INTRODUCTION

Since new discoveries for conventional oil and gas reservoirs have been steadily declining over the past few decades, rising energy demand has resulted in increasing attention on exploiting unconventional resources and developing these resources to their maximum potential. Oil shale, an impermeable sedimentary rock with complicated composition throughout its mineral matrix, is of interest to researchers for exploiting it without extra pollution. To develop oil shale, kerogen must be heated at the appropriate temperature for days to months by in situ heating methods.

The microstructure of oil shale plays a vital role in the seepage and production of shale oil and gas and can be modified by microwave irradiation. In this study, the experimental work aims to visualize and analyze the microstructure of oil shale under microwave heating with different heating parameters by traditional two-dimensional (optical microscope and scanning electron microscope) and advanced three-dimensional (micro-CT) methods. Volumetric reconstructions of oil shale before and after microwave heating were completed to directly visualize the pore structure and fracture network of the samples. The similarities and differences between microwave heating and conventional heating were also investigated. Finally, based on the three-dimensional CT data, the porosity and the degree of anisotropy of the samples were calculated. Traditional two-dimensional imaging methods showed that microwave heating led to more pores and fractures due to the differential thermal stress caused by rapid heating, which differed from conventional heating. Porosity calculations based on the CT image data indicated that a long heating time and a high output power could lead to a large porosity value. Oil shale obtained by drilling horizontally into the bedding plane showed the largest porosity and lowest heterogeneity of pore distribution after heating and had a different fracture morphology. The results from this study are important for the exploitation of oil shale using microwave heating, and the application of these analytical techniques is useful for the evaluation of the flow behavior of transformation products.

KEYWORDS
efficiency enhancement, micro-CT, microstructure, microwave heating, oil shale
situ heating methods, the microwave heating method has the advantage of heating oil shale layers with high efficiency. Many researchers have already proposed and investigated microwave heating to overcome the difficulties in reservoir development. Wang et al. used numerical simulation analysis to show that microwave heating could efficiently remove water and create microfractures in tight gas sand. Liu et al. performed numerical simulations to prove the possibility of microwave heating for enhanced shale gas recovery. Microwave heating has also been analyzed experimentally for developing oil shale. It has been shown that microwave heating could rapidly heat oil shale and obtain shale oil with high efficiency. Although oil shale has a low dielectric constant, enhancing its microwave absorption could be accomplished by adding materials such as carbon and iron oxide nanoparticles.

The transformation of oil shale during microwave treatment involves complex physical and chemical reactions. In recent years, much progress has been made comparing the similarities and differences between microwave heating and conventional heating with different fundamental parameters. These parameters include heating rate, temperature distribution, shale oil production and quality, gas volume and composition. The results have shown that microwave heating not only accelerates the heating rate but also improves the production and quality of shale oil compared with conventional heating. Microwave heating, in contrast to heat convection and heat conduction, induces the fragmentation of large molecules and promotes the decomposition of chemical bonds.

In addition to the above investigations, it is also important to comprehensively explore the changes in microstructure of oil shale including the development of the pore network and the creation of fractures. The microstructure of oil shale directly influences heat transfer and mass transport, particularly in the flow behavior of shale oil and gas after in situ conversion. Many experimental studies have used nitrogen adsorption-desorption isotherms, mercury intrusion porosimetry (MIP), and scanning electron microscope (SEM) to evaluate the pore structure of oil shale after conventional heating. However, there are few investigations about the effects of microwave irradiation on the visualization of pore networks, the creation of fractures and the quantification of the porosity of oil shale, not to mention the influence of microwave heating parameters. Wang et al. calculated the specific surface area and specific pore volume of oil shale particles by the low-temperature adsorption of nitrogen, but this method did not involve the direct observation of individual pores or address the connectivity of the pore space. Because pores and fractures in oil shale are the main channels through which the shale oil and gas generated from the thermal transformation of oil shale can seep, analyzing the microstructure of oil shale after microwave irradiation is of great importance. Moreover, the porous characteristics of oil shale after heating have a great influence on both the emission of pollutants and their recycling selection.

Micro-CT is a nondestructive tool to investigate the internal microstructure of objects and provide quantitative data about the morphology of three-dimensional (3-D) samples. There are several investigations of the pore structure of oil shale under conventional heating using the micro-CT tool. Zhao et al. analyzed the changes in internal structure in a high-temperature electric furnace by performing CT scanning of oil shale. Micro-CT was also applied by Tiwari et al. to characterize oil shale samples before and after pyrolysis at different reaction temperatures under a hot N₂ flow. Saif et al. used micro-CT to image the time evolution of the pore and microfracture networks during oil shale pyrolysis in a custom-built furnace. These studies have demonstrated that micro-CT is a powerful tool to quantify the changes in microstructure under microwave irradiation, and comparing the similarities and differences between microwave and conventional heating can also be fulfilled by this method.

In this study, the effects of heating time (10, 20, 30 minutes), output power (600, 800, 1000 W), and reaction temperature (550, 650, 750°C) on the microstructure of oil shale were studied. Traditional optical microscope and scanning electron microscope (SEM) were used to reveal the surface and two-dimensional morphology of oil shale. Visualization of the internal structure of pores and fractures was achieved using micro-CT. Porosity and the degree of anisotropy of oil shale samples after microwave and conventional heating have been quantified based on micro-CT data. Our experimental results provide a valuable way to understand the change in the microstructure of oil shale after microwave irradiation and to analyze the flow behavior of transformation products in the future.

2 MATERIALS AND METHODS

2.1 The preparation and characterization of oil shale samples

Raw oil shale was stemmed from the Jintang mining area in the Maoming Basin, Guangdong Province, China. Since the Jintang mining area is an opencast, the oil shale samples were collected from the surface. Cylinder samples with a diameter of 7.5 mm and length of 10-12 mm were obtained by drilling perpendicularly and horizontally to the bedding plane of oil shale. These small cylinders were observed by microscopes and scanned by micro-CT before and after heat treatment. Other particle samples were crushed and sieved to a size range of 0.1-1.0 mm and were used to conduct X-ray
diffraction analysis (D8 ADVANCE, Bruker, Germany), ultimate analysis (EA3000, EuroVector, Italy) and total organic carbon analysis (2100NC, Analytik Jena, Germany) experiments. Based on the composition analysis, geological thin sections were prepared by hand grinding and polishing. Slices with 20 μm thickness were examined by an optical microscope (Axio Imager A2m, Zeiss, Germany). The height, diameter, and mass data of the cylinder oil shale samples are shown in Table 1. Each cylinder sample was labeled in order.

2.2 Heating experiments

Under nonisothermal conditions, cylinder samples were irradiated as different microwave parameters were varied, including heating time, output power, and reaction temperature. Deionized water (0.02 mL) with 0.1 wt% iron oxide nanoparticles prepared in the laboratory was added to the heating system to enhance the microwave heating efficiency. Based on our previous study, nanoparticles could be injected with the fracturing fluid, which can enhance the permeability of the shale reservoir.38 Microwave heating experiments were performed in a homemade microwave apparatus shown in Figure 1. This unique experimental setup comprises multiple generators at a frequency of 2450 MHz with a maximum output power of 1600 W. The dimensions of the apparatus are 471 mm × 670 mm. Quartz glass, which can bear the maximum temperature of 1200°C, is used as the sample container. The top port and bottom holes of the container are used for gas effluent. A thermocouple is used to test surface temperature of the samples. If the oil shale reached the set temperature, the temperature of samples would remain unaltered. In addition, all the experiments were conducted under ambient air because oxygen exists in the oil shale reservoir. After microwave heating, the reaction cavity was naturally cooled to room temperature and the solid products were determined on a weight basis. To study the influence of the heterogeneity of oil shale, one cylinder obtained by drilling horizontally to the bedding plane was also heated in the microwave apparatus (labeled M8 in Table 1). To determine the similarities and differences between microwave heating and conventional heating, the sample labeled as C1 was also heated in an electric oven at the reaction temperature of 750°C. More experimental information is shown in Table 2.

2.3 Microscopic observation and analysis

A stereo microscope (XLT-165, Jiangxi Phoenix Optical Technology CO., Ltd, China) equipped with a color camera was
used to acquire the two-dimensional (2-D) images of oil shale. The working distance is from 3 to 16 cm. An oil immersion objective lens of 4.5 × with continuous variable times and eyepiece of 10 × were used for magnification. The stage and movement of the microscope were controlled by horizontal and vertical supports. Induced pores, fractures and their orientation after microwave irradiation were obtained from digital image analysis.

Scanning electron microscope (SEM) using energy dispersive spectroscopy was conducted to observe the microstructure of the samples and to analyze the elemental composition of the special minerals. After microwave heating, the small cylinder samples were first prepared to be conductive by surface treatment. After being sputter-coated with gold, the samples were placed in the vacuum chamber of a scanning electron microscope (Quanta 450, FEI Company, USA) featuring lenses with a maximum magnification of 100 000×.

X-ray micro CT is a noninvasive and nondestructive method to characterize induced pores and fractures in oil shale. An industrial X-ray CT (MICROXCT-400, XRADIA company, USA) scanner was used to obtain high-resolution oil shale volumetric reconstructions at a maximum voltage of 150 kV and maximum light power of 10 W with three different magnification lenses (4x, 10x, 20x). The projection data of the small cylinder samples before and after microwave irradiation were transformed into hundreds of two-dimensional gray scale cross-sectional images.

Image analysis was performed based on the polished oil shale surfaces and the volumetric reconstructions from X-ray CT data before and after microwave irradiation. The X-ray CT data consisted of approximately 900 2-D slices. After the 2-D slices were obtained, a 3-D model was reconstructed by using the Avizo software from the FEI Company. The model was processed by using a nonlocal means filter and segmented to generate a binarized representation of the pore space so that the pore network and fracture propagation could be visualized in the model. The degree of anisotropy of samples was determined based on the calculation module within the Avizo software. In particular, sample porosity was calculated by exporting the binarized images and importing them into the software programmed in the C++ language.

### 3 | RESULTS AND DISCUSSION

#### 3.1 | Characterization of raw oil shale

The results from XRD, TOC and ultimate analysis are summarized in Table 3, which shows that clay minerals and quartz compose the majority of the oil shale composition. Quartz is very stable under high temperatures while other minerals undergo thermal decomposition with increasing temperature. Furthermore, the existence of pyrite, a microwave absorber, should not be ignored because it can be quickly heated under microwave irradiation. The thin slices of Maoming oil shale shown in Figure 2 illustrate the lamellar structure in which minerals are distributed. The kerogen-rich layers are the dark colored region while the mineral-rich layers are the light color. Although kerogen is mostly associated with clay layers, the distribution of kerogen and minerals is not uniform, which may influence the thermal stress within the oil shale and pore network formation during microwave irradiation.

#### 3.2 | Similarities and differences between the two heating methods

Figure 3 shows the temperature distributions of oil shale under microwave and conventional heating. It can be

| Label | Output power (W) | Heating time (min) | Reaction temperature (°C) |
|-------|----------------|-------------------|--------------------------|
| M1    | 800            | 30                | 750                      |
| M2    | 800            | 20                | 750                      |
| M3    | 800            | 10                | 750                      |
| M4    | 800            | 30                | 650                      |
| M5    | 800            | 30                | 550                      |
| M6    | 1000           | 30                | 750                      |
| M7    | 600            | 30                | 750                      |
| M8    | 800            | 30                | 750                      |
| C1    | \              | 54                | 750                      |

| Mineral analysis (wt%) |
|------------------------|
| Clay                   | 42.4                  |
| Quartz                 | 42.8                  |
| Potash feldspar        | 4.7                   |
| Plagioclase            | 3.3                   |
| Calcite                | 1.9                   |
| Pyrite                 | 4.9                   |

| Clay mineral analysis (wt%) |
|-----------------------------|
| Illite and smectite mixed layer | 47                  |
| Illite                      | 14                    |
| Kaolinite                   | 28                    |
| Chlorite                    | 11                    |

| Ultimate analysis (wt%) |
|-------------------------|
| Hydrogen                | 2.70                  |
| Oxygen                  | 5.38                  |
| Nitrogen                | 0.44                  |
| Sulfur                  | 1.01                  |
| Total carbon            | 15.96                 |
| Organic carbon          | 14.8                  |

### TABLE 2 Oil shale experiment information with different heating parameters

### TABLE 3 Characteristics of Maoming oil shale
ZHU et al. have seen that high output power was an important factor affecting the heating rate. An output power of 1000 W resulted in the shortest time to reach 750°C. In contrast, the heating rate obtained by conventional heating was much lower than that of microwave heating. The efficiency of microwave heating depends on the high dielectric constant of materials such as water, pyrite and iron oxide nanoparticles. Under microwave irradiation, bulk water and bound water within oil shale would be evaporated, resulting in the creation of steam. During the discharge process, steam gushing from inside the oil shale to outside the shale would inevitably impact the oil shale, generating additional micro fractures. In addition, pyrite and iron oxide nanoparticles were also highly receptive to microwave energy and were heated quickly as “hot spots,” which resulted in local areas with much higher temperatures than adjacent areas. This characteristic, called selective heating, makes it possible to cause differential thermal stress within the oil shale matrix, inducing new fractures and causing changes in pore structure. This phenomenon would be more evident under higher output power. In contrast, conventional heating methods heated oil shale from outside to inside, so the heating process was slower than microwave heating. Moreover, the effect of differential thermal stress was weakened under conventional heating. To confirm our views, more investigations combined with traditional 2-D and advanced 3-D visualization methods were applied.

### 3.3 Visualization of microstructure by 2-D images

Figure 4 shows optical microscope images of oil shale before and after microwave heating with different heating parameters. Additional images of other samples can be seen in Supporting Information. For the C1 oil shale shown in Figure 4H, the relatively low heating rate obtained by conventional heating corresponded to the small number of fractures on the surface of oil shale. Although the time of microwave heating was only 30 minutes, Figure 4B displays high numbers of large and small fractures acquired by microwave irradiation. The comparison between the images of C1 and M1 validated the utility of microwave heating, which induced micro fractures and enlarged the pore volume of oil shale. In addition, the M8 oil shale acquired by drilling horizontally to the bedding plane was unique in morphology after microwave irradiation. The tendency and direction of fractures were along the bedding plane and a large number of relatively uniform microfractures were developed inside the kerogen-rich lamellar structures, showing that the M1 sample was more heterogeneous in the longitudinal direction. The weight loss of nine oil shale samples after heat treatment is shown in Figure 5, revealing a significant variation with different heating parameters. It is easily understood that high reaction temperatures and long heating times would cause high weight loss in the samples. Furthermore, the output power determines the heating rate, which is responsible for the complex physical and chemical reactions. Regardless of composition, both minerals and clay minerals would undergo thermal decomposition during a long heating time, but conventional heating within 55 minutes heating time did not show the highest weight loss. This phenomenon resulted from different heating mechanisms. At the same time, we
should quantitatively determine if the increase in weight loss of samples corresponds to the increase in porosity. This question will be discussed later.

When the inter-vapor pressure caused by steam increases beyond the mechanical strength of the samples, oil shale easily crumbles. After microwave irradiation, the M2 sample accidentally broke into two segments along fracture plane in the laboratory, and the cross section of the M2 sample was analyzed in an SEM experiment. The image shown in Figure 6A reveals the formation of pore space under microwave irradiation at a temperature of 750°C. Because the pore volume is also dependent on the distribution and content of kerogen, it is not appropriate to draw the conclusion that the pore volume shown in Figure 6A is generated by microwave induction instead of thermal decomposition of kerogen. Figure 6B shows a high resolution image of pyrite after microwave heating. As a microwave absorber, pyrite first absorbs a great deal of microwave energy and thus forms a local high temperature area, leading to huge differential thermal stress and decomposition of the pyrite. Since microwaves have the ability to penetrate and excite materials with high dielectric constants from the interior, they promote the formation of small pore spaces of the oil shale, but large pore spaces resulted from the thermal decomposition of kerogen. The pore network, therefore, was created by the synergistic effect of above two aspects.

Although the samples were imaged by optical microscope and SEM to characterize the microstructure of oil shale before and after heat treatment, traditional 2-D images only show the surface changes and not the internal structure of
samples. To better understand the transformation process of oil shale under microwave irradiation, a comprehensive tool is required to explore 3-D microstructure of samples before and after heat treatment.

3.4 | Visualization and quantification of microstructure by 3-D images

The structure and connectivity of pore spaces in oil shale after microwave irradiation directly influences the seepage and production of oil and gas after the complete transformation of kerogen. Therefore, it is vital to comprehensively explore and quantify changes in porosity of oil shale after microwave irradiation with different heating parameters. Moreover, microwave heating also has the characteristic of inducing micro fractures, which differs from conventional heating.

Oil shale has compositional and structural heterogeneity, so the pore structure of oil shale after heat treatment not only depends on the heating parameters but also is affected by the inhomogeneous distribution of kerogen. Photographs of oil shale samples of different sections before and after microwave heating are shown in Figure 7. Additional pictures with different sections of other samples can be seen in the Supporting Information. Initially, Figure 7A reveals that raw oil shale had limited pore volume with few microscale fractures. This compact structure makes it difficult for liquid and gas to flow. When exposed to microwave energy, the pore volume of oil shale increases significantly with multiple newly induced micro fractures, which differ from conventional heating.
fractures as shown in Figure 7D. The fracture network was so evident that many pores were effectively connected, benefiting the flow behavior of shale oil and gas through the pore channels. The results of the micro-CT images showed that the generated fractures tended to distribute horizontally in the bedding direction. Similarly, in Figure 7G-I, the preferential orientation of fractures is also associated with the direction of the bedding plane. However, the pore space of the M8 sample was greater than that of the M1 sample, demonstrating that the M8 sample acquired by drilling horizontally to the bedding plane contained more kerogen. Thermal transformation of kerogen-rich regions could lead to a significant increase in pore space and even the collapse of the samples.

Tiwari et al.\textsuperscript{44} estimated the porosity of oil shale under a hot gas environment, but it is proper to determine the porosity of oil shale after heat treatment from different angles and directions due to the serious heterogeneity within oil shale. In this study, when volumetric reconstructions of
samples were completed (see Figure 8A), the cubic model was saved as hundreds of binarized images in the computer. In Figure 8B and (c), black represents the matrix and white represents pores. All binarized images were input into the software programmed in C++. The function of this software is to calculate the porosity with increasing calculated volume until the calculated volume reaches 2/3 of the volume of the cubic model. The starting points of the calculation are A, B, C and D, as shown in Figure 8A. Points A, B and C are the vertices of the cubic model while point D is the center of the cubic model. This calculating method is more reliable and convincing for determining the overall variation in porosity with increasing calculated volume.

Depicting the porosity distribution within oil shale, Figure 9 reveals significant variation in porosity with increasing calculating volume. At first, the porosity data strongly fluctuated, indicating that a small calculating volume did not reflect the true porosity of the samples. When porosity data eventually became stable, the calculating volume was defined as a representative elementary volume (REV) which was dependent on the distribution of kerogen and minerals. In Figure 9, it can be seen that heating time highly influences the ultimate porosity of oil shale under microwave irradiation. Within 10 minutes of heating time, kerogen could not transform completely and part of it still remained in the oil shale. However, there was only a slight distinction in the ultimate porosity between 20 minutes and 30 minutes, demonstrating that kerogen could decompose under 20 minutes of microwave irradiation at an output power of 800 W. Based on the temperature profile shown in Figure 3, it only takes approximately 14 minutes to reach 750°C under microwave heating at an output power of 800 W. After reaching the set point of the reaction temperature, the oil shale mostly underwent the decomposition of minerals and clay minerals, so pore space would not obviously increase after 20 minutes of heating time. The effect of reaction temperature on the ultimate porosity of oil shale is demonstrated in Figure 9C, F and G. Generally, higher reaction temperature led to larger porosity, but the difference between the three porosity values at different temperatures was not evident. At 550°C, kerogen was able to transform into shale oil, which revealed that the content of kerogen was a dominant factor on pore volume. However, the high heating rate obtained by microwave heating makes it possible to trigger complex physical and chemical reactions under high temperature. Therefore, the porosity value shown in Figure 9C is large. Figure 9D,E illustrate that 1000 W output power corresponds to an ultimate porosity of 12.49% while 600 W of output power only leads to an ultimate porosity of <4% at the same reaction temperature and heating time. There was also large difference in the porosity data as the output power was varied. High power levels resulted in rapid emission of volatile matter, which increased the internal vapor pressure inside the pore network. When the pressure exceeded the mechanical strength of the oil shale, micro fractures and pore space were created. Moreover, high power levels accelerated the movement of molecules inside absorbers, and thus formed the local high temperature area shown in Figure 6B. Therefore, the largest difference in data resulted from the above two aspects. For conventional heating, the calculated porosity is relatively low, as shown in Figure 9I. Although the heating time for conventional heating was close to 55 minutes, the constant and low heating rates made it difficult to generate micro fractures. Neither local high temperature areas, nor rapidly increasing internal pressure were formed by conventional heating. This phenomenon revealed that there was no correlation between porosity and weight loss when it came to the two different heating methods. To explore the effect of heterogeneity of oil shale, the M8 sample acquired by drilling horizontally into the bedding plane was also irradiated. In Figure 9H, the ultimate porosity of the M8 oil shale after microwave irradiation is the highest, showing that the M8 sample contained a large amount of kerogen. In Figure 7G, the pore space and fracture networks of the M8 oil shale after microwave irradiation are also evident but the morphology corresponding to Figure 4E is very different from other samples. Under such circumstance, weight loss was responsible for the porosity, and the kerogen content drove the results when the microwave heating method was applied. This phenomenon suggests that the drilling well should be designed based on the distribution of kerogen when applying the microwave heating method for the in situ exploitation of oil shale.

After the investigation of porosity, the degree of anisotropy of the 3-D model was analyzed to study the structural

![Figure 10](image-url)
alignment of the pore network based on classical eigenvalue analysis. The degree of anisotropy was determined as follows:

\[
\text{The degree of anisotropy} = \left( 1 - \frac{\text{min eigenvalue}}{\text{max eigenvalue}} \right)
\]  

(1)

This analysis was based on extracting a minimal and a maximal eigenvalue. The closer these values are, the less structural anisotropy there is. The degree of anisotropy was 0 for total isotropy and 1 for total anisotropy. In Figure 10, the heterogeneity of the distribution of kerogen and minerals is evident in samples obtained by drilling perpendicularly to the bedding plane. This result shows that the degree of anisotropy was not controlled by heating parameters but was mainly dependent on the properties of oil shales themselves. For the M8 sample, the degree of anisotropy was lower than that of other samples, demonstrating that the distribution of kerogen in the bedding direction was relatively uniform.

3.5 | Potential application of microwave energy for the exploitation of oil shale

In China, abundant oil shale deposits make it possible to recover a large amount of shale oil, meet energy requirement and promote environmental protection. Microwave energy may effectively contribute to the in situ transformation of oil shale and create seepage channels for produced shale oil and gas. Drilling and production wells should be designed based on the distribution of kerogen. In this study, the experimental results showed that a horizontal well along the bedding plane could contain more kerogen, so the conceptual design for the in situ exploitation of oil shale by microwave heating is shown in Figure 11.

A horizontal well is used for the drilling and production wells where a production tube can extract shale oil and gas. The completion scheme of the horizontal well is specially designed for the application of microwave heating. A microwave heating system is composed of high power microwave sources, downhole transmission lines, a temperature sensor and multiple bottom-hole waveguide antennas. Under microwave irradiation, oil shale starts to transform into shale oil and gas. In the presence of a large pore volume and many fractures, products are able to be exploited as useful fuel. However, more laboratory experiments and numerical simulations are required in the future.

The results of this study showed that high output power levels promoted the creation of pore space and micro fractures, but more work is required to determine the optimization of the microwave heating parameters. The production and quality of shale oil and gas should also be considered at the same time. Since investigations conducted by Yang et al. showed that output power of 1000 W inevitably reduced the production of shale oil due to secondary reactions at high temperature, there are both advantages and disadvantages to applying high output power for the in situ exploitation of oil shale. Moreover, in order to upgrade microwave heating to an industrial scale, several potential challenges and difficulties including choosing optimal heating parameters, designing an industrial microwave generator and analyzing economic feasibility should also be overcome in the future.

4 | CONCLUSIONS

Microwave heating technology has been proven to be an effective and environmental-friendly method for the exploitation of
oil shale. The change in microstructure is an important aspect on the seepage and production of shale oil and gas after microwave heating. In this study, raw oil shale showed a high degree of anisotropy and complex compositions in the experiments of XRD, TOC, ultimate analysis and thin slice analysis. Optical microscope observations, SEM experiments and micro-CT analysis were applied to visualize and quantify the changes in morphology, porosity, and fracture network of oil shale before and after heat treatment. By combining traditional 2-D and advanced 3-D visualization methods, the pore network and fracture generation were analyzed comprehensively. After heating, all samples in the optical images showed obvious fractures on the surfaces of the oil shale due to the transformation of kerogen, but microwave heating led to additional pores and fractures. SEM analysis also illustrated that pore space around pyrite resulted from the differential thermal stress caused by the selective heating characteristic of microwave heating. Porosity calculations based on CT data showed different results for the different microwave heating parameters. Long heating times were responsible for the complete transformation of kerogen. High output power contributed to complex physical and chemical reactions, which included the elevation of internal pressure and decomposition of minerals. Therefore, large porosity was obtained in sample M1 while conventional heating only led to very low porosity values. Moreover, in order to explore the heterogeneity of oil shale, sample M8 was obtained by drilling horizontally to the bedding plane, and it showed high porosity and uniform pore distribution after microwave heating. This result could enlighten us about well design for the exploitation of oil shale.

The experimental data presented on oil shale before and after microwave heating serve as the valuable information on the issue of the in situ exploitation process. Based on this study, future analysis into flow behavior of shale oil and gas could be used for well simulation.

ACKNOWLEDGMENTS

This work was financially supported by Qihang Project of Southwest Petroleum University (2015QHZ006). We sincerely thank State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation at Southwest Petroleum University for the access to testing and analyzing oil shale samples. The authors are also grateful to Quantang Fang for conducting the CT experiments.

CONFLICT OF INTEREST

The authors declare no competing financial interest.

ORCID

Zhaozhong Yang https://orcid.org/0000-0002-7999-5025

REFERENCES

1. Hashemi R, Nassar NN, Almao PP. Nanoparticle technology for heavy oil in-situ upgrading and recovery enhancement: opportunities and challenges. Appl Energy. 2014;133(1):374-387.
2. Hascakir B, Babadagli T, Akin S. Experimental and numerical simulation of oil recovery from oil shales by electrical heating. Energy Fuels. 2008;22(6):3976-3985.
3. Chen C, Gao S, Sun Y, Guo W, Li Q. Research on underground dynamic fluid pressure balance in the process of oil shale in-situ fracturing-nitrogen injection exploitation. J Energy Resour Technol. 2017;139(3):1-7.
4. Pan Y, Chen C, Yang S, Ma G. Development of radio frequency heating technology for shale oil extraction. Open J Appl Sci. 2012;2(2):66-69.
5. Brandt AR. Converting oil shale to liquid fuels: energy inputs and greenhouse gas emissions of the Shell in situ conversion process. Environ Sci Technol. 2008;42(19):7489-7495.
6. Lee KJ, Moridis GJ, Ehlig-Economides CA. Numerical simulation of diverse thermal in situ upgrading processes for the hydrocarbon production from kerogen in oil shale reservoirs. Energy Explor Exploit. 2017;35(3):315-337.
7. Bientinesi M, Petarca L, Cerutti A, et al. A radiofrequency/microwave heating method for thermal heavy oil recovery based on a novel tight-shell conceptual design. J Pet Sci Technol Eng. 2013;107(7):18-30.
8. Hakala JA, Stanchina W, Soong Y. Hedges S. Influence of frequency, grade, moisture and temperature on Green River oil shale dielectric properties and electromagnetic heating processes. Fuel Process Technol. 2011;92(1):1-12.
9. Li H, Lin B, Yang W, Hong Y, Wang Z. A fully coupled electromagnetic-thermal-mechanical model for coalbed methane extraction with microwave heating. J Nat Gas Sci Eng. 2017;46(10):830-844.
10. Bera A, Babadagli T. Effect of native and injected nanoparticles on the efficiency of heavy oil recovery by radio frequency electromagnetic heating. J Pet Sci Eng. 2017;153(5):244-256.
11. Wang H, Rezaee R, Saeedi A, Josh M. Numerical modelling of microwave heating treatment for tight gas sand reservoirs. J Pet Sci Eng. 2017;152(4):495-504.
12. Liu J, Wang J, Leung C, Gao F. A fully coupled numerical model for microwave heating enhanced shale gas recovery. Energies. 2018;11(6):1608.
13. Zhu J, Yang Z, Li X, Wang N, Jia M. Evaluation of different microwave heating parameters on the pore structure of oil shale samples. Energy Sci Eng. 2018;6(6):797-809.
14. Asakuma Y, Nakata R, Saptoro A. Bubble formation in water with magnetite nanoparticles during microwave irradiation. Chem Eng Process Process Intensification. 2017;119(9):101-105.
15. Li K, Hou B, Wang L, Cui Y. Application of carbon nanocatalysts in upgrading heavy crude oil assisted with microwave heating. Nano Lett. 2014;14(6):3002-3008.
16. Zhang Y, Han Z, Wu H, Lai D, Glarborg P, Xu G. Interactive matching between the temperature profile and secondary reactions of oil shale pyrolysis. Energy Fuels. 2016;30(4):2865-2873.
17. Mutyal S, Fairbrige C, Pare JR, Belanger JM, Ng S, Hawkins R. Microwave applications to oil sands and petroleum: a review. Fuel Process Technol. 2018;9(2):127-135.
18. Hascakir B, Akin S. Recovery of Turkish oil shales by electromagnetic heating and determination of the dielectric properties of oil shales by an analytical method. Energy Fuels. 2009;24(1):503-509.
19. Chanaa MB, Lallemant M, Mokhlisse A. Pyrolysis of Timahdit, Morocco, oil shales under microwave field. Fuel. 1994;73(10):1643-1649.
20. Mokhlisse A, Chanaa MB, Outzourhit A. Pyrolysis of the Moroccan (Tarfaya) oil shales under microwave irradiation. Fuel. 2000;79(5):733-742.
21. Neto A, Thomas S, Bond G, Thibault-Starzyk F, Ribeiro F, Henriques C. The oil shale transformation in the presence of an acidic BEA zeolite under microwave irradiation. Energy Fuels. 2014;28(4):2365-2377.
22. Ribas L, Neto JM, França AB. The behavior of Irdi oil shale before and after the pyrolysis process. J Pet Sci Eng. 2017;152(4):156-164.
23. Schrodt JT, Ocampo A. Variations in the pore structure of oil shales during retorting and combustion. Fuel. 1984;63(11):1523-1527.
24. Han X, Jiang X, Yu L, Cui Z. Change of pore structure of oil shale particles during combustion. Part 1. Evolution mechanism. Energy Fuels 2006;20(6):2408-2412.
25. Bai F, Sun Y, Liu Y, Guo M. Evaluation of the porous structure of Huadian oil shale during pyrolysis using multiple approaches. Fuel. 2017;187(1):1-8.
26. Han X, Jiang X, Cui Z. Change of pore structure of oil shale particles during combustion. 2. Pore structure of oil-shale ash. Energy Fuels. 2008;22(2):972-975.
27. Wang Q, Jiao G, Liu H, Bai J, Li S. Variation of the pore structure during microwave pyrolysis of oil shale. Oil Shale. 2010;27(2):135-146.
28. Freidin C. Influence of variability of oil shale fly ash on compressive strength of cementless building compounds. Constr Build Mater. 2005;19(2):127-133.
29. Hong YD, Lin BQ, Li H, Dai HM, Zhu CJ, Yao H. Three-dimensional simulation of microwave heating coal sample with varying parameters. Appl Therm Eng. 2016;91(1):1145-1154.
30. Hassani F, Nekoovaght PM, Gharib N. The influence of microwave irradiation on rocks for microwave-assisted underground excavation. J Rock Mech Geotechnical Eng. 2016;8(1):1-15.
31. Hartlieb P, Toifl M, Kuchar F, Meisels R, Antretter T. Thermophysical properties of selected hard rocks and their relation to microwave-assisted comminution. Miner Eng 2016;91(5):34-41.
32. Tiwari P. Oil shale pyrolysis, Bench scale experimental studies and modeling. PhD dissertation. University of Utah, Salt Lake City, Utah, 2012.
33. Mathews JP, Pone JN, Mitchell GD, Halleck P. High-resolution X-ray computed tomography observations of the thermal drying of lump-sized subbituminous coal. Fuel Process Technol. 2011;92(1):58-64.
34. Guo X, Shen Y, He S. Quantitative pore characterization and the relationship between pore distributions and organic matter in shale based on Nano-CT image analysis: a case study for a lacustrine shale reservoir in the Triassic Chang 7 member, Ordos Basin, China. J Nat Gas Sci Eng. 2015;27(11):1630-1640.
35. Zhao J, Yang D, Kang Z, Feng Z. A micro-CT study of changes in the internal structure of Daqing and Yan’an oil shales at high temperatures. Oil Shale. 2012;29(4):357-367.
36. Tiwari P, Deo M, Lin CL, Miller JD. Characterization of oil shale pore structure before and after pyrolysis by using X-ray micro CT. Fuel. 2013;107(5):547-554.
37. Saif T, Lin Q, Bijeljic B, Blunt MJ. Microstructural imaging and characterization of oil shale before and after pyrolysis. Fuel. 2017;197(6):562-574.
38. Zhu J, Yang Z, Li X, Qi S, Jia M. Application of microwave heating with iron oxide nanoparticles in the in-situ exploitation of oil shale. Energy Sci Eng. 2018;6(5):548-562.
39. Uslu T, Atalay Ü, Arol Aİ. Effect of microwave heating on magnetic separation of pyrite. Colloids Surf A. 2003;3:161-167.
40. Burnham AK. Porosity and permeability of Green River oil shale and their changes during retorting. Fuel. 2017;203(9):208-213.
41. Hong YD, Lin BQ, Li H, Dai HM, Zhu CJ, Yao H. Three-dimensional simulation of microwave heating coal sample with varying parameters. Appl Therm Eng. 2016;91(1):1145-1154.
42. Hassani F, Nekoovaght PM, Gharib N. The influence of microwave irradiation on rocks for microwave-assisted underground excavation. J Rock Mech Geotechnical Eng. 2016;8(1):1-15.
43. Hartlieb P, Toifl M, Kuchar F, Meisels R, Antretter T. Thermophysical properties of selected hard rocks and their relation to microwave-assisted comminution. Miner Eng 2016;91(5):34-41.
44. Tiwari P. Oil shale pyrolysis, Bench scale experimental studies and modeling. PhD dissertation. University of Utah, Salt Lake City, Utah, 2012.
45. Hamdan MA, Qubbaj A. Inhibition effect of inert compounds on oil shale dust explosion. Appl Therm Eng. 1998;18(5):221-229.
46. Yang Z, Zhu J, Li X, Luo D, Qi S, Jia M. Experimental investigation of the transformation of oil shale with fracturing fluids under microwave heating in the presence of nanoparticles. Energy Fuels. 2017;31(10):10348-10357.

SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of the article.

How to cite this article: Zhu J, Yang Z, Li X, Qi S, Fang Q, Ding Y. The experimental study of microwave heating on the microstructure of oil shale samples. Energy Sci Eng. 2019;7:809–820.
https://doi.org/10.1002/esee.3.311