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COMPOSITION OF THE ORGANIC CONSTITUENTS OF DAHUANGSHAN OIL SHALE AT THE NORTHERN FOOT OF BOGDA MOUNTAIN, CHINA

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Abstract. *Modern analytical methods of organic petrology and organic geochemistry were used to carry out different tests like, Rock-Eval pyrolysis, Gray-King low-temperature dry distillation, and gas chromatography mass spectrometry (GC/MS) to analyse Dahuangshan oil shale. This paper also studies the abundance, the type and maturity of the organic matter, the content of total organic carbon (TOC), and hydrocarbon generating tendency of Dahuangshan oil shale. Chloroform bitumen A and its group composition were also studied. The results show that Dahuangshan oil shale is rich in organic matter and dominated by Type II_1 kerogen, which has high oil generating potential. However, the degree of transformation of hydrocarbons into oil and gas is low. Group composition of chloroform bitumen A shows high content of saturated hydrocarbons and heavy components, but the content of aromatic hydrocarbons is relatively low. The composition and distribution of saturated hydrocarbons' fractions show that the sedimentary environment of Dahuangshan oil shale was moderately brackish.*

Keywords: *Dahuangshan, oil shale, organic matter, hydrocarbon, sedimentary environment.*

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1. Introduction

Nowadays, the cause, occurrence, composition, structure, and evolution of sedimentary organic matter are mainly studied by the way of organic petrology [1–2], which have made outstanding contributions to the evaluation of organic petrological characteristics, organic geochemical characteristics, and hydrocarbon potential of source rocks [3–4]. Therefore, it is very important to develop a clean and efficient retorting process of oil shale. However, having a detailed understanding of organic composition of oil shale is a base for establishing retorting systems.

As the overall situation of conventional oil and gas resources becomes increasingly severe, oil shale resources start to get more and more attention. Since oil shale is characterized by beneficial features, economic values, and large resources, it is considered as an important substitutional resource for the 21st century [5–6]. Bogda Mountain oil shale has been hot topic over the last 40 years, and many scholars have done a lot to describe the resources, oil yield, sedimentary, and metallogenic characteristics. Hu et al. [7] conducted a survey to make a simple discussion on oil shale properties, and predicted the resources. Li et al. [8] proposed the genetic type of the Bogda Mountain oil shale. Li et al. [9] investigated the sedimentary characteristics of Dahuangshan oil shale in study area. Tao et al. [10] provided new ways to assess the quality and industrial grade of Bogda Mountain oil shale. Tao et al. [11] used step-by-step pyrolysis gas chromatography technique to evaluate the oil retorting process of oil shale samples from the northern foot of Bogda Mountain.

From above studies, we can see that the resource quantity of Bogda Mountain oil shale is very large, and has a tremendous potential for developing and utilizing [5, 12]. In this study, the organic geochemical characteristics, containing information about the organic matter content, maturity, type, and chloroform bitumen A and its group composition, the sedimentary environment, the biogenic composition, and the hydrocarbon generating potential of Dahuangshan oil shale are discussed. This information is significant to develop retorting systems for Dahuangshan oil shale.

2. Samples and tests

A total of 60 oil shale samples were collected from three Dahuangshan profiles, which are located in the northern foot of Bogda Mountain (Fig. 1). All samples come from Lucaogou Formation in Permian, whose lithology is dominated by sandstone, marl, dolostone, and oil shale. The average thickness of the whole formation is about 845 m, and the average thickness of total oil shale ore blocks is about 638 m.

30 oil shale samples were selected for testing and analyzing. The tests include analysing chloroform bitumen A, group composition, Rock-Eval

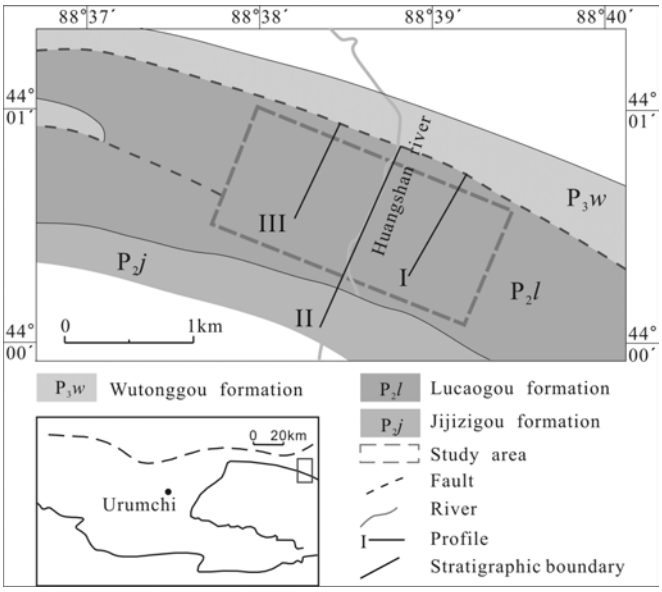

Fig. 1. Location of three profiles.

pyrolysis, *total organic carbon* (TOC), Gray-King low-temperature dry distillation, and *gas chromatography mass spectrometry* (GC-MS). The former four tests were carried out by the Petroleum Geology Research Center, China Petroleum Exploration and Development Research Institute. The applicable standards were SY/T5118-2005, SY/T5119-1995, GB/T 18602-2001, GB/T 19145-2003, respectively. The test instruments were IATROSCAN NEW MK-5, Rock-Eval 2 plus, and LECO CS-400. Gray–King temperature pyrolysis experiment was conducted at the Xinjiang Institute of Coal Science and Coal Testing Laboratory, and the implementation standard was GB/T 1341-2001. The GC-MS test was made by the State Key Laboratory Testing Center of Heavy Oil, China University of Petroleum (Beijing). The implementation standard was GB/T18606-2001, using the equipment of FISONS MD800. Test results are summarized in Table 1.

3. Results and discussion

3.1. Rock-Eval pyrolysis

The information of organic matter abundance, type, maturity etc. can be quickly got from Rock-Eval pyrolysis. The TOC content of Dahuangshan oil shale varies between 8.5%–34.8%, and in about 90% of the samples the content of TOC is more than 10%. The average hydrocarbon potential (S_1+S_2) of the samples is 104.7 mg HC/g. The HI of most samples is more than 600 mg HC/gTOC. The dominating kerogen type is type II_1 kerogen,

Table 1. Test data of Dahuangshan oil shale

Sample No.	Tar, %	TOC, wt%	HI[a], mg HC (g TOC)$^{-1}$	OI[b], mg HC (g TOC)$^{-1}$	T_{max}[c], °C	S_1[d]+S_2[e], mg HC[f]·g^{-1}	Chloroform bitumen A, %	Chloroform bitumen A/TOC, %	SaturatedHC, %	Aromatic HC, %	Non HC, %	Asphaltene, %	Total HC, ppm	Saturated HC / Aromatic HC	Types of organic matter
1	11.5	21.87	630	26	443	139.69	0.36	1.63	20.94	2.78	69.23	7.05	846	7.53	II_1
2	15.5	22.80	612	32	440	142.38	0.67	2.95	5.92	1.64	62.40	30.04	508	3.61	II_1
3	20.5	29.02	622	18	445	183.33	0.27	0.92	5.01	2.12	80.36	12.51	190	2.36	II_1
4	17.5	32.23	571	20	454	185.74	0.23	0.73	18.17	7.59	66.45	7.79	605	2.39	II_1
5	7.0	12.24	580	48	443	72.10	0.44	3.60	21.77	2.23	60.90	15.10	1057	9.76	II_1
6	7.4	13.45	483	18	438	65.74	0.36	2.68	2.78	1.52	89.15	6.55	155	1.8	II_1
7	7.5	13.20	725	48	441	97.37	0.43	3.28	4.36	4.33	75.44	15.87	376	1.01	II_1
8	7.8	21.12	630	18	440	134.06	0.19	0.90	5.07	11.94	73.15	9.84	323	0.42	II_1
9	5.1	11.00	487	24	441	53.97	0.28	2.59	24.39	3.28	69.72	2.61	787	7.44	II_1
10	10.8	13.92	540	23	437	76.33	0.53	3.80	12.23	1.63	82.01	4.13	732	7.50	II_1
11	9.9	17.77	498	19	441	89.96	0.20	1.14	22.24	3.78	69.28	4.70	529	5.88	II_1
12	5.7	14.94	632	8	438	95.32	0.30	2.02	12.65	2.15	79.41	5.79	447	5.88	I
13	7.3	12.35	559	15	439	70.78	0.27	2.19	33.14	28.04	36.26	2.56	1652	1.18	II_1
14	9.6	15.11	540	15	441	82.68	0.34	2.24	14.69	4.97	74.29	6.05	666	2.96	II_1
15	12.2	17.81	609	25	440	111.35	0.46	2.59	3.85	1.98	85.68	8.49	269	1.94	II_1
16	9.9	14.39	798	24	441	115.53	0.24	1.69	11.46	9.74	70.78	8.02	517	1.18	I
17	8.0	9.38	571	26	443	54.27	0.30	3.17	21.83	2.68	73.28	2.21	729	8.15	II_1
18	26.6	27.06	535	17	440	147.82	0.63	2.34	6.67	1.32	81.91	10.10	506	5.05	II_1

Table 1. Continued

Sample No.	Tar, %	TOC, wt%	HI[a], mg HC (g TOC)$^{-1}$	OI[b], mg HC (g TOC)$^{-1}$	T_{max}[c], °C	S_1[d]+S_2[e], mg HC[f]·g^{-1}	Chloroform bitumen A, %	Chloroform bitumen A/TOC, %	SaturatedHC, %	Aromatic HC, %	Non HC, %	Asphaltene, %	Total HC, ppm	Saturated HC / Aromatic HC	Types of organic matter
19	8.1	11.85	605	16	438	72.90	0.16	1.35	16.65	2.39	72.85	8.11	305	6.97	II_1
20	9.4	15.69	696	27	435	111.30	1.25	7.95	7.41	4.54	63.46	24.59	1491	1.63	II_1
21	5.9	11.49	560	10	439	65.80	0.37	3.23	29.23	19.93	47.59	3.25	1823	1.47	II_1
22	7.4	10.22	1068	39	441	109.79	0.24	2.38	16.29	3.63	76.10	3.98	485	4.49	II_1
23	6.6	13.33	431	21	433	59.68	0.81	6.07	21.39	12.22	56.66	9.73	2721	1.75	II_1
24	26.2	34.75	559	11	453	54.27	0.14	0.40	17.91	24.48	54.41	3.20	583	0.73	II_1
25	15.4	13.59	550	49	443	76.65	0.27	1.99	15.66	1.82	78.02	4.50	473	8.60	II_1
26	9.1	15.59	603	40	440	94.78	0.28	1.77	3.90	1.48	84.21	10.41	149	2.64	II_1
27	10.2	8.45	573	37	444	128.19	0.52	6.12	7.95	1.63	79.02	11.40	495	4.88	II_1
28	15.1	8.55	639	19	447	201.14	0.24	2.86	4.43	1.17	88.45	5.95	137	3.79	II_1
29	5.3	23.97	520	50	441	44.99	0.26	1.09	19.69	2.65	74.03	3.63	583	7.43	II_1
30	4.8	32.23	622	5	454	202.17	0.45	1.41	28.83	12.93	52.41	5.83	1895	2.23	II_1
Aver.	10.8	17.31	602	25	442	104.67	0.38	2.57	14.55	6.09	70.90	8.47	735	4.09	

[a] HI = hydrogen index
[b] OI = oxygen index
[c] T_{max} = temperature of maximum S2
[d] S_1 = f ree hydrocarbons
[e] S_2 = pyrolysable hydrocarbons
[f] HC = hydrocarbon.

indicating that Dahuangshan oil shale is rich in organic matter and has high oil generating potential (Table 1).

Pyrolysis parameters HI and OI are important indexes in reflecting the type of organic matter [13–14]. High HI and low OI value in Dahuangshan oil shale reflect a sapropelic organic component (Fig. 2).

3.2. Chloroform bitumen A and its group composition

The content of chloroform bitumen A in Dahuangshan oil shale is between 0.14–1.25%. The ratio of chloroform bitumen A content to organic carbon is 0.40–7.95%, and the average value is 2.57%. So the yield of soluble organic matter is relatively high, which also shows a good hydrocarbon-generating potential (Table 1).

Figure 3 and Table 1 show two remarkable characteristics of the group composition of chloroform bitumen A in Dahuangshan oil shale. One is high yields of total hydrocarbons (saturated hydrocarbons plus aromatic hydrocarbons) of 137–2721 ppm, with an average of 734 ppm, mainly composed by saturated hydrocarbons. Generally, the ratio of saturated hydrocarbons to aromatic hydrocarbons in Type I kerogen is above 1, whereas the ratio in Type III kerogen is below 1. In 30 tested samples, most of the ratios are above 1, and nearly half of the ratios reach up to 3, showing that the sapropelic parent material occupies absolute predominance. The other is the content of heavy components (asphaltenes plus nonhydrocarbons). Their content varies between 38.8–95.7% with an average value of 79.4%, and non-hydrocarbons take large proportion. Generally, the high ratio of non-hydrocarbons to asphaltenes shows a low organic maturity, which also means that the polymerization degree of lipid hetero atomic compounds is low. Through above mentioned

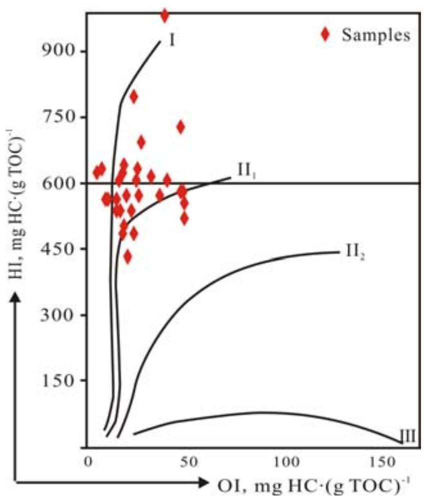

Fig. 2. Relationship between HI and OI of Dahuangshan oil shale.

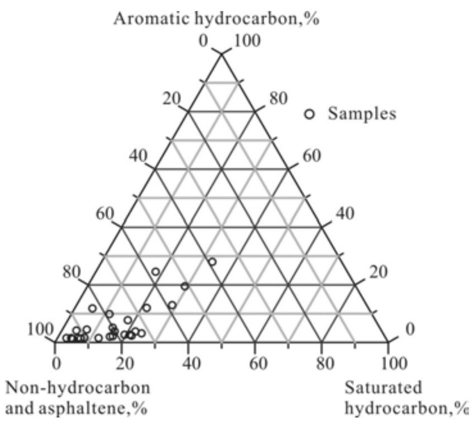

Fig. 3. Distribution of soluble organic matter in oil shale.

analysis, we can see that Dahuangshan oil shale is rich in organic matter, and soluble organic matter is heavier. The transformation degree of hydrocarbon source to oil and gas is low, which can be verified by the T_{max} value, which is approximately 440 °C. Therefore, the hydrocarbon generating potential of kerogen is not largely released.

3.3. Saturated hydrocarbon fractions

Saturated hydrocarbon fractions of chloroform bitumen A are mainly composed of n-alkanes, branched alkanes, isoprenoid alkanes, alkyl cyclohexane, cyclic sterane, terpane etc. Most of these compounds have important geological and geochemical significance, such as biogenic composition, sedimentary environment, and thermal maturity. Meanwhile, it also has significance in indicating the composition of oil products of oil shale.

Chain alkanes are the most abundant compounds in saturated hydrocarbon fraction, which occupy 50–80% in general (Fig. 4). N-alkanes are widely distributed in chain alkanes, which always have three types of distribution patterns. The first type has double-peak structure. The former peak is composed of nC_{16}-nC_{18}, and some samples consist of nC_{15}, nC_{17} or nC_{19}, which reflects the presence of homonemeae microorganisms. The later peak consists of nC_{25}-nC_{33}, and has often obvious odd-even predominance. It means the input of waxiness from higher plants and the low organic matter maturity of source rocks [13]. There are many kinds of biogenic origin inputting in continental lake basin source rocks, therefore, double-peak structure is the most common for n-alkanes in low maturity source rocks. The second type has three peaks, which barely exists in low maturity source rocks of brackish-saline environment. What is different from the type one is that there is one more peak between nC_{22} and nC_{23}. The third type has only one peak, which is common in source rocks of brackish-saline environment, and its main peak is nC_{22} or nC_{23}.

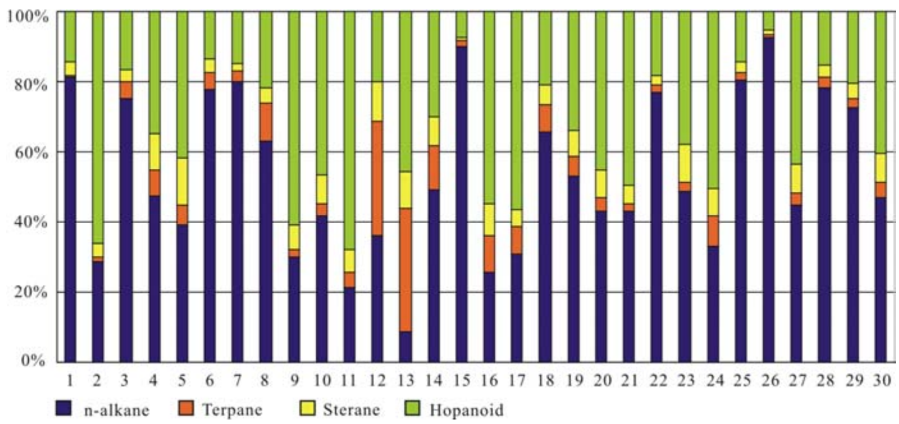

Fig. 4. Saturated hydrocarbon composition of chloroform bitumen A in Dahuangshan oil shale.

From the above analysis, we can see that the carbon number distribution of n-alkanes is influenced by different biogenic origins and evolution of parent materials.

The Carbon preference index (CPI) is a measure of the predominance of *n*-alkanes with an odd number of carbon atoms, which was first proposed by Bray and Evans [15]. The CPI is expressed as a summation of the odd number homologues within a specified range of carbon numbers divided by a summation of the even number homologues within the same range [16]. Dahuangshan oil shale has the characteristics of odd even predominance, which is not significant (Fig. 5), and has just one peak with lower carbon number (from nC_{19} to nC_{22}) (Fig. 6). It shows that most of homonemeae microorganisms are of biogenic origin, and the deposition condition were in partially saline environment.

Isoprenoid alkane in saturated hydrocarbon fractions is primarily composed of phytane series. The pristane/phytane (Pr/Ph) ratio is one of the most commonly used geochemical parameters and has been widely invoked as an indicator of the redox conditions in the depositional environment and source of organic matter [17–18]. Organic matter originating predominantly from land plants would be expected to contain high Pr/Ph > 3.0 (oxidizing conditions). Low values of (Pr/Ph) ratio (<0.6) indicate anoxic conditions and values between 1.0 and 3.0 suggest intermediate conditions (suboxic conditions) [19]. In study area, the value of Pr/Ph in most tested samples is between 1 and 3, which reflects a reducing sedimentary environment, and the sedimentary aqueous media under moderate salinization (Fig. 7).

As shown in Fig. 4, the sterides and terpenoids are very common in saturated hydrocarbon fractions. From Fig. 8 and Table 2, we can see that 17α(H)-22,29,31-trisnorhopane (Tm), 17α(H),21β(H)-30-norhopane, 17α(H),21β(H)-hopane, 17β(H),21α(H)-hopane, and 17α(H),21β(H)-29-homohopane 22S are the subjects of terpenoids. These pentacyclic triterpenoids principally come from prokaryotic organisms, and only partially from plants.

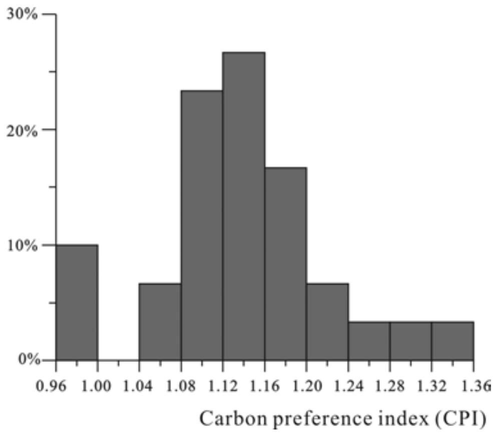

Fig. 5. CPI of n-alkane in Dahuangshan oil shale.

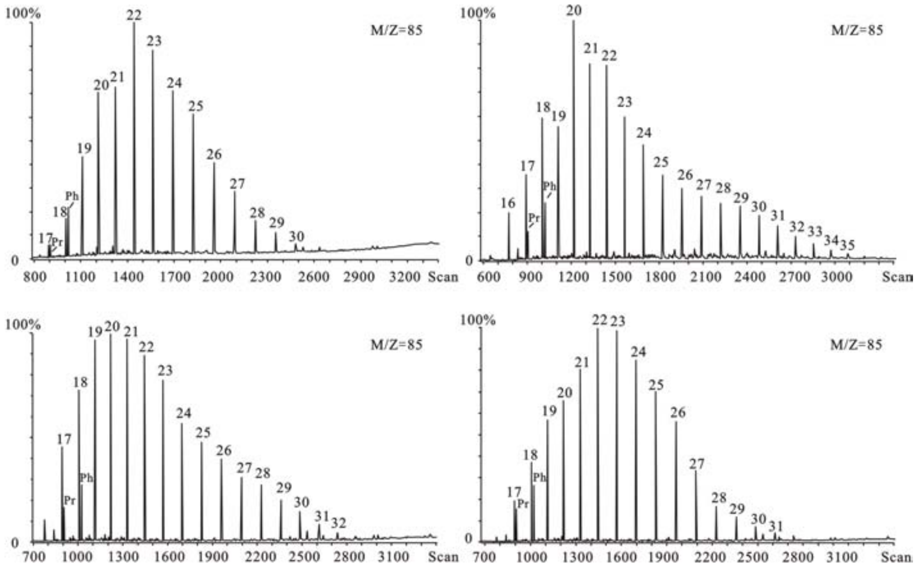

Fig. 6. Distribution of n-alkane in Mass-to-Charge (M/Z) 85 mass chromatogram.

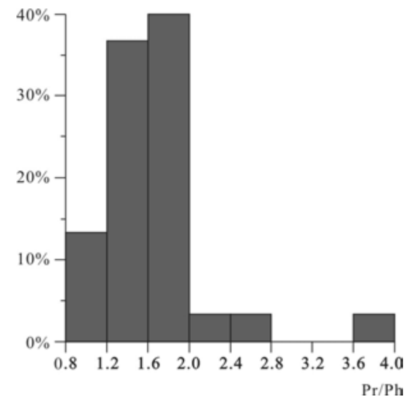

Fig. 7. The value of Pr/Ph of Dahuangshan oil shale.

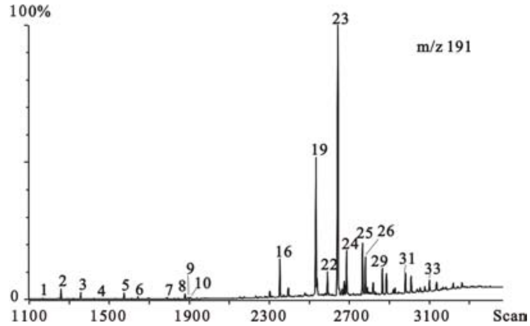

Fig. 8. Distribution of terpane compounds.

Table 2. Content of terpane compounds (m/z 191)

Peak number	Name of the compound	Percentage, %
16	17α(H)-22,29,31-trisnorhopane (Tm)	0.0219
19	17α(H),21β(H)-30-norhopane	0.0788
22	17β(H),21α(H)-30-norhopane	0.0138
23	17α(H),21β(H)-hopane	0.1840
24	17β(H),21α(H)-hopane	0.0249
25	17α(H),21β(H)-29-homohopane 22S	0.0286
26	17α(H),21β(H)-29-homohopane 22R	0.0216
27	Gammacerane	0.0037

As shown in Fig. 9 and Table 3, 5α,14α,17α-cholestane 20R, 5α,14α,17α-ergostane 20R, and 5α,14α,17α-sitostane 20R are the subjects of steroids. Steroids may come from terrestrial plant, phytoplankton or zooplankton. Reduction, hydrogenation, and biodegradation are helpful to form sterane in sedimentary rocks. So the relative enrichment of steroids and terpenoids reflect the accumulation of zooplankton and phytoplankton, which is similar to Green River oil shale, rich in both steroids and pentacyclic triterpenoids (hopane) [20].

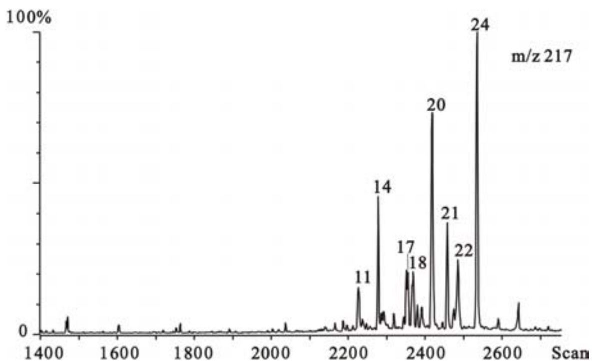

Fig. 9. Distribution of sterane compounds.

Table 3. Content of sterane compounds (m/z 217)

Peak number	Name of the compound name	Percentage, %
11	5α,14α,17α-cholestane 20S	0.0023
14	5α,14α,17α- cholestane 20R	0.0044
17	5α,14α,17α-ergostane 20S	0.0019
18	5α,14β,17β- ergostane 20R	0.0033
20	5α,14α,17α- ergostane 20R	0.0119
21	5α,14α,17α- sitostane 20S	0.0047
22	5α,14β,17β- sitostane 20R	0.0047
24	5α,14α,17α- sitostane 20R	0.0140

3.3.1. Aromatic fractions

There are many kinds of compounds of aromatic fraction, with complicated composition, which bear plenty of geochemical information. They can also be used to indicate the biogenic origin, evolution, and other features of source rocks. Because of the volatilization of low-ring aromatics during the process of extraction, the tested aromatic compounds are always polycyclic aromatic hydrocarbons, including naphthalene, phenanthrene, pyrene, chrysene, biphenyl, fluorine, fluoranthene, and so on. Most of them are the aromatized degradation products from steroids and terpenoids with different biogenic origin, so they do not have significance in indicating the biogenic origin.

Triaromatics in aromatic fraction are mainly composed of phenanthrene and C1-C5-alkylated phenanthrenes. There are some differences in phenanthrene series composition in genetically different source rocks. The content of phenanthrene series increases gradually with the increase in maturity of source rocks, therefore, the content of phenanthrene series is usually higher in mature source rocks compared to immature and low mature source rocks. Tetraaromatics mainly come from the aromatization during the diagenesis of organic parent material, and consist mainly of chrysene, pyrene, and its alkyl derivatives (Fig. 10). From the above analysis, due to the input of simpler organisms, and low evolution degree of organic matter, the content of aromatic hydrocarbon products of Dahuangshan oil shale is lower compared to the content of saturated hydrocarbons.

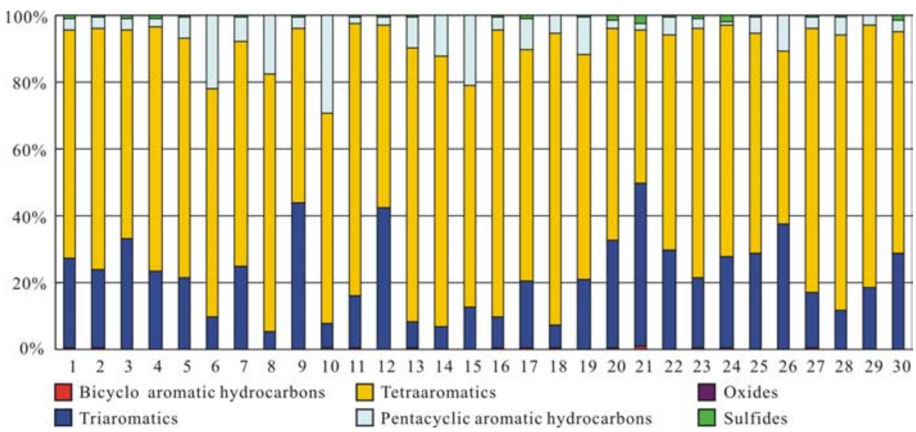

Fig. 10. Composition of aromatic hydrocarbons of chloroform bitumen A in Dahuangshan oil shale.

4. Conclusions

The content of TOC is over 10% in approximately 90% of Dahuangshan oil shale samples; the average values of S_1+S_2 and chloroform bitumen A are 104.7 mg HC/g and 2.57%, respectively; dominating kerogen type is type II_1 kerogen; the T_{max} value is around 440 °C. All those parameters indicate that Dahuangshan oil shale is rich in organic matter with a low evolution degree, and has a strong tendency of oil generation. Group composition of chloroform bitumen A shows high content of saturated hydrocarbons and heavy components and relatively low content of aromatic hydrocarbons. The composition and distribution features of saturated hydrocarbon fractions show that sedimentation conditions of Dahuangshan oils shale were moderately saline, with the accumulation of zooplankton and phytoplankton.

5. Acknowledgements

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