Application of Biomarkers to Quantitative Source Assessment of Oil Pools

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Abstract Recent detailed organic geochemical and geological investigation indicate that oils of the Bamianhe oilfield, Bohai Bay Basin, East China are the mixture of less mature oils and normal oils derived from the Es_4 mudstones and shales with a wide range of thermal maturity from immature to middle-maturity, and most of the oils were proved to be sourced from the depocenter of the Niuzhuang Sag immediately adjacent to the Bamianhe oilfield. Two approaches to quantify the amount of immature oils mixed through quantitative biomarkers were established. One is a relatively simple way only through organic geochemical analysis while the other is to be combined with basin modeling. Selecting biomarkers as proxies is the crucial point in both of them. The results show that the less mature oils mixed in the Bamianhe oilfield is less than 10% and 18% respectively based on the two approaches, which coincide with the results of oil-source rock correlation.

Key words: Biomarkers, immature oil, mixed oil, source, quantitative assessment

1 Introduction

Less mature oils were discovered in almost every Mesozoic and Cenozoic rift basins in East China such as the Bohai Bay Basin and North Jiangsu-South Yellow Sea Basin, and considerable immature resources were reported in the basins. It has been argued that the genetic mechanisms of immature oils are different from the traditional kinetics of kerogen degradation (Zhang and Zhang, 1999; Huang, 1999), and several mechanisms were suggested worldwide (Khorasani and Michelsen, 1991; Snowdon and Powell, 1982; Snowdon, 1991). In China, immature oils were reported associated with hypersaline environments where sulfur-rich kerogen, which generate hydrocarbons in low temperature, is usually formed (Peters et al., 1996). It seems that the best recognized genetic mechanism of immature oil is that soluble organic matter in lipids such as alga deposited in brackish and saline, anoxic lacustrine settings generates hydrocarbons above the normal oil-window (Huang, 1996; Zhang and Zhang, 1999). The Bamianhe oilfield in the Bohai Bay Basin and Jinhu Depression in the North Jiangsu-South Yellow Sea Basin were thought to be typical regions bearing large amount of immature resources.

However, it was recently found that the so-called immature oils in the Bamianhe oilfield, Bohai Bay Basin and Jinhu Depression, North Jiangsu-South Yellow Sea Basin are mostly derived from the deep part of the adjacent depocenter (Pang et al., 2003a and b), and re-investigations showed that immature characteristics, i.e. relatively low level of C_{29} sterane $\alpha\alpha\alpha$20S/(S+R), the presence of biomarkers with low thermal stability including 5β(H)-sterane, methylated chromans and β-carotane as well as high values of gammacerane content, can be the results of the mixing of mature oils with immature oils or bitumen during migration (Pang et al., 2001; Pang et al., 2003a, b; Li et al., 2002). It is very important to recognize the proportion of the mixed immature oils from views of both further exploration and classification of the exact kinetics of the so-called immature oils.

The variation of the composition and distribution of oils and rock extracts with maturity has been well known. By using standard co-injection before GC-MS analysis, we can obtain the absolute abundance of a specific compound in the oils and rock extracts and its varying trend with thermal maturity. In this study, the Bamianhe oilfield was selected as a case for calculating the relative contribution of immature oils according to the quantification of biomarkers. Previously, Wang et al. (1999) involved in a similar research by experiment, and Jiang et al. (2001) and Boreham et al. (1994) also carried out some work in a quantification study to solve geological issues. To identify the proportion of oils with different maturities in an oil pool by biomarkers is still a new direction though biomarkers have been used in many aspects of petroleum exploration (Wang et al., 2001; Huang and Pearson, 1999).

2 Geological Setting

The Bohai Bay Basin is a complex, rift basin formed in the late Jurassic and early Tertiary (Hu et al., 1986). Significant petroleum reserves in this basin are distributed in various depressions, including the Dongying Depression (Fan and Wang, 1997). The Teriary strata of the Dongying Depression is comprised of the Paleogene Kongdian, Shahejie and Dongying formations, and the Neogene Guantao and Minghuazhen formations. The Paleogene is a period of downfaulting when the Kongdian and Shahejie formations were deposited, whereas the Neogene is a period for the formation of basin-wide caprocks. The Kongdian Formation consists mainly of coarse clastic redbeds, unconformably covering the Mesozoic strata. The Shahejie Formation can be further divided into four members, among which the Es_3 and Es_4 members contain the main petroleum source rocks in the basin. Detailed contour mapping by SINOPEC in-house study indicates that the most likely source rocks in the Dongying Depression are the oil shales and dark mudstones in the upper part of Es_4 and the middle-lower parts of Es_3. The total thickness of the oil shales and dark mudstones in the upper part of Es_4 is around 40 m and 300 m, respectively. In addition to 40 m of oil shales, Es_3 contains up to 800 m of dark mudstones. A brackish to saline lacustrine setting for the deposition of the Es_4 member is indicated by the presence of such fossils as *Discobis sp.*, *Ammonia* and *Nonion* (Hong et al., 1997; Zhang and Zhang, 1999). The Es_3 member is composed of dark-grey and grey mudstones, oil shales, siltstones and fine sandstones deposited in a deep to semi-deep lake environment. The Tertiary stratigraphy of the Niuzhuang South Slope including the study area is similar to that of the Dongying Depression except the absence of the lower part of the Es_3 member and the thinning of the Dongying Formation.

Significant oils have been found in the Bamianhe oilfield (Zhang and Zhang, 1999). The field is located in the eastern segment of the Niuzhuang South Slope (Fig. 1). The Niuzhuang Sag to the northwest of the field covers an area of over 900 km^2 with over 4000 m of Tertiary sediments. Between the Niuzhuang Sag and the Bamianhe field lays the Niuzhuang South Slope, also with an area of around 900 km^2.

3 Samples and Experiment

Twenty samples of oils and 8 of oil sands from Bamianhe were selected for detailed geochemical characterization (Li, 2002; Pang et al., 2003a). A large number of potential source rock samples were also collected for analysis. The sampling locations are shown in Fig. 1. Detailed results on the Es_3 and Es_4 source rock samples were reported by Li et al., 2003.

After asphaltene precipitation, crude oils were fractionated on a neutral alumina chromatographic column into saturate, aromatic hydrocarbons and a polar fraction, by sequential elution with *n*-hexane, toluene and chloroform. For the quantitative determination of absolute concentrations of aliphatic and aromatic hydrocarbons, known amounts of standard compounds, d_4-$C_{29}\alpha\alpha\alpha$(20R) ethylcholestane and d_8-dibenzothiophene were added to the concentrated extracts prior to fractionation steps. Similar procedures were used for the preparation of rock extracts.

Gas chromatography (GC) of saturate fractions was

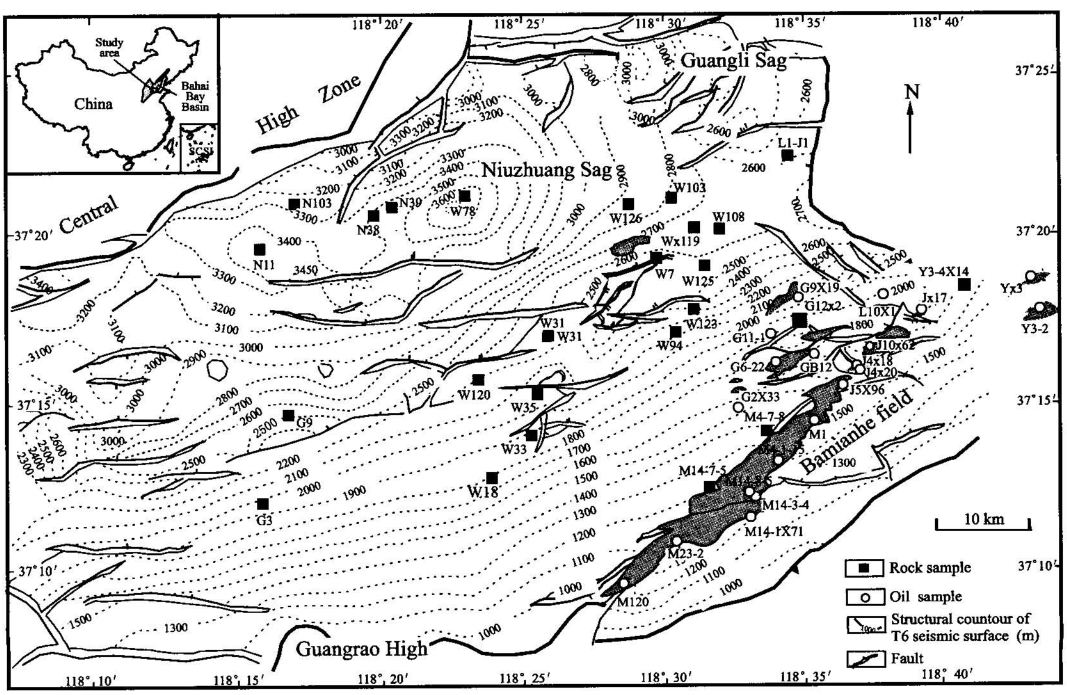


Fig. 1. Location of the Bamianhe Oilfield and samples collected.

performed on a Hewlett-Packard 5160 GC fitted with an SE54 fused silica column (25 m×0.25 mm i.d.). The GC oven was initially set at 50°C for 2 min, and then programmed to 300°C at 4°C/min. Gas chromatography-mass spectrometric analyses (GC-MS) of saturates and aromatic hydrocarbons were carried out on a Hewlett-Packard 5980 Mass Selective Detector (MSD) fitted with a 30 m × 0.25 mm i.d. HP-1 MS capillary column (with 0.25 mm film thickness). For analyzing saturate fractions, the GC oven was initially set at 50°C for 2 min, programmed to 100°C at 2°C/min and then to 310°C at 3°C/min, with a final holding time of 15 min. For analyzing aromatic hydrocarbons, the GC oven was initially set at 60°C, programmed to 150°C at 8°C/min and then to 320°C at 4 °C/min, with a final holding time of 10 min. The mass spectrometer was operated in selected ion monitoring mode. Peak identifications and quantitative methods were reported in Jiang et al. (2001).

4 Results and Discussion

The Bamianhe oilfield was considered as one of the most typical regions producing immature oils (Zhang and Zhang, 1999) in China for the majority of the oils have relatively low levels of thermal maturity, e.g. C_{29} sterane isomerization ratios, 20S/(20S+20R) (0.24–0.44) and $\alpha\beta\beta/(\alpha\beta\beta+\alpha\alpha\alpha)$, of the oils having not attained the equilibrium values (0.5 and 0.55, respectively), and the presence of biomarkers with low thermal maturity. The alga-rich Es_4 shale above the normal window developed in a brackish lacustrine setting in the South Slope of the Niuzhuang Sag was suggested as the main source rock accounting for the oils of the field (Zhang and Zhang, 1999). The shale was also normally regarded as a kind of special source rocks with good petroleum potential because of high level of the total organic carbon (TOC) and the hydrocarbon generative potential (S_1+S_2). The less mature oils derived from soluble lipid directly in the diagenesis stage was proposed as the hydrocarbon genetic mechanism of the area studied (Huang, 1996). However, a recent investigation showed that most oils of the Bamianhe oilfield were contributed by the Es_4 interval within the normal oil window, and the Niuzhuang Sag is the key source kitchen of the oils (Pang et al., 2003a).

4.1 Geological model for resource assessment

The geological model adopted for the assessment of the proportion of the oils produced from the South Slope above the normal oil window and the depocenter of the Niuzhuang Sag within the normal oil window was shown in Fig. 2. Three tectonic units were divided standing for source kitchen (Niuzhuang Sag), the South Slope and the oil reservoir respectively. Two approaches can be utilized for the assessment of the immature hydrocarbons mixed. One is a simple way only through the organic geochemical method; the other is a combined way of basin modeling and the organic geochemical method.

4.2 Organic geochemical approach

As mentioned above, the composition and distribution of chemical constitution of the non-mixed oils and rock extracts indicate the thermal maturity of their source rocks. Little change, especially in the maturity of the oils, is expected if there is no secondary alteration once the oils migrated and accumulated in the traps located in high tectonic positions. Therefore, through quantitative analysis of the fractions of the immature and normal oils as well as the mixed oils respectively, the relative contribution of the two parts of the Niuzhuang Sag and its South Slope, standing for the oils generated in different thermal stages can be calculated. The reservoir of the Bamianhe oilfield is relatively shallow, and the oils in the field are unlikely to suffer extensive thermal evolution. Two rock samples in the lowest and other positions of the South Slope of the Niuzhuang Sag were selected and the rock extracts are utilized as two end members standing for immature oils respectively. In this study, we use rock extracts as immature oils for it is difficult to collect the actual oils generated by the rocks within low thermal maturity located in the South Slope of the Niuzhuang Sag. We selected several samples including oil sands and rock extracts, and the oils with relatively high values of maturity as mature oils. After a comprehensive correlation, steranes were chosen as indicators/proxies for they have relatively high chemical stability and good correlation of abundance with thermal maturity in the range of the maturity of total samples in this study. The following formulae were used in calculating the proportion of the oils with different sources,

$$m=[K\cdot(S_{oS}+S_{oR})-S_{oS}]/[S_{rS}-S_{oS}+K\cdot(S_{oS}+S_{oR}-S_{rS}-S_{rR})]\cdot 100\% \quad (1)$$

$$S_{mix}=S_{mat}\cdot(1-m)+S_{imm}\cdot m \quad (2)$$

where S_{oS} and S_{oR} are the absolute abundance of $C_{29}\alpha\alpha\alpha$ sterane 20S and 20R in mature oil; S_{rS} and S_{rR} are the absolute abundance of $C_{29}\alpha\alpha\alpha$ sterane 20S and 20R in

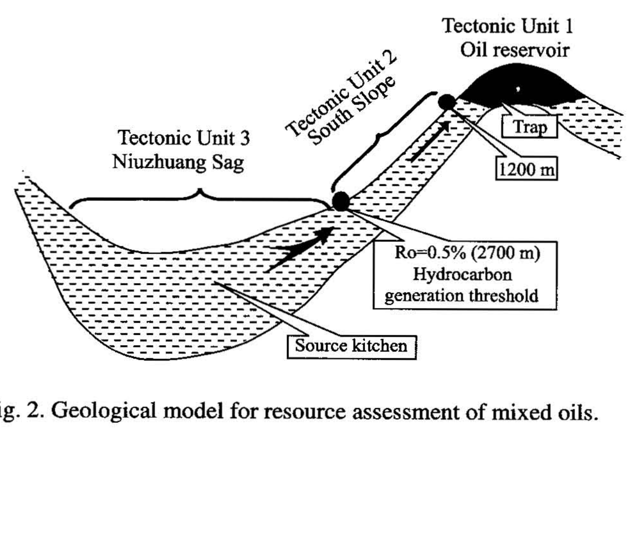


Fig. 2. Geological model for resource assessment of mixed oils.

immature oil; K is $C_{29}\alpha\alpha\alpha$ sterane 20S/(S+R) of mixed oils; m is the content of immature oils mixed (%). S_{mix}, S_{mat}, and S_{imm} are the absolute abundance of $(C_{27}+C_{28}+C_{29})\alpha\alpha\alpha$ sterane20R of mixed oils, mature and immature oils respectively.

Immature oils (end member B and end member C) and mature oils (end member Ai; Totally 15 samples were chosen for there is a range of maturity of mature oils.) selected for the assessment were listed in Table 1. It was expected that the primary and secondary migrations might have an effect on the relative abundance of chemical components, i.e. polar fractions such as nonhydrocarbons and asphaltene are readily adsorbed by clay minerals in rocks. Thus, the absolute abundance of selected biomarkers relative to hydrocarbons (saturated and aromatic hydrocarbons) were used to reduce the difference between crude oils and rock extracts. It was assumed that the absolute abundance of steranes in rock extracts in Table 1 is similar to the crude oils expelled by the rocks with the same maturity. Two theoretical diagrams were shown in Fig. 3 based on calculation. The oils of the Bamianhe oilfield are drawn in the region with <10% immature oils mixed.

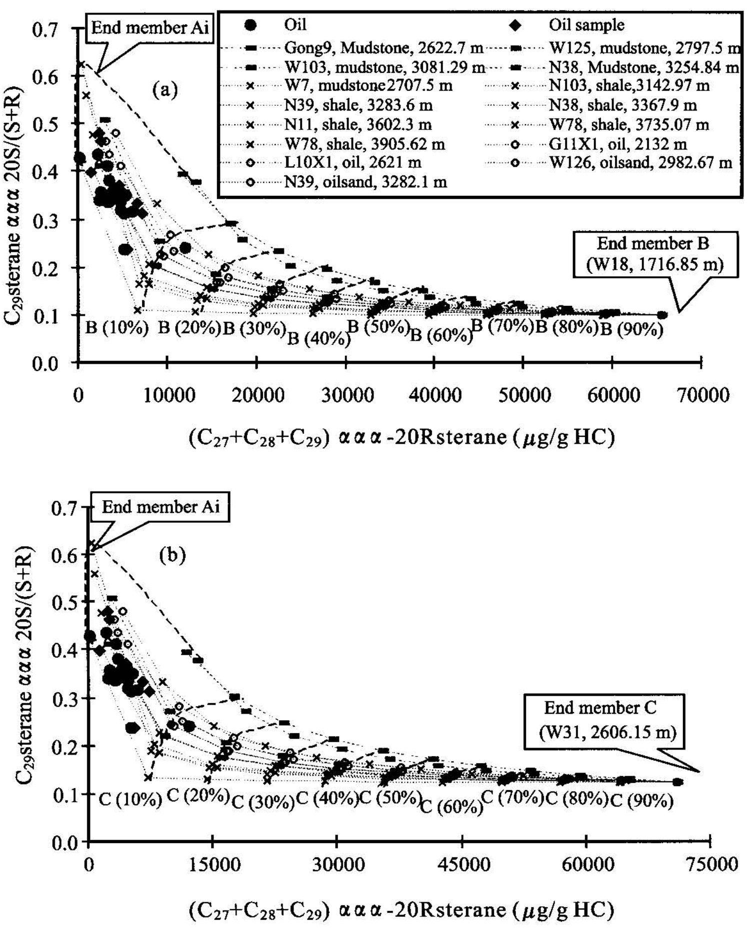


Fig. 3. Theoretical diagrams illustrating immature oils mixed in the Bamianhe oilfield.

4.3 Geological-organic geochemical approach

The approach discussed above is a relatively simple way to assess the relative contribution from the two parts with differential evolution stages. The assessment can be conducted even though only 2 to 3 samples are used. However, there are some disadvantages in it. For example, the composition of the 2 or 3 samples selected can not reflect the average component of the crude oils generated by a continuous range of maturity of source rocks in earlier or later thermal stages. The only way to solve the issue is to calculate the total hydrocarbons expelled. Hydrocarbons expelled by source rocks with continuous maturity range were take into consideration in approach II through the basin modeling method whose geological model is shown in Fig. 4. The total hydrocarbons expelled during earlier and later thermal stages respectively can be obtained. Then, select similar geochemical proxies as approach I and obtain theoretical curves of the proxies with depth/ maturity, which can be attained through regression of natural samples collected (Fig. 5). Finally, using the basin modeling results and the theoretical curves established, formulae about the relation of the proxies in the mixture with the proportion of immature oils mixed can be acquired, and a standard diagram can be constructed for the evaluation of the proportion of different sources. The theoretical curves of the selected geochemical proxies can be obtained through the regression of natural samples collected (Fig. 5). Then, a standard diagram can be constructed for the evaluation of the proportion of different sources.

The boundary of immature and mature stages of source rock in Fig. 4 is identified according to vitrinite reflectance values (R_o%) and maturity parameters of hydrocarbons (Li et al., 2003). It was proved that R_o=0.5% is the point where the distribution of steranes and terpanes in the studied samples exhibit significant variation, suggesting the beginning of large amount hydrocarbon generation. The 0.5% R_o value corresponds to a burial depth of approximately 2700 m in the Bamihe area. The determination of hydrocarbons expelled is based on the geological model in Fig. 4. Rock-Eval parameters S_1 and S_2 stand for soluble and pyrolysis hydrocarbons respectively. (S_1+S_2)/TOC could be used to reflect the total potential of remaining hydrocarbons. The decreasing of (S_1+S_2)/TOC is the result of hydrocarbons expulsion (Zhou and Pang, 2002). We assume the decreasing of (S_1+S_2)/TOC before 0.5% R_o for source rocks is a result of generation of less mature oils. It was suggested that the kinetics of immature oils in the Bamianhe area is different from that of kerogen degradation, and soluble organic matter

Table 1 Absolute abundance of single biomarkers in oils and rock extracts of the Bamianhe oilfield

Catalogue	Well	Depth (m)	Strata	Type	$C_{29}\alpha\alpha\alpha$20S (μg/g HC)	$C_{29}\alpha\alpha\alpha$20R (μg/g HC)	$(C_{27-29})\alpha\alpha\alpha$ 20(R)(μg/g HC)	$C_{29}\alpha\alpha\alpha$ 20S/(S+R)
	W103	3081.29	Es_4	Marly shale	788	769	1844	0.51
	N11	3602.3	Es_4	Shale	389	308	716	0.56
	W78	3735.07	Es_4	shale	247	149	307	0.62
	W78	3905.62	Es_4	shale	50	69	140	0.42
	W125	2797.5	Es_4	mudstone	1998	3046	8108	0.40
	Gong9	2622.7	Es_4	Calc. mudstone	1559	2585	10129	0.38
	N103	3142.97	Es_3	shale	308	457	1132	0.40
End M-Ai	N38	3367.9	Es_3	shale	418	458	897	0.48
	N38	3254.84	Es_3	mudstone	500	718	1464	0.41
	N39	3283.6	Es_3	mudstone	418	564	1240	0.43
	W7	2707.5	Es_4	shale	1017	2031	5636	0.33
	G11-1	2132	Es_4	oil	653	845	2274	0.44
	L10x1	2621	Es_4	oil	868	1233	3333	0.41
	W126	2982.67	Es_4	Oil sand	829	963	2577	0.46
	牛39	3281.3	Es_3	Oil sand	842	909	2396	0.48
End M-B	W18	1716.85	Es_4	Calc. shale	1131	10243	20728	0.10
End M-C	W31	2606.15	Es_4	shale	1397	9882	22775	0.12
	G2x33	1690	Es_4	oil	872	1710	4774	0.34
	G6-22	1680	Es_3	oil	801	1464	4090	0.35
	G9-19	2380	Es_4	oil	766	1247	3618	0.38
	G12x1	1873.2–1877.2	Es_4	oil	603	1196	3268	0.34
	M120	948.5–969.9	Es_4	oil	870	1921	5157	0.31
	M23-2	1109.3–1145	Es_4	oil	940	1647	4543	0.36
	M14-8-5	1197.9–1228.5	Es_4	oil	1020	2199	6038	0.32
Mixed oil	M4-1-15	1060.2–1066	Es_3	oil	776	1400	3927	0.36
	M1	1210–1239	Es_3	oil	972	1740	4844	0.36
	M14-3-4	872.4–894	Guantao Fm.	oil	528	950	2643	0.36
	M14-1-X71	1580–1600	Ordovician	oil	71	94	199	0.43
	J5-x96	1530	Es_3	oil	1039	1940	5407	0.35
	J4-x18	1440	Es_3	oil	689	1316	3784	0.34
	J10-x62	1736–1740	Es_3	oil	455	885	2454	0.34
	Jx17	1930	Es_3	oil	817	1757	4768	0.32
	J4-x20	1695	Es_4	oil	657	1290	3485	0.34
	Yx3	1760	Es_3	oil	587	1895	5252	0.24
	Y3-2	1462–1484	Es_3	oil	1395	4425	12134	0.24
	M14-7-5	1225.34	Es_4	Oil sand	1230	2695	7306	0.31
	W18	1724.87	Es_4	Oil sand	623	2002	5549	0.24
Oil sand	W123	2276.1	Es_4	Oil sand	1268	2549	6659	0.33
	W7	2708	Es_4	Oil sand	386	581	1434	0.40
	L1-J1	2582	Es_4	Oil sand	926	1716	4864	0.35
	G12x2	1991	Es_3	Oil sand	997	1690	4676	0.37

End M-Ai: end member Ai; End M-B: end member B; End M-C: end member C.

can form immature oils under low thermal maturity (Zhang and Zhang, 1999). Proper quantitative geochemical indices were selected after detailed correlation among a number of potential proxies. Finally, the absolute abundance of $C_{27-29}\alpha\alpha\alpha$20R steranes and alkanes was chosen for they have comparatively good correlation with burial depth through the maturity range of source rocks studied (Fig. 5).

Formulas used are as the following,

$$Q_{HE} = \int q_e(z)\cdot H\cdot S\cdot \rho(z)\cdot TOC \qquad (3)$$

$$Q_{BIO} = \int q_e(z)\cdot H\cdot S\cdot \rho(z)\cdot TOC\cdot y(z) \qquad (4)$$

where Q_{HE} is the total amount of hydrocarbon expelled (mg), Q_{BIO} is the total amount of a specific biomarker expelled (mg), q_e(z) is the hydrocarbon expulsion intensity (varying with depth) (mg/gc), ρ(z) is the density of source rock (varying with depth) (g/cm^3), y(z) is the abundance of biomarker (varying with depth) (mg/gc) (Fig. 5), TOC is the total organic carbon, H is the depth of source rock (m) and S is the area of source rock (m^2).

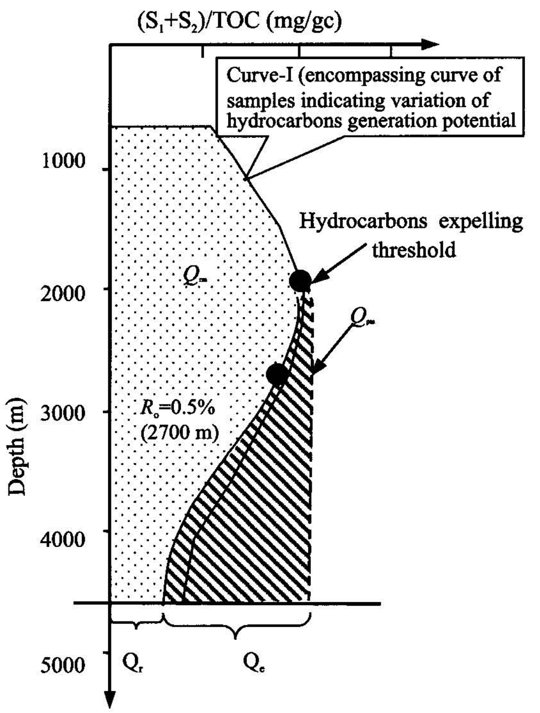
The absolute abundance of specific biomarkers (C_{27-29}sterane $\alpha\alpha\alpha$20R, alkanes in this study) varying with the proportion of less mature oils expelled can be established in the following formulae

$$S\ (C_{27-29}\text{sterane-}\alpha\alpha\alpha\text{20R}) = 129.96m + 1781.9 \qquad (5)$$

$$S\ (\text{alkanes}) = -0.6593m + 135.36 \qquad (6)$$

where S(C_{27-29}sterane-$\alpha\alpha\alpha$20R) is the absolute abundance of C_{27-29}sterane $\alpha\alpha\alpha$20R in mixed oils (mg/g HC), S (alkanes) is the absolute abundance of alkanes in mixed oils (mg/g HC) and m is the relative concentration of immature oils mixed (%).

Standard diagrams showing the relationship between the absolute abundance of sterans in mixed oils and percentage of immature oils mixed were shown in Fig. 6. The absolute abundance of C_{27-29}sterane-$\alpha\alpha\alpha$20R in mixed oils can be obtained directly according to the standard co-injected. Then the percentage of immature oils mixed can be calculated based on formula (5). It is apparent that the less mature oils mixed in the Bamianhe oilfield is about 4%–33% without considering the influence of biodegradation



(Q_{rm}=residual hydrocarbons generation intensity of source rock;
Q_{pm}=maximum value of hydrocarbons generation intensity of source rock;
Q_r=quantity of hydrocarbons resided;
Q_e=quantity of hydrocarbons expelled)

Fig. 4. Geological model for calculating hydrocarbons expelled.

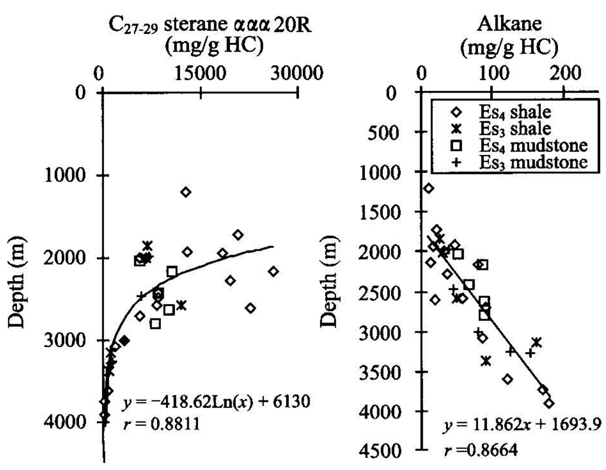


Fig. 5. Variation of absolute abundance of biomarkers in rock extracts with buried depth.

and/or water washing (Fig. 6a), whereas less then 12% if correction is conducted (Fig. 6b). Biodegradation and/or water washing have led to the depletion of hydrocarbons with low molecular weight. The correction is based on the concentration of the compounds in rock extracts with the same maturity (Li et al., 2002). Similarly, the percentage of immature oils mixed can be calculated based on formula (6). Standard diagrams showing the relationship between the absolute abundance of alkanes in mixed oils and percentage of immature oils mixed were shown in Fig. 7. Correction is also performed, which is necessary for the secondary modification that has much more influence on alkanes than sterans.

5 Conclusions

According to the impressive difference of the

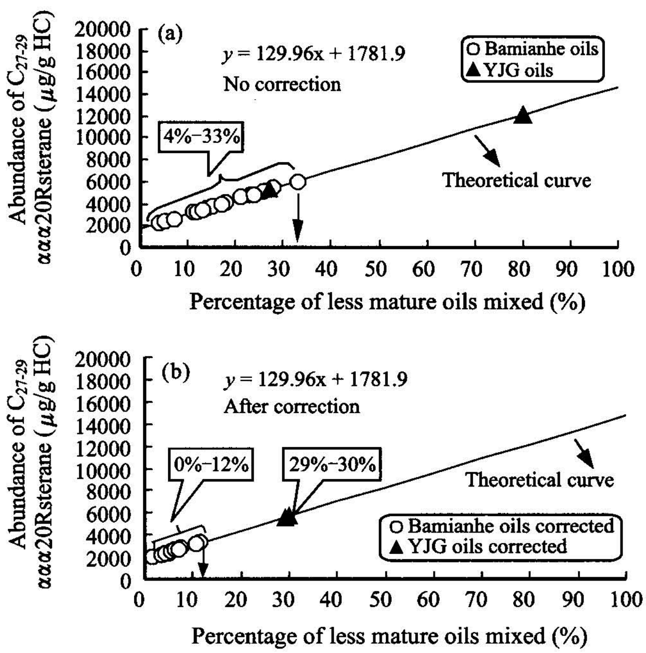


Fig. 6. Theoretical diagrams of steranes and immature oils mixed in the Bamianhe oilfield.

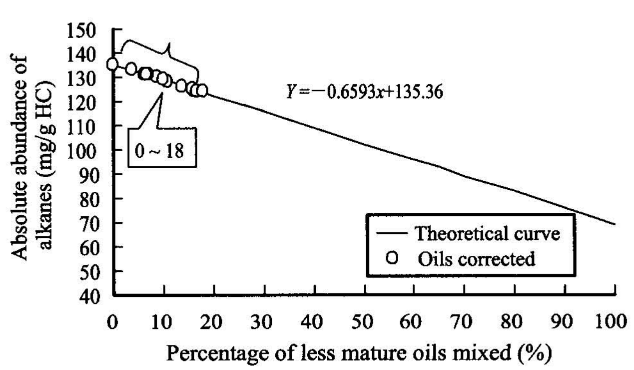


Fig. 7. Theoretical diagrams of alkanes and immature oils mixed in the Bamihe oilfield.

concentration of biomarkers such as *n*-alkane and sterane fractions in rock extracts with varying thermal maturities, the proportion of mixed oils derived from two sources with different maturity can be calculated. Two approaches were adopted in this study, among which one is a relatively simple way only through quantifying biomarkers in crude oils and rock extracts directly and the other one is combined with basin modeling involving complicated calculation. A quantitative assessment shows that less than 18% immature oils are mixed in the oils of the Bamianhe oilfield. Therefore the alga-rich source rock of Es_4 formed in a brackish paleoenvironment developed in the south slope of the Niuzhuang Sag has only a little contribution to the oils discovered because of the low thermal evolution of this source rock. The quantitative assessment agrees with a recent re-investigation of the source rocks of the oils.

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