# Mineral characteristics and their geological significance of black shales in southeastern Ordos Basin by X-ray diffraction analysis

YAO Zhigang\*, YANG Yang, YING Huawei, and DONG Yunpeng

School of Earth Sciences and Engineering, Xi'an Petroleum University, Xi'an 710065, China \* Corresponding author, E-mail: yzg123-68@163.com

Received May 28, 2013; accepted July 29, 2013 © Science Press and Institute of Geochemistry, CAS and Springer-Verlag Berlin Heidelberg 2014

**Abstract** X-ray diffraction analysis of black shale of Upper Triassic Member Chang 7 of the Yanchang Formation in southeastern Ordos Basin showed that black shales were deposited in brackish, strongly reducing, semi-deep-deep lacustrine facies, and mainly composed of quartz, feldspar, carbonate (dolomite), clay minerals (illite and il-lite/smectite) and a certain amount of pyrite. The mineral composition characteristics of this set of black shales are similar to those of highly productive shale gas in North America, for example shallow burial, low clay mineral and abundant brittle mineral, so the strata are conducive to the development of cracks and fractures. Thus, this area is favorable for shale oil/gas exploration and development.

Key words mineral composition; shale gas/oil; southeastern Ordos Basin

## **1** Introduction

Member Chang 7 of the Upper Triassic Yanchang Formation in southeastern Ordos Basin is a set of thick, black, dark gray mudstones formed during the largest expansion of lake basin in deep water, which are the main source rocks of the Ordos Basin in Mesozoic. The study area is located in Tongchuan City (southeastern Ordos Basin). In the lower-middle section of the Triassic Member Chang 7 of the Yanchang Formation are extensively developed shallow and organic-rich black shales favorable for shale oil/gas exploration and development. Samples for this analysis were selected from core rocks of Member Chang 7. By using X-ray diffraction instrument this paper systematically analyzed the mineral composition of black shales (according to SY/T 6210-1996), and explored the geological significance.

# 2 Sampling

The black shales in the Tongchuan area are black, dark brown thin-layered shales in surface, but show black massive mud (shale) underground. Eleven samples were selected for this analysis, and they were analyzed in a whole-rock quantitative manner using X-ray diffraction instruments for their mineral compositions (Table 1).

#### **3** Mineral compositions

There is no general classification scheme for mineral compositions of source rocks in oil industry at present time (Fu Xiaodong et al., 2011). For the convenience of analysis, this paper divided major minerals in black shales into four categories: (1) detrital minerals, including quartz, plagioclase, potassium feldspar, etc.; (2) carbonate minerals, including calcite and dolomite; (3) clay minerals, including illite, smectite, chlorite, kaolinite and mixed-layer minerals; and (4) other minerals, such as pyrite (Table 1).

### 3.1 Detrital minerals

Table 1 and Fig. 1 show that samples of Member Chang 7 black shales generally contain a high content of detrital minerals, mainly quartz and feldspar. In the analyzed samples, quartz content ranges from 20% to 30%, or more than 35%, with the average content of 36%, and the maximum value of 64.3%. And for plagioclase, its content ranges mainly from 10.0% to 15.0%, or more than 35% with the average content of 16.7%, and the maximum value of 29.5%. The content of potassium feldspar varies from 6.3% to 17.3%.



	Sample				Mineral composition of the whole rock									
No.	Well	Depth (m)	Quartz	Plagioclase	Potassium feldspar	Calcite	Ankerite	Siderite	Pyrite	Illite/ Smectite	Illite	Illite 1 Md	Chlorite	Kaolinite
1	683. 690.4 TH 3 695.4 698. 701.4	683.1	32.0	29.5	11.0		1.2		11.3	13.0			2.0	
2		690.8	39.0	14.2	17.3		4.6		13.9	10.0			1.0	
3		695.0	42.7	26.2	11.6		2.0		9.5	7.0			1.0	
4		698.1	28.7	22.9			1.7		16.7			18.0	2.0	1.0
5		701.5	34.9	16.0			3.7		19.4		2.0	15.0	3.0	6.0
6		252.5	64.3	8.3	9.6		1.2	1.0	3.6		1.0	10.0	1.0	
7	25 TH 2 26 26	253.5	23.8	11.0				1.4	36.8	20.0			4.0	3.0
8		260.9	25.8	11.4	6.3		0.6	1.0	26.9		3.0	23.0	2.0	
9		262.5	57.6	15.3			3.7		4.0			8.0	1.0	
10		268.8	19.3	17.0			0.8		23.6		1.0	17.0		4.0
11		2723	27 /	12.0		0.6		26	26.4		2.0	26.0	3.0	

 Table 1
 Mineral composition of black shales in the Tongchuan area



Fig. 1. Distribution histograms of the content of main detrital minerals in the Tongchuan area.

Quartz content is an important factor affecting the fracture development, thus the quartz-rich black shale section is more brittle (Nie Haikuan et al., 2009). Take the famous high-yield Barnett Shales of the Fort Worth Basin for example. Their quartz content varies mainly from 20.0% to 70.0% (Loucks and Ruppel, 2007; Jarvie et al., 2007), close to 19.3%– 64.3% in the study area. Therefore, black shales in the Tongchuan area are brittle and easy to develop fractures.

#### 3.2 Carbonate minerals

Earlier studies showed that mudstone with a higher content of carbonate minerals should be one of the characteristic features of lacustrine argillaceous sediments (Miao Jianyu et al., 2003). Black shales in the Tongchuan area contain carbonate minerals in various degrees (Table 1, Fig. 2). Their ankerite content varies mainly from 0.5% to 1.5%, accounting for more than 30% of the analyzed samples; siderite content varies from 1.0% to 2.6%, and calcite is rarely seen. According to previous geological and geochemical studies (Miao Jianyu et al., 2005) the depositional environment of black shales in the Tongchuan area is of semi-deep-deep lacustrine facies. Nelson (1985) considered that feldspar and dolomite were also brittle components in shales besides quartz. Brit-

tle minerals such as quartz, feldspar, carbonate minerals, etc. rich in shales are prone to create fractures (Li Xinjing et al., 2007). As seen in Fig. 3, the total content of brittle minerals mainly varies from 30% to 40% accounting for more than 25% of the samples, and for another 25% samples the content varies from 70% to 80%. Thus, brittle mineral content of black shales in the Tongchuan area is high.

#### 3.3 Clay minerals

Clay minerals are the main constituents of argillaceous sediments and argillaceous rocks (Zhang Yonggang et al., 2004). Analytical results of core samples show that clay mineral content of black shales in the Tongchuan is within the range of 8.0% to 31.0%, mainly from 10% to 15% and 25% to 30%, accounting for more than 50% of the analyzed samples (Fig. 4). Clay minerals are chiefly illite/smectite mixture and illite, with minor kaolinite and chlorite. Illite/smectite content ranges from 7% to 20%; those of illite, 1% to 13%, with the average content of illite 1Md (illite crystals have 1 M, 2 M, 1 Md and 3 T variants) of 16.7%; those of kaolinite, 1% to 6%; and those of chlorite, 1% to 4% (Fig. 5). This reflects the black shales were formed in the middle period of digenesis (CNPC criteria, 2003), and clay mineral content is generally not high.

Figure 6 shows that the correlation between total clay minerals and quartz content is inverse correlation, and ankerite is fairly weak, indicating that the total amount of clay minerals in the black shales is mainly controlled by sedimentary environment (Li Zhiming et al., 2010).

# 3.4 Other minerals

Some of the analyzed core rocks (not black oil shale) in the Tongchuan area contain a small amount of halite, suggesting that the deposition environment there is characterized by brackish lake water. In addition, most of the samples contain pyrite whose content is within the range of 4.0%–36.8% (Fig. 7), indicating that the black shales in the study area were deposited in a strongly reducing environment. This conclusion is in line with other analyses of sedimentary facies in this area (Yang Hua et al., 2010).

## 4 Comparison with North American shales

Mineral compositions of black shales are compared between the study area and the main North American shale area (Table 2), showing similar characteristics: high content of brittle minerals (quartz, feldspar, etc.) and low content of total clay minerals (usually below 30%).



Table 2 Mineral composition comparison between the Tongchuan area and North American shales

Shale	Clay mineral content (%)	Quartz content (%)
Barnett shale, Fort Worth Basin (Loucks et al., 2007; Jarvie et al., 2007)	20%-60%	20%-70%
Devonian-Mississippian mud rock, Seaga Basin (Ross et al., 2008)	5%-80%	48.9%
Green River shale, Uinta Basin (Hunt, 1996)	<10%	
Heather shale, North Sea region (Hunt, 1996)	<5%	53%-57%
Woodford shale, Oklahoma area (Isaacs, 1987)		85%-95%
Bakken shale, Willistton Basin (Hao Fang, 2005)	$<\!\!20\%$	
Tongchuan black shale, Ordos Basin	8%-31%	19%-64%



Fig. 7. Pyrite content distribution histogram in the Tongchuan area.

# **5** Geological significance

# 5.1 Shale gas show

In the exploration and development of southeastern Ordos Basin, shale gas outflow was encountered during well drilling process. When the core was put into the tube, big bubbles and many gas outflow points were observed (Fig. 8), especially along the bedding plane.

Test Analysis shows that bubbles releasing from the well core are not only adsorbed gas, but also free gas. Gas composition analysis indicates it is mainly hydrocarbon gas, namely, the average content of 72.24% (methane), 11.51% (ethane), 5.91% (propane), 0.54% (*n*-butane), and a small amount of pentane and hexane.

#### 5.2 Gas-bearing characteristics

Gas-bearing capability of shales is an important parameter to evaluate shale gas resources (Zhang Jinchuan et al., 2004). Recently, there are many reports on Yanchang Formation shale gas, such as small fracturing test and successful ignition of Member Chang 7 of Well Liuping 177 in the Xiasiwan region of Ganquan County by Yanchang Petroleum Corporation, while Well Xin 57 succeeds in gas producing by fracturing. In addition, black shales of Members Chang 7 and 8 (910-960 m) of Well Zhongfu 18 in the Fuxian area show obviously abnormal gas survey and high impedance of deep response curve; Member Chang 7 muddy shale gas of Well Zhuang 167 and Well Zhuang 171 also shows abnormal gas survey (Xu Shilin et al., 2009), which indicates good gas bearing, and favorable exploration environment of Member Chang 7 black shale oil and gas.

#### 5.3 Mineral composition and shale exploration

Brittle mineral content is an important factor of matrix porosity, micro-fracture development, gas bearing and fracturing reformation of shales. Low content of clay minerals and high content of brittle minerals make rocks become more brittle. In such circumstances, rocks are more easily to create natural fractures and induced fractures under artificial fracturing forces to form structural joints with tree network, which is conducive to shale gas exploration (Zou Caineng et al., 2010). Black shales in the Tongchuan area are rich in brittle minerals, thus conducive to fracturing.



Fig. 8. Water immersion test of Chang 7 well core.

#### 5.4 Mineral composition and cap rock sealing

Cap rocks of natural gas reservoir are mainly argiloids. 60% of 34 large- and medium-sized gas fields in China are in mud shale cap rocks (Cheng Rongshu, 1989; Zhang Houfu, 1999; Hu Guoyi et al., 2009). Rich organic argillaceous rocks are not only high-quality source rocks, but also prominent cap rocks. Sealing ability of argiloid cap rocks is inseparable with mineral compositions which can affect the plasticity of clay shales, wettability and water absorption, mineral grain structure and orientation, as well as porosity and permeability (Huang Haiping et al., 1995). Usually clay minerals, in plasticity-reducing order of smectite, illite/smectite, illite, and chlorite, have greater plasticity than detrital minerals, (Zhang Changjiang et al., 2008). Argillaceous rocks of the Yanchang Formation in the Tongchuan region, Ordos Basin, extend widely and are relatively thick (Miao Jianyu et al., 2005). However, the content of clay minerals in the study area ranges from 8% to 31%, rich in clay minerals, mainly illite/smectite mixed layer, illite and illite 1Md crystal variant, but less in kaolinite and chlorite. As a result, the sealing ability of black shales is preferable in the Tongchuan area.

# 6 Resource assessment and exploration potential forecast

Shale gas reservoirs are of the self-generation and self-storage type (Zhang Jinchuan et al., 2008). The accumulation mode requires thick shale bed, wide distribution area, moderate burial depth, high total organic carbon content, good organic matter maturity, as well as relatively good reservoir porosity, permeability, and micro-fracture (Zhang Jinchuan et al., 2008; Pu Boling et al., 2008; Chen Gengsheng et al., 2009). After detailed study of Well Chang 7 core, well logging, analysis and test, it is estimated that shale gas resources in southeastern Ordos Basin are  $4.8 \times 10^{12}$ – $6.3 \times 10^{12}$  m<sup>3</sup>. The potential of shale gas exploration and development is huge.

# 7 Conclusions

(1) Black shales in the Tongchuan area are dominated by detrital minerals (quartz, plagioclase, and potassium feldspar), clay minerals (mainly illite and illite/smectite) and carbonate minerals (dolomite, siderite). The pyrite content is high and partially, there is small amount of rock salt in black shale samples, which indicates black shales in the study area are deposited in semi-deep-deep brackish lacustrine reducing water. This conclusion provides another piece of evidence of X-ray diffraction for sedimentary facies analysis in this area.

(2) Mineralogical characteristics of black shales in the Tongchuan area are similar to those in North America with regard to high-yielding shale gas, rich brittle minerals and low clay minerals. Therefore, it is favorable for hydrocarbon exploration.

(3) Black shales in the study area have various clay minerals, such as illite/smectite mixed layer and illite 1 Md crystal variant, kaolinite and chlorite with strong plasticity. So black shales in the Tongchuan area have a preferable sealing ability.

(4) Member Chang 7 shale gas of the Yanchang Formation in southeastern Ordos Basin shows good gas survey and high gas content, so the preliminary evaluation of Member Chang 7 black shales is: high gas-bearing shale gas reservoir. Moreover, gas test has obtained industrial gas flow, and indicates favorable exploration prospects.

Acknowledgements This research project was financially supported by China Geological Survey and the Ministry of Land and Resources of China (No. 1212011120963). X-ray diffraction analysis of all samples was carried out at the State Key Laboratory of Continental Dynamics. The field work in Tongchuan, Ordos Basin, was greatly assisted by Li Yuhong, Li Jinchao and Zhang Huiyuan. The paper has benefited from instructive discussion with Prof. Wu Fuli. Thanks are due to the reviewers for their helpful suggestions.

#### References

- Chen Gengsheng, Dong Dazhong, and Wang Shiqian (2009) Formation mechanism and concentration patterns of shale gas [J]. Gas Industry. 29, 17–21 (in Chinese).
- Cheng Rongshu (1989) *Natural Gas Geology* [M]. China University of Geosciences Press, Wuhan (in Chinese).
- Fu Xiaodong, Qin Jianzhong, Tenger, and Wang Xiaofang (2011) Mineral components of source rocks and their petroleum significance: A case from Paleozoic marine source rocks in the Middle-Upper Yangtze region [J]. *Petroleum Exploration and Development*. 38, 671–684 (in Chinese).
- Hao Fang (2005) Hydrocarbon-generating Dynamics and Formation Mechanism of Oil and Gas Reservoirs for Overpressure Basin [M]. Science Press, Beijing (in Chinese).
- Hu Guoyi, Wang Xiaobo, and Wang Yifeng (2009) Cap rock characteristics of medium and large gas fields in China [J]. *Natural Gas Geoscience*. 20, 162–166 (in Chinese).
- Huang Haiping and Deng Hongwen (1995) Confining behavior of shale cap and its control factors [J]. *Natural Gas Geoscience*. 6, 20–26 (in Chinese).
- Hunt J.M. (1996) Petroleum Geolgoy and Geochemistry [M]. Freeman Company, San Fransisco.
- Isaacs C.M. (1987) Source and Deposition of Organic Matter in the Monterey Formation, South-Central Coastal Basins of California [C]. pp.93–205. AAPG. Meyer R.F. Exploration for heavy crude oil and natural bitumen, Tulsa.
- Jarvie D.M., Hill R.J., and Ruble T.E. (2007) Unconventional shale-gas systems: The Mississippian Barnett Shale of north-central Texas as one model for thermogenic shale-gas assessment [J]. AAPG Bulletin. 91, 475–499.
- Li Xinjing, Hu Suyun, and Cheng Keming (2007) Suggestions from the development of fractured shale gas in North America [J]. *Petroleum Exploration and Development.* **34**, 392–400 (in Chinese).
- Li Zhiming and Yu Xiaolu (2010) The Characteristics of mineral components for effective source rocks from dongying depression of Bohai Bay Basin and its significance [J]. *Petroleum Geology & Experiment*. 32, 270–275 (in Chinese).
- Loucks R.G. and Ruppel S.C. (2007) Mississippian Barnett shale: Lithofacies and depositional setting of a deep-water shale-gas succession in the Fort Worth Basin, Texas [J]. *AAPG Bulletin.* **91**, 523-533.
- Miao Jianyu, Zhu Zongqi, and Liu Wenrong (2003) The pore structure characteristics of Paleogene-Neogene mudstone in Jiyang depression [J]. *Geological Review.* 49, 330–336 (in Chinese).
- Miao Jianyu, Zhao Jianshe, and Li Wenhou (2005) Reseach on the diposite environments about source rocks in south Ordos Basin [J]. Journal of Northwest University (Natural Science Edition). 35, 771–776
- Nelson R.A. (1985) Geologic Analysis of Naturally Fractured Reservoirs: Contributions in Petroleum Geology and Engineering [M]. Gulf Publishing Company, Houston.
- Nie Haikuan, Tang Xuan, and Bian Ruikang (2009) Controlling factors for shale gas accumulation and prediction of potential development area in shale gas reservoir of South China [J]. *Journal of Petroleum Institute*. **30**, 484–491 (in Chinese).
- Pu Boling, Bao Shujing, and Wang Yi (2008) Accumulation condition of shale gas reservoir—U.S. shale gas basin [J]. Petroleum Geology and

Engineering. 22, 33-36 (in Chinese).

- Professional Standardization Committee for Petroleum Exploration (1996) Quantitative Analysis Method by X-Ray Diffraction of Total Clay Minerals Content and Non-clay Minerals in Sedimentary Rocks [S]. China National Petroleum Corporation, Beijing (in Chinese).
- Ross D.J.K. and Bustin R.M. (2008) Characterizing the shale gas resource potential of Devonian-Mississippian strata in the Western Canada sedimentary basin: Application of an integrated formation evaluation [J]. AAPG Bulletin. 92, 87–125.
- Xu Shilin and Bao Shujing (2009) Preliminary analysis of shale gas resource potential and favorable areas in Ordos Basin [J]. *Natural Gas Geoscience*. 20, 460–465 (in Chinese).
- Yang Hua, Dou Wei Tan, and Liu Xianyang (2010) Triassic Yanchang seven sedimentary facies analysis [J]. *Journal of Deposition Institute*. 28, 254–263 (in Chinese).
- Zhang Changjiang, Pan Wenlei, and Liu Guangxiang (2008) Dynamic evaluation to the cap formation of Silurian argillaceous rock, southern

China [J]. Natural Gas Geoscience. 19, 301–310 (in Chinese).

- Zhang Houfu (1999) *Oil Geology* [M]. Petroleum Industry Press, Beijing (in Chinese).
- Zhang Jinchuan, Jin Zhijun, and Yuan Mingsheng (2004) Reservoir mechanism and distribution of shale gas [J]. *Nature Gas Industry*. **24**, 15–18 (in Chinese).
- Zhang Jinchuan, Wang Zongyu, and Nie Haikuan (2008) Shale gas and the exploration significance [J]. Modern Geology. 22, 640–645 (in Chinese).
- Zhang Jinchuan, Xu Bo, and Nie Haikuan (2008) The potential of shale gas resource exploration [J]. *Natural Gas Industry*. 28, 136–140 (in Chinese).
- Zhang Yonggang and Cai Jingong (2004) *The Mechanism of Organic Concentration in Mudstone* [M]. Science Press, Beijing (in Chinese).
- Zou Caineng, Dong Dazhong, and Wang Shejiao (2010) Geological characteristics, formation mechanism and resource potential of shale gas in China [J]. *Petroleum Exploration and Development*. **37**, 641–653 (in Chinese).