

Thermodynamic Characteristics of Adsorption-Desorption of Methane in 3[#] Coal Seam of Sihe

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Abstract

A series of methane adsorption-desorption isotherm experiments on anthracite of No. 3 Sihe coal mine were conducted at 20°C, 25°C, 30°C, 35°C and 40°C respectively. Based on Clausius-Clapeyron equation, isosteric heat of adsorption and maximum heat of adsorption has been calculated. These calculations indicate that the maximum heat of adsorption in process of elevated pressure (adsorption) and lowered stress (desorption) is 23.31 KJ/mol and 24.02 KJ/mol, so it belongs to physical adsorption. However, the latter is higher than the former. From the point of view of thermodynamics, in the adsorption-desorption equilibrium system, dropping pressure alone does not lead to desorption, but it improves adsorption of water vapor molecules on the surface of the coal pores. The adsorption heat of water vapor molecules is greater than 40 KJ/mol, so the methane on the surface of coal pores will be easily replaced by water vapor, and the desorption of methane occurs eventually. Thus, the gas production in coalbed methane well by pressure reduction is consistent with the negative pressure for gas extraction.

Keywords

Methane, Adsorption, Desorption, Thermodynamics

1. Introduction

Coal methane mostly exists in the coal seams in the form of adsorption. Currently, the approach of coal methane desorption by drainage and lowering pressure for its ground production is widely adopted both at home and abroad. The method of negative pressure production with wells up and down connected is employed by Mine

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Gas Prevention and Control Center of Ningxia Bureau of Coal Geology at Shaqu Mine of Liulin County in Shanxi Province of China, and it has made great achievement with the production more than 10,000 m³ per day. The new approach has shed light on the theory of methane production, and demands careful exploration of the nature of drainage and lowering pressure production. Through the experiments on methane adsorption-desorption isotherm [1]-[3], by the calculations of isosteric heat of adsorption and maximum heat of adsorption with Clausius-Clapeyron equation, and by the contrast of the adsorption heat between methane and water vapor, the paper probes into the function and mechanism of water vapor in the process of water drainage and gas production.

2. Samples and Experimental Apparatus

Experimental samples are fresh raw coal collected by channel method from Sihe No. 3 coal (WY) in Shanxi Group of Permo-carboniferous system.

The collected samples, after being crushed, ground and screened, were processed into 60 - 80 mesh and made into moisture-equilibrated samples according to the standards of ASTM (American Society for Testing Material).

The experiments were accomplished by employing AST-2000 simulation experimental apparatus for bulk sample coalbed methane adsorption and desorption. The temperatures were set at 20°C, 25°C, 30°C, 35°C and 40°C respectively.

3. Experimental Results

Figure 1 and **Figure 2** are the isothermal adsorption-desorption data by making use of the fitting to simulate mathematics software of the moisture-equilibrated samples at different temperatures.

To express the action process of adsorption-desorption and pressure, the experimental data of adsorption at the five temperatures are Langmuir fitted [4] [5], and the experimental data of desorption are descriptively fitted [6] [7]. The results are shown in **Table 1**, **Figure 1** and **Figure 2**.

$$\text{Langmuir equation: } V = \frac{a \cdot b \cdot p}{1 + b \cdot p}$$

$$\text{Desorption equation: } V_d = \frac{a \cdot b \cdot p}{1 + b \cdot p} + c$$

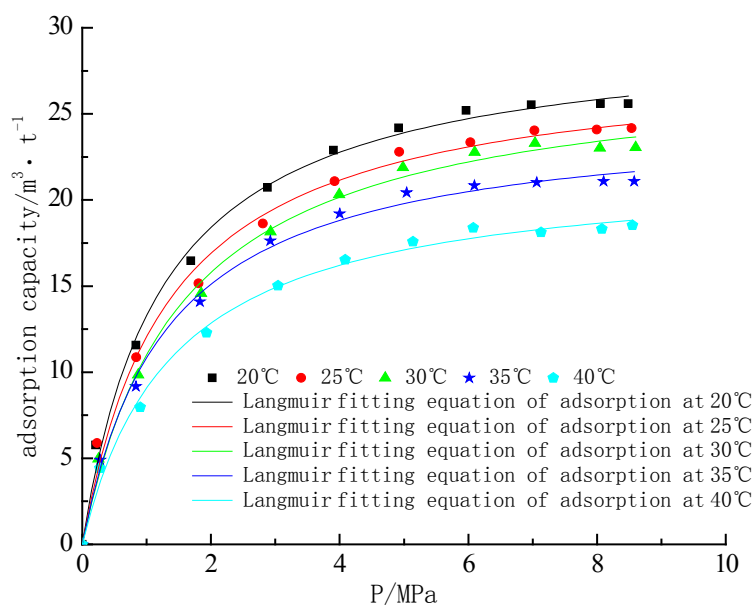


Figure 1. Fitted curves on experimental adsorption data by Langmuir equation of Sihe No. 3 WY.

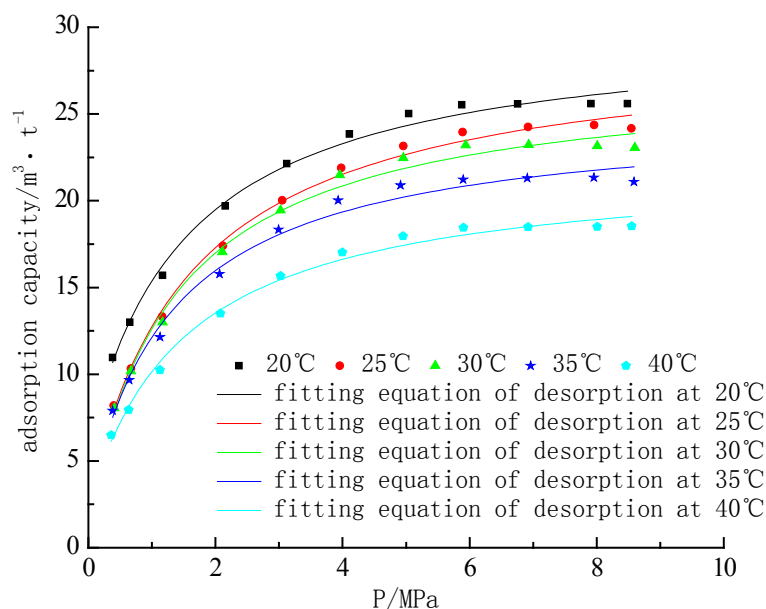


Figure 2. Fitted curves on experimental desorption data of Sihe No. 3 WY.

Table 1. Fitted parameters of the experimental data at five temperatures.

Temperature (°C)	Langmuir fitting of adsorption data			Descriptive fitting			
	<i>a</i>	<i>b</i>	<i>R</i> ²	<i>a</i>	<i>b</i>	<i>c</i>	<i>R</i> ²
20	29.84	0.80	0.996	24.08	0.64	5.97	0.993
25	28.24	0.75	0.993	26.51	0.62	2.64	0.995
30	27.87	0.65	0.996	25.48	0.72	1.93	0.992
35	24.93	0.77	0.996	22.42	0.74	2.57	0.988
40	21.94	0.71	0.995	19.71	0.65	2.39	0.992

4. Isotheric Heat of Adsorption

Isotheric heat of adsorption, also called differential heat of adsorption [8], is the heat released as the infinitesimal methane molecules are adsorbed, with the adsorption capacity being constant. It is the enthalpy change [9] at the moment of adsorption.

Isotheric heat of adsorption is expressed as q^{st} , calculated with Clausius-Clapeyron equation [10] [11]:

$$\frac{d \ln P}{dT} = \frac{q^{st}}{RT} \quad (1)$$

In the equation: q^{st} stands for isotheric heat of adsorption; P for pressure; T for absolute temperature; R for gas constant, 8.314.

Integrate Equation (1) and get [12]:

$$\ln P = -\frac{q^{st}}{RT} + C \quad (2)$$

Calculation process: $\ln P$ correlates with n , adsorption capacity; fit $\ln P - n$ data and get $\ln P - n$ relation. Have several fixed n , and the corresponding $\ln P$ can be got with fitting formulas. Then, plot $\ln P$ with $1/T$ at fixed adsorption capacity and have linear fitting, so adsorption isostere is achieved, based on whose slope isotheric heat of adsorption is calculated.

Calculation of isotheric heat of adsorption in process of adsorption of moisture-equilibrated samples of Sihe

No. 3 WY.

1) Based on the experimental data of isosteric adsorption, draw a $\ln P$ (KPa)– n (mmol/g) scatter diagram at different temperatures.

2) Have $\ln P$ – n linear fitting, and the fitting equation is shown in **Figure 3**.

3) Based on the above fitting formula, calculate $\ln P$ of n —fixed adsorption—(0.1, 0.2, ..., 1.2 mmol/g) at different temperatures (20°C - 40°C), shown by **Table 2**.

4) Plot $\ln P \sim T^{-1}$ data graph.

5) At different fixed adsorption capacity, linearly fit the relational data of $\ln P$ and T^{-1} . The fitting equation is shown by **Figure 4**.

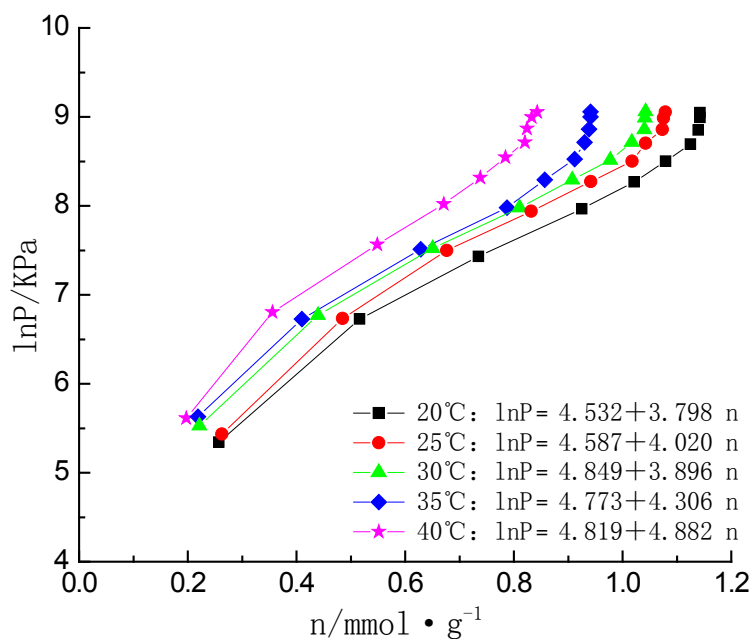


Figure 3. Relationship of pressures (logarithms) and adsorption capacity in the adsorption process.

Table 2. Calculations of $\ln P$ on different adsorption capacity in the adsorption process.

Temperature (°C)	20°C	25°C	30°C	35°C	40°C
Adsorption (mmol/g)					
0.1	4.91	4.99	5.24	5.20	5.31
0.2	5.29	5.39	5.63	5.63	5.80
0.3	5.67	5.79	6.02	6.06	6.28
0.4	6.05	6.20	6.41	6.50	6.77
0.5	6.43	6.60	6.80	6.93	7.26
0.6	6.81	7.00	7.19	7.36	7.75
0.7	7.19	7.40	7.58	7.79	8.24
0.8	7.57	7.80	7.97	8.22	8.72
0.9	7.95	8.21	8.36	8.65	9.21
1.0	8.33	8.61	8.75	9.08	9.70
1.1	8.71	9.01	9.13	9.51	10.19
1.2	9.09	9.41	9.52	9.94	10.68

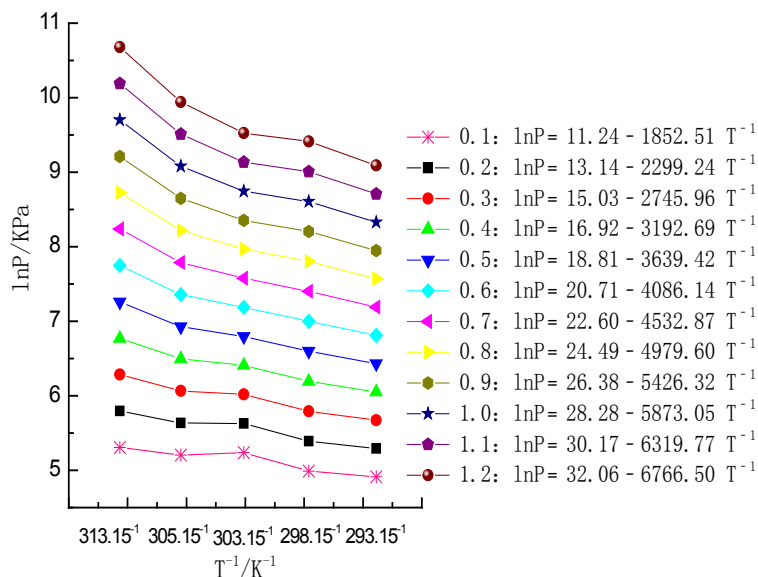


Figure 4. Isosteric adsorption line in the adsorption process.

6) Based on the slope of linear fitting, find isosteric heat of adsorption at different adsorption capacity by Equation (3).

$$q^{st} = -R \cdot B \tag{3}$$

In the equation, B is the slope of linear fitting equation.

The calculations are shown in Table 3.

7) Figure 5 is the curve of isosteric heat of adsorption with the change of adsorption capacity.

In the same way, calculate the isosteric heat of adsorption in process of desorption of moisture-equilibrated samples of Sihe No. 3 WY.

1) Based on the experimental data of isosteric desorption at different temperatures, Figure 6 is created by relating $\ln P$ (KPa)—the natural logarithm of pressure to n (mmol/g)—adsorption capacity.

2) Fit the data with linear $\ln P-n$. The fitting equation is shown by Figure 6.

3) Based on the above fitting formula, calculate the value of $\ln P$ on n —fixed adsorption—(0.2, 0.4, ..., 1.0 mmol/g) at different temperatures (20°C - 40°C), shown by Table 4.

4) Plot $\ln P \sim T^{-1}$ data graph.

5) At different fixed adsorption capacity, linearly fit the relational data of $\ln P$ and T^{-1} . The fitting equation is shown by Figure 7.

6) Based on the slope of linear fitting, *i.e.* B , find isosteric heat of adsorption at different adsorption capacity by Equation (3). The results are shown by Table 5.

7) Figure 8 is the curve of isosteric heat of adsorption with the change of adsorption capacity.

5. Maximum Heat of Adsorption

Maximum heat of adsorption is the isosteric heat of adsorption when the pressure tends to zero [13] [14].

Usually Virial equation is used to calculate maximum heat of adsorption. In the condition of extremely low pressure, adsorption isotherm should conform to Henry Law [15]-[17]:

$$n = K'P \tag{4}$$

In the equation: n , adsorption capacity, $\text{mmol} \cdot \text{g}^{-1}$; P , equilibrium pressure, KPa; K' , Henry constant, $\text{mmol} \cdot \text{g}^{-1}/\text{KPa}$.

Virial Equation is used to describe the adsorption isotherm in the whole process of experimental scope, and by the way of extrapolation, K' , Henry Law constant, can be found when the pressure is towards zero in the low pressure area. Then, q_0^{st} , maximum heat of adsorption, can be found by Vant Hoff Equation which K'

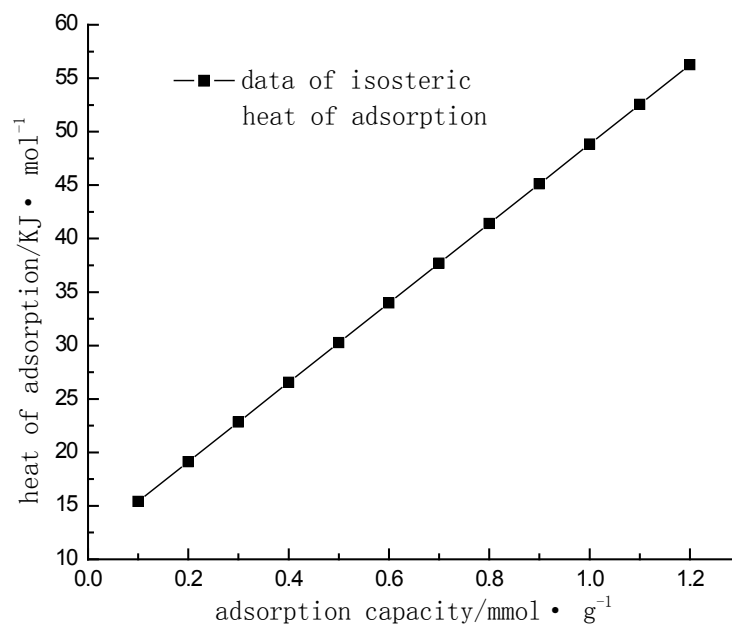


Figure 5. Change of isosteric heat of adsorption with adsorption capacity in adsorption process of Sihe No. 3 WY.

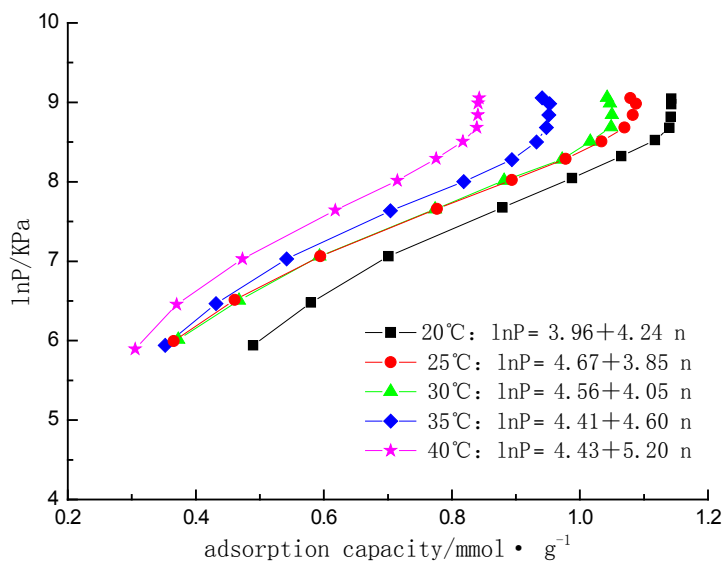


Figure 6. Relationship of pressures (logarithms) and adsorption capacity in the desorption process.

Table 3. Calculations of the isosteric heat of adsorption in the adsorption process.

Adsorption (mmol/g)	Heat of adsorption (KJ/mol)	Adsorption (mmol/g)	Heat of adsorption (KJ/mol)
0.1	15.40	0.2	19.12
0.3	22.83	0.4	26.54
0.5	30.26	0.6	33.97
0.7	37.69	0.8	41.40
0.9	45.11	1.0	48.83
1.1	52.54	1.2	56.26

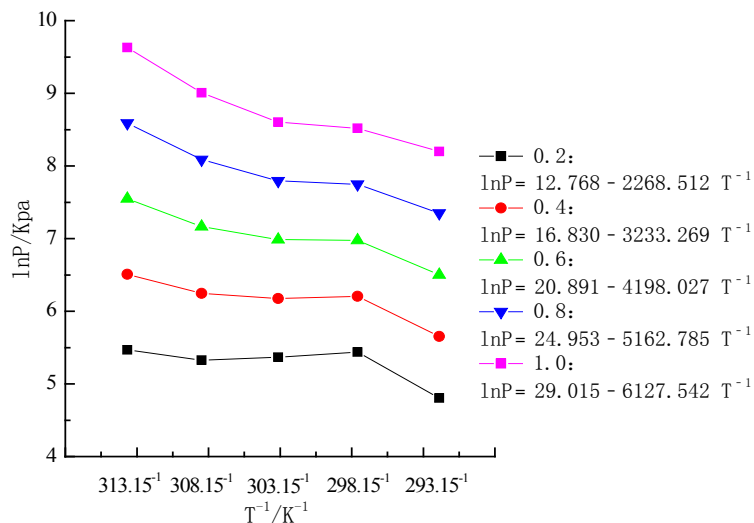


Figure 7. Isothermic adsorption line in the desorption process.

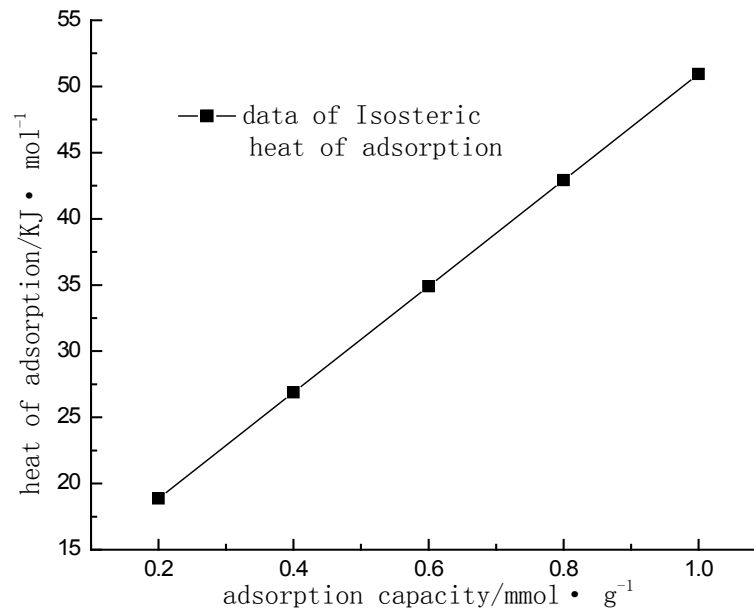


Figure 8. Isothermic heat of adsorption with adsorption capacity in desorption process of Sihe No. 3 WY.

Table 4. Calculations of $\ln P$ on different adsorption capacity in the desorption process.

Adsorption (mmol/g)	Temperature (°C)	20°C	25°C	30°C	35°C	40°C
	0.2		4.80	6.21	5.37	5.33
0.4		5.65	6.98	6.18	6.25	6.51
0.6		6.50	7.75	6.99	7.17	7.55
0.8		7.35	8.52	7.79	8.09	8.59
1.0		8.20	6.21	8.60	9.01	9.63

Table 5. Calculations of the isosteric heat of adsorption in the desorption process.

Adsorption (mmol/g)	Isosteric heat of adsorption (KJ/mol)
0.2	18.86
0.4	26.88
0.6	34.90
0.8	42.92
1.0	50.94

and T abide by. Vant Hoff Equation goes as follows:

$$\frac{d \ln K'}{dT} = \frac{\Delta H_0}{RT^2} \quad (5)$$

In the equation, the physical interpretation of ΔH_0 is the mole enthalpy difference between adsorption state and gas state when the pressure tends to zero, so ΔH_0 is actually q_0^{st} , maximum heat of adsorption.

Find Henry Law constant K' [18] [19] by Virial Equation

Virial Equation below is used to describe adsorption isotherm when adsorption is in equilibrium.

$$\frac{\pi}{nRT} = 1 + c_1 n + c_2 n^2 + c_3 n^3 + \dots \quad (6)$$

In the equation: π , two-dimensional dispersion pressure; c , Virial constant.

Equation (6) can be transformed by Gibbs Equation to Virial adsorption isotherm:

$$\frac{aP}{n} = \exp\left(2c_1 n + \frac{3}{2}c_2 n^2 + \dots\right) \quad (7)$$

When the pressure is very low, n is relatively small, and high order term can be neglected. Then, the plotting of $\ln(P/n)$ to n should be linear. In other words, Henry Law is tenable, so the value of K' , Henry Law constant, can be determined by the extrapolation value when $n=0$. From the above equation, when $n \rightarrow 0$,

$$n = aP \quad (8)$$

Therefore, Henry Law constant $K' = a$. In this condition, the following equation is found:

$$\ln\left(\frac{P}{n}\right) = \ln\left(\frac{1}{a}\right) = \ln\left(\frac{1}{K'}\right) \quad (9)$$

Suppose the intersection value of plotting and vertical axis is ζ , and $\ln(1/a) = \ln(1/K') = \zeta$. Then,

$$K' = \exp(-\zeta) \quad (10)$$

Therefore, Henry constant K' can be found based on the result of fitting straight line in low pressure.

The method of finding Henry constant K' by extrapolation is reliable because the experimental data in the low pressure area are linear, which guarantees the validity of ζ . Therefore, the calculated value of K' is also accurate.

Calculation of maximum heat of adsorption by Henry constant

The relationship between the known Henry Law constant K' and temperature abide by Vant Hoff Equation. If the interphase heat capacity difference is neglected, the result below can be got by integral:

$$K' = K'_0 \exp\left(-\frac{\Delta H_0}{RT}\right) = \exp(-\zeta) \quad (11)$$

Usually the plotting of $\ln K'$ and $1/T$ is linear, q_0^{st} , maximum heat of adsorption, is determined by its slope.

Calculation of maximum heat of adsorption in process of adsorption of moisture-equilibrated samples of Sihe

No. 3 WY.

1) Based on the experimental data of isosteric adsorption at different temperatures, plot with Virial $\ln(P/n)$ (KPa/mmol·g⁻¹) and adsorption capacity n (mol·g⁻¹). (See Figure 9)

2) Linearly fit Virial plotting by $\ln(P/n) = A + Bn$. (See Table 6)

3) Based on fitting result intercept, *i.e.* the value of A , calculate Henry Law constant K' by $K' = \exp(-A)$. The results are shown in Table 7.

4) Plot $\ln K'$ and $1/T$ and have linear fitting.

A : Vant Hoff plotting (See Figure 10)

B : Linear fitting results:

$$\ln K' = -15.67 + 2803.25 T^{-1}$$

5) Calculate maximum heat of adsorption based on B , the linear fitting slope. The equation is as follows:

$$q_0^{st} = -R \cdot B \tag{12}$$

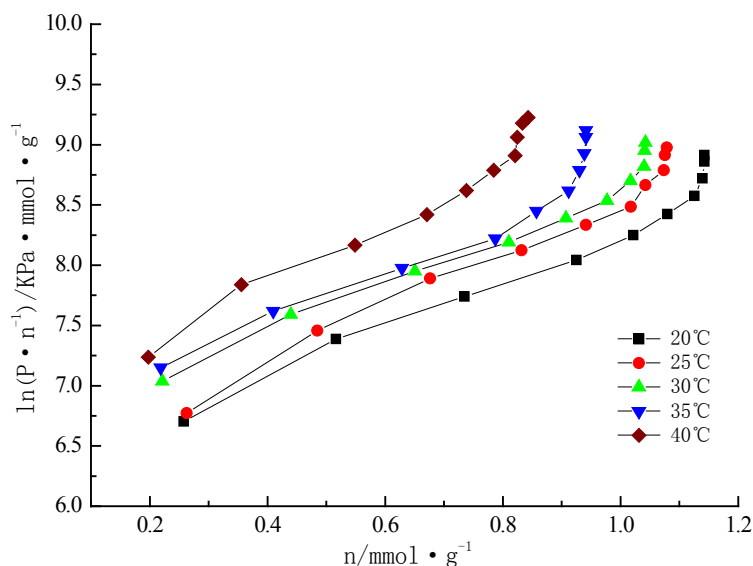


Figure 9. Cross plotting with Virial for adsorption data of Sihe No. 3 WY.

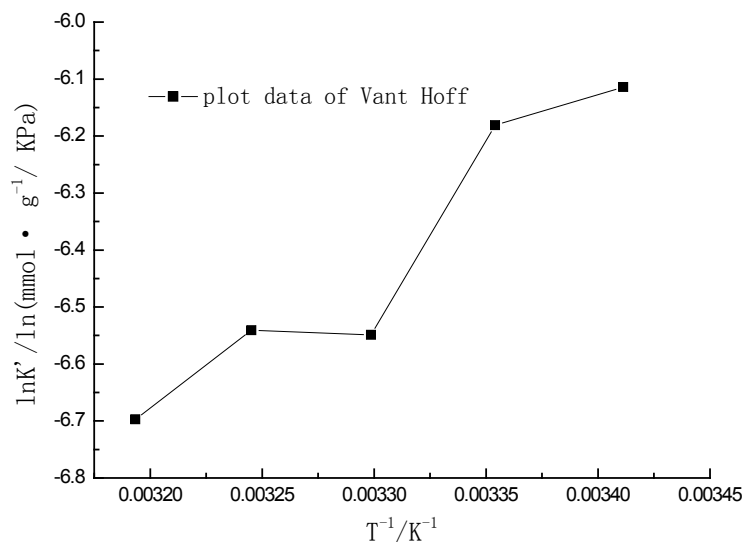


Figure 10. Cross plot with Vant Hoff for adsorption data of Sihe No. 3 WY.

Table 6. Fitted parameters of the Virial data in the adsorption process.

Temperature (°C)	T (K)	A	B
20	293.15	6.11	2.25
25	298.15	6.18	2.43
30	303.15	6.55	2.17
35	308.15	6.54	2.45
40	313.15	6.70	2.79

Table 7. Calculations of Henry Law constant K' in the adsorption process.

T (K)	K' (mmol·g ⁻¹ /KPa)	$1/T$ (K ⁻¹)	$\ln K'$ ln(mmol·g ⁻¹ /KPa)
293.15	0.002212	0.003411	-6.114
298.15	0.002068	0.003354	-6.181
303.15	0.001432	0.003299	-6.549
308.15	0.001443	0.003245	-6.541
313.15	0.001235	0.003193	-6.697

It is found $q_0^{st} = -23.30619$ KJ/mol, the minus indicating that adsorption is an exothermic process.

Calculation of maximum heat of adsorption in process of desorption of moisture-equilibrated samples of Sihe No. 3 WY.

1) Based on the experimental data of isosteric desorption at different temperatures, plot $\ln(P/n)$ and n , adsorption capacity. (See **Figure 11**)

2) Fit Virial plotting by linear $\ln(P/n) = A + Bn$. The results are shown in **Table 8**.

3) Based on fitting result intercept, *i.e.* the value of A , calculate Henry Law constant K' by $K' = \exp(-A)$. The results are shown in **Table 9**.

4) Plot $\ln K'$ and $1/T$ and have linear fitting.

A : Vant Hoff plotting (See **Figure 12**)

B : Linear fitting results:

$$\ln(P/n) = -15.40 + 2888.73n$$

5) Calculate maximum heat of adsorption based on B , the linear fitting slope.

Find: $q_0^{st} = -24.02$ KJ/mol.

6. Contrast of Adsorption Heat Results

Table 10 is the isosteric heat of adsorption calculations result. The maximum heat of desorption process is higher than adsorption.

7. Conclusions

1) Adsorption heat and adsorption capacity are positively correlative.

2) Adsorption heat in rising pressure is less than that in dropping pressure. When the fixed adsorption capacity is 0.2 - 1.0 mmol/g, adsorption heat is 19 - 51 KJ/mol. Methane-coal adsorption belongs to physical adsorption [20]-[24].

3) Desorption rate increases as the temperature rises. Adsorption heat in rising pressure is less than that in dropping pressure. To make desorption occur, heat supply is needed, and the adsorption heat of water vapor is larger than 40 KJ/mol. Therefore, as long as water vapor exists, desorption probably occurs in the system of methane-coal adsorption. We believe that pressure reduction results in the partial pressure increase of water va-

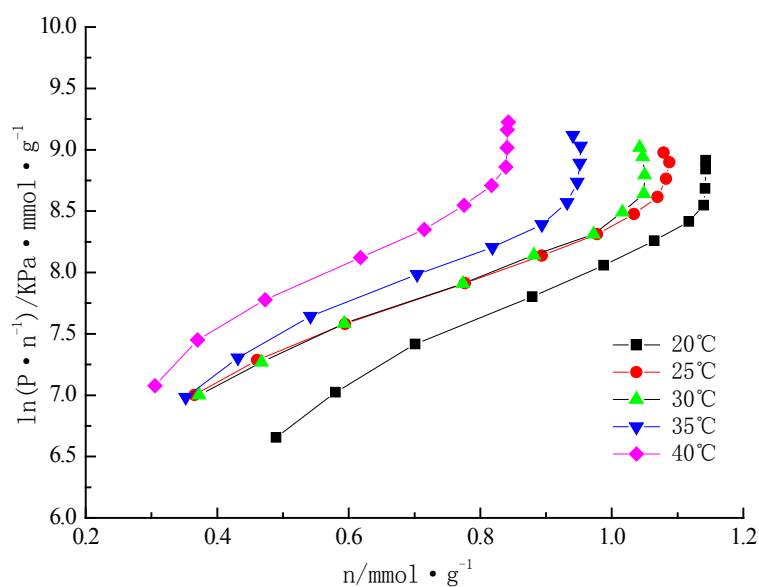


Figure 11. Cross plot with Virial for desorption data of Sihe No. 3 WY.

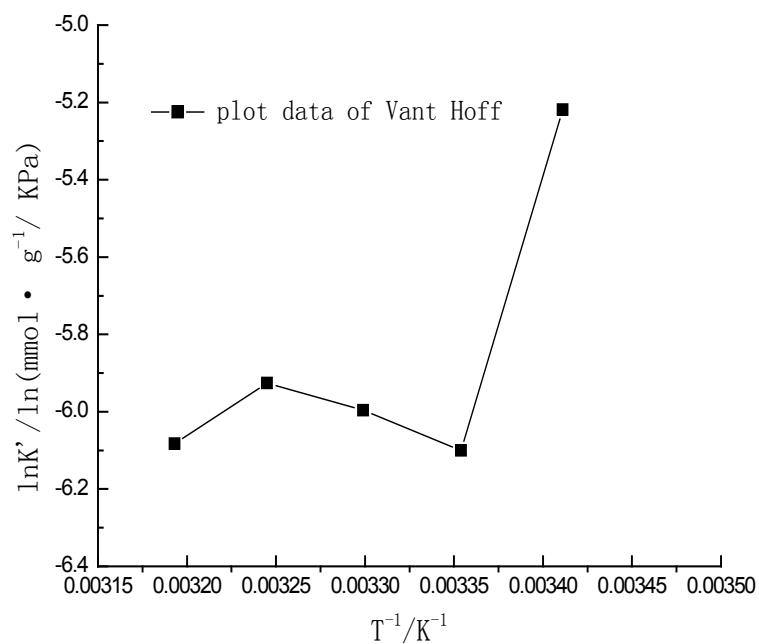


Figure 12. Cross plot with Vant Hoff for desorption data of Sihe No. 3 WY.

Table 8. Fitted parameters of the Virial data in the desorption process.

Temperature (°C)	T (K)	A	B
20	293.15	5.22	3.00
25	298.15	6.10	2.43
30	303.15	6.00	2.61
35	308.15	5.93	3.03
40	313.15	6.08	3.41

Table 9. Calculations of henry law constant K' in the desorption process.

T (K)	K' (mmol·g ⁻¹ /KPa)	$1/T$ (K ⁻¹)	$\ln K'$ ln(mmol·g ⁻¹ /KPa)
293.15	0.005413	0.003411	-5.219
298.15	0.002243	0.003354	-6.1
303.15	0.002489	0.003299	-5.996
308.15	0.002669	0.003245	-5.926
313.15	0.002281	0.003193	-6.083

Table 10. Calculations of isosteric heat of adsorption.

Adsorption capacity (mmol/g)	Isosteric heat of adsorption (KJ/mol)		Maximum heat of adsorption (KJ/mol)	
	Adsorption process	Desorption process	Adsorption process	Desorption process
0.2	19.12	18.86		
0.4	26.54	26.88		
0.6	33.97	34.90	-23.31	24.02
0.8	41.40	42.92		
1.0	48.83	50.94		

por in the local space, and heat is released while water vapor adsorbs, and thus the methane molecules in the corresponding places of the pore surface turn into free state. In the methane-coal-water interface interaction, with micro-environmental pressure decreasing, methane's desorption on the pore surface of coal is actually the result of replacement of methane molecules by water vapor (adsorption) in the corresponding adsorption sites.

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