



## Research article

# Evolution of pore structure, submaceral composition and produced gases of two Chinese coals during thermal treatment



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## ABSTRACT

To better understand coal performance during underground coal gasification, coal combustion and in-situ enhancement of coalbed methane recovery by heating, the variable gases generation and pore structure of subbituminous coal and bituminous coal with thermal treatment related to organic petrology are investigated. Multiple experiments including organic petrology analysis from an optical microscope, gases analysis from thermogravimetry coupled with mass spectrometry (TG-MS), pore structure analysis using scanning electron microscopy (SEM), mercury intrusion porosimetry (MIP) and nuclear magnetic resonance (NMR) were constructed to simulate the thermal treatment of coal at elevated temperatures. The coal petrology results exhibit that the submacerals that contribute to the gases generation were significantly reduced when temperature was over 400 °C, especially for collinite, cutinite, resinite and telalginite. And there was only a slight difference in CO<sub>2</sub> content between subbituminous coal and bituminous coal, which was reduced in the bituminous coal. On the other hand, thermal decomposition of functional groups of submacerals at over 400 °C also creates more seepage pores and fractures. Pore structure analysis indicates that the adsorption pores were observed with aggregates of plate-like particles leading to slit-shaped pores below 200 °C. However, the massive seepage pores and fissures (over 35.42% in volume) were created in the high-temperature-treated coals, especially between 400 °C and 600 °C. Furthermore, the dominant seepage pores and fissures formed at high temperature (>400 °C) are due to the pyrolysis of semifusinite and collinite submacerals. These investigations may serve to characterize gases generation and pore evolution of coal during thermal treatment.

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## 1. Introduction

Currently, China has a vast amount of unrecoverable coal resources due to depth, access and other factors, which may provide an opportunity for an in situ technology to exploit potential energy (e.g., coalbed methane) from coal. Coalbed methane (CBM) recovery, which produces <1% of the total coal seam's energy, is feasible in this environment [1]. Although CBM has less contribution to energy, it is essential in the context of reducing greenhouse gas emissions, contributing to the development of the gas industry and maintaining the safety and economic advantages of mining [2]. Furthermore, enhancing CBM recovery from underground coal with in situ thermal treatment may provide the possibility for more massive gases recovery [3,4]. For the past decade, thermal treatment technology has observed promising acceptance as an effective tool for improving reservoir properties and increasing gases content in coal seams. Existing researches [4,5] have been conducted to simulate coal thermal treatment with the goal of improving reservoir properties and increasing gases content, which differs significantly from underground coal gasification (UCG) [6–8]; it indirectly heats the coal to

pyrolysis temperatures rather than injecting air/oxygen mixtures to directly gasify the coal. This process produces CH<sub>4</sub>, H<sub>2</sub> and CO<sub>2</sub> due to the thermochemical decomposition reaction and gasification of organics without oxygen as Kerogen pyrolysis [9]. Therefore, an improved capability for suspending operations and reducing the risk of subsidence for in situ coal thermal treatment offers substantial merits, compared with UCG. By heating coal in situ, the coal is decomposed from long chain organics to synthetic gases, liquids, and char. This process has the potential to produce large portions of the available fuels from the coal in situ. Although in situ coal thermal treatment requires extra energy [4, 10,11], these additional energy resources may prove that this process is worthwhile. Different property transformations occur during coal pyrolysis: (1) the first stage (25–300 °C) is the dry gas stage. In this stage, moisture and adsorbed gases are desorbed until ~200 °C. The coal structure keeps steady. When the temperature is increased from 200 °C to 300 °C, thermal decomposition occurs for the low-rank coals; (2) the second stage (300–550 °C) is the coal pyrolysis stage, which involves forming gases and tars. Before 400 °C, the coal turned softer and congealed into a viscous mass. In the range of 400 to 550 °C, gases increase and coal tar precipitates. The residual coal mass increasingly stiffens and solidifies just as char. Gas produced during 450–550 °C includes light aromatic hydrocarbon and a long chain fatty substance.

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### Nomenclature

TG-MS	thermogravimetry coupled with mass spectrometry
SEM	scanning electron microscopy
MIP	mercury intrusion porosimetry
NMR	nuclear magnetic resonance
C	collinite
Cu	cutinite
R	resinite
Ta	telauginite
MiS	microsporinite
MaS	macrosporinite
LD	liptodetrinite
CBM	coalbed methane
UCG	underground coal gasification
$T_E$	time interval of echoes, ms
$T_W$	a waiting time, ms
$T_2$	transverse relaxation time, ms

The petrophysical properties of coals (including pore fractures, adsorption and permeability, etc.) significantly change in this stage [5,12,14]. The condensation reaction is not common (3) in the third stage (550–1000 °C) or the carbocoal stage [15], in which polycondensation reactions occur. The aromatic layers often become even more ordered, realistic density increases and a breakthrough degree of aromatization exists. The gas generation is due to thermal decomposition of the functional groups. Generated gases will flow/burst out of the mass, which produce more gas-burst pores. The gases/liquids generation and petrophysical variation are the keys for energy extraction. Although existing research has investigated the process of gases generation and petrophysical variation during coal pyrolysis [4,7,13,14], the pore changes and gases generation related to coal submacerals are still unclear.

In response to the findings above, the objective of the research presented here is to investigate the pores and gases change of the coals (a subbituminous coal and a low volatile bituminous coal) with thermal treatment related to coal submacerals. The NMR and modified MIP could be the promising methods to investigate the pore structure. Pore structure is the key to CBM preservation and production. Therefore we put a lot of work into understanding the pore development. Recently, a few researchers [11,13,14] have conducted the experiments of heating on coal reservoir from the perspective of enhancing CBM production. In this research, firstly, thermogravimetry coupled with mass spectrometry (TG-MS) was employed to investigate the decomposed products from these two rank coals during pyrolysis (25–1200 °C) as related to coal petrology. Although only two Chinese coals (a subbituminous coal and a low volatile bituminous coal) were analyzed, the detailed and systematic investigations would provide a better way to

**Table 2**

Proximate analysis of the subbituminous coal and bituminous coal at elevated temperatures.

Temperatures (°C)	Proximate analysis (wt.%)	Sub. coal	Bit. coal
200	$M_{ad}$	0.93	1.28
	$A_{ad}$	21.96	14.49
	$V_{ad}$	15.01	7.99
400	$C_{ad}$	62.1	76.24
	$M_{ad}$	1.09	0.59
	$A_{ad}$	19.85	26.11
600	$V_{ad}$	10.97	14.74
	$C_{ad}$	68.09	58.56
	$M_{ad}$	0.72	0.71
	$A_{ad}$	23.45	21.4
	$V_{ad}$	15.69	12.39
	$C_{ad}$	60.14	65.5

Note: Sub. Coal-Subbituminous coal; Bit. Coal-Low volatile bituminous coal;  $M_{ad}$ -Moisture at air dry basis;  $A_{ad}$ -Ash at air dry basis;  $V_{ad}$ -Volatile at air dry basis;  $C_{ad}$ -Carbon at air dry basis.

understand the variable gases generation, pores development and submacerals evolution of two Chinese coals with thermal treatment. Therefore, it is meaningful to know well the effects of submacerals on pore development and valuable gases generation. Secondly, the pathways including pores (e.g., pore shape, pore surface, pore volume/size distribution) and fractures features of coals during pyrolysis were studied with raising temperatures up to 600 °C. An investigation into petrophysics of the two thermal treated coals by multiple methods (SEM, MIP,  $N_2$  adsorption, NMR etc.) were systematically and detailed conducted. Finally, the effects of the parameters (coal rank, particle size, pyrolysis temperature, and coal submacerals) from the adopted experiments on pore structure development of coals with varying ranks were discussed.

## 2. Experiment and methodology

### 2.1. Samples and coal basic analyses

A subbituminous coal ( $R_{o,m}$  0.57%) from the southern Junggar Basin and a low volatile bituminous coal ( $R_{o,m}$  1.68%) from the eastern Ordos Basin were gathered from active mines (Table 1). Blocks of these two samples, which were approximately 3 kg each, were carefully collected and wrapped and then delivered immediately to the laboratory. The standard coal analysis, optical microscope analysis and TG-MS analysis were executed for both of these two samples. SEM, MIP,  $N_2$  adsorption and NMR analyses were conducted for the low volatile bituminous coal to investigate the pore changes by thermal treatment targeted at 25 °C, 200 °C, 400 °C and 600 °C. The maximum vitrinite reflectance ( $R_{o,m}$ ), proximate analysis and macerals analysis for these two thermal treated coals were measured in a previous study [16], and these figures were provided in Table 2.

**Table 1**

Variation of coal composition at elevated temperatures.

Coal rank	Sampling location	$R_{o,max}$ (%)	Coal mass composition (vol.%)					Proximate analysis (wt.%)			
			Minerals		Exinite	Vitrinite	Inertinite	$M_{ad}$	$A_{ad}$	$V_{ad}$	$C_{ad}$
Pyrite	Others										
Sub. coal	SCB	0.57	0.6	7.8	8.9	83.1	8.5	0.7	6.6	21.1	71.6
Bit. coal	EOB	1.68	0.5	13.4	0.0	93.1	6.9	1.0	7.4	14.1	77.5

Note: Sub. Coal-Subbituminous coal; Bit. Coal-Low volatile bituminous coal; SJB-Southern Junggar Basin; EOB-Eastern Ordos Basin;  $M_{ad}$ -Moisture at air dry basis;  $A_{ad}$ -Ash at air dry basis;  $V_{ad}$ -Volatile at air dry basis;  $C_{ad}$ -Carbon at air dry basis.

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