

Geochemical characteristics of Lower Permian coal-measure source rocks in Northeast Ordos Basin

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Abstract General characteristics and biomarker distributions of Lower Permian coal-measure source rocks in northeastern Ordos Basin have been analyzed in this paper. The results show that the source rocks are type III kerogen, thermally mature, with high content of total organic carbon. The extracts of the source rock samples with different lithologies from Well Su 27 feature high Pr/Ph ratios, high C₁₉TT and C₂₄Te, high rearranged hopanes, a predominance of C₂₉ sterane in regular steranes, and the absence of C₃₀ 4-methylsteranes. These data suggest that the source rocks were deposited in suboxic to oxic conditions with dominantly terrigenous higher plant input. In contrast, the source rocks from wells Shuang 1 and Yu 20, are characterized by low Pr/Ph ratios, low C₁₉TT and C₂₄Te, high C₂₃TT, similar content of C₂₇ sterane and C₂₉ sterane, and the presence of C₃₀ 4-methylsteranes, showing that the source rocks were deposited in reducing environment with algae and/or microorganisms and terrigenous higher plant input.

Key words coal-measure source rock; biomarker; Permian; Ordos Basin

1 Introduction

The coal system of Upper Paleozoic is the primary source rocks of the Upper Paleozoic gas reservoirs in the Ordos Basin (Xue et al., 2010; Dai et al., 2005). This coal-measure gas source rock is mainly consisted of coal, carbonaceous mudstone, and mudstone, which were formed in peat swamp environment (Wang et al., 2002).

At present, there is limited literature on the geochemical characteristics of coal measure source rocks from Upper Paleozoic, which only focus on basic geochemical evaluation such as the abundance, type and thermal maturity of organic matter, or the geochemical characteristics of the gas derived from the coal-measure source rocks (Dai et al., 2005; Miao et al., 2007; Hu et al., 2007; Li et al., 2008; Xue et al., 2010; Li et al., 2012). Research on the characteristics of biomarkers from coal measure source rocks and their molecular geochemical significance are seldom

reported.

Therefore, coal, carbonaceous mudstone, and mudstone samples from Shanxi and Shangshihezi formations in northeastern Ordos Basin were systematically collected. The molecular geochemical characteristics were studied in detail to illustrate various types of humic coal biomarker assemblages, and reveal the diversity of organic matter sources and sedimentary environments.

2 Geological setting

The Ordos Basin, lies in western North China Platform, is the second biggest depositional basin in NW China. Covering 370000 km², it has multiple tectonic systems, sedimentary evolution cycles and sedimentary types. According to the geologic evolution history and Mesozoic tectonics characteristics, the basin can primarily be divided into six secondary structure units, namely, Tianhuan Syncline, fault-fold

belt of west margin, Yimeng Uplift, Yishan Slope, Jinxi Fault-fold Belt, and Weibei Uplift (Zhang et al., 1997; Hou et al., 2004) (Fig. 1). It underwent the transformation history from offshore basin after epeiric sea basin to inland depression basin, and developed several sets of source rocks vertically: from bottom to up exhibits successively Upper Carboniferous Benxi Formation, Lower Permian Taiyuan Formation, Shanxi Formation, Upper Permian Lower Shihezi Formation, Upper Shihezi Formation, and Shiqianfeng Formation in Late Paleozoic.

3 Samples and methods

Twenty-five samples from Permian coal-measure source rocks were taken from drill cuttings of wells Shuang 1, Yu 20 and Su 27 in Northeast Ordos Basin. According to TOC content classification (Chen et al., 1997) and petrographical features, the samples in this study are referred as mudstones (<6%), carbonaceous mudstones (6%–40%) and coals (>40%). The burial depths of samples in the three wells range from

2594.43 to 2697.77, 2611.71 to 2763.9, 3155.03 to 3185.38 m, respectively. The geographic locations of the wells chosen for the study are shown in Fig. 1.

Rock-eval pyrolysis analysis and total organic carbon content were performed on all rock samples. Maceral composition, vitrinite reflectance, extraction and gas chromatography-mass spectrometry (GC-MS) analyses were conducted on eight typical source rock samples. Samples were extracted in a Soxhlet apparatus for 72 hours using chloroform. Extracts were decomposed into saturated hydrocarbons, aromatic hydrocarbons and NSO (nitrogen, sulfur and oxygen) compound fractions by liquid column chromatography. Saturated fractions were analyzed using an HP 6890 gas chromatograph and a 5973 mass spectrometer equipped with a HP-5MS fused silica capillary column (30 m×0.25 mm×0.25 μm). The GC for the analysis was temperature-programmed as being heated for 1 minute at 50°C, then increasing from 50°C to 100°C at a rate of 20°C/min, and from 100 to 315°C at a rate of 3°C/min and at 315°C for 18 minutes. Helium was used as the carrier gas with a rate of 1.0 mL/min and the ionization source was operated at 70 eV.

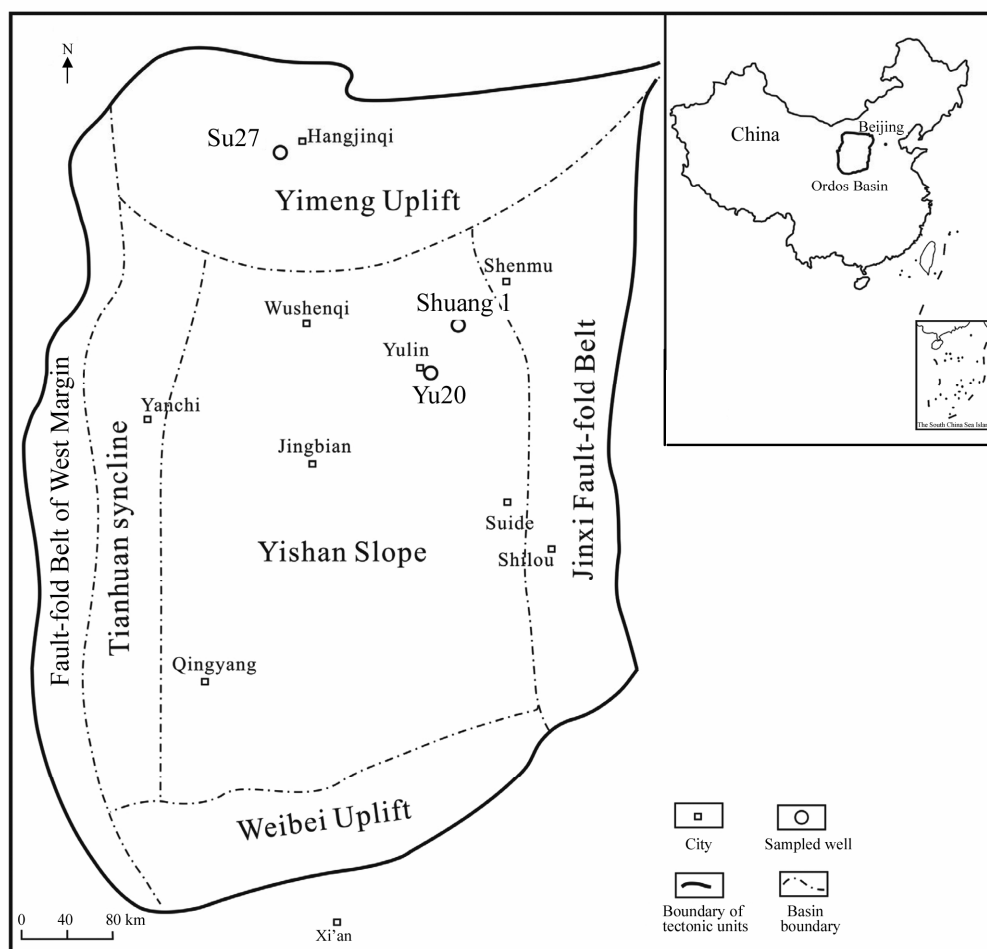


Fig. 1. Map showing the location of the sampled wells and structural divisions of Ordos Basin.

4 Results and discussion

4.1 Bulk composition and properties

The mudstones of Shanxi and Shangshihezi formations show variable TOC values, ranging from 0.11% to 3.04%, with a mean value of 1.49%. Their hydrocarbon-generating potential (S_1+S_2) values vary between 0.05 and 2.05 mg/g, with an average of 0.85 mg/g. The carbonaceous mudstones and coals display higher amounts of TOC (6.85%–72.62%) and S_1+S_2 (7.87–266.74 mg/g) than those of the mudstones. Coals and carbonaceous mudstone have hydrogen index (HI) values in the range of 159.21 to 338.07 mg/g, while the mudstone samples mainly lower than 50 mg/g (Table 1), implying a slightly hydrogen-rich kerogen and a relatively greater hydrocarbon-generating potential.

Vitrinite, generally classified as type III kerogen, is the most abundant macerals in all of the source rock samples (68.6%–99.08% by volume). The inertinite comes second, which ranges from 0% to 23.22%. The amount of liptinite and sapropelinite are relatively small. The kerogen type indices of all samples are less than 0, suggesting that the rock samples are type III kerogens.

The mean vitrinite reflectance data (R_o) for the rock samples from Su 27 vary between 0.82% and 0.86%, indicating that the samples are thermally mature. The R_o of the source rock samples selected from wells Shuang 1 and Yu 20 are higher, which range from 1.03% to 1.20%, suggesting the samples are thermally mature and have entered the peak mature stage of oil window.

4.2 *n*-alkanes and isoprenoids

The carbon numbers of *n*-alkanes in the extracts of coal-measure source rocks mainly range from C_{15} to C_{32} . The investigated samples are characterized by OEP and CPI values of 1.01–1.82 (mainly from 1.01 to 1.28) and 0.91–1.12, respectively, indicating that these rock samples are thermally mature.

The Pr/Ph ratio can be used to reflect the redox condition of depositional environment despite that it may be influenced by some other factors (Didyk et al., 1978). For instance, unsaturated isoprenoids in zooplankton (Blumer et al., 1963), higher animals (Blumer and Thomas, 1965), and tocopherols (Goossens et al., 1984) in the sediments may give rise to pristine and/or phytane. Crude oil Pr/Ph generally increases with increasing thermal evolution degree (Connan, 1974) while Ph/*n*- C_{18} ratio decreases (Ten et al., 1987). However, Pr/Ph ratio is still regarded as an effective macroscopic palaeoenvironment parameter (Wang, 1990).

Pr/Ph ratios of the coal-measure source rocks from Shanxi Formation are listed in Table 2. The source rock samples from well Su 27 are represented obviously by high Pr/Ph ratios with different lithologies (include coal and mudstone). It ranges from 0.87 to 3.35, mainly from 2.07 to 3.35. Whereas, the coal, carbonaceous mudstone, and mudstone samples from wells Shuang 1 and Yu 20 have low Pr/Ph ratios (<0.5). The maturity of the source rocks from Well Su 27 was lower than that of wells Shuang 1 and Yu 20, as mentioned above. So, the differences of Pr/Ph ratios are independent of maturation in this paper (Connan, 1974). The diversity of Pr/Ph ratios imply that the source rocks from Well Su 27 were deposited under oxic conditions, while the source rocks from wells Shuang 1 and Yu 20 were deposited in reducing environment.

4.3 Terpanes distribution

4.3.1 Tricyclic terpanes and tetracyclic terpanes

In addition to hopanes, tricyclic terpanes with high abundance are also observed in *m/z* 191 mass chromatograms of the saturated hydrocarbon fraction of the 8 source rock samples (Fig. 2).

Tricyclic terpanes are generally believed to be originated from algae or microorganisms (Aquino et al., 1983), and have strong thermal stability (Peters et al., 1990). Tricyclic terpanes with the carbon number below C_{21} are considered to be derived mostly from higher plants (Simoneit, 1977; Bao et al., 1999). Tricyclic terpanes demonstrate very similar distribution characteristic in the source rock samples with different lithologies from wells Shuang 1 and Yu 20, which are featured by the predominance of C_{23} compound (Fig. 2). In contrast, the tricyclic terpanes in coal and mudstone samples from well Su 27 are dominated by low molecular weight compounds (C_{19} or C_{20}).

Philp and Gilbert (1986) proposed that the proportion of tetracyclic terpanes relative to tricyclic terpanes is facies-dependent, and have relatively high concentrations in source rocks and oils with terrigenous organic matter input. C_{24} tetracyclic terpanes show low concentrations in extracts of the samples from wells Shuang 1 well and Yu 20, with $C_{24}Te/C_{26}TT$ ratios less than 0.65 (Fig. 3, Table 2). In contrast, $C_{24}Te/C_{26}TT$ ratios of the samples from well Su 27 demonstrate high values, mainly higher than 2.39.

Different distribution characteristics of tricyclic terpanes and C_{24} tetracyclic terpanes are in different wells (Figs. 2 and 3), interpret that source rocks from wells Yu 20 and Shuang 1 have high contribution of algae and/or microorganisms. While, source rocks from well Su 27 have high contribution of terrigenous organic matter.

Table 1 Pyrolysis data for source rock samples in this study

Well	Fm.	Depth (m)	Lithology	TOC (%)	S1 (mg/g)	S2 (mg/g)	T _{max} (°C)	S1+S2 (mg/g)	HI (mg/g)
Shuang 1	P ₂ sh	2594.43–2594.52	Mudstone	0.11	0.01	0.04	493	0.05	35.87
Shuang 1	P ₁ s	2619.35–2619.47	Mudstone	1.14	0.04	0.43	478	0.47	37.75
Shuang 1	P ₁ s	2651.48–2651.58	Mudstone	3.04	0.12	1.56	490	1.68	51.25
Shuang 1	P ₁ s	2653.40–2653.52	Coal	64.08	5.36	113.9	475	119.26	177.75
Shuang 1	P ₁ s	2654.3	Mudstone	0.23	0.01	0.05	492	0.06	21.73
Shuang 1	P ₁ s	2697.25–2697.37	Mudstone	0.30	0.02	0.07	522	0.09	23.22
Shuang 1	P ₁ s	2697.68–2697.77	Coal	33.05	3.24	66.21	478	69.45	200.33
Yu 20	P ₂ sh	2611.71–2613.69	Mudstone	2.44	0.09	1.61	477	1.70	65.90
Yu 20	P ₂ sh	2696.11–2701.95	Mudstone	2.20	0.11	0.99	486	1.10	45.10
Yu 20	P ₁ s	2703–2704.94	Mudstone	0.89	0.03	0.20	523	0.23	22.56
Yu 20	P ₁ s	2726.74–2730.62	Coal	55.78	4.58	88.81	476	93.39	159.21
Yu 20	P ₁ s	2732.2–2733.6	Mudstone	0.23	0.01	0.10	423	0.11	43.52
Yu 20	P ₁ s	2733.6–2734.84	Coal	62.22	1.07	6.80	475	7.87	10.93
Yu 20	P ₁ s	2734.84–2737.24	Mudstone	2.53	0.16	1.02	490	1.18	40.30
Yu 20	P ₁ s	2737.24–2742.45	Mudstone	1.47	0.07	0.52	499	0.59	35.30
Yu 20	P ₁ s	2744.4–2749.41	Carbonaceous mudstone	23.54	2.43	50.7	474	53.13	215.38
Yu 20	P ₁ s	2744.4–2749.41	Mudstone	1.59	0.06	0.95	481	1.01	59.82
Yu 20	P ₁ s	2744.4–2749.41	Mudstone	1.50	0.09	0.89	478	0.98	59.10
Yu 20	P ₁ s	2761.16–2763.9	Coal	65.67	2.34	56.14	490	58.48	85.49
Su 27	P ₁ s	3155.03–3155.97	Mudstone	2.505	0.13	1.92	455	2.05	76.65
Su 27	P ₁ s	3157.37–3158.08	Coal	44.09	6.46	130.06	446	136.52	294.99
Su 27	P ₁ s	3175.03–3175.49	Coal	72.62	21.23	245.51	445	266.74	338.07
Su 27	P ₁ s	3175.49–3177.42	Mudstone	2.20	0.19	1.26	466	1.45	57.40
Su 27	P ₁ s	3184.46–3185.06	Coal	63.14	13.52	196.11	450	209.63	310.60
Su 27	P ₁ s	3185.06–3185.38	Mudstone	6.848	0.78	16.52	450	17.30	241.24

Table 2 Geochemical parameters of typical coal-measure source rocks from Shanxi Formation in Northeast Ordos Basin

Well	Depth (m)	Fm	Lithology	a	b	c	d	e	f	g	h	i
Shuang1	2619.35–2619.47	P ₁ s	Mudstone	1.03	0.89	0.45	0.42	0.53	0.18	0.20	1.13	0.18
Shuang1	2697.68–2697.77	P ₁ s	Coal	1.15	1.02	0.48	0.54	0.14	0.14	0.31	0.83	0.08
Yu 20	2733.6–2734.84	P ₁ s	Coal	1.12	0.98	0.39	0.50	0.30	0.26	0.25	1.14	0.14
Yu 20	2744.4–2749.41	P ₁ s	Carbonaceous mudstone	1.20	0.86	0.38	0.62	0.16	0.19	0.29	1.20	0.11
Su 27	3155.03–3155.97	P ₁ s	Mudstone	0.82	1.30	0.83	0.77	1.72	0.72	0.17	0.64	0.03
Su 27	3157.37–3158.08	P ₁ s	Coal	-	4.16	2.10	7.07	1.10	0.82	0.09	0.29	0.00
Su 27	3184.46–3185.06	P ₁ s	Coal	0.86	1.79	2.07	3.53	0.72	1.56	0.17	0.48	0.00
Su 27	3185.06–3185.38	P ₁ s	Mudstone	-	2.50	3.35	2.39	0.54	1.10	0.20	0.53	0.00

Note: a. R_o (%); b. $\sum C_{19}-C_{21}TT/(\sum C_{23}TT+\sum C_{24}TT)$; c. Pr/Ph; d. C_{24} tetracyclic/ C_{26} tricyclic terpene; e. the absolute concentration of C_{30} diahopane ($\mu\text{g}/\text{mg}$); f. C_{30}^*/C_{30} hopane; g. $(C_{21}+C_{22})/\sum C_{27}-C_{29}\alpha\alpha R$ sterane; h. $C_{27}\alpha\alpha R/C_{29}\alpha\alpha R$ sterane; i. C_{30} 4-methylsteranes/ C_{29} steranes; - no data.

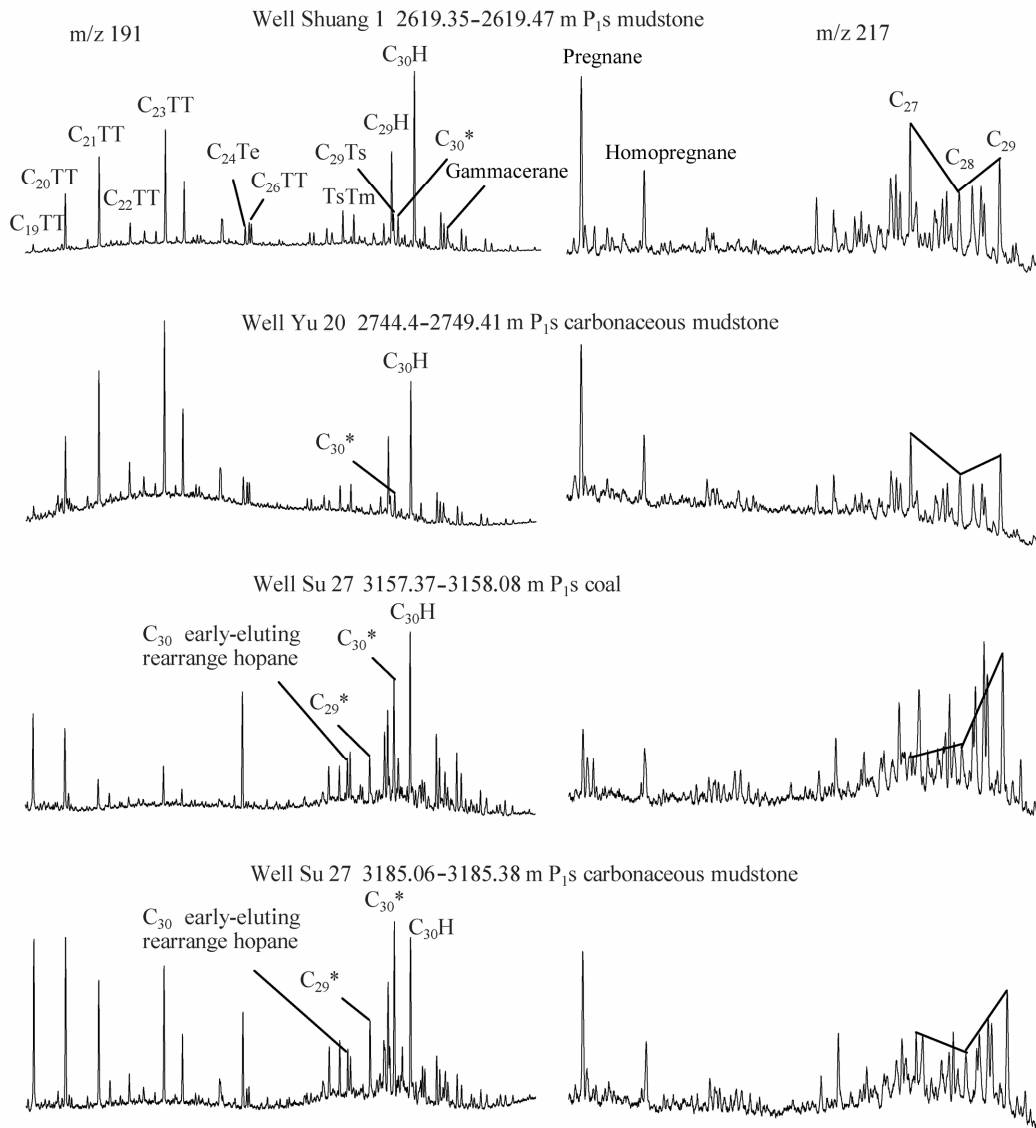


Fig. 2. The distribution of steranes and terpanes in typical source rocks from research area.

4.3.2 Pentacyclic triterpanes

Overall, the observed distributions of regular hopanes are similar in the extracts of the 8 coal measure source rock samples. The most distinctive features of the pentacyclic triterpanes in the source rock samples are the distribution patterns of rearranged hopanes (Fig. 2).

Philp and Gilbert (1986), Volkman et al. (1983), Zhang and Zhu (1996) regarded the $17\alpha(\text{H})$ -diahopane as a possible terrigenous biomarker. Moldowan et al. (1991) believed that the $17\alpha(\text{H})$ -diahopane is derived from bacterial hopanoid precursors in clay-bearing sediments deposited in oxic or suboxic conditions. Zhang et al. (2009) proposed that environmental factors such as redox settings and lithology are key factors that control the concentration of C_{30} rearranged

hopane in source rocks, while organic types and maturity may be minor factors. Liu et al. (2014) regarded that the algae in saline water environment may be a kind of origin of $17\alpha(\text{H})$ -diahopanes; the maturity and redox settings have little effect on the formation of $17\alpha(\text{H})$ -diahopanes. Though the origin and genesis is yet unclear (Zhang, 2013), the key factors that control the abundance of $17\alpha(\text{H})$ -diahopanes are mainly depositional environment, diagenetic conditions, and parent material sources. While, the formation of $17\alpha(\text{H})$ -diahopanes appears to be independent of maturation.

High abundance of $17\alpha(\text{H})$ -diahopane (include C_{29} and C_{30} diahopane) and a C_{30} early-eluting rearranged hopane were detected in coals and mudstones from well Su 27. Even the pentacyclic triterpanes are dominated by C_{30} diahopane (C_{30}^*) in some samples

(Fig. 2). The C_{30}^*/C_{30} hopane ratios are generally higher than 0.72, and the absolute concentrations of C_{30}^* are mainly higher than 0.7 $\mu\text{g}/\text{mg}$. While, the source rocks from wells Shuang 1 and Yu 20 have low contents of rearranged hopanes, with the C_{30}^*/C_{30} hopane ratios ranging from 0.14 to 0.26, and the absolute concentrations of C_{30}^* are lower than 0.53 $\mu\text{g}/\text{mg}$.

Fig. 4 shows Pr/Ph versus C_{30}^*/C_{30} hopane ratios for the source rock samples which are calculated from m/z191 fragmentograms. These two ratios for the source rocks from wells Shuang 1 and Yu 20 are similar, and are clearly separated from well Su 27, indicating that the source rocks from the first two wells have similar depositional environment and parent material sources that are obviously different from the source rock from well Su 27.

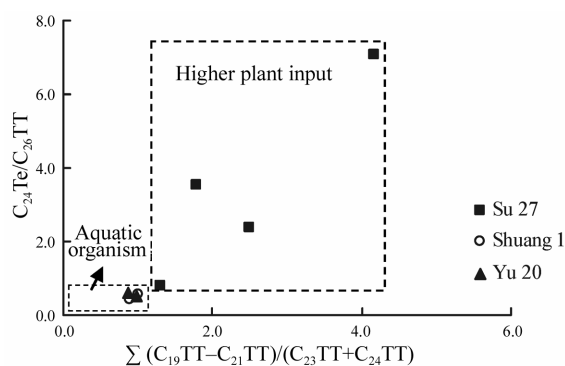


Fig. 3. Cross-plot of the $C_{24}\text{Te}/C_{26}\text{TT}$ versus $\Sigma(C_{19}\text{TT}-C_{21}\text{TT})/(C_{23}\text{TT}+C_{24}\text{TT})$ ratios for the typical source rock samples in research area.

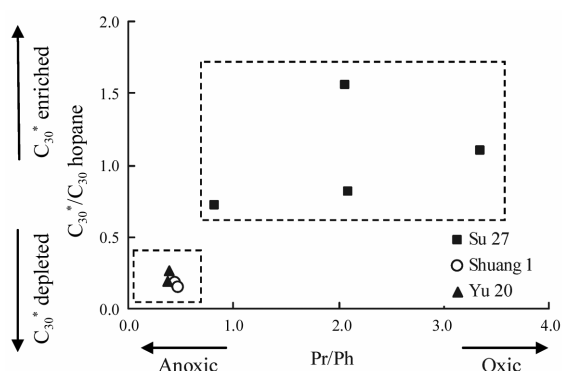


Fig. 4. Cross-plot of the Pr/Ph versus C_{30}^*/C_{30} hopane ratios for the typical source rock samples in research area.

4.4 Steranes distribution

The m/z 217 mass chromatograms of the coal-measure source rock samples (Fig. 2) show a clear pregnanes, regular steranes and diasteranes distribution. Relatively high pregnane and homopregnane were detected in all source rock samples. The $(C_{21}+C_{22})/(C_{27}+C_{28}+C_{29})$ steranes ratio increases in

petroleums with maturation and correlates with $20\text{S}/(20\text{S}+20\text{R})$ C_{29} sterane parameter were shown by Sakata et al. (1988). Huang and Meinschein (1989) proposed that the pregnane and homopregnane are mainly generated by thermal decomposition. The $(C_{21}+C_{22})/(C_{27}+C_{28}+C_{29})$ steranes ratios of the source rocks from well Su 27 range from 0.09 to 0.20, whereas the source rock samples from wells Shuang 1 and Yu 20 have relatively higher values, ranging from 0.20 to 0.31. These data suggest that the maturity of the source rocks from wells Shuang 1 and Yu 20 are slightly higher than that well Su 27, which are consistent with their vitrinite reflectance values.

Huang and Meinschein (1979) proposed that a predominance of C_{29} steranes would indicate a strong contribution from organic matter of higher plants; whereas, a dominance of C_{27} would indicate a dominance of algae. The relatively molecular composition of C_{27} - C_{29} steranes ($\alpha\alpha\alpha\text{R}$) appear to be independent of maturation in the coal-bearing source rocks (Zhu et al., 2012). The source rock samples from the study area demonstrate two different distribution patterns of regular steranes. As illustrated in Fig. 2, the carbon number predominance of steranes in the source rock samples from well Su 27 are $C_{29}>C_{27}$ and $C_{29}>C_{28}$. The C_{27}/C_{29} ratios of $5\alpha(\text{H})$, $14\alpha(\text{H})$, $17\alpha(\text{H})$ -20R steranes of the source rocks from 27 Well range from 0.29 to 0.64, indicating a strong contribution of terrigenous higher plants. In contrast, the regular steranes of the source rocks from wells Yu 20 and Shuang 1 exhibit a V-shape pattern, namely $C_{27}>C_{28}<C_{29}$, with the C_{27}/C_{29} ratios ranging from 0.83 to 1.20, suggesting the contribution of algae and higher plants. The correlation between Pr/Ph and C_{27}/C_{29} sterane ratios also indicates the differences of source rock depositional environments and parent material sources among wells Shuang 1, Yu 20 and well Su 27 (Fig. 5).

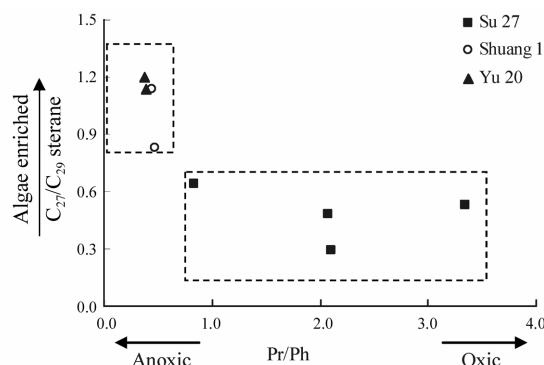


Fig. 5. Cross-plot of the Pr/Ph versus C_{27}/C_{29} sterane ratios for the source rock samples in research area.

The 4-methylsteranes in petroleum probably originate from 4α -methylsterols in living dinoflagellates (Wolff et al., 1986). However, 4α -methylsterols

occur in prymnesiophyte microalgae of the genus Pavlova (Volkman et al., 1990). In general, the 4-methylsteranes are originated from the lower aquatic organisms. The C₃₀ 4-methylsteranes are not detected in the source rock samples from well Su 27, except for one sample (Table 2), an observation that is consistent with their dominantly higher plant organic matter input. While, the C₃₀ 4-methylsteranes are detected with low abundance in the source rock samples from wells Yu 20 and Shuang 1, which are consistent with their higher contribution of aquatic plankton.

5 Conclusions

The Lower Permian coal-bearing interval in Northeast Ordos Basin is a set of coal-bearing gas source rock with relatively high thermally mature, relatively low HI, and type III kerogen.

Notable differences in biomarker assemblage exist between the investigated samples from wells Su 27 and Yu 20 as well as Shuang 1. On the whole, the molecular compositions of coal and mudstone samples from well Su 27 are generally characterized by abundant C₁₉ and C₂₀ tricyclic terpanes, a predominance of C₂₉ sterane over C₂₇ and C₂₈ steranes, unusually high content of 17 α (H)-diahopane, slight concentration of C₃₀ 4-methylsteranes, and high Pr/Ph ratios (mainly > 2). These characteristics suggest that the coal measure source rocks were deposited in suboxic to oxic swamp environment, and dominated by the input of terrigenous higher plants.

Compared with source rocks from well Su 27, the coals, carbonaceous mudstone, and mudstones from wells Yu 20 and Shuang 1 have relatively higher C₂₇ sterane, poor 17 α (H)-diahopane, the absent of C₃₀ 4-methylsteranes, and low Pr/Ph ratios (<0.5). These characteristics suggest that the coal measure source rocks were deposited in reducing conditions with mixing organic matter input.

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