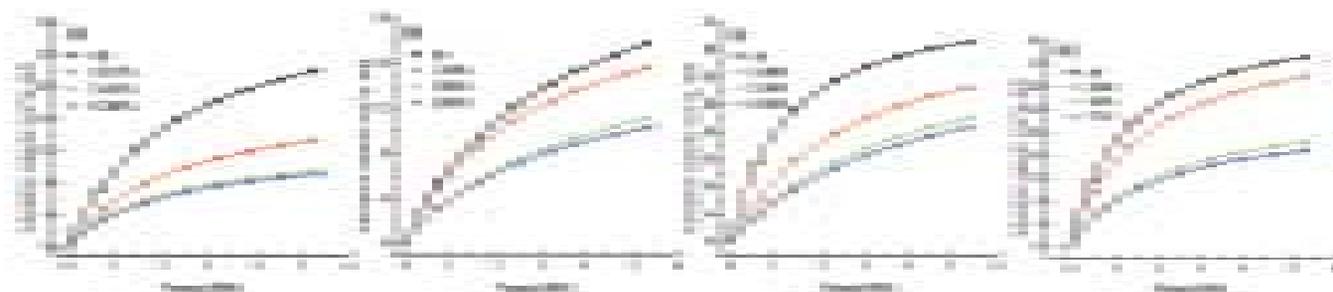


Fig.5 Methane adsorption isotherms of different rank coal at 30°C with different moisture contents (a - GC; b - TS; c - HL; d - CC)

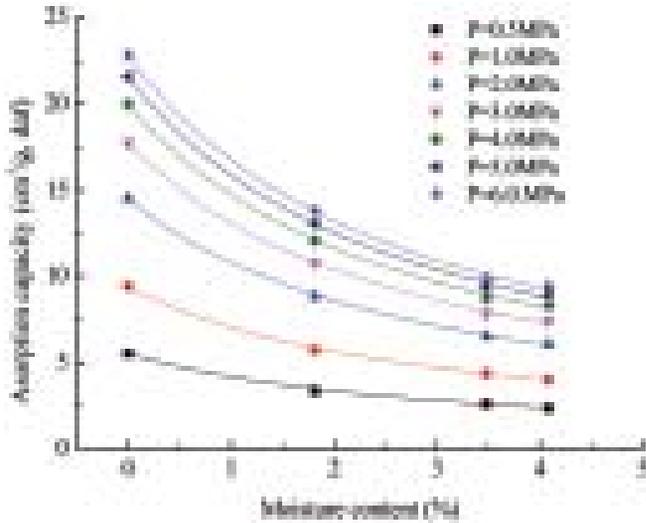


Fig.6 The relationship between moisture and adsorbed gas content at different pressure

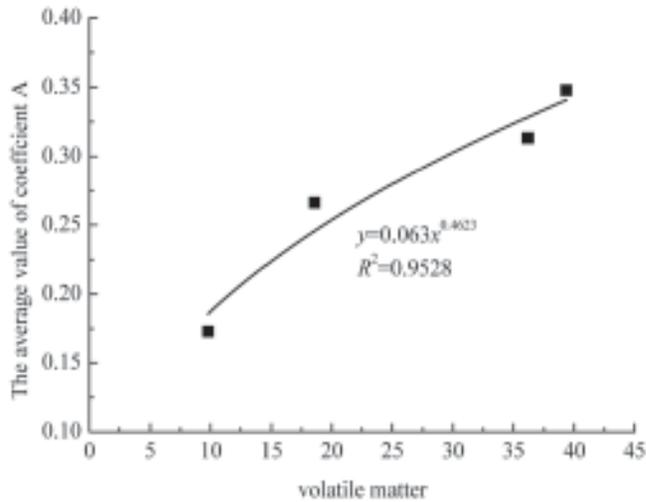


Fig.7 The relationship between  $A_{avg}$  and volatile matter

value of coefficient A at different pressure recorded as  $A_{avg}$  was calculated. The coefficient  $A_{avg}$  of the four coal samples were calculated. The values of  $A_{avg}$  are 0.347 for GC, 0.313 for TS, 0.266 for HL and 0.173 for CC. The relationship between  $A_{avg}$  and volatile matter is shown in Fig.7, the data can fitted by power function formula as follows:

$$A_{avg} = aV_{daf}^b \quad \dots \quad (5)$$

where  $V_{daf}$  is the volatile matter,  $a$  and  $b$  are constants. Hence, the relationship between moisture content and adsorption capacity can be expressed as:

$$V_w/V_d = 1/(1 + aV_{daf}^b m) \quad \dots \quad (6)$$

Fig.7 shows that  $A_{avg}$  increases with the increase of volatile matter. According to formula (4), when the moisture condition is invariable,  $V_w/V_d$  decreases with the increase of

volatile matter.  $V_w/V_d$  indicates the reduction degree of adsorption capacity caused by moisture. As the volatile matter increases with decrease of coal rank, it can be concluded that the lower rank the coal is, the greater impact the moisture has on the methane adsorption capacity. The formula explains the effect of coal rank on the adsorption capacity of wet coal from the perspective of mathematical theory.

## 5. Conclusions

Isothermal adsorption experiments were carried on the four coals of different rank in this study. The results of this work show that the coal rank has a significant impact on the capacity of the coal in presence of moisture. The conclusions are as follows:

- (1) The moisture content of coals varying with volatile matter ranking from lignite to anthracite shows a U-shape function under same relative humidity and temperature. It was found that the moisture content of coals reach the minimum at about 36% volatile matter when under the same humidity and temperature condition.
- (2) The Langmuir mode provides an excellent fit to the isothermal adsorption experimental data of four different rank coals. The results of this study show that Langmuir volume decreases with moisture content, but there is no specific relationship between Langmuir pressure and moisture content.
- (3) The critical moisture content depends on the rank of the coal. High rank coal usually has higher critical moisture than the low rank coal. Low rank coal is more affected by the presence of moisture than high rank coal. The higher proportion of oxygen containing functional groups is the reason why the methane capacity of low rank coal decreases more than high rank coal in the presence of moisture.
- (4) The adsorption capacity of different moisture content could well fitted by the formula of  $V_w/V_d = 1/(1 + aV_{daf}^b m)$ . The formula explains the effect of coal rank on the adsorption capacity of wet coal from mathematical theory.

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