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A comparison of the distributions of carbazoles and dibenzothiophenes in western Qaidam Basin, Northwest China

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Abstract Variations in the distributions of carbazoles and dibenzothiophenes were investigated in a set of source rocks, which differ mainly in their maturity levels during immature-mature stages. A comparison of the distributions of carbazoles and dibenzothiophenes has revealed the main results as follows: ① variations in the relative amounts of C_0-, C_1-, C_2-dibenzothiophenes show a low correlation with that of the corresponding carbazoles, with the former's being much higher than the latter's; ② variations in the relative amounts of methyldibenzothiophene isomers also display a low correlation with that of the corresponding methylcarbazoles, with 4-/4- +1-methyldibenzothiophene ratio ranging from 0.52 to 0.96, while the corresponding carbazole ratio of 1-/1- +4-methylcarbazole only being 0.71±0.05; ③ the maturity parameter for 4,6-/4,6- +1,4-dimethyldibenzothiohene, ranging from 0.34 to 0.75, shows a remarkable linear correlation with the corresponding ratio of 1,8-/1,8- +1,4-dimethylcarbazole (R^2＞0.84). The un-correlation may indicate some different geological-geochemical fates for some isomers of dibenzothiophenes and carbazoles. The high correlation may reveal a strong maturation dependence on the dimethylcarbazole distributions, indicating that attention should be paid when 1,8-/1,8- +1,4-dimethylcarbazole is used as a petroleum migration indicator.

Key words carbazole; alkylcarbazole; dibenzothiophene; alkyldibenzothiophene; maturity

1 Introduction

Carbazoles and dibenzothiophenes are typical non-hydrocarbon components in crude oils and also the main subjects of non-hydrocarbon geochemical studies. Approaches used in separating carbazole compounds from oils (Li et al., 1995; Bastow et al., 2003) have greatly promoted geochemical research on pyrrolic nitrogen compounds. Understanding the physical-chemical properties of carbazoles, as well as the pyrrolic compound distributions in petroleum systems, can reveal the fractionation mechanism of carbazoles during petroleum migration (Li et al., 1995; Later et al., 1996), which was successfully applied to research on petroleum migration and accumulation (Li et al., 1995; Later et al., 1996; Bennet et al., 2002; Wang et al., 2004a; Zhang et al., 2004). Dibenzothiophenes and carbazoles may have similar geochemical behaviors in the depositional systems owing to their similar molecular structures. Therefore, the distribution of dibenzothiophenes in oils from a given petroleum system may be potentially used as migration indicators as carbazoles (Wang et al., 2004b; Li et al., 2008). It is well-known that molecular indicators for petroleum migration should exclude effects from non-migration fractionation, such as maturity. Previous studies have clearly indicated that the distributions of carbazoles and dibenzothiophenes are related to many factors, and thermal evolution is the major one (Radke et al., 1986; Radke, 1988; Chakhmakhchev et

al., 1995, 1997; Clegg et al., 1998; Li et al., 1999). Particularly, the distribution of alkyldibenzothiophenes was demonstrated to be strong maturity-dependant in a wider maturation span, which was well proved by the application of maturity parameters such as 4-/1-methyldibenzothiophene (4-/1-MDBT), 4, 6–/1, 4-dimethyldibenzothiophene (4,6–/1, 4-DMDBT), and 2, 4–/1, 4-dimethyl-dibenzothiophene (2,4–/1,4-DMDBT) (Li, 2000; Huo et al., 2008). Although the fact that maturation depends on carbazoles distributions has also been noticed, it still entails further work for improvement. In this paper, we made a comparison of carbazoles and dibenzothiophenes from a typical saline depositional system in western Qaidam Basin, to reveal potential correlations between two compounds with potential maturity-dependance on carbazoles.

As polynuclear aromatic heterocyclic compounds, it should be noticed that carbazoles and dibenzothiophenes have different numbering orders in nomenclature in spite of their similar molecular structures (Fig. 1). Therefore, similar alkyl substituted structures for carbazoles and dibenzothiophenes should have different names: such as 1-methylcarbazole structurally corresponding to 4-methyldibenzothiophene, and 1, 8-dimethylcarbazole corresponding to 4,6- dimethyldibenzothiophene (Fig. 1).

2 Sampling and experimental

General geological characteristics of the Qaidam Basin were well documented (Jin and Zha, 2000; Qiu et al., 2000; Xia et al., 2001; Pang et al., 2004). Briefly, the basin is a Mesozoic-Cenozoic interior sedimentary basin developed from the pre-Jurassic Qaidam Massif, located at the northeastern corner of the Tibetan Plateau. The basin, as an irregular rhombus in shape, is surrounded by mountains, with the Qilian Mountains in the north, the Altun Mountains in the west, and the Kunlun Mountains in the south. A complex deposition history of the basin resulted in different geological features in different areas. Therefore, the basin can be divided into three structural areas: the western depression area, the northern broken block belt and the eastern depression area (Zhu et al., 2005). Three oil and gas systems have been discovered in the corresponded areas: the Jurassic fresh-water lacustrine system in the north (Ritts et al., 1999), the Tertiary saline depositional system in the west (Hanson et al., 2001), and the Quaternary biogas system in the east (Pang et al., 2005). Of the three oil-gas systems, the Tertiary saline depositional system is the major one located in the west, generally called western Qaidam Basin. The Tertiary saline sediments are widely developed in the western area, covering about 30000 km^2 with the maximum thickness up to 7000 m, which can be divided into six formations: the Lulehe Formation, the Lower Ganchaigou Formation, the Upper Ganchaigou Formation, the Lower Youshashan Formation, the Upper Youshashan Formation, and the Shizigou Formation. The main source bed is located in the Lower Ganchaigou Formation, with parts located in the Lulehe and Upper Ganchaigou formations. The source bed consists mainly of laminated-massive calcareous mudstone. Generally, the source rocks mainly contain Type-II kerogens and cover maturity levels in marginally mature to oil-window stages. The distributions of typical biomarkers indicate saline depositional settings (Hanson et al., 2001; Zhu et al., 2005; Zhang et al., 2008a, b).

The source rocks studied here were sampled from the Tertiary formations in the basin. General geochemical properties of the samples were reported in the previous studies (Zhang et al., 2008). Abundant carbazoles and dibenzothiophenes were detected in the samples. So, it is a good case for comparison.

Experimental analysis was conducted as follows: the source rock samples were crushed and then Soxhlet extracted for 72 hours with CH_2Cl_2. The isolation for nitrogen-compounds was adopted as the method reported by Li et al. (1995). The GC-MS method was used to analyze saturated, aromatic, neutral nitrogen fractions (Zhang et al., 2008). The quantification of C_0-, C_1-, C_2-dibenothiophenes in aromatic fractions was based on the peak areas in m/z 184, 198, 212 chromatograms. The quantification of neutral nitrogen compounds was based on the peak areas of related chromatograms of molecular ions (Li et al., 1995; Bowler et al., 1997).

3 Results and discussion

3.1 Biomarkers and maturities

Biomarker compositions of oils and source rocks from the Qaidam Basin were well documented (Hanson et al., 2001; Zhu et al., 2005; Zhang et al., 2008a). Briefly, the typical composition characteristics of biomarkers from western Qaidam Basin are presented as follows: lower Pr/Ph value, even-over-odd predominance in n-alkanes (nC_{18}–nC_{28}), high gammacerane and C_{35} homohopane abundances, and abundant arylisoprenoids (Zhang et al., 2011), indicating strong reducibility in the saline lacustrine depositional systems.

Due to the lack of vitrinite in the source rock samples, C_{29} ααα sterane isomerization parameter (20S/20S+20R) was used to measure the maturity levels of organic matter. GC-MS analysis of saturated

hydrocarbons reveals that the C_{29} sterane maturity parameters range from 0.06 to 0.58, indicating a wide maturity span from low maturity to main oil window stages (Zhang et al., 2008b). As the geochemical characteristics of the depositional system were relatively unchanged, variations in carbazoles and dibenzothiophenes may be mainly related to maturities. Shown in Fig. 2 is a typical distribution pattern of carbazoles and dibenzothiophenes with different maturities.

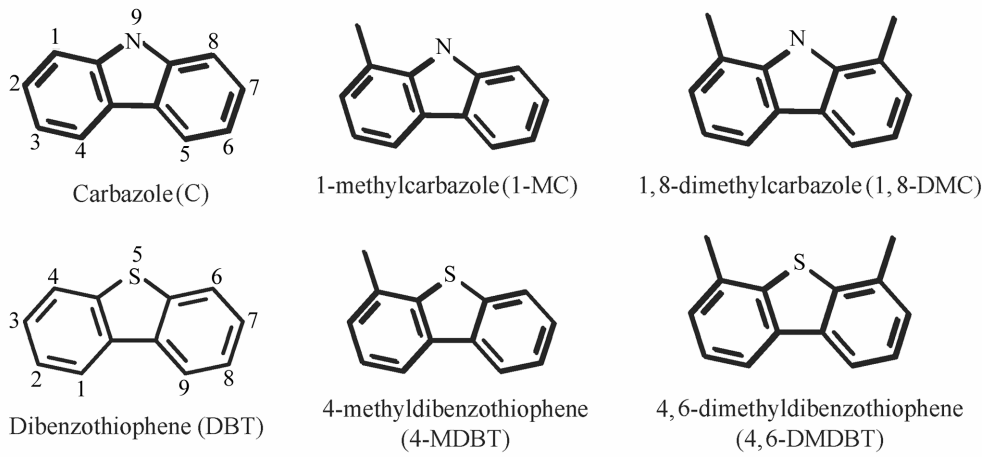

Fig. 1. Molecular structures of carbazoles and dibenzothiophenes.

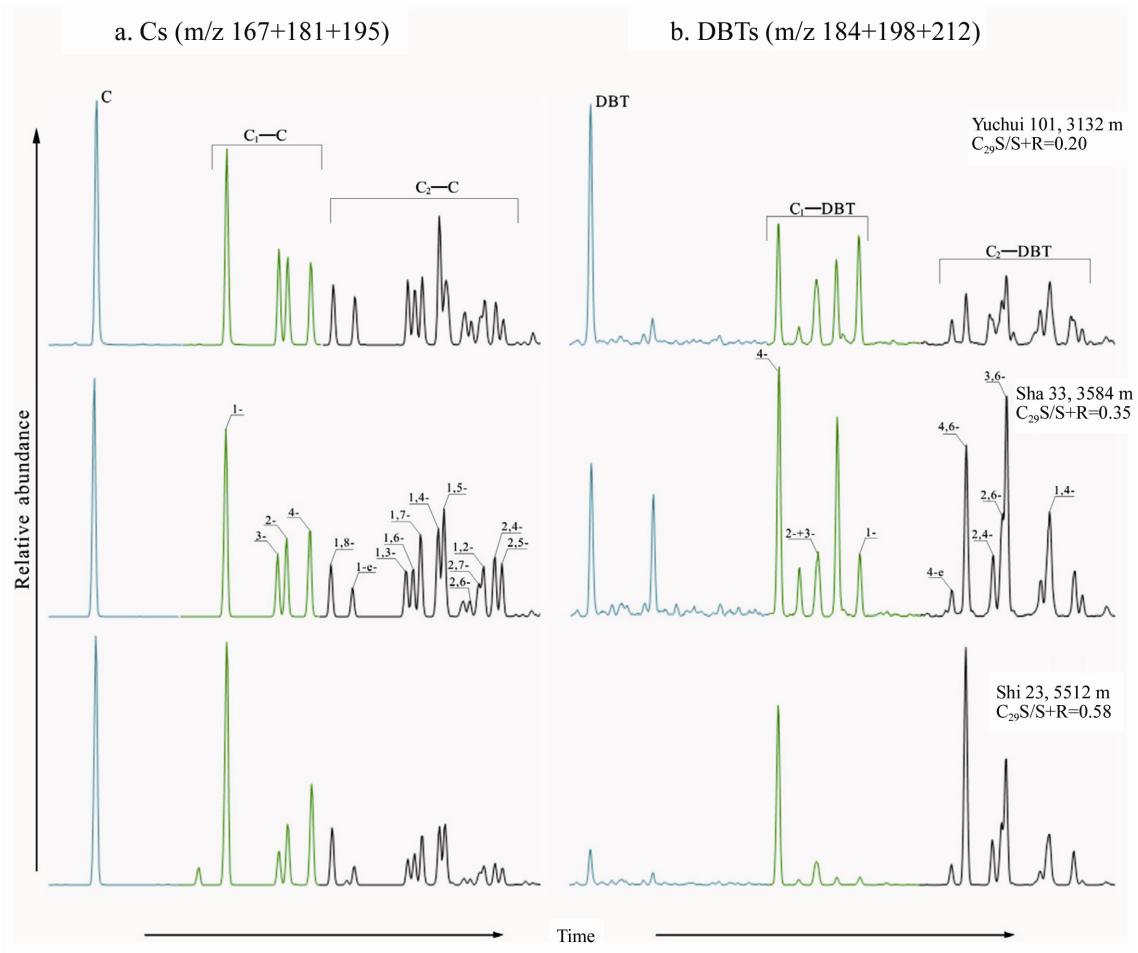

Fig. 2. Comparison of carbazoles and dibenzothiophenes at different maturity levels.

3.2 C_0-, C_1- and C_2-substituted compounds

As shown in Fig. 2, the distribution of dibenzothiophenes with different substitution groups shows an obvious variation with maturity. With increasing maturity, the abundance of dibenzothiophene decreases progressively, while the relative amounts of 4, 6-DMDBT increase obviously. Similar maturity-dependance was detected in oils from Russia (Chakhmakhchev and Suzuki, 1995). Therefore, significant variations in relative amounts of DBT, C_1-DBTs and C_2-DBTs should be attributed to maturation. In comparison, maturation effect on the relative amounts of carbazoles, C_1-carbazoles and C_2-carbazoles seems to be much lower than that on the relative amounts of dibenzothiophenes.

A detailed comparison was made of variations in the relative amounts of carbazoles and dibenothiophenes with different substitution groups. Carbazole, 1-MC and 1, 8-DMC were selected as typical compounds structurally related to C_0-, C_1- and C_2-substituted components, respectively. The relative amounts displayed interesting variations as shown in Fig. 3: the relative amounts of carbazoles are within the range of 36%–50%; those of 1-MC, 38%–53%; and those of 1, 8-DMC, 9%–12%. By contrast, different variations were apparently observed in the relative amounts of DBT, 4-MDBT and 4, 6-DMDBT (structurally corresponding to carbazole, 1-MC and 1, 8-DMC) in Fig. 3: DBT, 5%–58%, 4-MDBT, 29%–60%; and 4, 6-DMDBT, 12%–52%. Figure 3 clearly shows that the abundance data for carbazoles are concentratedin a relatively small area, while the dibenzothiophenes are scattered in a much greater area.

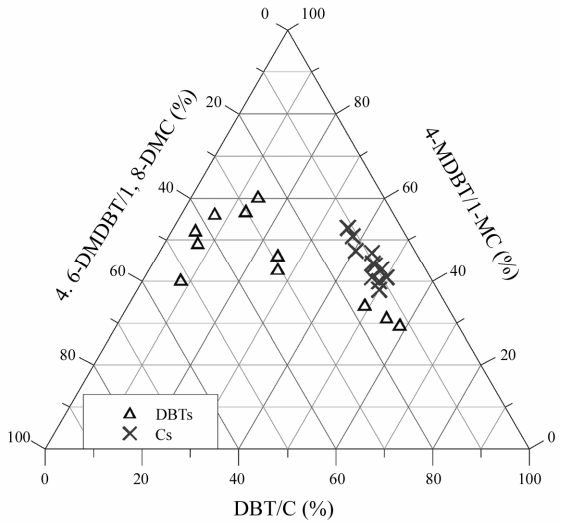

Fig. 3. Ternary plot of the relative amounts of C, 1-MC, 1,8-DMC vs. DBT, 4-MDBT, 4, 6-DMDBT.

As shown in Fig. 4a, 4-MDBT/(4-MDBT+DBT) displays significant changes, over a wider range (0.34–0.91). In comparison, the corresponding ratio of 1-MC/(1-MC+C) for carbazoles shows a strong difference: the ratio is limited in a much narrower span (0.44–0.60), with the major data being fixed at about 0.5. This variation further indicates that the relative amounts of DBT and MDBTs show much higher variations than those of carbazole and MCs in a similar maturity span. In addition, similar variations are also shown in Fig. 4b for the parameters 4-MBDT/(4-MDBT+4, 6-DMDBT) and 1-MC/(1-MC +1, 8-DMC), with dibenzothiophene parameters changing more significantly than carbazole parameters.

The relative amounts of C_0-, C_1- and C_2-compounds show quite a difference between dibenzothiophenes and carbazoles within a certain maturity span, showing a different maturity-dependance for the two kinds of compounds. The variations reveal that maturation effects on the relative amounts of C_0-, C_1- and C_2-DBTs are much stronger than those on the relative amounts of C_0-, C_1- and C_2-carbazoles, indicating little correlation between C_0-, C_1- and C_2-compounds of carbazoles and DBTs.

3.3 C_1-carbazoles vs. C_1-dibenzothiophenes

The common methyl substituted isomers of carbazoles are 1-, 2-, 3- and 4-methylcarbazole and the corresponding isomers for dibenothiophenes are 4-, 3-, 2- and 1-MDBT, respectively. Three peaks in the M/z 198 mass chromatogram were identified as 4-MDBT, 2- +3-MDBT and 1-MDBT with 2-MDBT co-eluting with 3-MDBT under general chromatographic conditions (Fig. 2). Therefore, the comparison of the distributions of methylcarbazoles and methyldibenzothiophenes was focused on the relative amounts of 4-, 2- +3- and 1-methyl substituted compounds. The relative amounts of methylcarbazole isomers are 43%–58% for 1-MC, 22%–40% for 2- +3-MC, and 16%–23% for 4-MC (Fig. 5a), while variations in the relative amounts of methyldibenzothiophenes show that 4-MDBT varies within the range of 39%–92%; 2- +3-MDBT, 5%–36%; and 1-MDBT, 3%–34% (Fig. 5a). The relative amounts of methylcarbazoles are mainly concentrated in a narrower area, while those of methyldibenzothiophenes are in a wider area, as shown in Fig. 5a. It is shown that variations in the relative amounts of methyldibenzothiophenes are much greater than those in the relative amounts of methylcarbazoles.

The ratio of 4-/1-MDBT was noticed to be of maturity-dependence and used as a maturity indicator

(Radke et al., 1986). For comparison, the relative ratio of 4-/4- +1-MDBT was used in the following discussion. Significant variations in the ratio of 4-/4- +1-MDBT were observed in the samples. The samples with low C_{29}ααα sterane 20S/20S+20R show relatively low values of 4-/4- +1-MDBT (0.52–0.77), and the samples with high C_{29}ααα 20S/20S+20R ratios have relatively high ratios of 4-/4- +1-MBDT (0.80–0.96). However, the corresponding methylcarbazole parameter (1-/1- +4-MC) ranges from 0.66 to 0.76 for all of the samples with no obvious variation between the immature and mature stages. Methyldibenzothiophene parameter varies remarkably, while the methylcarbazole ratio varies within a limited range (Fig. 5b). It is demonstrated that methyldibenzothiophene parameter (4-/4- +1-MDBT) is closely related to maturity (Radke et al., 1986). Therefore, as shown in Fig. 5b, maturity may have much less affect on methylcarbazoles than on methyldibenzothiophenes, indicating little correlation between methylcarbazoles and methyldibenzothiophenes.

3.4 C_2-Carbazoles vs. C_2-dibenzothiphenes

As for C_2-carbazoles and C_2-dibenzothiophenes, some typical isomers are selected for the following discussion. In previous geochemical studies on DMDBTs, the parameter 4, 6-/1, 4-DMDBT was proposed to be a maturity indicator (Chakhmakhchev et al., 1997). For comparison, the relative ratio of 4, 6-/4, 6- +1, 4-DMDBT was used here. Significant variations were observed in the ratio of (4, 6-/4, 6- +1, 4-DMDBT) ranging from 0.34 to 0.75. In addition, the ratio of 4, 6-/4, 6- +1, 4-DMDBT shows a good correlation with C_{29} sterane maturity parameter 20S/20S+20R, indicating that the parameter (4, 6-/4, 6- +1, 4-DMDBT) is a good maturity indicator. The corresponding parameter for carbazoles (1, 8-/1, 8- +1, 4-DMC) also shows significant variations ranging from 0.27 to 0.53, slightly narrower than that of 4, 6-/4, 6- +1, 4-DMDBT. The comparison reveals a remarkable linear correlation (R^2>0.84) between the two parameters in spite of their differences in value (Fig. 6a). The remarkable correlation between the two kinds of compounds may indicate some similar geogeochemical controls on the related isomers of C_2-dibenzothiophenes and C_2-carbazoles and the ratio of 1, 8-/1, 8- +1, 4-DMC, just like the parameter 4, 6-/4, 6- +1, 4-DMDBT, should have a strong maturity-dependence.

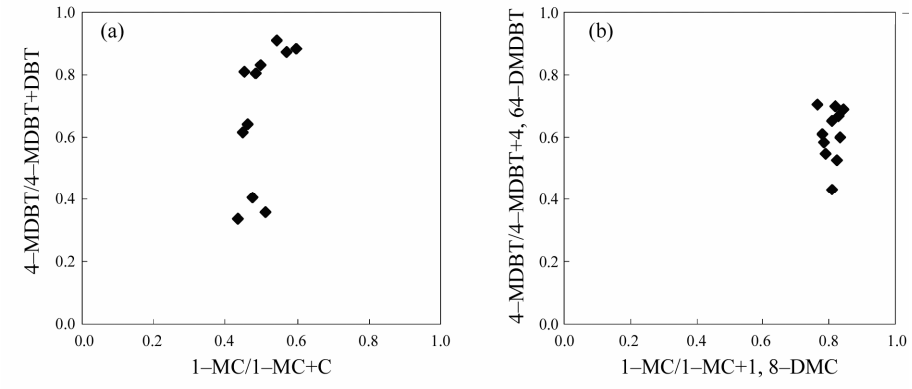

Fig. 4. Comparison of the ratios of C, MC, and DMC with those of the corresponding DBTs.

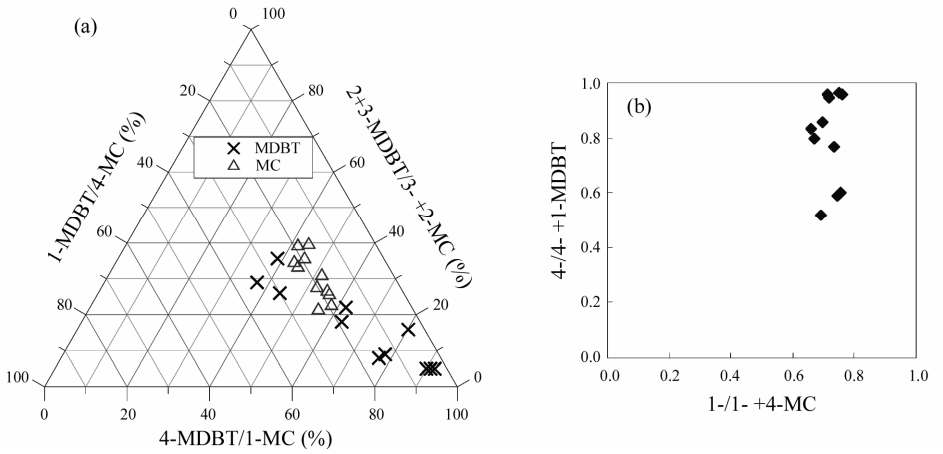

Fig. 5 (a) Ternary plots of the relative amounts of methyl compound isomers; (b) plots of 4-/4- +1-MDBT and 1-/1- +4-MC.

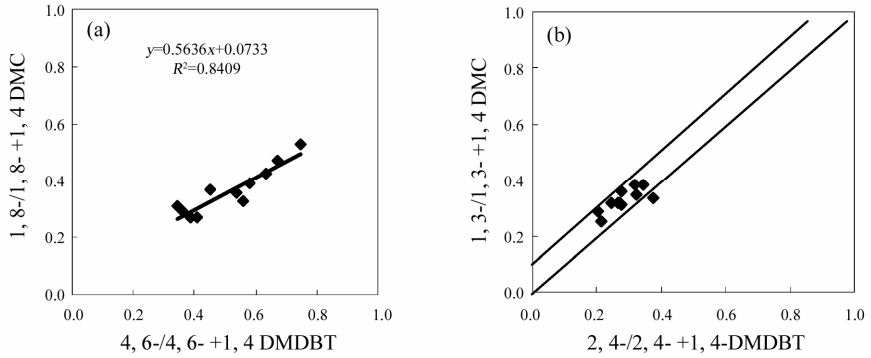

Fig. 6. Comparison of the maturity parameters for DMDBTs and ratios in the corresponding DMCs.

The ratio of 2, 4–/1, 4-DMDBT is another maturity parameter for dimethyldibenzothiophenes (Cakhmakhchev et al., 1997). The ratio of 2, 4-/2, 4-+1, 4-DMDBT ranges from 0.20 to 0.37 in the samples and the parameters for the corresponding carbazoles (1, 3-/1, 3- +1, 4-DMC) range from 0.25 to 0.39. The parameter 2, 4-/2, 4- +1, 4-DMDBT also shows a good linear correlation with the corresponding ratio of carbazoles despite only a small variation range in the ratio values (Fig. 6b).

From the above, the comparison of the distributions of carbazoles and dibenothiophenes in the saline depositional source rocks in immature-mature stages reveals some similarities and differences, which may indicate some similarities and differences in the genetic mechanism or geo-geochemical controls for the two kinds of compounds. Poor correlations of the distributions of C_0-, C_1-, and C_2-carbazoles with corresponding substituted dibenzothiophenes may indicate that alkylation for isomers with different substituted groups could be controlled by different geo-geochemical factors for the two compounds. Budzinski et al. (1991) proposed that the thermodynamic stability order of alkyldibenzothiophenes was C-4> C-2-> C-3> C-1. As for methyldibenzothiophenes, parameter 4-/1-MDBT is a typical maturity indicator (Radke et al., 1986; Radke, 1988). From the above comparison, an apparent variation was observed for 4-/1-MDBT, but 1-/4-MC didn't show any corresponding change, indicating different geochemical controls on the two kinds of methyl compounds. In addition, as for the 4 methylcarbazole isomers, the abundance of 3-MC tends to be reduced with maturity, indicating its low thermodynamic stability, which may show some different thermodynamic stabilities between methylcarbazoles and methyldibenzothiophenes (Budzinski et al., 1991). As for dimethyldibenzothiophenes, C-4 and C-6 were considered to be the most thermodynamic stable positions (Budzinski et al., 1991). Therefore, 4, 6-/1, 4-DMDBT was proposed to be a maturity indicator (Chakhmakhchev et al., 1997). From the above comparison, a remarkable correlation was observed between the dimethyldibenzothiophene maturity parameter and the corresponding dimethylcarbazole ratio, indicating that some similar alkylation or similar geo-geochemical controls for the dimethyl isomers. However, this research entails further research work.

4 Conclusions

The relative amounts of C_0-, C_1-, C_2-carbazoles show little correlation with those of the corresponding dibenzothiophenes. Variations in dibenzothiophenes show a high maturity-dependence, while those in carbazoles show a much lower maturity-dependence.

The relative amounts of methylcarbazole isomers show little correlation with those of the corresponding methyldibenzothiophenes. Parameter 4-/1-MDBT shows a strong maturity-dependence, while ratio 1-/4-MC shows a much lower maturity-dependence. This may indicate that the ratio of 1-/4-MC, usually used as a petroleum migration indicator, is affected little by thermal maturation.

A remarkable correlation was observed between the maturity parameter (4, 6-/1, 4-DMDBT) and the corresponding ratio of carbazoles, indicating a similar genetic or evolution mechanism. The shield/semi-shield parameter (1, 8-/1, 4-DMC) for carbazoles shows a strong maturity-dependence, therefore attention should be paid when 1, 8-/1,4-DMC is used as a migration indicator for oils.

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