Isotopic composition characteristics and identification of immature and low-mature oils

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Abstract **Isotopic composition characteristics and the significance of immature and low-mature oils are first systematically discussed. The carbon isotopes of the whole oil can be divided into two groups, one has $\delta^{13}C$ main peak values ranging from −30‰ to −29 and the other from −27‰ to −25‰, they are related to lacustrine and salt-lake facies or swamp facies, respectively. The carbon isotopic fractionation among different group components is relatively small, usually less than 2‰ and the biggest difference in fractionation often occurs between saturated and aromatic fractions. Their δD values vary between −180‰ and −130‰. The main peak of their δD values concentrates between −170‰ and −150‰, suggesting a domination of lacustrine facies. However, the secondary peak ranges from −160‰ to −150‰, showing a frequent salinization of paleo-water bodies. The average $\delta^{13}C$ values of the methane vary between −50‰ and −52‰, about 10‰ lighter than those of mature oils. There is a relatively good correlationship between immature and low-mature oils and their source rocks in carbon isotopic compositions of group fractions and monohydrocarbons; moreover, compared with the source rocks of mature oils, that of immature oils is often relatively depleted in ^{13}C, which is one of the characteristics of immature oils, differing from those of mature oils.**

Keywords: immature and low-mature oil, isotope, associated gas.

The study on the hydrocarbon formation, migration and accumulation of organic matter at the low evolutionary stage to form an immature and low-mature petroleum reservoir is one of the frontiers and hot points in the current oil-gas geochemistry[1−3]. Relatively speaking, the isotopic study on immature oils and gases, especially on immature oils, falls behind and few data are available.

Based on the isotopic data of 39 immature and low-mature oils systematically analysed in recent years and of 4 mature oils used for the comparison, the present note discusses the isotopic composition characteristics of immature and low-mature oils and their identifying indicators, meanwhile some data of previous studies are employed. The isotopic measurement of whole oils and different group components was performed in the Shengli Petroleum Geology Institution and the Lanzhou Institute of Geology, and the analytical error is less than 0.3‰. The isotopes of saturated monomer hydrocarbons were mainly measured on Mat-252 at the State Key Laboratory of Mineralization, Nanjing University, the hydrogen isotopes were measured on MAT-251 with the analytical error less than 5‰.

The carbon isotopes of crude oils and corresponding Pr/Ph ratios are listed in table 1.

1 Distribution frequency of carbon isotopes of whole oils

The $\delta^{13}C$ frequency distribution of whole oils of immature and low-mature oils from different areas in China is shown in fig. 1, from which one can see that the frequency of the $\delta^{13}C$ values shows a doublet distribution, the first peak group is the main peak group, ranging from −34‰ to −28‰ and with −30‰ to −29‰ as the main peak values, the second peak group locates between −28‰ and −25‰ with −27‰ to −26‰ as the main peak values.

The immature and low-mature oils of the first peak group have rather light carbon isotopes and the depositional environment of their hydrocarbon-forming parent materials is related to lacustrine facies[5]. The parent materials are comparatively good, usually Type-Ⅰ or Type-Ⅱ kerogen, constituting the major part of immature and low-mature oils.

The immature and low-mature oils of the second peak group have relatively heavier carbon isotopic compositions and they are mainly related to two depositional environments of oil-forming parent materials, namely salt-lake and swamp facies. However, the Type-Ⅲ kerogen is not the only parent material of these immature and low-mature oils. Since oils and kerogens from salt-lake facies often have heavier carbon isotopic compositions, their original organic matter may be Type-Ⅲ or even Type-Ⅰ kerogen, which characterizes the salt-lake kerogen.

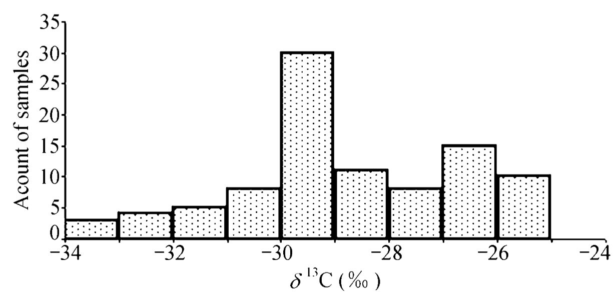

Fig. 1. Frequency distribution of $\delta^{13}C$ values of immature and mature oils.

2 Correlation between $\delta^{13}C$ values and Pr/Ph ratios of whole oils

Two dotted lines, referring herein to $\delta^{13}C$ = −28‰, according to the demarcation in fig. 1 and Pr/Ph=1.5, respectively divide all of symbols in fig. 2 into four zones representing four different depositional environments of oil-forming parent materials.

(i) Zone a. More samples concentrate in this zone whose $\delta^{13}C$ values are less than or equal to −28‰ and Pr/Ph ratios less than 1.5, suggesting a lacustrine environment with relatively strong reducing conditions and a better kerogen type.

(ii) Zone b. Samples in this zone are of similar oil-forming conditions to those in zone a. Their $\delta^{13}C$ values are less than or equal to −28‰ but Pr/Ph ratios greater than 1.5, i.e. most of them are 2±. Compared with the oil-forming environment of zone a, it has mildly reducing conditions and all of samples here come from the Jinggu Basin that is known to be of lacustrine fresh-water source rocks, above and beneath which there are coal beds and no wonder their reducing conditions are relatively weak. Organic petrological study has shown that in the Jinggu Basin exinite and sapropelinite account for 70%—95% of the maceral in the source rocks, being advantageous to oil

Table 1 Carbon isotopic compositions of immature and low-mature crude oils in China

Location	Sample No.	Well No.	$\delta^{13}C$ (‰)					Pr/Ph
			whole oil	saturated	aromatics	nonhydrocarbon	asphaltene	
Liaohe	1	Gao1-6-14		−31.1	−28.8	−29.8	−29.4	2.1
	2	Gao81	−29.6	−29.8	−28.4	−28.4	−29.2	1.06
	3	Gao3-0-041	−29.9	−31.0	−29.3	−29.0	−29.2	
	4	Tuo12	−29.9	−29.0	−28.1	−28.2	−28.3	1.02
	5	Lei11	−30.4	−30.9	−29.0	−29.4	−29.4	0.6
	6	Du126	−29.6	−29.6	−28.4	−28.4	−28.0	1.18
Jiyang	7	Guangbei6-Xie2	−29.0	−29.0	−28.1	−28.0	−26.6	
	8	Gunan24-2	−28.1	−27.9	−26.5	−27.3	−26.0	1.02
	9	Chengbei36	−27.8	−27.1	−26.3	−26.0	−26.5	2.7
Jianghan	10	Tan342	−25.6	−26.5	−26.2	−25.7	−26.3	0.4
	11	Guang33		−27.5	−25.9	−25.7	−25.7	0.35
	12	Wang8-1	−25.6	−26.1	−24.0	−25.8	−25.8	0.33
	13	Hong3-2	−26.6	−27.1	−26.8	−27.2	−28.0	0.87
	14	Wangsi4-1	−26.8	−28.7	−27.4	−27.4	−26.8	0.37
Huanghua	15	Bansheng56	−27.2	−26.9	−25.5	−26.7	−26.7	
	16	Guan128		−34.2	−29.9	−27.3	−29.1	0.43
	17	Guan143	−32.6	−33.2	−30.9	−30.0	−31.0	0.53
	18	Zhang18-28	−28.0					0.31
	19	Ban25	−25.1	−25.2	−23.9	−23.0	−24.1	1.71
Jizhong	20	Zhao27	−25.6	−26.7	−25.0	−27.2	−25.8	0.62
	21	Xiliu101	−28.5	−29.0	−27.7	−27.8	−28.5	0.21
Erlian	22	Ba1		−33.5	−31.5	−31.4	−30.4	0.39
	23	Ba9	−30.7	−34.5	−29.8	−30.6	−32.8	0.78
Subei	24	Ta5	−29.7	−29.7	−28.8	−29.2	−29.5	0.21
	25	Lin1	−29.7	−31.9	−29.8	−30.1	−29.3	0.25
Anhui	26	Wang16	−29.3	−29.4	−28.3	−28.9	−28.9	0.46
Henan	27	Mi103	−29.8	−29.4	−28.3	−29.9	−29.0	0.29
	28	Yun1	−28.1	−29.9	−28.3	−27.9	−29.8	0.52
	29	Zhao1		−30.1	−29.7	−28.7	−28.1	0.48
	30	Wei135	−27.2	−27.5	−26.5	−27.8	−28.9	0.69
Songliao	31	Lai64	−29.9	−30.2	−29.9	−29.0	−27.2	0.82
	32	Chang23	−31.8	−31.6	−30.4	−27.4	−30.5	0.88
	33	Da407	−30.0	−30.2	−29.1	−29.9	−30.0	0.93
Jinggu	34	Niu2	−32.1	−32.4	−31.1	−30.2	−28.7	1.9
	35	Niu2-1	−30.0					2.0
	36	Niu2-6	−29.3					1.89
	37	Niu5-1	−31.4					1.95
	38	Niu7	−29.1	−31.4	−30.9	−29.6	−31.0	2.85
Qaidam	39	HuaS1-103	−24.9					0.58
	40	Yao319	−25.7					0.33
	41	Qidong1	−25.3					0.38
	42	Qi6-3	−25.2					0.74
	43	Yao24	−26.3					0.51

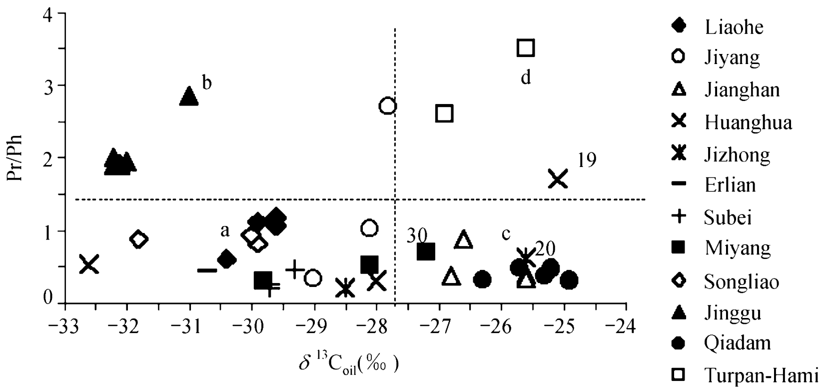

Fig. 2. δ^{13}C-Pr/Ph relationship of immature and low-mature oils.

formation.

(iii) Zone c. In this zone δ^{13}C values should be greater than –28‰ but Pr/Ph ratios less than 1.5. In fact, the samples distributed currently in this zone have δ^{13}C values ranging from –27‰ to –24‰ and Pr/Ph ratios less than 1. The depositional environment of their parent materials is very strongly reducing, the type of organic matter is not Type Ⅲ though their kerogens and crude oils possess heavy carbon isotopic compositions. In general, the δ^{13}C value of kerogens cannot be used to identify the types of organic matter of salt-lake facies.

(iv) Zone d. The division line for this zone is δ^{13}C values greater than –28‰ and Pr/Ph ratios greater than 1.5. Samples in this zone are of poor types of organic matter and their depositional environments are usually weakly reducing or oxydizing. Typical coal-formed oils from the Turpan-Hami Basin are distributed in this zone and their Pr/Ph ratios are generally greater than 3. A distinct difference from common immature and low-mature oils is that those oils from the Turpan-Hami Basin are light oils, which is one of the important indexes of the immature and low-mature coal-formed oil. Sample No.19 from Well Ban-25 is located in this zone and its source rock is formed in a proximal open lake basin that receives plenty of terrestrial organic debris, such as pine and cypress input. After biological decomposition and reworking, light oils characterized by bicyclic sesquiterpanes are formed. Maybe, this kind of light oils can be regarded as a transient product from the immature and low-mature oil of lacustrine facies to that of swamp facies.

3 Carbon isotopic characteristics of group components of crude oils

The carbon isotopic compositions of group components for the same crude oil show a tendency of the ^{12}C depletion with the increase of polarity, and the type curve drawn according to δ^{13}C values of saturated hydrocarbons, aromatics, non-hydrocarbons and asphatenes is often employed in the oil-oil and oil-source correlations[4,5]. Crude oils of the same source usually possess the similar type curves because of the isotopic inherited effect of parent materials and a certain point of the tendency line is the δ^{13}C value of kerogens of source rocks. Based on the data shown in table 1 the following conclusions can be drawn.

(1) In the data of group components of 34 samples there are 6 samples whose ^{13}C increases basically with the increase of polarity, namely their group components have a normal type curve of δ^{13}C values. They, amounting to 17% of the total samples, are Nos. 7, 22, 25, 29, 31 and 34. Moreover, such a tendency can also be found in other 5 samples, which, accounting for 15% of the total samples, are Nos. 8, 16, 17, 32 and 38. The samples whose carbon isotopic compositions of the aromatics are heavier than and similar to those of the non-hydrocarbons and asphaltenes are 14 and 17, respectively, i.e. about 21 samples, amounting to 62% of the total samples, have relatively heavier carbon isotopic compositions of aromatics, which may be regarded as one of the characteristics of immature and low-mature oils.

(2) The biggest fractionation of carbon isotopes generally exists between saturated hydrocarbons and asphaltenes for a normal crude oil, if the carbon isotopic compositions are relatively light, this fractionation is often greater than 2‰. However, in the case of immature and low-mature oils the situation is more complicated and the total fractionation, in general, is smaller than that of normal crude oils. The fractonations of 23 samples among the 43 samples listed in table 1 are smaller than 2‰ and this proportion is about 68%; morever, quite a number of the samples possess the greatest fractionation between saturated hydrocarbons and aromatics, which, to some extent, can be regarded as one of the characteristics of immature and low-mature oils.

4 Corretationship of $\delta^{13}C_{sat}$ -$\delta^{13}C_{aro}$

It can be seen in fig. 3 that most of the points are basically distributed along a band with 45° gradient and can be roughly divided into two zones.

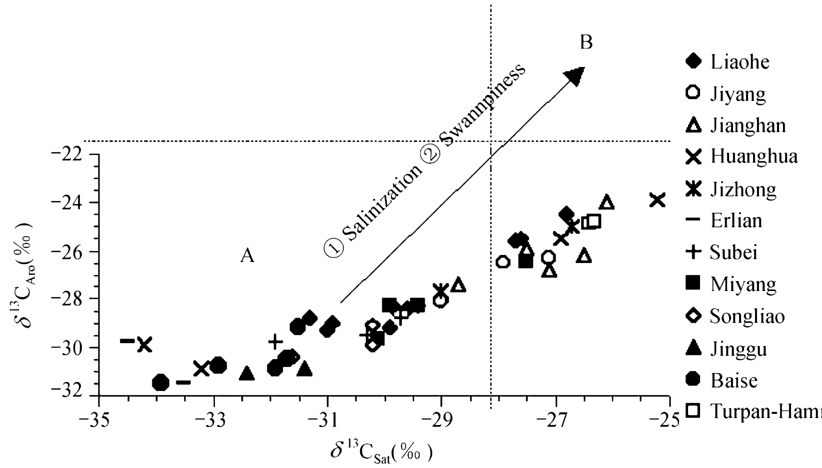

Fig. 3. $\delta^{13}C_{Sat}$-$\delta^{13}C_{Aro}$ relationship of immature and low-mature oils.

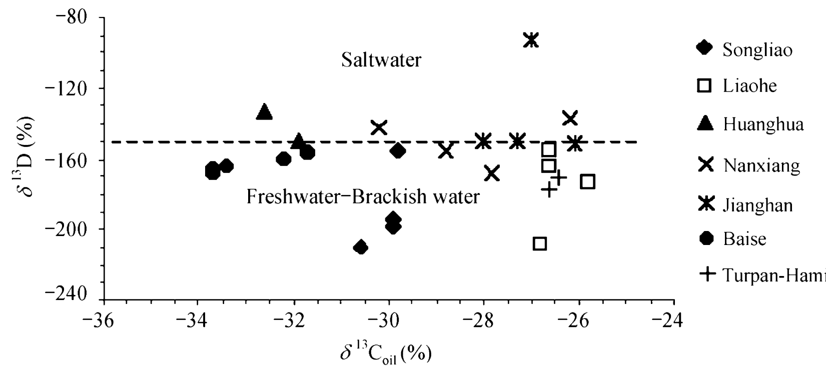

Fig. 4. $\delta^{13}C_{oil}$-δD_{oil} relationship of immature and low-mature oils.

(1) Zone A basically includes all of oils formed in lacustrine facies and equals zone a in fig. 2.

(2) Zone B mainly consists of oils formed in salt-lake and swamp facies and corresponds, on the whole, to zones c and d in fig. 2.

(3) The 45° gradient line indicates two directions of lake evolution. As for immature and low-mature oils, it principally means salinization, and oils from salt-lake facies of the Jianghan and Qaidam Basins are distributed in zone B. The other direction of the tendency line implies swampiness to form coal-type immature and low-mature oils, such as oils from the Turpan-Hami Basin.

5 Carbon isotopic characteristics of hydrogen isotopes and associated gases in immature and low-mature oils

The hydrogen isotopic composition of crude oils is mainly related with the salinity of depositional medium when oil-forming parent materials are formed. By studying hydrogen isotopes of crude oils from terrestrial basins in China, Shen Ping[5] delimited the $\delta D=-150‰$ of whole oils as a boundary between fresh, brackish water facies and saline water facies.

Fig. 4 shows a $\delta^{13}C$ - δD correlationship of immature and low-mature oils. A more distinct concept can be drawn out from fig. 4 due to the concrete indication of oil production areas. For example, the δD values of the crude oil samples from the Baise, Nanxiang and Liaohe Basins approach −150‰, suggesting a certain salinization of the oil-forming environment; however, the oils from the Songliao Basin are mainly formed under a freshwater lacustrine facies. Moreover, the oils from the Turpan-Hami Basin seem to be formed in a freshwater facies as well, in combination with their carbon isotope data and other information it can be drawn that their oil-forming environment should be a freshwater swamp facies.

From the data of refs. [6, 7], it can be seen that except for the Turpan-Hami Basin, the $\delta^{13}C$ values of methane associated with immature and low-mature oils range from −46‰ to −58‰; for example, the $\delta^{13}C_1$ values of 12 samples from the Liaohe Basin vary from −47.2‰ to

–52.4‰, averaging to –50‰, those of 6 samples from the Jiyang Basin range from –47.1‰ to –55.4‰, averaging to –51.9‰, 17 samples from the Subei Basin possess the $\delta^{13}C_1$ values of –46.2‰ to –57.6‰ with an average of –52‰, and 4 samples from the Jianghan Basin have $\delta^{13}C_1$ values range from –46.9‰ to –55.5‰ with –49.7‰ as the mean value. Fig. 5 is a $\delta^{13}C_1$ frequency distribution of typical gases associated with immature and low-mature oils, from which it can be seen that their $\delta^{13}C_1$ values range from –46‰ to –58‰ while those of gases associated with normal crude oils vary generally from –40‰ to –48‰[6,7]. Therefore, in the case of carbon isotopes, the $\delta^{13}C_1$ value of an associated gas can be regarded as one of the indicators to its associated immature and low-mature oils if the value ranges from –50‰ to –54‰.

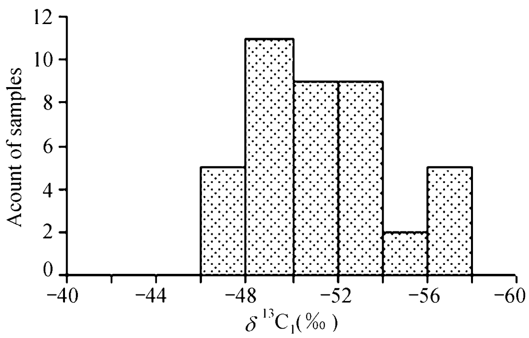

Fig. 5. $\delta^{13}C_1$ frequency distribution of gases associated with immature and low-mature oils.

6 Correlation of oil-source rock and monomer hydrocarbons by carbon isotopes

Six groups of type curves are drawn in fig. 6 according to the carbon isotopic data of oils and corresponding source rocks, and three types of correlations are delineated as follows:

(i) Type of lightness in source rocks. There are five $\delta^{13}C$ values of each sample, namely for saturated hydrocarbon, whole oil or asphaltene A, aromatics, non-hydrocarbon and asphaltene. If four values of a source rock show that it is rich in ^{12}C, this source rock belongs to the type of lightness. For example, the $\delta^{13}C$ values of the source rock from Well Guan 996 of the Huanghua Sag are lighter than those of the crude oil from Well Guan 143 (fig. 6(a)), so does the source rock from Well Gudong 23-1 of the Jiyang Sag compared with the crude oil from Well Guan 24 (fig. 6(b)). Except for the asphaltene of the crude oil from Well Yun 1 of the Miyang Sag, the $\delta^{13}C$ values of other components are heavier than those of the source rock from the same well (fig. 6(c)), so its source rock can also be regarded as belonging to the type of lightness. Generally speaking, carbon isotopic compositions of source rocks are heavier than those of crude oils, which is considered as the results of the mass discrimination effect happening during the hydrocarbon migration to enable lighter isotopes to be expelled out preferentially and heavier ones as well as of the isotopic fractionation left behind during the hydrocarbon formation. Among six combinations between the immature and low-mature source rock and oil there are three combinations that have such a reverse correlation, accounting for 50%. If counting in Well Ba 9 of the Erlian Basin (fig. 6(d)) and Well Tou 12 of the Liaohe Basin (fig. 6(e)), in which three $\delta^{13}C$ values of the source rocks are heavier than their counterparts of the crude oils, one can reasonablly draw a conclusion that it is very common for the correlation between immature and low-mature source rocks and their corresponding oils that the $\delta^{13}C$ values of the soluble organic matter extracted from source rocks are less than those of crude oils and their group components. Another feature of the type of lightness is that the tendency of the type curve towards the ^{13}C enrichment becomes clear with increasing the polarity of group components.

(ii) Type of crossing. The two combinations in fig. 6(d) and fig. 6(e) belong to this type. Isotopic type curves of source rocks and oils are mutually across; on an average, the type of lightness is still dominant, i.e. among the five values those of source rocks with ^{12}C enrichment account for 60%. One of the characteristics for the type of crossing is that the carbon isotopic composition of aromatics is depleted in ^{12}C compared with the counterparts of non-hydrocarbons and asphaltenes.

(iii) Type of heaviness in source rocks. Four out of the five components in this type possess a feature that the $\delta^{13}C$ value of source rocks is greater than that of oils. As mentioned above, this is the main type for normal oils. The ^{13}C enrichment of source rocks is identical with the fractionation mechanism caused by the fractionation effect of thermo-evolutionary dynamics and hydrocarbon migration. However, among the comparison diagrams of type curves for immature and low-mature oils and source rocks there is only one typical group, namely that shown by fig. 6(f), in which the two type curves of the Well Lei36 source rock and Well Lei11 oil from the Liaohe Basin demonstrate that all the components of the crude oil are rich in ^{12}C compared with their counterparts of the source rock except for the $\delta^{13}C$ value of the saturated hydrocarbon of the oil that is similar to but slightly heavier than its counterpart of the source rock. In addition, a similar relationship exists between the Well Gudong 23-1 source rock and the Well Guangbei-6-Xie2 oil of the Jiyang Sag (fig. 6(b)), but their fractionation value is much smaller than that in fig. 6(f).

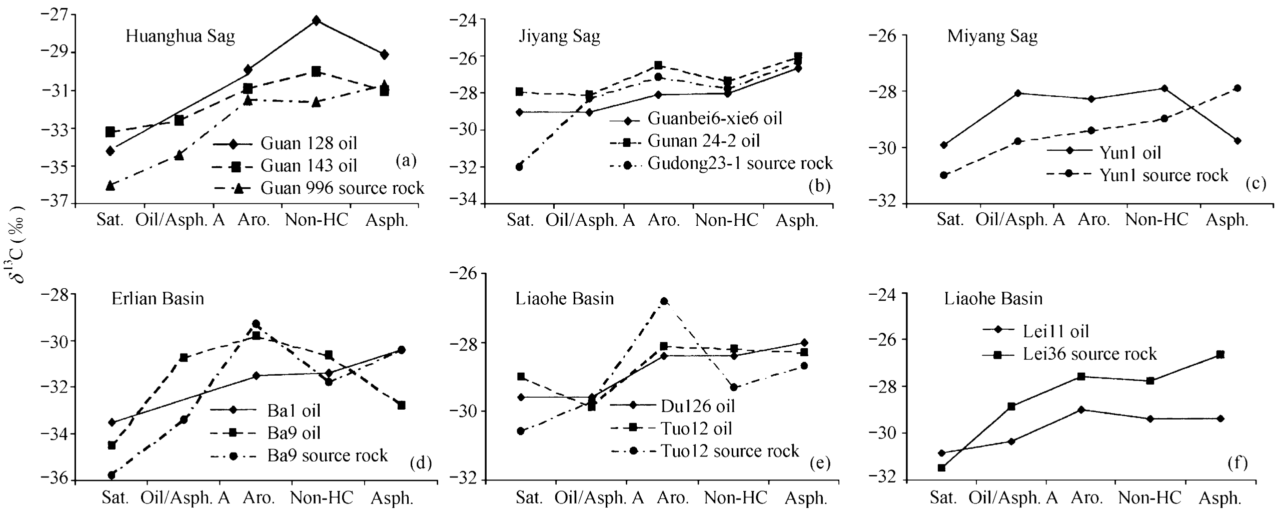

Fig. 6. Comparison of carbon isotopic type curves of oils and source rocks.

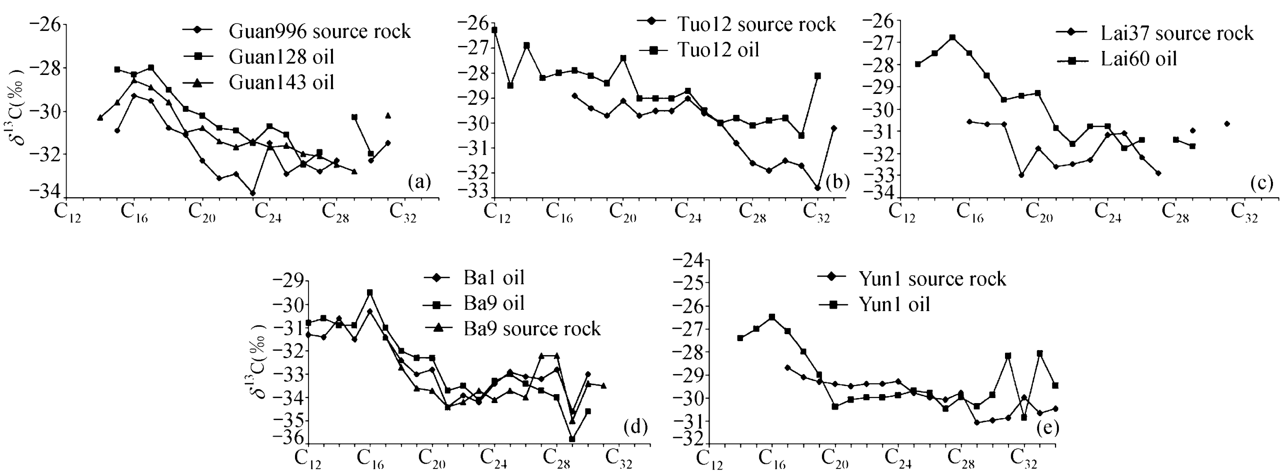

Fig. 7. Carbon isotopic correlation of monomer hydrocarbons between crude oils and source rocks.

Carbon isotopic correlation of monomer hydrocarbons among the five groups of source rocks and crude oils is demonstrated in fig. 7, from which the following can be deduced:

(1) Source rocks and crude oils possess a similar shape of carbon isotopic curves of monomer hydrocarbons, approximately before C_{16} the δ^{13}C values show a tendency to enrich ^{13}C, at C_{16} they are of the ^{13}C peak, and afterward they have a tendency of ^{12}C enrichment with the increase of carbon numbers. The similarity of δ^{13}C curve shapes of monomer hydrocarbons indicates a constituent likeness of precursors, namely there is a comparability between oils and source rocks.

(2) Like the δ^{13}C type curve of oils and source rocks, the δ^{13}C value of monomer hydrocarbons of source rocks is more depleted in ^{13}C than that of crude oils. As mentioned above, the type of lightness in source rocks is the characteristic of carbon isotopic compositions of monomer hydrocarbons from immature and low-mature oils, like what is shown in fig. 7(a, b); however, fig. 7(c, d) may belong to the above-mentioned type of crossing. Except fig. 7(e), all of them can also be regarded as taking on the character of the type of lightness in source rocks, speaking precisely, before C_{24} they all possess this character except the case as shown by fig. 7(e).

7 Conclusions

The δ^{13}C frequency histogram of whole oils shows a doublet distribution, indicating that immature and low-mature oils consist of two groups of crude oils. One constitutes the major part of immature and low-mature oils and its $\delta^{13}C$ values of the whole oil mainly range from –34‰ to –28‰ with the main peak values from –30‰ to –29‰. Combined with Pr/Ph ratios and other data, this type of crude oils is thought to form mainly in reducing environments related with lacustrine facies and to have chiefly types Ⅰ and Ⅱ kerogens. The other group has $\delta^{13}C$ values varying from –28‰ to –25‰ with main peak values from –27‰ to –26‰. According to Pr/Ph ratios and other data, this kind of crude oils is deduced to form in two depositional environments, i.e. salt-lake and swamp facies. Oils formed under both of the conditions are of $\delta^{13}C$ values of whole heavier oils, the salt-lake oil is formed in a strongly reducing environment and often possesses a Pr/Ph ratio less than 0.5, usually its kerogen types are good though their $\delta^{13}C$ values are of heavier oils. However, the coal-type immature and low-mature oils of swamp facies is formed under a comparatively oxydizing condition, its Pr/Ph ratio is often greater than 3 and its kerogen types are relatively poor. It is a light oil.

In crude oils $\delta^{13}C$ values of group components, such as saturated hydrocarbons, aromatics non-hydrocarbons and asphaltenes, generally show a little difference, namely the carbon isotopic fractionations among various components are small with the increase of polarity, their mutual difference is usually less the 0.3‰; this may be one of the characteristics of immature and low-mature oils. With the increase of maturity, group components gradually show a tendency of ^{13}C enrichment as the polarity increases; however, their type curves sometimes are not regular due to the heavier carbon isotopic composition of aromatics.

The hydrogen isotopic composition of immature and low-mature oils ranges from −180‰ to −130‰ and often shows a normal distribution, indicating the variety of their paleo-salinity, such as freshwater, brackish and salt-lake facies. The main peak groups of their δD values vary from −170‰ to −160‰, suggesting the domination of lacustrine facies for the formation of immature and low-mature oils in China. The δD values ranging from −160‰ to −150‰ as the secondary peak indicate a frequent salinization of paleowater bodies during the formation of lacustrine immature and low-mature oils.

The carbon isotopic value of whole oils can hardly be an indicator to the identification of immature and low-mature oils; however, the $\delta^{13}C$ value of the methane associated with oils is able to play an important role in distinguishing immature and low-mature oils from normal ones. In general, the $\delta^{13}C$ value of the methane associated with mature oils ranges from −48‰ to −40‰ with an average from −41‰ to −42‰; however, the methane associated with immature and low-mature oils mainly has a $\delta^{13}C$ value varying from −48‰ to −58‰ and a medium value ranging from –50‰ to –51‰. The difference in $\delta^{13}C$ values between the two different oil-associated methanes is usually as big as 8‰—10‰, thus, it can be a powerful identification indicator.

Correlations by the $\delta^{13}C$ type curves of group components from source rocks and oils as well as by the $\delta^{13}C$ curves of monomer hydrocarbons from source rocks and oils have shown that a relatively good comparability exists between immature and low-mature oils and their possible source rocks. On the basis of the comparable $\delta^{13}C$ features between immature and low-mature oils and source rocks, the present note divides them into three different types, i.e. the type of lightness in source rocks, the type of crossing and the type of heaviness in source rocks. Among six groups of $\delta^{13}C$ data, the type of lightness in source rocks amounts to 50%. The feature of the type of crossing is that three out of the five components, such as asphaltene A, saturated hydrocarbon, aromatics, non-hydrocarbon and asphaltene, belong to the type of lightness in source rocks, namely the relative ^{12}C enrichment still dominates the $\delta^{13}C$ values of source rocks and this type of oil and source rock combination accounts for 33% of the total data. There is only one, typical type of heaviness in source rocks, among the six groups of data and its proportion makes up 16% of the total data. The lighter $\delta^{13}C$ value for the various components of the soluble organic matter from source rocks in comparison with that from oils is probably one of the carbon isotopic characteristics of immature and low-mature oils.

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