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Effects of Organic Maceral on Biogenic Coalbed Gas Generation from Bituminous Coal

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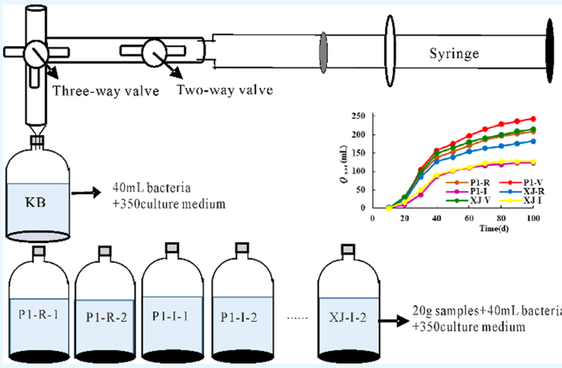
ABSTRACT: Clarifying the effect of organic maceral on biogenic coalbed gas generation is important to understand the mechanism of biogenic coalbed gas generation and to develop bioengineering of coalbed gas. Bituminous coals in the Huainan mining area of China were selected as the research object, and the organic macerals were enriched through manual separation and floatation−sedimentation experiments first. Then, the simulated biogas generation experiments were carried out by using raw coal, single vitrinite, and inertinite, respectively. The results showed that all the bituminous coal, vitrinite, and inertinite could be biodegraded to generate biogas. The gas production yield of vitrinite was11.5 mL/g, which was more than that of raw coal (9.8 mL/g) and inertinite (6.26 mL/g). The production processes showed the stage characteristics of rapid increase and continuous decrease, but the gas production peak of inertinite lagged behind that of raw coal and vitrinite. Vitrinite content was positively correlated with total gas production, while inertinite could inhibit biogas production. CH_4 composition in simulated biogas from vitrinite was the most, and that from inertinite was the least, while there was a positive correlation between vitrinite content and CH_4 composition. The above evidence showed that vitrinite in bituminous coal is more easily biodegradable. There were significant positive correlations between chloroform bitumen "A", H, and H/C to total gas production, and they can be used as important indicators to evaluate the output of coalbed biogas.



1. INTRODUCTION

As a new kind of unconventional natural gas, biogenic coalbed gas has been found in many coal-bearing basins all over the world.[1−4] The exploration and development of biogenic coalbed gas has achieved great success in the Powder River basin, U.S.A., and Surat basin, Australia.[5−7] Under suitable conditions, fluidization (gasification) mining of coal resources can be realized by injecting microorganisms into coal seams, which had been studied and explored internationally for many years, and field tests have been carried out.[8−11] Understanding the process and mechanism of biogenic coalbed gas generation is the basis for evaluating the potential of coalbed gas resources, and it is also the core foundation for the technology of in situ fluidized mining of underground coal resources.

The process of biogenic coalbed gas generation is the decomposition, polymerization, and transformation of available organic matter in coal through a series of functional microorganisms to generate gas. From the perspective of chemistry, coalbed biogas generation is a process in which complex organic compounds in coal are decomposed by microorganisms into organic acids or alcohols, esters, carbohydrates, hydrogen, and carbon dioxide. This process is mainly completed by anaerobic and facultative anaerobic hydrolytic bacteria or fermentative bacteria. Then the above organic molecules are decomposed into organic acids, hydrogen, and CO_2 by hydrogen and acid producing bacteria. Finally, strictly anaerobic methanogens use acetic acid or CO_2 and H_2 to form CH_4. The former is called acetic acid fermentation pathway and the latter is called CO_2 reduction pathway. In recent years, it has been founded that a class of methanogens can directly use benzoxy in coal to generate methane, which is the third methanogenic pathway at present.[12]

The effect factors of biogenic coalbed gas include temperature, salinity, pH, trace elements, and so on, but the organic matter in coal is the main controlling factor. Some researchers examined the possible metabolic process of aromatic and aliphatic components under anaerobic conditions,[11,13] and some researchers speculated the metabolic mechanism of microbial degradation of coal by detecting the concentration changes of alkanes, aromatic compounds, and heteratomic compounds.[14,15] These results indicated that organic com-

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pounds with small molecular weight and good water solubility were effective substrates for microorganisms.

Coal petrology defines organic materials in coal as organic macerals, including vitrinite, inertinite, and exinite. Different macerals have different chemical compositions, so their resistances to microbial degradation are different. In general, vitrinite is formed by gelation of coal-forming plants, which is relatively weak in resistance to microbial degradation and easily generates biogas. The aromatic ring in inertinite is highly concentrated and resistant to microbial degradation. Exinite is composed of lipids with a stable chemical structure and is not easy to biodegrade.[16] However, the cork body has relatively weak resistance to microbial decomposition due to its high lignin and fiber composition.

Most of the research on the mechanism of biogenic coalbed gas generation focused on coal chemistry, and few researchers payed attention to coal petrology. To study the effect of organic macerals on biogenic coalbed gas generation from the perspective of coal petrology is an important part of systematically understanding the mechanism of biogenic coalbed gas generation. Due to the weak coalification, the coal structure of middle and low grade coals retains more smaller molecule side branches and chains and has large porosity, so it has the advantage of generating biogas.[17,18] In this paper, bituminous coal and its single maceral were used as substrates to carry out simulation experiments of biogas generation. By analyzing the gas production, gas generation rate, and biogas composition, the effect of maceral on biogas generation was studied. The results provide theoretical support for understanding the process and mechanism of coal biogas generation.

2. MATERIALS AND METHODS

2.1. Sample Collection and Basic Properties. Two bituminous coal samples were collected from Panyi Coal Mine and Xinji Coal Mine in the Huainan area of China, and the sample numbers were P1 and XJ in sequence. The samples collected under the mine were immediately wrapped in a thin film and sealed in bags. The samples were crushed uniformly, and the pure coal samples were selected manually. Each coal sample was divided into two parts, one for basic property testing and the other for biogas generation simulations.

Maximum vitrinite reflectance ($R_{o,max}$) was achieved using a Zeiss imaging Mim microscope (Carl Zeiss Company, Germany). Proximate analysis was performed in accordance with ASTM standards D3173-11, D3175-11, and D3174-11.

The coal samples were in the stage of gas-fat coal and belonged to high volatile coal (Table 1). The volatiles mainly come from small molecular compounds after the break of fatty side chains and oxygen-containing functional groups. The higher volatile content of XJ coal indicated that the coal sample had greater biodegradability.

Table 1. Basic Properties of Coal Samples[a]

coal sample source	serial number	$R_{o,max}$ (%)	proximate analysis (%)			
			M_{ad}	A_d	V_{daf}	FC_d
Panyi Coal Mining	P1	1.02	1.24	12.94	38.07	53.92
Xieji Coal Mining	XJ	0.96	1.88	12.19	42.28	50.68

[a] $R_{o,max}$ is the maximum vitrinite reflectance; M_{ad} is moisture on an air-dry basis; A_d is ash on a dry basis; V_{daf} is volatile matter on a dry and ash-free basis; and FC_d is fixed carbon content on a dry basis.

Coal samples were released naturally first, and specific methods were shown in the literature.[19] To prevent coal samples from being contaminated by environmental bacteria, the flask, the breaker, and the glass catheter were all sterilized in an autoclave before the natural release.

2.2. Maceral Enrichment and Separation. First, vitrinite and inertinite visible in coal samples were separated by hand, and then the samples were crushed to 60 mesh. Using inorganic zinc chloride as the specific gravity liquid, maceral separation was carried out according to the coal float and sediment test method (GB/T478-2008). In particular, the crushed samples were added into 1.35 g/cm^3 zinc chloride solution and centrifuged at 6000 rpm for 20 min to obtain vitrinite enrichment, using a high-speed centrifuge (TG16-WS, China Xiangyi Instrument Co. Ltd.). Inertinite enrichment was obtained by adding the sediment to 1.45 g/cm^3 zinc chloride solution and centrifugation at 6000 rpm for 20 min.

2.3. Properties of Raw Coal and Single Maceral. *2.3.1. Maceral Statistical Analysis.* The raw coal and single macerals were rinsed and filtered with distilled water several times until no white silver chloride precipitated when silver nitrate was added. Then according to the preparation method of the coal and rock analysis sample (GB/T16773-2008), the polished coal section was prepared. Using a Zeiss SIP No. MC02139 polarizing microscope (Carl Zeiss Company, Germany), each sample was observed under a 50× microscope for the statistical analysis of the maceral composition.

2.3.2. Elemental Analysis and Organic Geochemical Characteristics Analysis. Elemental analysis was performed in accordance with ASTM standard D5373-08. The total organic carbon (TOC) content was tested with a LECO SD-230, C and S analyzer (U.S.A.). The test conditions were as follows: temperature of 23 °C, 40% relative humidity, and sample crushing to 200 mesh. Chloroform bitumen "A" was measured by Soxhlet extraction. The extraction agent was trichloromethane, and the extraction temperature was set at 80 °C for 72 h.

2.4. Biogas Generation Simulated Experiment. *2.4.1. Microbial Resource and Culture Medium.* The bacterial solution was enriched and purified from mine water from the Dananhu Coal Mine of the Hami coalfield in China, which was supplied by the Chinese Academy of Science.

The composition and concentrations of the trace element solution were 6.0 g/L $FeCl_2 \cdot 4H_2O$, 5.0 g/L $MgCl_2 \cdot 6H_2O$, 5.0 g/L $MnCl_2 \cdot 4H_2O$, 0.64 g/L $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$, 0.45 g/L $Ni_2SO_4 \cdot 6H_2O$, 0.15 g/L $CoCl_2 \cdot 6H_2O$, 0.1 g/L $ZnSO_4 \cdot 7H_2O$, 0.5 g/L $CuSO_4 \cdot 5H_2O$, 0.1 g/L H_3BO_3, and 0.001g/L resazurin.[19]

The composition and concentrations of the culture medium were 4.27 g/L $MgCl_2 \cdot 6H_2O$, 3.32 g/L $CH_3COONa_3 \cdot H_2O$, 0.55 g/L $K_2HPO_4 \cdot 6H_2O$, 2.0 g/L NaCl, 1.0 g/L NH_4Cl, 0.4 g/L KH_2PO_4, 0.2 g/L KCl, 1.0 g/L yeast extract, 0.5 g/L cyusteine, and 0.001 g/L resazurin.[19]

2.4.2. Simulated Experiment. The 500 mL brine bottles with butyl rubber stoppers and wax seals were selected as the simulation experiment device to ensure good airtightness. The experiment device was composed of a culture bottle, a needle, a three-way valve, a two-way valve, and a 10 mL screw syringe. The gas was collected by downward discharge of saturated salt water. The culture flask was filled with 20 g of raw coal or a single maceral sample, 40 mL of bacterial liquid, and 350 mL

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Table 2. Maceral Composition, Organic Matter, and Element Test Results of Coal and Single Maceral[a]

sample	maceral content (%)			TOC (%)	chloroform asphalt "A" (%)	element content and ratio				
	V	I	E			C_{daf} (%)	H_{daf} (%)	O_{daf} (%)	H/C	O/C
P1-R	70.8	12.2	11.6	74.38	2.04	83.54	5.37	9.00	0.77	0.08
P1-V	91.35	6.60	2.0	76.48	2.08	76.98	5.83	10.88	0.91	0.11
P1-I	46.75	45.32	7.90	73.36	1.64	77.87	5.07	10.39	0.78	0.10
XJ-R	63.6	14.4	18.6	79.96	2.20	78.92	4.93	14.30	0.75	0.14
XJ-V	89.12	7.33	3.55	80.42	2.95	76.01	5.68	15.01	0.90	0.15
XJ-I	47.43	42.67	10.0	79.31	1.76	78.24	4.07	14.39	0.62	0.14

[a]Note: R, V, I, and E mean raw coal, vitrinite, inertinite, and exinite in coal, respectively.

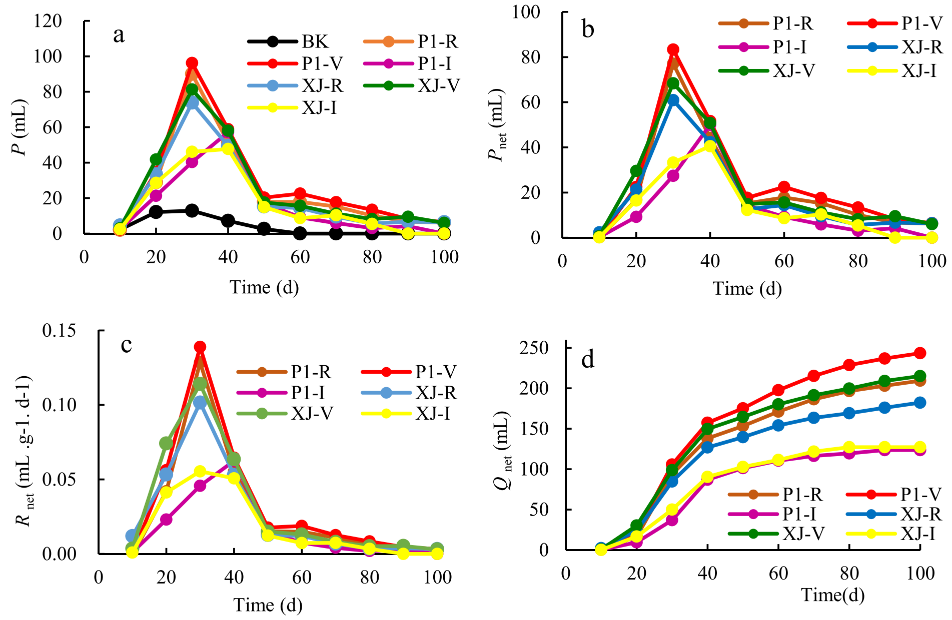

Figure 1. Change in the simulated biogas generation. (a) Change in biogas generation of BK and experimental groups; (b) change in net productions of experiment groups; (c) change in net biogas production yield; and (d) change in total net biogas production.

of medium. The whole process of inoculation was completed in an SYQX-II anaerobic incubator, and the flasks after inoculations were put in an HZQ-F160 constant temperature oscillation incubator. The temperature was set at 37 °C, and the speed was 50 rpm. The continuous culture lasted for 100 days. The blank experimental group (BK group) with no coal or maceral samples was set to compare with the experimental groups. Two parallel experiments were conducted for the experimental groups.

2.5. Gas Composition Analysis. Headspace gas chromatography (GC-4000A) was used to determine the gas composition. The gasification chamber temperature was 120 °C, the column temperature was 90 °C, and the detector temperature was 140 °C. A series flame ionization detector (FID) was used for organic gas compounds, and a thermal conductivity detector (TCD) was used for inorganic gas compounds. N_2 was the carrier gas, and the flow rate was 30 mL/min.

3. RESULTS AND DISCUSSION

3.1. Separation Effects and Geochemical Characteristics of Maceral. The average content of exinite was 15.6%, and it rarely existed as a monomer. Thus, only vitrinite and inertinite were obtained in this study. The sample numbers and test results are shown in Table 2.

The contents of vitrinite in P1-V and XJ-V increased by 29.02% and 40.12%, respectively, compared to raw coal. The contents of inertinite in P1-I and XJ-I increased by 3.71 and 2.69 times, respectively, compared to raw coal. After the separation, the vitrinite content increased in proportion, while the inertinite content increased greatly. The inertinite enrichment effect was much better than that of vitrinite.

TOC contents of single vitrinite and inertinite changed little compared with raw coal, but they showed the characteristics of vitrinite > raw coal > inertinite. Compared with raw coal, the content of chloroform bitumen "A" in vitrinite changed little, but in inertinite it decreased obviously. In P1-I and XJ-I, the contents were 19.6% and 20% lower than that in raw coal, respectively. Compared to raw coal, H contents in P1-V and XJ-V increased by 8.56% and 15.2%, respectively. C contents showed the characteristics of raw coal > inertinite > vitrinite, while O contents showed vitrinite > inertinite > raw coal. The H/C (average of 0.9%) and O/C (average of 0.11%) in vitrinite were the maximum, indicating that vitrinite concentrated more H and O but less C. The above characteristics indicated that the vitrinite contained more functional groups containing H and O, so its soluble organic matter content was more, while inertinite had a higher degree of aromatization and poor solubility. These results were consistent with the results of previous studies.[20,21]

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3.2. Simulation Results of the Biogas Generation Experiment. The gas in the BK group indicated that the nutrient solution in the culture medium could be used to produce biogas, which had been reported already.[19,22] Therefore, it is necessary to deduct the biogas production of the nutrient solution as shown in Formula 1. Previous studies showed that N_2 did not belong to biogas[19,23] but existed because it was used to replace air during the inoculation. We removed N_2 from the gas composition by Formula 2.

$$P_{net} = P(E) - P(BK) \quad (1)$$

$$P = P_t \times (100 - C(N_2))/100 \quad (2)$$

Notes: P_t is the origin gas production, mL; P is the net production after N_2 is deducted, mL; C (N_2) is the content of N_2, %; $P(E)$ is the gas production of experiment groups, mL; $P(BK)$ is the gas production of BK groups, mL; and P_{net} is the net production of the experiment groups after deducting the gas production of the BK groups, mL.

According to P_{net}, Formula 3 was used to calculate the gas production rate, R_{net}. The total gas production, Q_{net}, was obtained by adding stage gas production through Formula 4.

$$R_{net} = P_{net}/w/\Delta t \quad (3)$$

$$Q_{net} = \sum P_{net} \quad (4)$$

Notes: R_{net} is the stage net biogas production yield, mL/g/d; w is the weight of the coal samples, g; Δt is the time of gas collection, d; and Q_{net} is the total net biogas production, mL.

3.3. Effect of Maceral on Biogas Production. The simulated biogas productions are shown in Figure 1. The BK group had biogas generated before 50 d, and after that biogas was no longer produced, showing that nutrients in the medium had been used completely then. The gas productions of experimental groups were higher than that of the BK group throughout the whole experimental process, and gas was still collected after 50 d. This indicated that methanogens can effectively use raw coal and single maceral samples to generate biogas.

After deducting the BK gas production, the total biogas productions of P1-R and XJ-R were 209.32 and 182.23 mL, respectively. The total gas productions of P1-V and XJ-V were 243.36 and 215.08 mL, respectively. The total gas productions of P1-I and XJ-I were 123.57 and 127.12 mL, respectively. The average gas production rates of raw coal, vitrinite, and inertimite were 9.8 mL/g, 11.5 mL/g, and 6.26 mL/g, respectively. Vitrinite had the maximum gas production, followed by raw coal and inertinite (Figure 1d).

The gas productions and gas production rates were basically the same, showing the staged characteristics of rapid increase and then continuous decrease. The gas production rates of raw coal and vitrinite reached the peaks at 30 d, with averages of 0.12 mL/g/d and 0.13 mL/g/d, respectively. Inertinite groups reached the peak value (average of 0.06 mL/g/d) at 40 d and then experienced a slow declination and rapid decline (Figure 1a−c). These results showed that vitrinite was biodegraded earlier. Inertinite could also be biodegraded, but it lagged behind vitrinite.

The vitrinite in coal concentrates more aliphatic hydrocarbons.[24] Chloroform bitumen “A” and H/C in vitrinite are both more than inertinite, indicating the higher content of soluble organic matter. Studies have shown that soluble organic matter, such as aliphatic hydrocarbon, can be effectively utilized by methanogens and can produce more biogas.[18] Once the soluble components were decomposed, at about 40−50 d as shown in Figure 1, the gas productions decreased rapidly. However, gas was still generated at 60−80 days in the experimental groups, indicating that after soluble alkanes were degraded, polycyclic hydrocarbons in coal could also be effectively degraded to produce biogas.

Vitrinite content was positively correlated with the total gas production, while inertinite content was negatively correlated with the total gas production (Figure 2). This shows that vitrinite was an effective substrate for biogas production, while inertinite inhibited biogas production, which was consistent with previous research results.[25]

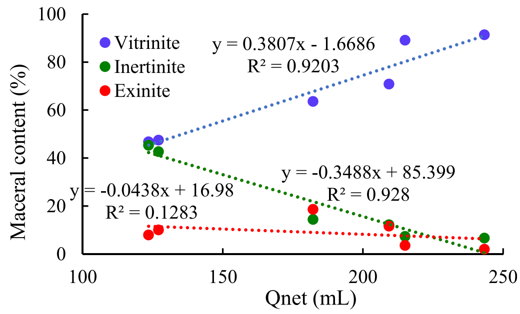

Figure 2. Effects of maceral content on total gas production.

The above analysis shows that the content of vitrinite influences greatly the biogas production potential of bituminous coal. Inertinite has fewer C and H contents and more aromatic structures, while vitrinite has more fatty C content and the aryl carbon content of vitrinite is lower than that of inertinite,[26,27] which is easier to biodegrade than an aromatic ring. These results may explain how a high vitrinite content can promote the generation of biogas.

3.4. Influence of Macerals on Biogas Composition. Changes of the CH_4 and CO_2 contents in simulated biogas are shown in Figure 3a,b. The average CO_2 content of two raw coal groups was 57.97% at 10 d, then decreased to 19.67% at 20 d, and remained at about 30% after 30 d. The average CO_2 contents of vitrinite groups and inertinite groups were 65.28% and 66.14% at 10 d and decreased to 22.12% and 47.11% at 20 d, respectively. Because CH_4 and CO_2 were the main components of the simulated biogas, the change trends of CH_4 content were basically opposite that of CO_2 content, showing a trend of rapid increasing (10−20 d)−decreasing (20−30 d) and then remaining steady (30−100 d). For example, the average CH_4 content from raw coal groups rapidly increased from 42.03% at 10 d to 80.33% at 20 d and decreased to 60.09% at 30 d. The variations of CH_4 content of the vitrinite and inertinite groups were similar to those of the raw coal groups.

The total CH_4 amount had obvious characteristics of vitrinite > raw coal > inertinite (Figure 3c). Raw coal and vitrinite generated similar amounts of CO_2, while inertinite produced the least. The previous studies showed that the changes and thresholds of CO_2 and CH_4 content could indicate the pathway of biomethane formation.[19] The result in this study indicated that some CH_4 might come from the pathway of CO_2 reduction. In the initial stage (before 30 d), a large amount of CO_2 was generated, and since the 40 d, the CO_2 contents were lower than 38% in all experimental groups.

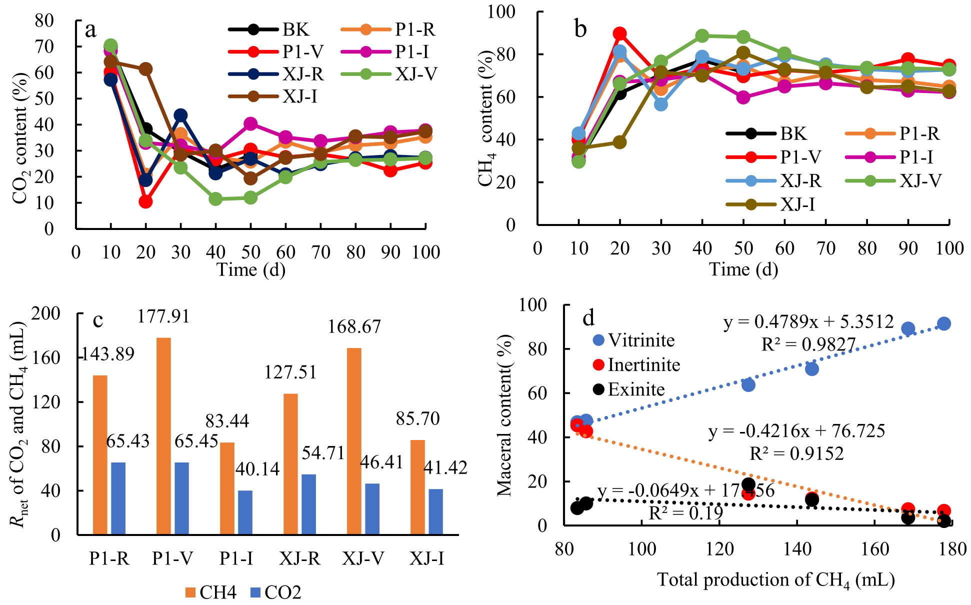

Figure 3. Change of the simulated biogas composition. (a) Change of CO_2 contents of BK and experimental groups; (b) change of CH_4 contents of BK and experimental groups; (c) total R_{net} of CO_2 and CH_4 in the experiment; and (d) the correlations between maceral content and total production of CH_4.

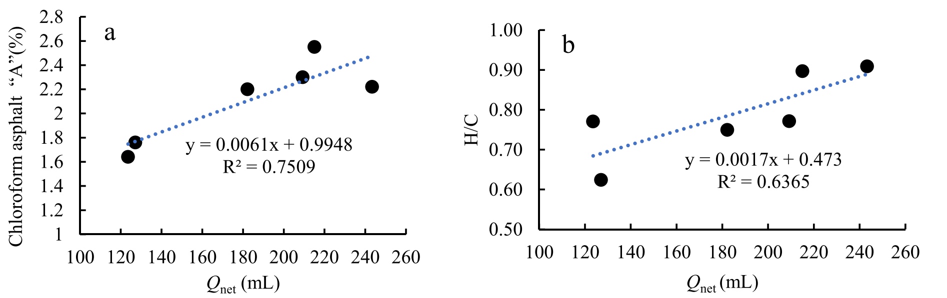

Figure 4. Correlations between chloroform asphalt "A" and H/C and total gas production. (a) The relationship between chloroform asphalt "A" and total biogas production and (b) the relationship between ratio of H/C and total biogas production.

Therefore, it was inferred that the CO_2 reduction pathway was involved in CH_4 generation at the later stage of the experiment. However, the CO_2 contents were relatively low throughout the experiment (lower than 32.96% after 20 d), which was not enough to provide all the carbon sources for biological CH_4. Therefore, the simulated CH_4 should be a mixed genetic type of CO_2 reduction and acetic acid fermentation.

The effect of maceral on CH_4 production was shown in Figure 3d. There was an apparent positive correlation between vitrinite content and CH_4 production, while CH_4 content decreased linearly with the increase of inertinite content. The relationship between exinite and CH_4 production was discrete. There was no obvious linear relationship between each maceral and the total CO_2 production.

3.5. Relationship between Maceral Properties and Biogas Generation. The Q_{net} value was selected as the evaluation index to analyze the effects of raw coal and single maceral properties on biogas. It was found that the content of chloroform bitumen "A", H, and H/C showed significant positive correlations to Q_{net} (Figure 4). High H/C could provide an abundant gas-producing substrate and generate more biogas and CH_4 concentration.[25] Chloroform bitumen "A" represents soluble organic matter in coal and is the parent precursor of biogas generation in coal.[28] Consequently, the chloroform asphalt "A" and H/C can be used as important indexes to evaluate the generation potential of biogenic coalbed gas.

There was no obvious correlation between TOC and Q_{net} (Figure 4 c), as well as contents of C and O. TOC is a traditional evaluation index of source rocks, but coal is different from traditional source rocks, because almost all C in coal is organic carbon, whether soluble or not. Therefore, the relationships among the TOC, C, and Q_{net} were not obvious. The oxygen functional groups of coal macerals are mainly carbonyl, carboxyl, aromatic, and aliphatic oxygen functional groups.[16,24] The latest research results showed that aromatic compounds in lower rank coals contain the most oxygen functional groups, while aliphatic compounds contain the fewest oxygen functional groups.[24] The structure of aromatic compounds is relatively stable, which is not conducive to biodegradation, so the increase of O content cannot effectively promote biogas generation.

4. CONCLUSIONS

(1) Bituminous coal and its single vitrinite and inertinite could all be utilized by methanogenic bacteria to generate biogas. The biogas production and composition showed the common variation rules, indicating that both vitrinite and inertinite in

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bituminous maceral contribute to biogenic coalbed gas generation. The gas production yield of vitrinite was 11.5 mL/g, which was more than those of raw coal (9.8 mL/g) and inertinite (6.26 mL/g).

(2) The total gas production and CH_4 concentration of vitrinite were obviously more than those of raw coal and inertinite, and the gas production peak of inertinite lagged behind those of raw coal and vitrinite, indicating that vitrinite contains more functional groups that can be easily degraded into gas and is a better biogenic gas substrate. Therefore, more attention should be paid to bituminous coal with high vitrinite content in the evaluation of biogenic coalbed gas generation potential.

(3) Chloroform bitumen "A", H, and H/C were significantly positively correlated with total gas production. In particular, the chloroform bitumen "A" and H/C can be used as an important index to evaluate biogenic bituminous coalbed gas production.

(4) Bituminous coal with high vitrinite content should be given attention during biogenic coalbed gas exploration. In addition, a special coal species, commonly known as "bark coal", is widely developed in the Late Permian Longtan coal measures in South China. Its H/C ratio is greater than 0.9,[29] and its biogenic coalbed gas generation potential deserves attention.

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Notes

The authors declare no competing financial interest.

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