

## THE INFLUENCE OF MICROWAVE DRYING ON PHYSICOCHEMICAL PROPERTIES OF LIUSHUHE OIL SHALE

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*In this paper, microwave drying of Liushuhe oil shale was studied and compared with conventional drying process. Oil shale was treated in a microwave oven at four different microwave energy levels. The drying trial showed that the drying rate of microwave drying was much higher than that of conventional hot air drying. Attention has been given to the influence of microwave treatment on the physicochemical properties of oil shale. Samples dried at 550 W for 15 min (MD3) were chosen as the representative ones for microwave drying. The grindability of oil shale samples was characterized by Hardgroove grindability index (HGI). It was found that significant increases in oil shale grindability could be achieved after exposure to microwave energy. The pore structure of raw oil shale (OS) and dried samples (CD and MD3) was measured by the method of low-temperature adsorption of nitrogen. Specific surface area was calculated based on Brunauer–Emmett–Teller (BET) equation, and pore size distribution was calculated by Barrette–Joynere–Halenda (BJH) method. The results showed that the specific surface area and specific pore volume of CD and MD3 increased considerably, and microwave drying led to the formation of mesopores. Fourier transform infrared (FTIR) spectra indicated that microwave drying had no significant effect on the chemical property of oil shale.*

### Introduction

Oil shale, a sedimentary rock, often contains a proportionally small amount of kerogen, which can be converted into oil by thermal degradation. World-wide deposits of oil in shale are estimated at 2.8 trillion barrels of recoverable oil, which is a conservative figure given that oil shale resources from some countries are not reported or the deposits are not fully investigated. The world's largest oil shale deposits are found in Australia, China and

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the United States, though currently only Israel, Russia, Germany, Brazil, Estonia and China utilize their reserves [1]. There are two conventional technologies available for utilizing oil shale resources: one is retorting oil shale to produce shale oil and fuel gas, and the other is burning oil shale to generate electricity [2, 3].

Natural shale contains various non-negligible amounts of water. The high moisture content is well-known adverse to the energy efficiency of power generation and other utilization processes. Hence, it is necessary to dry oil shale to reduce the water content. Several workers have paid attention to the drying process of oil shale. B. C. Dickson and L. A. Baker [4] studied the rotary drying of Stuart oil shale. They described the operating conditions and provided valuable scaleup information. D. S. Lane et al. [5] investigated kinetics of drying Stuart oil shale over the temperature range 100–250 °C in both fluidized bed and convection oven heating. A. Tamimi and B. Z. Uysal [6] analyzed and modeled the tray-drying process of oil shale.

Different methods are used for drying. In particular, conventional hot air drying is the most common drying technique. Microwave drying is capable of a much higher drying rate and energy efficiency than conventional hot air drying and is widely applied to treat various kinds of raw materials and products, including food, vegetables, fruits, wood, etc. [7–10]. In addition, many previous works deal with the microwave drying characteristics and the effect on the physicochemical properties of coal or other materials. X. H. Wang et al. [11] made a study of the influence of microwave drying on biomass pyrolysis. The results indicated that the specific surface area of biomass was increased greatly after microwave drying. S. Marland et al. [12] studied the effect of microwave radiation on coal grindability. They found that reduction of up to 50% relative grindability of the coals can be achieved after 5 min microwave exposure. F. P. Miknis et al. [13] investigated the effect of different drying methods on coal structure. The NMR measurements showed that, in general, there were no major structural changes in coals dried thermally or with microwaves other than partial decarboxylation. However, using microwave technique for drying oil shale has not been reported.

The aim of the present work was to investigate the microwave drying characteristics of oil shale and its influence on the physicochemical properties, including proximate composition, grindability index, pore structure and chemical change.

## Experimental

### Materials

The oil shale selected for study was taken from Liushuhe, Heilongjiang province, China. Master batch sample was prepared by grinding and sieved to a particle size range of 1–3 mm using a standard sieve. The samples were placed in a constant humidity chamber until aliquots were taken for different

drying tests. Based on National Standards of China (GB/T 212-2001 and GB/T 476-2001), proximate and ultimate properties of raw oil shale (OS) were analyzed respectively. The results were as follows (%):

Proximate analysis:

$$M_{ad} - 5.52, V_{ad} - 46.63, A_{ad} - 24.75, FC_{ad} - 23.1, Q_{net,ar} - 18775.08 \text{ J/g};$$

Ultimate analysis:

$$C_{ad} - 45.17, H_{ad} - 2.49, N_{ad} - 0.75, O_{ad} - 21.18, S_{ad} - 0.14.$$

### Experimental equipment

A domestic digital microwave oven (Panasonic NN-GS575W, Japan) with a maximum output of 1000 W at 2450 MHz was used for the microwave drying experiment. In order to measure the temperature in the centre of a sample, a hole was drilled into which an infrared thermometer (ST-16A) was placed. The temperature was displayed and recorded by a data acquisition unit (Agilent, 34970A, USA). The weight of oil shale sample was recorded by means of a weighing system. It consists of an electronic balance (Shimadzu, AUW320), and the data was output by computer through RS-232C interface. As a comparison, the conventional hot air drying test was carried out in an electrothermal blowing dry box at  $100 \pm 5$  °C.

### Experimental process

Microwave drying trial was carried out at four different microwave generation power being 300, 400, 550 and 700 W. For each experiment, oil shale sample to be dried was  $50 \pm 0.5$  g and the drying time was 15 min. During the microwave drying process, the weight of the sample was recorded by the weighting system at 30 s intervals, and the temperature of sample was recorded by the temperature measuring system at 30 s intervals. Two different drying trials were conducted at each microwave generation power, and the values obtained from these trials were averaged and the parameters of drying were determined. The samples obtained were called MD1, MD2, MD3 and MD4, respectively. In addition, the conventional hot-air drying trial was performed in an electrothermal blowing dry box at  $100 \pm 5$  °C as the reference. The sample weight was also  $50 \pm 0.5$  g and the drying time was 2 h. The sample was removed from the dry box every 5 min, and the weight was determined using a digital balance with a sensitivity of  $\pm 0.01$  g, all weighing processes were completed in 5 s during the drying process. The dried sample was called CD.

### Analysis methods

The following common semi-empirical Page's equation [14, 15] (Eq. (1)) was used to describe the drying kinetics of oil shale, where  $MR$  is the moisture ratio;  $X$  is the moisture content db at time  $t$ ;  $X_0$  is the moisture

content of the raw oil shale;  $X_e$  is the equilibrium moisture content db;  $t$  is the time in min;  $k$  is the drying constant in  $\text{min}^{-1}$ ; and  $n$  is the dimensionless exponent. The equilibrium moisture content ( $X_e$ ) was assumed to be zero.

$$MR = \frac{X - X_e}{X_0 - X_e} = \exp(-kt^n). \quad (1)$$

The drying rate ( $R_D$ ) of oil shale was calculated using the following equation:

$$R_D = \frac{dMR}{dt}. \quad (2)$$

The grindability index of oil shale was determined using Hardgrove method. The  $\text{N}_2$  adsorption measurement was performed by an automated surface analyzer (Gemini 2380, Micromeritics, USA). The Brunauer–Emmett–Teller (BET) surface area and pore volumes were calculated by accessory software in the automated surface area and pore size analyzer, the pore size distributions were estimated by the Barrette–Joynere–Halenda (BJH) method. Infrared spectra of oil shale samples were taken using a Fourier transform infrared spectrometer (Tensor 27, Bruker, German) by KBr disk method.

## Results and discussion

### Temperature profile during microwave drying

Figure 1 shows the time dependences of the temperature for different microwave powers. One can observe that the sample temperature increases to a maximum value ( $T_{\text{max}}$ , 89 °C at 300 W, 97 °C at 400 W, 99 °C at 550 W and 94 °C at 700 W), after which it stays approximately constant (around  $T_{\text{max}}$ ). This may be attributed to the dielectric properties of oil shale. The permittivity of a material is a complex number and is composed of a real part ( $\epsilon'$ , conveniently termed dielectric constant) and an imaginary part ( $\epsilon''$ , termed dielectric loss factor). K. Rajeshwar and R. Inguva [16] estimated the dielectric constants  $\epsilon'$  for various constituents in Green River oil shale, the  $\epsilon'$  of water was 80, and it was much higher than that of other materials. R. Nottenburg et al. [17] studied the effect of pore water and adsorbed moisture on the dielectric properties of Green River oil shale, they noted a regular decrease in the relative dielectric constant  $\epsilon'$  and dielectric loss  $\epsilon''$  with decreasing amount of adsorbed moisture and pore water. The dielectric properties of oil shale change along with the decrease in moisture content during the microwave drying process. Due to the different values of microwave power, the dielectric properties of oil shale are different as well, which will result in different temperature profiles.

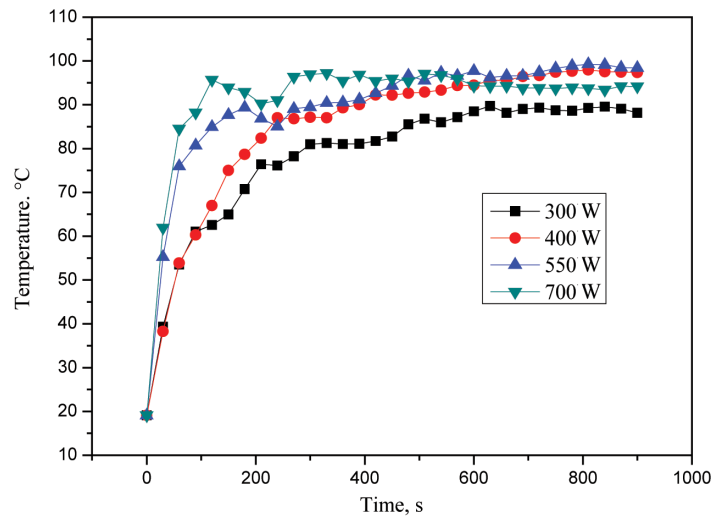


Fig. 1. Time dependences of the temperature during the microwave drying of oil shale samples.

### Drying curves

A graphical representation of the change in moisture content with drying time (drying curve) at different microwave output powers is illustrated in Fig. 2, in which symbols represent the data of experiment, lines represent the fitting drying curve based on Eq. (1). As it is shown in Fig. 2, the drying

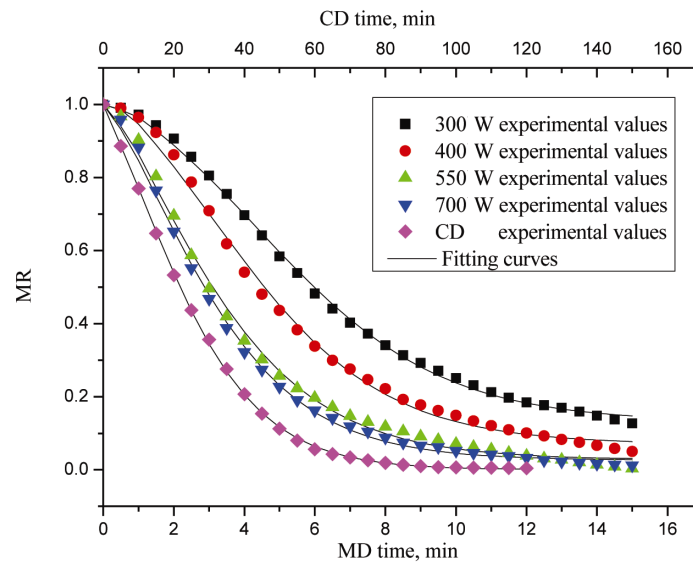


Fig. 2. Moisture ratio ( $MR$ ) versus time, comparing experimental values with the fitting curves through semi-empirical Page's equation (Eq. (1)) for oil shale.

time achieving the same  $MR$  for MD was much shorter than that for CD. A reduction in the drying time and  $MR$  occurred with the increase of the microwave power level. The time required for lowering the  $MR$  of oil shale from 1 to 0.1 varied between 12 and 7 min depending on the microwave output power, whereas, it was 54 min for CD.

The dependences experimental drying rate ( $R_D$ ) [g (H<sub>2</sub>O)/g (dry solid)·min] vs. moisture ratio ( $MR$ ) for MD and CD are shown in Fig. 3. It can be observed that the  $R_D$  of MD is much higher than that of CD. The maximum value of  $R_D$  at 700 W is 0.23 g (H<sub>2</sub>O)/g (dry solid)·min, as for CD, the value is 0.02 g (H<sub>2</sub>O)/g (dry solid)·min. Figure 3 also reveals that the shapes of drying rate curves of MD and CD differ markedly. This may be attributed to the different mechanism of heat transfer. Microwave heating is fundamentally different from conventional heating since microwaves take the form of electromagnetic energy; the polar molecule interacts with microwave electromagnetic field and converts the electromagnetic energy to heat. Conventional thermal processing will heat the sample from the outside inwards through standard heat transfer mechanisms, i.e. through convection, conduction and radiation. Previous literature on microwave drying of food products considered that the whole microwave drying process could be divided into three periods, namely warming-up, constant rate and falling rate periods. During the microwave drying of oil shale, the constant period is not revealed for high microwave power level. This is in agreement with a previous study. Al-Harshseh et al. [9] studied the microwave drying kinetics of tomato and observed that a constant period occurred only at low output power since the difference in vapor pressure between the center and the surface of the sample created by higher microwave power was larger.

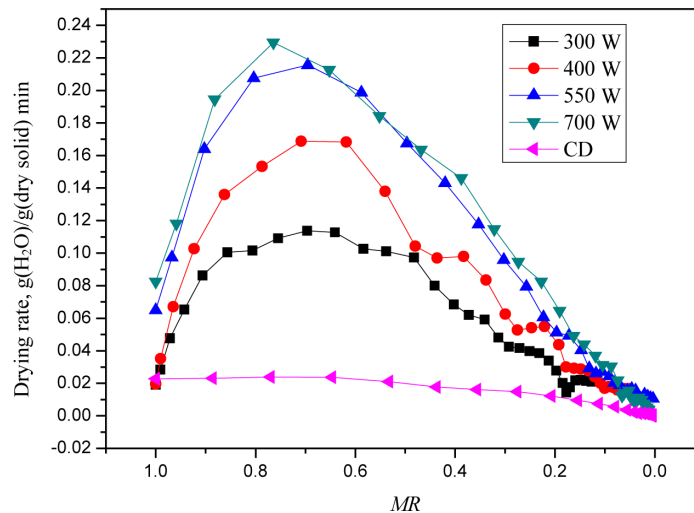


Fig. 3. Drying rate  $R_D$  (g (H<sub>2</sub>O)/g (dry solid)·min) vs. moisture ratio ( $MR$ ).

### Modeling drying data

Microwave drying kinetics of oil shale was described using the drying data. Non-linear regression technique was used to estimate the parameters  $k$  and  $n$  of semi-empirical Page's equation (Eq. (1)). The fitting drying curves are shown in Fig. 2, and the parameters of fitting drying curves are presented in Table 1. The model gave an excellent fit for the experimental data points with the regression correlation coefficient greater than 0.98. As seen in Fig. 2, measured moisture ratio values and calculated moisture ratio values were found to resemble each other. It was determined that the value of the MD constant  $k$  is bigger than that of CD and the increase in the drying constant  $k$  value with increasing microwave output power is consistent with the drying rate curves showed in Fig. 3.

Table 1. Parameters of fitting drying curves

| Drying method | Drying rate constant $k$ , $\text{min}^{-1}$ |                | Exponent $n$ |                | $R^2$  |
|---------------|--|----------------|--------------|----------------|--------|
|               | Value  | Standard Error | Value        | Standard Error |        |
| CD            | 0.01501                                      | 0.06685        | 1.26181      | 0.01681        | 0.9959 |
| MD, 300 W     | 0.03501                                      | 0.06124        | 1.58949      | 0.03092        | 0.9891 |
| MD, 400 W     | 0.04811                                      | 0.06351        | 1.61304      | 0.04586        | 0.9871 |
| MD, 550 W     | 0.12745                                      | 0.06700        | 1.35703      | 0.03383        | 0.9823 |
| MD, 700 W     | 0.15758                                      | 0.06530        | 1.28632      | 0.03297        | 0.9813 |

### Change in the chemical composition

The proximate analysis of oil shale samples after drying is shown in Table 2. The corresponding proximate analysis of the raw oil shale (OS) used in this study is illustrated above. It showed that the inherent moisture ( $M_{\text{ad}}$ ) and volatile ( $V_{\text{daf}}$ ) decrease with the increase of the microwave power level, only the MD4 did not follow the pattern in regard to the inherent moisture content. The higher inherent moisture content of MD4 is explained by the higher  $T_{\text{max}}$  at 550 W (99 °C) as compared to that at 700 W (94 °C) as shown in Fig. 1. Figure 2 reveals that the final MR at 700 W is lower than that at 550 W. It is in agreement with the proximate analysis. The inherent moisture

Table 2. Proximate analysis of oil shale samples after drying

| Sample | Proximate analysis, wt.% |                 |                 |                  |                  |                |                 |
|--------|--------------------------|-----------------|-----------------|------------------|------------------|----------------|-----------------|
|        | $M_{\text{ad}}$          | $V_{\text{ad}}$ | $A_{\text{ad}}$ | $FC_{\text{ad}}$ | $V_{\text{daf}}$ | $A_{\text{d}}$ | $FC_{\text{d}}$ |
| CD     | 0.58                     | 47.75           | 27.81           | 23.86            | 67.07            | 27.98          | 23.99           |
| MD1    | 3.54                     | 47.31           | 26.11           | 23.04            | 68.25            | 27.07          | 23.86           |
| MD2    | 2.57                     | 47.61           | 25.67           | 24.15            | 68.09            | 26.35          | 24.79           |
| MD3    | 1.40                     | 48.71           | 25.49           | 24.39            | 67.58            | 25.85          | 24.74           |
| MD4    | 1.53                     | 46.96           | 26.98           | 24.53            | 66.71            | 27.39          | 24.91           |

of CD is smaller than that of MD. It may be attributed to the long drying time. It also indicates from Table 2 that the volatile matter content ( $V_{\text{daf}}$ ) is affected after microwave treatment. S. Marland et al. [12] noted that the reaction between some volatile matter and air can occur and may contribute to fracture propagation due to release of gaseous products.

### Effect of different drying methods on HGI

The standard Hardgrove grindability index (HGI) tests were performed first to characterize the grindability of the oil shale samples. Grindability index is calculated to be  $\text{HGI} = 13 + 6.93 \times W$  (200 mesh), where  $W$  is the weight of material passing through (200 mesh). Figure 4 shows the effect of different drying methods on the HGI of oil shale samples.

As it is shown in Fig. 4, the grindability of oil shale samples has been improved after drying treatment. The influence of microwave pre-treatment on ore grindability has been investigated earlier. S. Marland *et al.* [12] studied the effect of microwave radiation on coal grindability. They found that reduction of relative grindability of coals up to 50% can be achieved after 5 min microwave exposure and suggested that evolution of gaseous products (water and volatile matter) as well as expansion of gangue minerals were the probable causes for the improvement of coal grindability and to what extent grindability depended on coal particular chemical properties. P. Kumar et al. [18] investigated the influence of microwave pre-treatment on grindability of iron ore (Orissa, India) by grindability tests. They noted that the grindability increased significantly as a result of microwave pre-treatment with the specific rate of breakage increasing by an average of 50%.

Oil shale contains solid, combustible organic matter in a mineral matrix. Due to different dielectric properties of different mineral components,

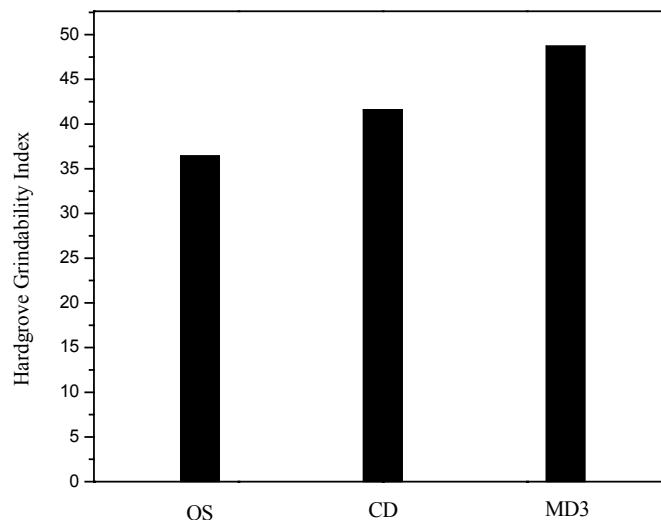


Fig. 4. Effect of different drying methods on HGI of oil shale samples.



differential expansion will occur during microwave drying process. At the same time, inherent moisture within the oil shale structure changes phase creating significant pressure. The two distinct fracture mechanisms are suggested to be the reason for the improvement of grindability of oil shale under microwave radiation.

### Specific surface area and specific pore volume

The adsorption/desorption isotherms of raw oil shale (OS) and dried samples (CD and MD3) are shown in Fig. 5. One can see that the overall shapes of these samples do not differ. They all show an invert “S”. A BET type-II [19] classification can be assigned to them, indicating a predominant micropore and mesopore structure. Specific pore features can be deduced through visual interpretation of the isotherms. At the low pressure stage ( $P/P_0:0\sim0.3$ ), the absorption isotherm rises slowly, it is mainly the adsorption of micropore wall. Steep curve rises where  $P/P_0 > 0.8$ , but it does not reach a stable state even when  $P/P_0 \approx 1.0$ , which indicates that nitrogen vapor confined in the pore of sample particles condenses at a pressure lower than its saturation pressure, which is called “capillary condensation”. This phenomenon indicates that there is a certain amount of mesopores and macropores in samples [20].

The pore size distribution is shown in Fig. 6. It shows that different drying methods have different effect on the development of pore. Pore size distribution of CD has two visible peaks near 2 nm and 10 nm, compared to OS, the micropores (2 nm) are slightly increased, and the mesopores (10 nm) are greatly increased, whereas the mesopores (2 nm) of MD3 are obviously reduced while the mesopores (10 nm) are increased significantly; compared with CD, the peak value near 10 nm of MD3 is larger.

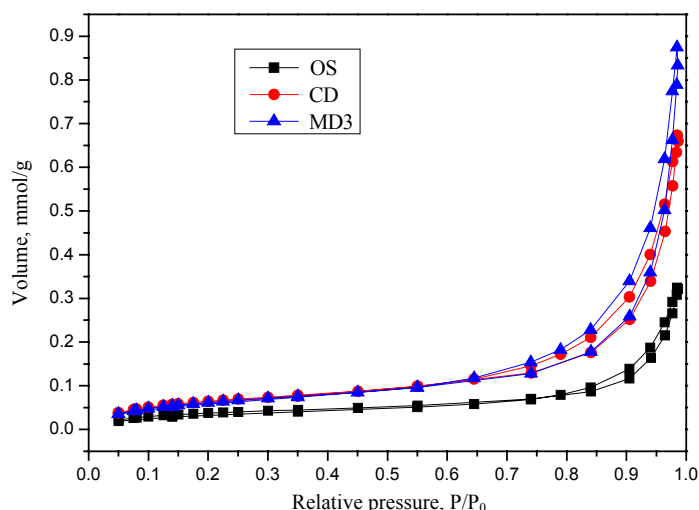


Fig. 5. The adsorption/desorption isotherms of oil shale samples.

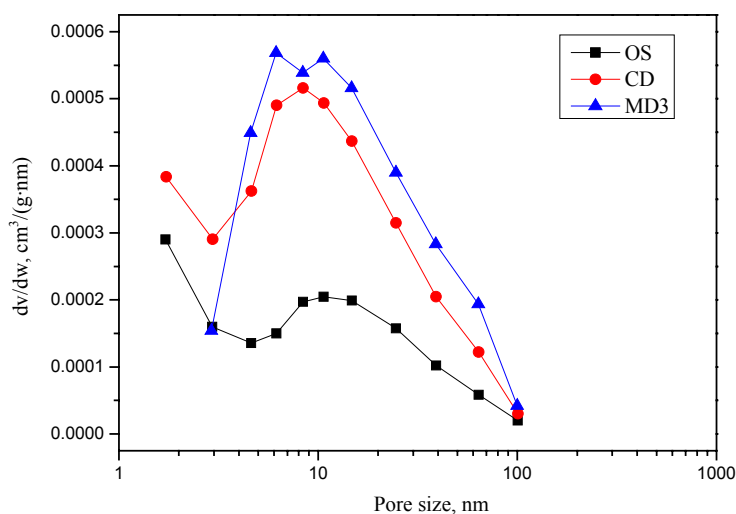


Fig. 6. Pore size distribution of oil shale samples.

Due to the different heating mechanism, the development of pores is different. Microwave drying creates an effect for moisture transfer, leading to a water vapor pressure gradient between the surface and internal part of the material [21]. As a result, a large number of micropores develop into mesopores; as mentioned above, the drying rate of MD was much higher than that of CD, in other words, a large amount of moisture was released from the inside of the sample in a very short time, which would create more inner paths. The mesopores are formed during the volatilization of moisture, so the peak value near 10 nm of MD3 is larger than that of CD, which would favor quick release of volatiles from oil shale during the pyrolysis process.

The specific surface area and pore properties are summarized in Table 3. As can be shown, the Brunauer–Emmett–Teller (BET) surface area and single point specific pore volume of CD and MD3 are greater than those of OS. This may be attributed to the release of moisture during the drying process, more new paths are broken. As the pore size distribution is different, the BET surface area of CD is somewhat bigger than that of MD3 since it has more micropores, and there are more mesopores in MD3, with

Table 3. Specific surface areas and pore properties of oil shale samples

| Sample | BET specific surface area, $\text{m}^2/\text{g}$ | Correlation coefficient of BET equation, R | Single point specific pore volume at $P/P_0 = 0.9846$ , $\text{cm}^3/\text{g}$ | Average pore size, nm |
|--------|--|--|--|-----------------------|
| OS     | 3.2478   | 0.9989                                     | 0.0112   | 14.3630               |
| CD     | 5.5659   | 0.9996                                     | 0.0233   | 14.6622               |
| MD3    | 5.3853   | 0.9997                                     | 0.0303   | 19.2616               |

the average pore size 19.2616, while it is 14.6622 for CD, as a result. Compared to CD, the specific pore volume of MD3 is much bigger.

### Effect of drying methods on chemical properties

Chemical changes in oil shale samples after drying were monitored by FTIR spectroscopy. The Fourier transform infrared (FTIR) spectra of oil shale samples are given in Fig. 7. The absorption bands at  $3698\text{ cm}^{-1}$  and  $3620\text{ cm}^{-1}$  correspond to free O–H stretching; the broad and strong band at  $3423\text{ cm}^{-1}$  shows the presence of both phenolic and carboxylic O–H groups. The adsorption bands with moderate intensities at  $2921\text{ cm}^{-1}$  and  $2851\text{ cm}^{-1}$  are due to the aliphatic stretching C–H vibrations of  $\text{CH}_2$  groups. The relatively broad band at  $1619\text{ cm}^{-1}$  belongs to aromatic C=C stretching. The peaks with relatively lower intensities at around  $1379\text{ cm}^{-1}$  correspond to the C–H vibrations of aliphatic groups ( $\text{CH}_3$ ,  $\text{CH}_2$ ). The strong adsorption band at  $1033\text{ cm}^{-1}$  and  $1009\text{ cm}^{-1}$  corresponds to the vibration of C–O group. The successive peaks with very low intensities between  $913\text{ cm}^{-1}$  and  $696\text{ cm}^{-1}$  are attributed to the aromatic out-of-plane C–H deformation vibrations [22–24].

In Fig. 7, it can be found that chemical properties of the oil shale samples after thermal and microwave drying seem to be unchanged. The FTIR spectra of CD and MD3 are very similar to that of OS. Considering also the analysis of the change in the chemical composition of oil shale samples, the moisture and volatile matter content are affected after drying treatment, but no significant changes in chemical properties were found for the samples studied here.

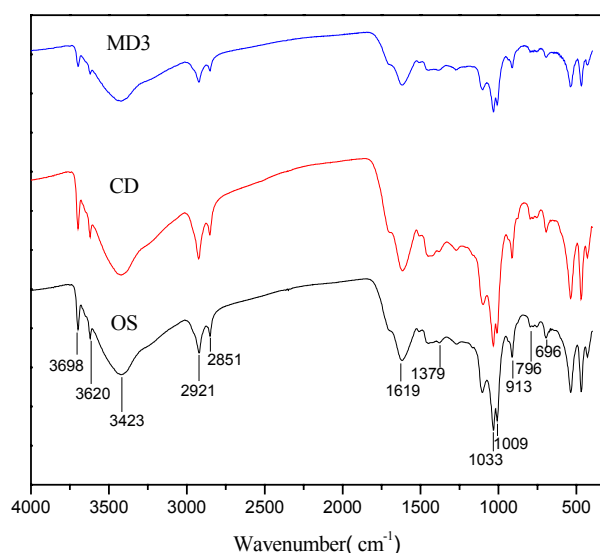


Fig. 7. Fourier transform infrared (FTIR) spectra of the oil shale samples.

## Conclusions

1. Microwave drying affords a much higher drying rate than conventional hot air drying, which indicates the feasibility of microwave drying of oil shale.
2. The moisture content of oil shale decreases significantly during the microwave drying process, and the volatile matter content is affected slightly after microwave treatment.
3. The grindability of oil shale samples is improved after microwave drying. The increases in grindability are suggested to be the result of two distinct fracture mechanisms, namely: the differential expansion of oil shale matrix and phase changes of inherent moisture within the oil shale structure creating significant pressure.
4. Both microwave drying and conventional drying increase the specific surface area and specific pore volume of oil shale. However, the pore size distribution is different owing to the different heating mechanism. The microwave drying leads to the formation of mesopores.
5. Microwave drying has no significant effect on the chemical properties of oil shale.

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