Research Article

Mineral Heterogeneity Characterization of the Lacustrine Yanchang Shales, Ordos Basin Using Micro-Fourier Transform Infrared Spectroscopy (Micro-FTIR) Technique

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Shale is a typical fine-grained sedimentary rock with small grain sizes of matrix components, significant lithofacies variation of rock texture and structure, and strong heterogeneity of organic matter and mineral compositions. Characterization of mineral compositions and their heterogeneity in micro- to nanoscale are the key parameters to gas shale pore structure and rock physical properties. In order to study the microscale mineralogy heterogeneity of the lacustrine shales in the Triassic Yanchang Formation in the Ordos Basin, the micro-Fourier transform infrared spectroscopy (micro-FTIR) technique was conducted. Based on the specific micro-FTIR spectra peaks, the abundance of mineral compositions can be quantitatively determined in the selected microscale areas. The results show that within the range of 80 μm micro-FTIR test interval, both massive argillaceous shale and silty interlayered shale show obvious heterogeneity; in particular, the relatively homogeneous shale observed by the naked eyes also has strong mineral heterogeneity. The results of micro-FTIR spectra are basically consistent with the bulk X-ray diffraction (XRD) data. The advantage of this micro-FTIR technique includes higher resolution (less than 100 μm) and in situ mineral characterization of shale samples at micro- and nanoscale.

1. Introduction

Shales have received worldwide attention due to their emergence as effective unconventional hydrocarbon reservoirs [1–5]. Shale gas is an unconventional gas resource which is generated and accumulated in organic-rich shales [6]. Shale gas is primarily found in free gas and adsorbed gas occurrences [7, 8]. Shales are typical fine-grained sedimentary rock with complex matrix components, multiscale heterogeneity of mineral and organic matter (OM) compositions, and significant lithofacies variation in rock texture and structure, which have important impacts on shale gas exploration and development [9–14]. Accurate determination and fine characterization of mineral composition and distribution in the micro- and nano-

scale is of great significance for pore structure characterization and petrophysical evaluation of gas shales [15–19]. At present, the evaluation of the mineral composition of gas shale mainly depends on bulk rock mineral analysis based on microscope observation on thin sections and X-ray detection. However, the existing techniques have limitations in higher resolution and in situ fine characterization, and it is difficult to evaluate the heterogeneous distribution characteristics of mineral compositions at high resolution using these methods [20–23]. Under actual geological conditions, the microscale heterogeneity of mineral compositions, distribution, and rock texture in shale is still significant [24–27]. Therefore, the characterization of shale heterogeneity needs improvement in the spatial resolution and in situ mineral composition measurement.

FIGURE 1: Regional tectonic profile and the location of the sampling well in the Ordos Basin, China.

Transmission Fourier transform infrared (FTIR) spectroscopy has been widely used for many years to effectively assess mineral compositions [28–30]. However, traditional FTIR experiments are time consuming and prone to problems. Diffuse reflectance FTIR spectroscopy (DRIFTS) requires the use of a diluent, such as potassium bromide (KBr), for high-quality reflectance results [31]. DRIFTS has the advantage of no physical contact with the analyte, and the technique can be automatically performed and compared with previous results. Attenuated total reflectance- (ATR-) FTIR has been used in a wide range of fields in the field of geology [32, 33]. Unlike transmission and DRIFTS, ATR-FTIR can be used to treat very few samples without dilution. However, ATR-FTIR still requires crushing and homogenization of the powder samples, resulting in loss of spatial and heterogeneity information. This approach requires that surfaces be highly polished, and even then, the quality of the DRIFTS spectra tends to be much lower than the spectra acquired using ATR. The novel micro-FTIR experiment provides a direct measurement technology of mineral distribution in micro- to nanoscales [22]. FTIR microscopy does not require the samples to be pulverized. The principle of micro-FTIR is based on the specific absorption characteristics of infrared rays by chemical bonds in macerals and minerals [34, 35]. Previous research has used spatially resolved FTIR to create three component mineralogy maps [28]. This allows for localized measurements to characterize composition heterogeneous in very small scales. Therefore, multiple micro-FTIR measurements across a sample can be performed rapidly and precisely.

Therefore, micro-FTIR experiment is suitable for higher-resolution characterization of mineral distribution heterogeneity of shale. Another advantage of micro-FTIR spectrum is that in situ measurements can be carried out at submicron scale, which is particularly effective for analyzing mineral heterogeneity in fine-grained gas shales [22].

In this study, two different lacustrine shale samples from the Triassic Yanchang Formation in Ordos Basin were analyzed by micro-FTIR, which provided more detailed data of heterogeneous mineral composition and rock texture and structure. The objectives of this paper are as follows: (1) to study the micromineral heterogeneity of shale by micro-FTIR experiments, (2) to quantitatively characterize the in situ mineral composition of shale by micro-FTIR spectra, and (3) to compare the micro-FTIR results with the traditional bulk shale XRD method.

2. Samples and Methods

Two lacustrine shale samples with different rock texture features from the Triassic Yanchang Formation, Ordos Basin, were selected after carefully examining more than 20 shale samples. These samples were collected in Well FY-2 (Figure 1). The study area is located on the southeast Yishan slope of the Ordos Basin (Figure 1(b)). The target reservoir is the 7th member of the Yanchang Formation (Chang-7 member) of the Upper Triassic strata (Figure 2).

The Ordos Basin, located on the northwestern North China plate, can be divided into six structural units: the

FIGURE 2: Stratigraphic column and depositional environment of the upper Triassic Yanchang Formation in the Ordos Basin, NW China.

Yimeng uplift zone in the north, the Weibei uplift zone in the south, the Jinxi flexural fold zone in the east, the Yishan slope, the Xiyuan obduction zone, and the Tianhuan depression in the west (Figure 1(a)). Late Triassic strata formed in a large-scale inland freshwater lacustrine environment in the internal part of the Ordos Basin. The Chang-7 member deposits consist of deep and semideep facies that are mostly black or blackish gray organic-rich shales, which are primary source rocks in the study area [36, 37].

Each sample was separated into several parts for bulk mineral composition, thin section petrological observation, and micro-FTIR spectra measurements. Thin sections of the lacustrine shale samples were prepared with a thickness of about 80 μm . Petrological observation on the thin sections

was performed on an OLYMPUS microscope with crossed polarized light and plane polarized light. Additional samples were prepared as blocks, then were polished for an ultrasmooth surface. A micro-FTIR spectra experiment was performed using a Bruker Lumos FTIR microscope (Bruker Optics, Billerica, MA). A total of 200 spectra (100 spectra for each sample) were collected. There was a micro-FTIR measurement spacing interval of 80 μm across each sample (for a total of 8 mm in length) to get the in situ heterogeneous characteristics in shale samples (red blocks in Figure 3).

Each spectrum ranges from 400 to 4000 cm^{-1} , with a resolution of 4 cm^{-1} . A nonnegative least squares (NNLS) curve-fitting method was used to quantify the matrix components in samples [21]. Standards for composition calibration

FIGURE 3: Photos of cores and thin sections of shale samples L-1 and L-2. (a) Silty laminated shale (sample ID L-1, 1400 m). (b) Massive argillaceous shale (sample ID L-2, 1625 m). (c) Thin section images of sample L-1, showing silty laminae in silty laminated shale that can be seen by naked eyes. (d) Thin section images of sample L-2, showing argillaceous shale without silty laminae.

TABLE 1: Mineral composition and geochemical characteristics of the Yanchang shale samples.

Sample ID	Lithology	Depth (m)	TOC (%)	Ro (%)	Mineral content (%)					
					Quartz	Feldspar	Calcite	Dolomite	Pyrite	Clay
L-1	Silty laminated shale	1400	1.7	0.93	35.2	11.1	7.1	2.2	3.1	41.1
L-2	Massive argillaceous shale	1625	3.5	1.02	25.3	15.8	5.2	0	4.2	48.8

followed published databases [21–23, 34–38]. Averaged spectra were used for the prediction of bulk mineral compositions and compared with bulk XRD results.

3. Results

3.1. Bulk Mineral Compositions and TOC Contents. Mineral compositions and TOC contents of the lacustrine Yanchang shale samples are listed in Table 1. The TOC of the massive argillaceous shale is 3.5%, relatively richer in OM than the silty laminated shale with a TOC value of 1.7%. The equivalent Ro values of the two samples are both in the oil generation window (0.9–1.1%). XRD results show that the mineral compositions in the Yanchang shale are dominated by quartz

and clay minerals. The silty laminated shale has higher quartz contents, while the massive argillaceous shale has higher feldspar and clay contents. Both samples have relatively high clay contents (41.1% and 48.8%). For the silty laminated shale, the quartz, feldspar, carbonate, and pyrite contents are 35.2%, 17.1%, 2.2%, and 3.1%. For the massive argillaceous shale, the quartz, feldspar, carbonate, and pyrite contents are 25.3%, 15.8%, 5.2%, and 4.2%.

3.2. Mineral Information Derived from Micro-FTIR. Laminae of the sample L-1 can be clearly seen (Figure 3(a)). Silty laminae are commonly grayish-white and are different from adjacent clayey laminae in grain size and mineral composition. Most grains in silty laminae range from 40 μm to 160 μm in size, while grains in clayey laminae are mostly less

FIGURE 4: Micro-FTIR spectra of selected measurement spots, see location in Figures 1(c) and 1(d).

than $6\text{ }\mu\text{m}$ in size (Figure 3(c)). The detrital grains and the mud matrix or organic matter are mixed and poorly sorted in the massive argillaceous shale, and the grain sizes are mostly less than $10\text{ }\mu\text{m}$ (Figure 3(d)).

The micro-FTIR spectra measured for ten selected spots on the polished surface of the shale samples L-1 and L-2 are shown in Figure 4.

The spectra are highly diverse even when the measured spots are closely located. Micro-FTIR is a surface technique involving specular reflectance, which can produce derivative-shaped peaks or apparent absorbance dips [39]. A previous study has reported the characteristic peaks in micro-FTIR spectra; for example, carbonates have characteristic peaks at 1630 cm^{-1} , 900 cm^{-1} , and 720 cm^{-1} ; phyllosilicates display characteristic peaks at $1280\text{--}1150\text{ cm}^{-1}$ and $1060\text{--}1020\text{ cm}^{-1}$; and quartz expresses a unique peak at 1360 cm^{-1} [21] (Table 2).

Mineral composition data of the selected measured spots obtained by NNLS processing are listed in Table 3. The sum of the mineral phases should ideally reach 100 vol.%, but actually accounted for 80%-90%. This indicates that some minor minerals like phosphates are ignored during the model prediction process. The selected spots in the silty laminated shale sample (L-1) have relatively high quartz (28.26-42.39 wt%), feldspar (11.95-14.24 wt%), and clay mineral (36.39-44.09 wt%) contents. A higher clay mineral content (44.01-49.64 wt%) was determined in the massive argilla-

ceous shale sample (L-2) than in the silty laminated shale sample (L-1). In addition, there are large variations in mineral content within a microscale region in both L-1 and L-2.

4. Discussion

4.1. Quantification of Mineralogy through Micro-FTIR Spectra. The KBr FTIR spectra of standard minerals and kerogen have been published [21]. Previous studies presented that the characteristic infrared absorption band range of carbonate minerals is between 1500 and 1400 cm^{-1} , mainly due to the vibration of the internal Co bond [21–23]. The infrared absorption characteristics of silicate minerals are between $1200\text{--}800\text{ cm}^{-1}$ and $600\text{--}400\text{ cm}^{-1}$. The peak at $798\text{--}780\text{ cm}^{-1}$ corresponds to the tetrahedral bridging bond in quartz. Phyllosilicates can be separated from quartz according to the occurrence of minerals. The results of tensile vibration of the hydrogen oxygen bond are $3750\text{--}3400\text{ cm}^{-1}$. The characteristic infrared absorption band of kerogen is in the wavelength range of $1800\text{--}1000\text{ cm}^{-1}$ (hydrocarbon bond stretching of aliphatic hydrocarbon) [21].

In order to constrain the quantitative analysis of mineral components in rocks, the FTIR spectra of shales and pure minerals have been carefully compared by previous studies, such as the relative contents of clay, quartz, carbonate, and organic matter in shales [20–23]. The overlapping of infrared bands of some analytes and the resulting errors are inevitable.

TABLE 2: Micro-FTIR spectra peaks of specific minerals.

Minerals	Carbonates	Phyllosilicates	Hydrogen oxygen	Kerogen	Quartz	Silicate
Micro-FTIR spectra peaks (cm ⁻¹)	1600, 900, and 720	1280–1150 and 1060–1020	3750–3400	1800–1000	1360	1200–800 and 600–400

TABLE 3: Mineral composition calculated from micro-FTIR spectra of the Yanchang shale samples.

Sample ID	Spot ID	Mineral composition (wt%)								
		Quartz	Feldspar	Calcite	Dolomite	Pyrite	Clay	Illite/smectite	Kaolinite	Chlorite
L-1	#1	32.07	14.24	—	1.3	—	42.54	32.66	—	8.36
	#2	28.26	11.95	—	—	3.2	39.75	24.71	6.54	7.27
	#3	42.39	12.68	—	2.2	—	36.39	24.80	—	10.80
	#4	38.60	13.80	4.95	—	2.05	44.09	31.57	—	11.79
	#5	37.94	13.64	4.1	—	3.11	43.06	26.18	—	15.77
L-2	#6	34.77	15.65	—	—	0.21	47.47	25.06	—	16.36
	#7	26.09	15.29	5.33	—	0.44	44.01	33.06	—	10.64
	#8	36.56	13.11	—	1.6	—	49.64	21.41	—	12.53
	#9	30.24	10.12	—	11.04	—	42.97	27.18	—	—
	#10	31.47	12.51	4.01	—	1.8	45.67	22.71	8.22	—

Some studies have shown that in the middle range of the infrared wavelength, the mineral concentration of an artificial mixture can be quantitatively obtained from the spectrum of mineral standards [34, 35]. The complexity of the matrix components of gas-bearing shale brings challenges to the quantitative study of FTIR minerals. The deviation between the standard spectral characteristics of a single mineral and the actual shale samples easily leads to systematic error, resulting in the total mass fraction of mineral components in shale less than 100 wt.% [21]. In addition, the omission of some trace minerals (such as anatase, phosphate, and pyrite) will also lead to the underestimation of FTIR [21–23]. Despite these potential problems, FTIR quantitative and independent parallel analysis by XRD and elemental chemistry yielded comparable data for quartz and carbonate content, which XRD showed was higher than FTIR and elemental analysis. The results of FTIR and XRD are also comparable in clay content. The chemical spectroscopy method for quantitative analysis of shale components is limited by its basic assumptions. The compounds are converted to mineral concentrations using equations based on the ratio of possible pure mineral elements.

4.2. In Situ Heterogeneity Characteristics. In the present study, the heterogeneous distribution of mineral compositions in the lacustrine shale samples (red blocks in Figure 3) were derived from the totally obtained 200 micro-FTIR spectra and shown in Figure 5.

The spacing of 80 μm for each micro-FTIR measurement spots were selected to get in situ heterogeneous characteristics in shale samples. Therefore, micro-FTIR spectra measurements are able to capture the specific information expressed by the heterogeneity at high spatial resolution [21].

In general, silty laminae with higher quartz content (40–60 wt %) had lower concentrations of clay minerals. It was observed that, despite general agreement between the averaged FTIR results and the XRD data (Table 1), there are large standard deviations for most minerals. The mean value determined from micro-FTIR predicted values are compared with the XRD-measured bulk sample values (Figure 6). Overall, the mineral compositions calculated from the micro-FTIR compared well to the bulk XRD results.

Recent studies have shown that shale has strong heterogeneity in mineral composition, structure, and pore structure [40–48]. The lighter layers contain more quartz and less clay minerals than the darker ones. Changes in mineralogical composition from microscopic FTIR spectra along the length of the sample are clearly shown (Figure 5). Color changes in the measurement area are noted in the mineral composition derived from microscopic FTIR, which is reflected in mineralogical changes along the measurement area. The quartz and clay mineral compositions most clearly show the layered structure of the L-1 sample (Figure 5). The distribution of quartz and clay shows the layered structure of the L-1 sample most clearly, showing alternating distribution of high concentration and low concentration along the y -axis perpendicular to the bedding plane. Laminae is attributed to seasonal cycles [49–50], which may explain fluctuations in organic and mineral content.

At the micron scale, the regular changes of mineral composition and distribution obtained by microscopic FTIR spectra (Figure 5) can be used to precisely understand the heterogeneity and high-quality reservoir characterization of shales. In addition, minerals are the result of sensitive changes in physical and chemical conditions due to depositional environment evolution. In the shale gas production process, large

FIGURE 5: Distribution of mineralogy composition along the measured region (red blocks in Figure 1) of the (a) silty laminated sample and (b) the massive argillaceous shale.

FIGURE 6: Histograms showing the maximum, minimum, and mean values of (a) silty laminated sample L-1 and (b) massive argillaceous shale L-2. The mean value determined from micro-FTIR-predicted values are compared with the XRD-measured bulk sample values.

changes may occur, which may lead to more changes in cross well productivity [2–4]. Therefore, a better understanding of the changes in development and production can provide important guidance for the efficient development of lacustrine shale gas. Considering the small mismatch value and good fit-

ting, we believe that the micro-FTIR technique provides an effective method for quantitative evaluation of the shale matrix at a micron scale. Two main advantages of the micro-FTIR are proposed. Firstly, this is an in situ technique to avoid destructive and potentially contaminated grinding of the sample. It

can be used to analyze in situ shale samples with constant kerogen mineral assemblage and spatial information. Secondly, the micro-FTIR spectra measurements can capture the abundant information expressed by heterogeneity at high spatial resolution.

5. Conclusions

Based on the above study, the following conclusions can be drawn:

- (1) Mineral compositions in the Yanchang shale are dominated by quartz and clay minerals. The massive argillaceous shale is OM richer than the silty laminated shale. The silty laminated shale has higher quartz contents, while the massive argillaceous shale has higher feldspar and clay contents
- (2) The micro-FTIR results are basically consistent with the bulk shale XRD results. The analyzed massive argillaceous shale and silty laminated shales in the study area show obvious heterogeneity within the measurement interval of 80 μm . The mineral composition of homogeneous samples observed by naked eyes is also strongly heterogeneous when measured by the micro-FTIR technique
- (3) The micro-FTIR technique provides a fast and non-destructive method to determine the heterogeneity of mineral components in shale samples. The mineral composition information of sample heterogeneity can be obtained by multiple measurements
- (4) The micro-FTIR techniques can precisely characterize the in situ compositional heterogeneity of shale samples and capture abundant information expressed by shale heterogeneity while providing resolution for shale mineralogy research

Data Availability

The mineral composition, geochemical characteristics, and micro-FTIR spectra experiment result data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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