INTRODUCTION

Because of low carbon emissions and economic benefits, large-scale extraction of coalbed methane has attracted widespread attention. However, the strong gas adsorption capacity and low permeability of coalbed methane reservoirs result in a long period of low production rates and low coalbed methane extraction efficiencies. Therefore, researchers and engineers have tried different methods to enhance the recovery of coalbed methane.

Coal is an extremely heterogeneous porous material and has a broad pore size distribution and various pore types. Both fractures and nanopores in coal provide basic channels for gas storage and transport. Generally, pores in coal can be classified into micropores (<2 nm), mesopores (2-50 nm), and macropores (>50 nm). The micropores and fine mesopores are predominant in anthracite. In addition, there are also many closed pores and semiclosed pores in coal, which trap gas, making gas recovery difficult. Thus, efficiently extracting coalbed methane from these nanopore systems and closed pores remains a challenge.

Most coalbed methane is stored in the form of adsorbed gas gathering in nanopore systems, as the nanopores have many exposed internal surface areas that can provide potential adsorption sites. Organic matter, especially vitrinite, has a positive impact on gas adsorption of coal, which...
contains many micropores for gas adsorption.\textsuperscript{12} Since organic matter is the major constituent of coal,\textsuperscript{13} coalbed methane reservoir has a high gas adsorption capacity. Therefore, methods of effectively promoting the desorption of coalbed gas molecules merit further research.

Gas transport in coalbed includes desorption from the surfaces of organic matter and clays, diffusion through the nanopore systems, and flow through the fracture networks.\textsuperscript{3} Several gas diffusion mechanisms occur at different conditions, such as bulk diffusion due to intermolecular collisions; Knudsen diffusion due to molecule-wall collisions; and surface diffusion when gas molecules are transported along the adsorbed layer.\textsuperscript{12} Additionally, irregularly distributed natural fracture networks provide the main channels for gas fluid migration, particularly Darcy flow.\textsuperscript{1,12,14} However, the permeability of most Chinese coal seams is too low for coalbed gas transport.\textsuperscript{15}

Several methods have been proposed to enhance the recovery of coalbed methane by increasing permeability and promoting gas desorption, such as hydraulic fracturing, CO\textsubscript{2} sequestration, ultrasonic wave treatment, and thermal recovery.\textsuperscript{16-20} Hydraulic fracturing consumes excessive amounts of water and has negative effects on ecosystems.\textsuperscript{3} Injection of CO\textsubscript{2} into coal reservoirs to replace methane has been tested as a means to enhance CH\textsubscript{4} production due to the high adsorption capacity of CO\textsubscript{2} on coal.\textsuperscript{21} However, large-scale utilization of CO\textsubscript{2} injection remains difficult because low permeability prevents CO\textsubscript{2} from replacing methane gas.

Thermal recovery methods have also been proposed for gas extraction in coal,\textsuperscript{22-24} which can provide energy for gas desorption and transport.\textsuperscript{25} Gas desorption is an endothermic process and could be promoted by thermal treatment, resulting in more gas desorbing into pore spaces.\textsuperscript{23} In addition, the physical and chemical changes of coal during thermal treatment also affect gas extraction.\textsuperscript{26} The increased temperature contributes to gas diffusion,\textsuperscript{10,27} by means of thermal deformation caused by thermal stress at low temperature and changes in the pore characteristics at high temperature.\textsuperscript{24,28} The generation and development of thermal fractures inside coal could provide channels for gas transport and further increase coal permeability.\textsuperscript{29,30} Moreover, pyrolysis leads to the generation of hydrocarbon gas from the coal.\textsuperscript{22} This additional gas source could further increase the methane yields. At present, there are limited studies focused on the effects of thermal treatment in promoting coalbed methane recovery by combining gas generation and transport conditions, which need further investigation.

In this study, the changes in coal properties during heating were analyzed, including the characterization of microstructural changes and the identification of gaseous products. The internal pressure variation of coal samples at different temperatures (280°C, 400°C, and 500°C) was investigated to analyze the gas generation and transport behaviors.

2 | EXPERIMENTAL AND METHODS

In order to better study the mechanisms of coal internal pressure changes (eg, gas desorption, and generation behaviors) and gas transport conditions changes (eg, microstructural changes) during heating process, the physical and chemical properties of coal, such as compositions, vitrinite reflectance, and pore characteristics, were measured.

2.1 | Coal rank and coal quality analyses

The coal samples used in this study were selected from Balougou #8 coalmine in the Shanxi Province of China. Figure 1 shows an example of a raw cylindrical coal core that has many natural fractures distributed on the surface.

For thermal recovery of natural gas, coal properties affect not only the gas generation and transport behaviors but also the changes of the microstructure under high-temperature heating conditions. The proximate analysis, ultimate analysis, vitrinite reflectance tests, and thermal conductivity measurement were performed by a commercial laboratory before the heating experiments. In proximate analysis, the content of moisture, ash, volatile matter, and fixed carbon was measured based on GB/T212-2008. In ultimate analysis, carbon and hydrogen were determined based on GB/T476-2008. The content of nitrogen and sulfur was determined based on GB/T19227-2008 and GB/T214-2007, respectively. The vitrinite reflectance ($R_o$) was measured based on GB/T 6948-2008. The analysis results are given in Table 1.

2.2 | TGA-FTIR analysis

Since elevated temperature has significant effects on coal properties and gas generation, a thermogravimetric analysis coupled with Fourier transform infrared spectrometry (TGA-FTIR, PerkinElmer STA6000-Frontier) analysis was conducted to study the coal pyrolysis and combustion behaviors. The coal samples were first divided into two parts and tested in dry air.
TABLE 1  Proximate analysis, ultimate analysis, and vitrinite reflectance results

| Proximate and ultimate analysis | As received |
|--------------------------------|-------------|
| Moisture                      | 2.15        |
| Ash                           | 13.94       |
| Volatile matter               | 7.17        |
| Fixed carbon                  | 76.74       |
| Carbon                        | 75.62       |
| Oxygen                        | 4.51        |
| Hydrogen                      | 2.42        |
| Nitrogen                      | 0.98        |
| \( R_o (%) \)                 | 2.67        |
| Thermal conductivity (W/(m K))| 0.148       |

and \( N_2 \) environments, respectively. The dry air was composed of 21% \( O_2 \) and 79% \( N_2 \) by volume in order to avoid the influence of water and \( CO_2 \) in air on FTIR spectra. Then, samples in these two environments were heated from 25°C to 800°C at a rate of 20°C/min. The mass loss curve and gaseous products released during the heating process were recorded. The test results of these samples were compared and analyzed.

2.3 | Scanning electron microscope (SEM) experiment

Heating alters the pore structure of coal and thus changes the coal permeability. To investigate microstructural changes in the coal, a morphology analysis of the raw and heated samples was performed using an SEM (HITACHI SU8010).

2.4 | Pore characteristics analysis

To characterize the pores in the raw and heated coal samples, low-temperature \( N_2 \) adsorption measurements were conducted using a Micromeritics TriStar II 3020 surface area analyzer. The samples were first crushed into 0.063-0.088 mm diameter powder particles, then dried, and degassed. The pore specific surface area was measured using the Brunauer-Emmett-Teller (BET) method. BET method is usually used to evaluate monolayer capacity and surface area of porous adsorbents according to the adsorption isotherm. The pore volume and pore size distribution were measured using the Barrett-Joyner-Halenda (BJH) method, in which method the relationship between pore volume and pore radius can be calculated based on the Kelvin equation. The description of BET and BJH methods was given in Appendix A.

2.5 | Thermal recovery experiment set up and procedure

In this study, three cylindrical coal samples (Figure 1) were combusted at 280°C, 400°C, and 500°C to observe the internal pressure changes. The setup of heating experiment is shown in Figure 2. To measure the transient internal pressure, a pressure sensor was placed in the center of the coal core at a depth of 3 cm. The gap between the hole and sensor was well-sealed with silicone, and the pressure sensor was calibrated to reduce the errors. The electric heater was first preheated to the desired temperatures. Then, the coal samples were heated on the heater. The thermal wave was propagated from the heater to the top of coal. The internal pressure varied due to the physical and chemical changes. To investigate the whole internal pressure variation process of coal samples, the experiments all took around 1.1 hours. During the combustion process, a forward-looking infrared (FLIR T440) camera was used to capture the surface transient temperature distribution of the coal samples.

2.6 | Gas transport inside coal

Because of the dual porosity systems, including the nanopores system and fractures system, the gas transport mechanism differs with different porosities. The Knudsen number has been widely used to determine the appropriateness of either the continuum model or molecular diffusion. The Knudsen number is defined as

\[
K_n = \frac{kT}{\sqrt{2\pi d_0^2} P}
\]

where \( k \) is the Boltzmann constant; \( T \) is the absolute temperature; \( d \) is the pore diameter; \( d_0 \) is the effective molecular diameter; and \( P \) is the gas pressure. When \( K_n \leq 0.001 \), the gas flow is governed by Darcy flow; when \( K_n \) values are in the range of 0.1-10, the flow stands within the transition flow; and at a high
Knudsen number regime \((0.001 \leq K_n \leq 0.1)\), the flow is mainly slip flow.\(^{32}\)

# RESULTS AND DISCUSSION

## 3.1 Coal properties

As shown in Table 1, the coal sample mainly consists of 13.94% low ash, 76.74% fixed carbon, 7.17% volatile matter, and 2.15% moisture. Both combustion and pyrolysis of coal can lead to the release of volatile matter and the generation of gaseous products inside coal matrix. The vitrinite reflectance \((R_o)\) was 2.67%, indicating that the coal sample is a high rank anthracite.\(^{33}\) It was found that high rank coal usually has high gas content potential.\(^1\) Organic matter, such as vitrinite, the amount of which increases with coal rank, has a high gas adsorptive capacity.\(^{10}\) It is because that the well-developed nanopores in organic matter could provide large surface area (adsorption sites) for gas adsorption.\(^{25,34}\)

Table 1 shows the moisture content in the coal sample is 2.15%. The presence of moisture in coal matrix leads to a decrease in gas adsorption capacity, sorption rate, and gas diffusivity in the coal.\(^{35,36}\) This is because the water molecules would occupy high energy sites of organic matter, competing with CO\(_2\) and CH\(_4\).\(^{37}\) The moisture is mainly released in the temperature range below 300°C.\(^{32}\) The loss of moisture can cause the shrinkage of coal by reducing the coal-water interaction.\(^{36}\) This microstructural change leads to the changes in its mechanical properties, contributing to the increase in coal permeability and gas diffusivity.\(^{35,38,39}\) The coal sample has a low thermal conductivity of 0.148 W/(m K). Therefore, it would be a challenge to improve the heat transfer in this coal seam.

## 3.2 Mass loss and gas generation

Figure 3 shows the mass loss process of the coal samples in air and N\(_2\) environments. At low temperatures (<110°C), the small amount of mass loss was because of moisture evaporation for both samples in air and N\(_2\), which accounts for approximately 2% of total sample mass. The release of moisture only led to slight microstructure changes. It is worth noting that, in air environment, the mass of coal sample slightly increased in the temperature range from 280°C to 410°C (Figure 3B). This is because of complex chemical transformations such as the slow oxidization of organic matter and the formation of oxygen-containing complexes on the coal’s surface.\(^{40}\) When the coal sample was heated up to 500°C, rapid mass loss occurred due to the intensive oxidization reactions including the oxidization of fixed carbon. The peak mass loss rate occurred at around 630°C.

Compared with coal sample in air, the sample in N\(_2\) had less mass loss. This is because organic constituents including volatile and fixed carbon were oxidized thoroughly under air environment at high temperature (>500°C), while only volatile matter was released during the pyrolysis process in N\(_2\) environment. The more thorough consumption of organic matter in air environment would result in more gas generation and significant structure changes, which can improve gas transport condition. To investigate the changes of coal properties at different temperature stages, the typical temperature points (280°C, 400°C, and 500°C) were selected to study the coal property changes.

The FTIR spectra of gaseous products from coal pyrolysis and combustion under different temperatures are presented in Figure 4. CO\(_2\) and gases containing C-H are the main products during pyrolysis and combustion processes. The peaks and infrared absorption bands in air atmosphere were mainly distributed at 900-1300 cm\(^{-1}\). These
bands are characteristic peaks of C-H functional groups associated with unsaturated aliphatic hydrocarbons. Therefore, a large number of gaseous products containing C-H functional groups were formed during combustion. CO$_2$ peaks in the range of 560-780 cm$^{-1}$ were also found from the combusted sample. Compounds containing C=C (1440-1600 cm$^{-1}$), C=O (1600-1850 cm$^{-1}$), and O-H (3500-4000 cm$^{-1}$) bonds were released at higher pyrolysis temperatures (Figure 4B-D, highlighted by the dotted line circles), and these peaks were not identified during combustion. In addition, the infrared absorbance intensity of functional groups from coal pyrolysis was generally lower than those from coal combustion because the thermal decomposition yield of organic matter in combustion is greater than in pyrolysis.

Furthermore, the infrared absorbance intensity of these functional groups increased with increasing temperature both under pyrolysis and combustion conditions, especially the C-H peaks at 900-1300 cm$^{-1}$ and CO$_2$ peaks at 2240-2400 cm$^{-1}$ (Figure 4C,D). Therefore, more gaseous products were generated due to the decomposition of organic matter at higher pyrolysis and combustion temperatures, which can result in an increase in the internal pressure of coal matrix.

### 3.3 Observation of coal matrix morphology

The matrix morphology of coal samples was observed using SEM to study the microstructural changes of coal during combustion. SEM micrographs in Figure 5A,B both showed flat surfaces, indicating that low temperatures did not have obvious effects on the morphology of coal samples. However, a rougher surface with fractures and pores was observed under 400°C (Figure 5C,D). At low heating temperatures, a small number of fractures could be generated due to the thermal deformation of heterogeneous coal. Thermal fracturing can greatly improve coal’s porosity and permeability. It is worth noting that there were numerous newly developed microfractures and pores in coal that was combusted at 500°C (Figure 5E,F). This significantly rougher matrix morphology is because of the thermal decomposition of organic matter and the generation of thermal fractures. The width of fractures generated at 400°C ranged from 0.7 μm to 0.9 μm (Figure 5C,D), while the fractures at 500°C had a larger width from 1.5 μm to 5.0 μm (Figure 5E,F). It indicates that higher temperature leads to larger thermal fractures, which can improve coal permeability and provide channels for gas transport.
3.4 | Pore characteristics of combusted coal

Pore characteristics, such as specific surface area and pore volume, were investigated to explore the effects of temperature on the porosity of coal. The specific surface areas of coal samples using the BET method are shown in Figure 6. The pore specific surface area increased from 11.08 m$^2$/g to 23.17 m$^2$/g when the temperature was increased from ambient temperature to 400°C. As the temperature increased (500°C), the pore specific area dramatically increased to 273.09 m$^2$/g. At low temperatures, the loss of moisture and volatile matter led to the enlargement and interconnection of nanopores.$^{26,30}$ Then, the significant increase in surface area at 500°C could be attributed to newly generated fractures and nanopores. The oxidation and decomposition of organic matter under high temperature can expand the nanopores and form more microfractures.$^{45}$ Moreover, the inaccessible internal pores can reconnect to the exterior open pore networks due to the generation of fractures.

To explore the effect of combustion temperature on different-sized pore structures, the volumes of partial micropore (1.7-2 nm), mesopore (2-50 nm), and macropore (50-300 nm) were investigated using BJH adsorption data (Figure 7). Overall, the total pore volume increased from $5.38 \times 10^{-3}$ cm$^3$/g to $66.91 \times 10^{-3}$ cm$^3$/g with the increase in temperature (ambient conditions to 500°C). The dramatic increase in total pore volume and mesopore volume that occurred under 500°C was

![Figure 5](image-url)  
**Figure 5** Surface morphology of coal samples observed by SEM. (A) Raw samples; (B) samples combusted at 280°C; (C, D) samples combusted at 400°C; and (E, F) samples combusted at 500°C

![Figure 6](image-url)  
**Figure 6** The specific surface areas of coal samples based on the BET method
related to the numerous pores that were either generated or became accessible from the surface in this temperature range. This observation reveals the significant effect of high combustion temperature, which could potentially improve the effective porosity and permeability of coal.

The pore size distributions of coal samples were analyzed and given in Figure 8. It is apparent that pores less than 5 nm dominate the porosity of both the raw and treated samples. The high percentage of micropores is obvious in coal samples under 400°C, which may be due to the opening or connection of many micropores. Moreover, the high-temperature thermal treatment directly changes the distribution of nanopores. As shown in Figure 8, for the samples combusted under 500°C, the proportion of pores in the range of 2-10 nm is larger. This result indicated the enlargement of micropores and mesopores, and thus the improvement of the gas transport capacity in the coal matrix. For nanopores in coal matrix, the gas flow was mainly diffusion flow.12,35 According to Equation (1), with the enlargement of nanopores, the Knudsen number decreased, and the gas transport shifted from diffusion flow to slip flow.

3.5  The surface temperature of combusted coal

The surface temperature distribution of coal sample heated to 500°C is shown in Figure 9. During heating process, the heat was transferred from the bottom to the top of coal. The temperature increased quickly during the early heating process and then gradually reached a steady state. A significant vertical temperature gradient was created in the coal sample due to the low thermal conductivity. The low thermal conductivity in gas reservoirs, such as coal and shale, has attracted widespread attention, as it is essential to the effectiveness of heat flux transfer in gas reservoirs.46 Yuan et al47 found that the greater thermal conductivity can improve the total gas production in early production. Although the improvement of thermal conductivity is of great significance to gas thermal recovery, limited ways were proposed including multiple heater wells and injecting conductive fluid.23 Therefore, the ways to promote heat transfer in coalbed formation need more exploration.

3.6  The internal pressure variation of combusted coal

As shown in Figure 10, the internal transient pressure of coal samples at different temperatures had similar trends with respect to time. Internal pressures all initially increased, reached a peak after 1000 seconds, and then gradually decreased.

At early heating stages, the rapid pressure increase was mainly due to the gas accumulation inside coal from gas desorption. Heating can promote the equilibrium of adsorption/desorption processes to favor desorption and make gas desorb from pore surface. The accumulation of gases led to a concentration gradient inside the coal. At a high temperature (500°C), the internal pressure increased more rapidly and reached a higher peak value of 1000 Pa. However, the peak pressure of coal heated to 280°C only reached approximately 200 Pa. This observation is because at low temperature, mostly desorbed gas and a small amount of pyrolysis gas accumulated. In contrast, substantial pyrolysis gases were generated within a short period of time at 500°C, with the rapid pyrolysis of coal.

It is also interesting that two significant pressure drops occurred after reaching the peak pressure value at 500°C, which may be due to the generation of thermal fractures. These fractures provide space and paths for the large-scale release of
gas from the coal core. With continued heating, the rate of organic matter pyrolysis decreased, and more accumulated gas escaped through the developed fractures. Then, the internal pressure gradually decreased and finally stabilized.

3.7 The gas generation and transport capacity of combusted coal

At low heating temperature (280°C), a slight amount of mass loss occurred, which was mainly because of gas desorption from the pore surface. The increase in mesopores and macropores volumes occurred at this temperature can promote the gas flow in coal matrix. When the temperature increased to 400°C, gases were generated inside the coal matrix due to the decomposition of volatile matter, which increased the internal pressure. The release of volatile matter resulted in the generation of numerous micropores for gas transport. With the heating temperature further increasing to 500°C, the rapid oxidization of fixed carbon occurred, and more gases (e.g., CO₂) were generated from the coal. The newly developed thermal fractures and enlarged pore sizes could potentially improve the coal permeability. In addition, high pressure gradient was existed in coal matrix at high combustion temperature, which can promote gas diffusion. Therefore, thermal treatment not only promotes gas desorption and generation, but also effectively improves the conditions for gas transport in coal.

4 CONCLUSIONS

In this study, coal samples were heated at different temperatures to investigate the gas generation behaviors and coal microstructure changes. The main conclusions reached are as follows:

1. The coal sample has 7.17% volatile matter and 76.74% fixed carbon. The high vitrinite reflectance ($R_o = 2.67\%$) indicated the high rank of coal. It has well-developed pore system inside the matrix, in which the micropores and fine mesopores were dominant nanoparticles.

2. According to LTNA measurements, the pore volume and specific surface area of different-sized nanoparticles in coal samples all increased with heating temperature. At

3. The gas generation and transport capacity of combusted coal

The internal transient pressure of combusted coal sample
280°C, the volume of mesopores and macropores was slightly increased due to the release of volatile matter. At 400°C, numerous micropores and fine mesopores were generated due to the further removal of volatile matter. When temperature increasing to 500°C, lager mesopores were generated due to the rapid oxidization of fixed carbon. In addition, thermal fractures were generated at high temperatures, providing channels for gas transport.

3. Based on TGA-FTIR tests, the main gases were unsaturated aliphatic hydrocarbons containing C-H functional groups at 280°C during pyrolysis process. With heating temperature increase to 400°C, more volatiles were decomposed and gases containing C=C and C=O functional groups were generated in pyrolysis process. More CO₂ released as heating temperature increased to 500°C. The internal pressure changed significantly at different heating conditions. At relatively low heating temperature (280°C), the increase in internal pressure was mainly due to gas desorption and mild thermal release of organic matter. At higher heating temperatures (400°C, 500°C), the rapid decomposition of organic matter resulted in the generation of a significant amount of hydrocarbon gases inside coal, which significantly increase the internal pressure.

ACKNOWLEDGMENT
The authors wish to acknowledge the financial support from The National Natural Science Foundation of China (Grant No. 51776132) and Natural Science Foundation of Jiangsu Province (Grant No. BK20181170).

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How to cite this article: Cao Y, Chen W, Yuan Y, Wang T, Sun J, Cai Y. Experimental study of coalbed methane thermal recovery. *Energy Sci Eng*. 2020;8:1857–1867. https://doi.org/10.1002/ese3.637
APPENDIX A

THE BET METHOD AND BJH METHOD

In this study, the low-temperature N\textsubscript{2} adsorption and desorption test was conducted to investigate the pore characteristics of the coal samples. The Brunauer-Emmett-Teller (BET) method was used to analyze the pore specific surface area of coal samples, and the Barrett-Joyner-Halenda (BJH) method was used to analyze the pore size distribution of the samples.

BET method is usually used to evaluate monolayer capacity and surface area of porous adsorbents according to adsorption isotherm. The BET equation is shown as\textsuperscript{48}:

\[
\frac{p}{n(p_0-p)} = \frac{1}{n_mC} + \frac{C-1}{n_mC} \cdot \frac{p}{p_0}
\]  

(A1)

where \(p\) is pressure, \(p_0\) is saturation pressure, \(C\) is BET constant, \(n\) is the specific amount of gas adsorbed at the equilibrium pressure \(p\), and \(n_m\) is the monolayer capacity.

The BET model is based on the following assumptions\textsuperscript{48,49}:

(a) Adsorption takes place on an array of fixed sites; (b) the sites are energetically identical; (c) the adsorbed molecules in one layer can act as adsorption sites for molecules in the next layer; (d) the multilayer has infinite thickness at \(p/p_0\); (e) no allowance is made for lateral interactions between adsorbed molecules; (f) the uppermost molecules in adsorbed layers are in dynamic equilibrium with the vapor; and (g) in the second and all higher layers, the adsorption energy has the same value as the liquefaction energy of the adsorbive.

The specific surface area, SSA, can be given as follows\textsuperscript{50}:

\[
SSA = \frac{n_m}{M \times m} \times N \times A
\]  

(A2)

where \(M\) is nitrogen molar mass; \(m\) is the sample mass; \(A\) is the nitrogen molecular cross-sectional area; and \(N\) is Avogadro’s number.

BJH method is widely used to calculate pore size distributions from adsorption and desorption isotherms. The BJH model is based on two assumptions that the shape of pore is cylindrical, and the adsorbed amount results from both physical adsorption on the pore walls and capillary condensation in pores.\textsuperscript{51}

The Kelvin equation is given as\textsuperscript{52}:

\[
\ln \frac{p}{p_0} = -\frac{2}{r_K} \cdot \frac{V_m \gamma}{RT}
\]  

(A3)

where \(p/p_0\) is the relative pressure on equilibrium with a meniscus; \(V_m\) is the molar volume of liquid, \(\gamma\) is the surface tension of the adsorbate in the liquid form; \(r_K\) is the radius of the meniscus formed in the pore; \(R\) is the universal gas constant; and the \(T\) is temperature.

The pore radius is equal to a sum of the Kelvin radius and the thickness of the adsorbed multilayer \(t\):\textsuperscript{52}

\[
r_p = r_K + t
\]  

(A4)

where \(r_p\) is the pore radius; \(t\) is statistical thickness of adsorbed multilayer, which can be derived from isotherms.

The volume desorbed in each step can be related to the pore volume and radius\textsuperscript{51}:

\[
\Delta V = \frac{(r_K + \Delta t)^2}{r_p^2} \cdot V_p
\]  

(A5)

Then, the change of adsorbed film thickness and the average diameter of the pore due to capillary evaporation can be calculated with the decrease of relative pressure in the desorption branch.\textsuperscript{50}