

ORGANIC GEOCHEMISTRY OF THE PALEOCENE-EOCENE OIL SHALES OF THE GONGJUE FORMATION, NANGQIAN BASIN, EAST-CENTRAL TIBETAN PLATEAU

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Abstract. *The Paleocene-Eocene oil shales of the Gongjue Formation in the Nangqian Basin, east-central Tibetan Plateau, were investigated for their petroleum potential by analysis of organic matter (OM) type, source, richness, thermal maturity, and depositional environment. According to the T_{max} and molecular indicators, the samples were found to be thermally immature or at the early stages of maturity. The hydrogen index (HI) values and atomic ratios of carbon, hydrogen and oxygen show that the organic matter of oil shales is primarily of Type I or II. The high share of gammacerane and the low pristane-to-phytane (Pr/Ph) ratio indicate that the organic matter was deposited in saline paleoenvironmental settings.*

Keywords: *Nangqian Basin, Gongjue Formation, Paleocene-Eocene oil shales, Tibetan Plateau, organic geochemistry.*

1. Introduction

The Tibetan Plateau is referred to as the last frontier in petroleum exploration in China because it is located in the eastern part of the Tethyan Hydrocarbon Province, one of the most significant regions of hydrocarbon resources worldwide [1, 2]. For decades, numerous studies have focused on the sedimentary basins distributed across the “roof of the world”, despite a number of challenges such as a complicated tectonic history and poor natural

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environmental conditions. In fact, some of these basins, like the Lunpola Basin [3, 4] and the Qiangtang Basin [5, 6], have proven to have promising prospects with industrial oil flow or oil shows.

The Nangqian Basin is located in the hinterland of the Tibetan Plateau. Previous research on the Nangqian Basin focused on the sedimentary characteristics of the basin fill [7–9], the basin's structural evolution [10], and potassium-rich volcanic rocks [11, 12]. However, a comprehensive study on the geochemistry of the oil shale of the Nangqian Basin has yet to be conducted. Oil shale deposits are widely distributed throughout several regions of China, with proven reserves of approximately 7199.37×10^8 tons (when converted to shale oil, approximately 476.44×10^8 t) [13]. The globally growing interest, especially in China, Estonia, Brazil and Australia, in oil shale over the past few decades has been related to its importance as a natural unconventional fuel resource that could be mined and converted to liquid fuel. In addition, the multi-purpose utilization of oil shale considering its energy potential and the minerals contained improves the competitiveness of the rock [14].

In this paper, we present the first organic geochemical data for oil shale from the Nangqian Basin, east-central Tibetan Plateau. The main objective of the study is to determine the type, proneness and maturity of organic matter, as well as the depositional environment of the shales in order to evaluate the hydrocarbon potential of the basin. We also aim at improving the understanding of the paleoenvironmental conditions during the deposition of the shale by using organic geochemical proxies.

2. Geological background

The Nangqian Basin, with an average elevation of over 4000 m, is a lacustrine-dominated Cenozoic sedimentary basin in the east-central Tibetan Plateau near the headwaters of the Lancangjiang River (also called the Mekong River). The NW-SE elongated basin is approximately 55 km long and 18 km wide [15]. The basin is situated in the middle part of the Qiangtang terrane, which is bounded by two major suture zones, the Jinshajiang Suture to the northeast and the Bangong-Nujiang Suture to the southwest (Fig. 1a).

The sedimentary basin fill is mainly composed of the Gongjue Formation, while the Nangqian Basin is similar to the nearby Gongjue Basin in terms of age, sedimentary fill and tectonic setting [17].

The Gongjue Formation of the Nangqian Basin, with a thickness of over 2000 m, unconformably overlies the Late Triassic strata. The measured sections A and B are located in the western margin of the Nangqian Basin (Fig. 1b). Based on the findings by Horton et al. [8] and our field observations, we divided the Gongjue Formation into three lithostratigraphic units (Fig. 2).

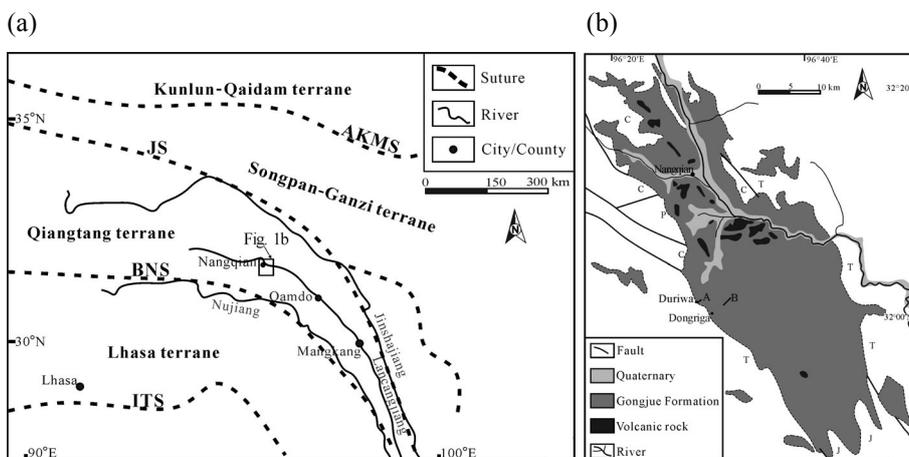


Fig. 1. (a) Simplified tectonic map of the study area and the adjacent region, including suture zones and major rivers (modified from [16]). Abbreviations: AKMS – Anyimaqin-Kunlun-Muztagh Suture, JS – Jinshajiang Suture, BNS – Bangong-Nuijiang Suture, ITS – Indus-Tsangpo Suture; (b) simplified geological map of the Nangqian Basin. Locations of the measured sections A and B are marked. Abbreviations: J – Jurassic, T – Triassic, P – Permian, C – Carboniferous.

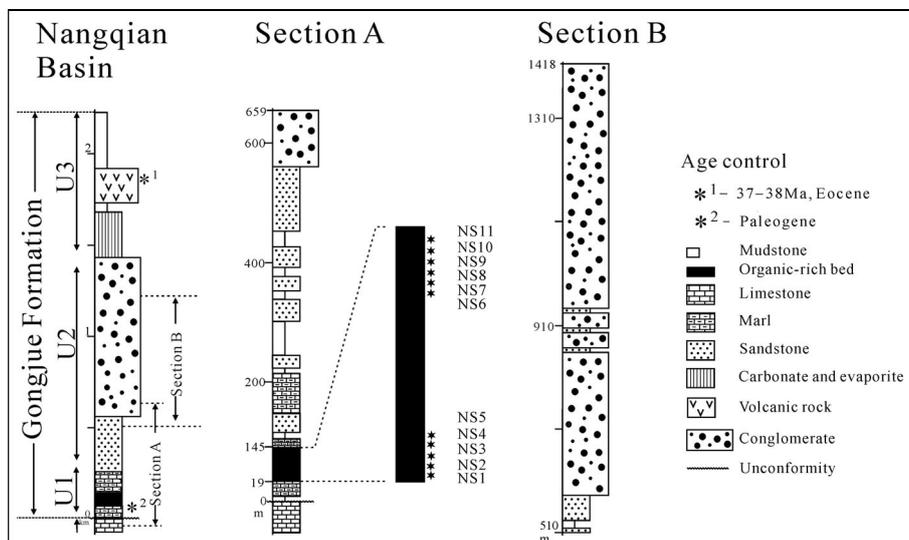


Fig. 2. Lithology log of sediments of the Nangqian Basin (based on [8] and our observations) and measured sections (sections A and B), including the location of the studied samples and age controls based on [8, 18].

The oldest of the lithostratigraphic units (U1) is well exposed at Duriwa and Dongriga villages along the southwestern part of the basin. This unit comprises gray mudstone and marl with thin interbeds of oil shale, which suggests deposition in an offshore lacustrine environment. The organic-rich oil shale can be ignited directly. Palynomorph and ostracod assemblages are present in this unit and indicate that it is likely of Paleocene to Eocene age [18].

Unit 2 (U2) is very thick, over 1000 m, and is primarily composed of alluvial sandstone and medium- to thick-bedded, light gray to red, cobble-boulder conglomerate, which suggests that deposition occurred proximal to its source areas and may be related to tectonism during the Cenozoic Indo-Asian collision. The uppermost unit (U3) is a lacustrine deposit which consists of mudstone, minor carbonates, evaporite and volcanic rock. The volcanic rock is well known for its widespread deposition in the Qamdo-Nangqian area. The Middle-Upper Eocene tuffs of this unit have $^{40}\text{Ar}/^{39}\text{Ar}$ ages of 37 to 38 Ma in the Nangqian Basin [10].

3. Materials and methods

A total of 11 outcrop oil shale samples were collected from section A of the Nangqian Basin (Fig. 2). The samples were collected after digging approximately 0.5 m into the subsurface and removing the uppermost layer to minimize the influence of surface weathering.

The samples were ground to analytical grain sizes (approximately 100 mg and 120 mesh) and dried at 60 °C prior to further treatment. After the removal of carbonates by acidification using hydrochloric acid (HCl), a Leco CS-200 carbon-sulfur elemental analyzer was used to determine the total organic carbon (TOC) values. Rock-Eval pyrolysis was carried out on all of the samples using a Rock-Eval II instrument. The crushed samples were extracted with chloroform in a Soxhlet apparatus for 72 h. The obtained extracts are a measure of the amount of soluble organic matter (SOM). The asphaltenes were precipitated with petroleum ether, and then the deasphalted extracts were further separated by column chromatography into saturated hydrocarbons, aromatic hydrocarbons and NSO compounds by using a silica gel alumina column (500 mm × 10 mm ID, over 12 h).

The saturated hydrocarbon fractions were analyzed on an Agilent 6890 N gas chromatograph (GC) equipped with a 30 m × 0.20 mm fused silica column (0.2 μm film). The oven was gradually heated from 70 to 300 °C at a rate of 8 °C/min, followed by an isothermal period of 20 min. An injection was performed in a split/splitless mode with an injector temperature of 300 °C. Helium was used as the carrier gas.

The gas chromatography-mass spectrometry (GC-MS) analysis of saturates was completed using a TRACE2000/SSQ-7000 mass spectrometer connected to the gas chromatograph. Helium was used as the carrier gas, and a 30 m × 0.20 mm Varina CP Sil-8CB fused silica column (0.2 μm film) was

applied. The operating temperature was programmed from 80 to 160 °C at a rate of 8 °C/min and further to 310 °C at 2.8 °C/min, with a final isothermal hold at 310 °C for 5 min.

Elemental analyses were performed on a FLASH EA-1112 Series elemental analyzer with an analytical precision of 0.3% for carbon and 0.5% for nitrogen [19].

All the experiments were performed at the Organic Geochemistry Laboratory, Institute of Exploration and Development, Huabei Oilfield Branch Company of China National Petroleum Corporation (CNPC).

4. Results and discussion

4.1. TOC, SOM and Rock-Eval

The TOC, SOM and Rock-Eval pyrolysis data for oil shale samples from the Gongjue Formation of the Nangqian Basin are summarized in Table 1. The TOC of the samples varies between 4.19 and 6.47 wt%, with the corresponding SOM values between 1193 and 2923 ppm. The Rock-Eval analysis of the bulk samples yielded hydrogen index (HI) values of 449 to 628 mg HC/g TOC. The hydrocarbon potential and free hydrocarbon content of the entire sample suite is indicated by parameters S_2 and S_1 , respectively. The average S_1 and S_2 values are 2.96 and 30.04 mg HC/g rock, respectively (Table 1). The calculated parameter PY ($S_1 + S_2$) is known as the potential yield and fluctuates between 28.30 and 42.43 mg HC/g rock.

Table 1. TOC, SOM and Rock-Eval pyrolysis data and calculated parameters for outcrop oil shale samples from the Gongjue Formation in the Nangqian Basin

Sample No.	TOC, wt%	SOM, ppm	T_{max} , °C	S_1 , mg HC/g rock	S_2 , mg HC/g rock	PY, mg HC/g rock	HI, mg HC/g TOC	PI
NS1	4.19	1193	419	2.13	26.17	28.30	625.25	0.08
NS2	5.07	1740	421	2.61	31.81	34.43	627.73	0.08
NS3	5.18	1421	421	2.47	31.07	33.53	599.83	0.07
NS4	4.26	2149	419	2.61	26.38	28.99	619.00	0.09
NS5	4.66	1668	418	2.63	26.73	29.36	573.90	0.09
NS6	6.47	2923	420	3.66	38.76	42.43	599.24	0.09
NS7	5.35	2051	414	3.40	28.37	31.77	530.03	0.11
NS8	5.93	2189	414	2.98	26.62	29.60	449.21	0.10
NS9	6.03	2516	416	3.62	33.55	37.17	556.08	0.10
NS10	5.36	2419	420	3.23	33.10	36.33	617.61	0.09
NS11	4.97	2434	413	3.22	27.94	31.16	562.44	0.10

Note: SOM – soluble organic matter; PY – potential yield ($S_1 + S_2$); HI – hydrogen index ($S_2 \times 100/TOC$); PI – production index [$S_1/(S_1 + S_2)$].

4.2. Thermal maturity

Besides vitrinite reflectance ($\%R_o$), T_{max} is another thermal stress parameter that can be used to estimate the thermal maturity of oil shale [20]. The T_{max} values for the Nangqian oil shale samples are similar and relatively low, ranging from 413 to 421 °C (Table 1). The calculated parameter $S_1/S_1 + S_2$ is known as the production index (PI), which is also related to the stage of petroleum generation. For the samples the Rock-Eval T_{max} is below 435 °C, and PI less than 0.1 (except for sample NS7), suggesting that these are immature [4, 20].

Biomarkers hold information about the origin of organic matter but also record the level of maturity of the host sample. The moretane/hopane (M/H) ratio is sensitive to maturity because the former compound is thermally less stable than the latter [21–23], which allows the ratio to serve as a maturity indicator, especially for samples in the immature-early mature stage. Peters et al. [20] suggested that the M/H ratio near 0.8 indicates the immature stage, whereas M/H less than 0.15 is typical of the mature stage. Table 2 presents the M/H ratios for all of the analyzed samples. The relatively high M/H ratios (0.83 to 1.11) suggest that the Nangqian oil shale samples are immature. Furthermore, the degree of isomerization of biological markers, such as steranes and hopanes, is now widely applied to indicate the maturity of source rocks and petroleum. Isomerization at C-22 in C_{32} homohopane gives rise to the change of the 22S/(22S + 22R) ratio from zero to the equilibrium value, 0.57–0.62, during maturation [22]. The Nangqian oil shale samples have 22S/(22S + 22R) ratios between 0.29 and 0.47 (Table 2), while the fact that all these values remain below 0.57 suggests that the samples are mostly immature.

These maturity indicators are all related to the increasing maturity and can be correlated with each other. Overall, the assessed thermal maturity level based on the presented parameters implies that the samples are immature to early mature and are just entering the oil window.

4.3. Organic matter type and source

4.3.1. Elemental analysis of kerogen

The organic matter in sedimentary rocks possesses different characteristics and compositions due to the different types of sources. Traditionally, there are distinguished three types of kerogen: I, II and III, which on the Van Krevelen diagram show diverse evolution paths [24]. The elemental analysis data for the Nangqian oil shale samples are listed in Table 3. The atomic H/C and O/C ratios range from 1.44 to 1.58 and from 0.13 to 0.27, respectively. Typically, a cross-plot of H/C vs O/C is used to determine the kerogen type. Figure 3 shows that the organic matter in the studied samples is mainly of Type I or II.

Table 2. Geochemical parameters for organic extracts of Nangqian oil shale samples

Sample No.	Pr/Ph	Pr/n-C ₁₇	Ph/n-C ₁₈	CPI	GI	22S/(22S + 22R)	M/H	C ₂₇ %	C ₂₈ %	C ₂₉ %
NS1	0.11	0.13	8.16	4.03	3.27	0.43	1.03	49	15	36
NS2	0.10	0.11	4.89	2.94	2.90	0.47	0.87	50	15	35
NS3	0.12	0.15	8.58	4.14	2.89	0.42	0.98	51	15	34
NS4	0.11	0.10	8.63	3.88	2.97	0.45	0.89	49	16	35
NS5	0.11	0.12	8.42	3.82	3.01	0.43	0.93	48	15	37
NS6	0.20	0.49	2.12	2.78	1.84	0.29	1.11	53	25	22
NS7	0.19	0.46	1.62	2.23	1.96	0.39	0.83	49	28	23
NS8	0.15	0.35	1.66	2.15	2.15	0.42	0.90	52	22	26
NS9	0.20	0.49	2.24	3.04	1.96	0.33	1.11	52	27	21
NS10	0.19	0.46	2.89	3.24	1.96	0.35	1.08	53	21	26
NS11	0.20	0.47	3.01	3.21	2.07	0.35	1.00	50	27	23

Note: Pr – pristane; Ph – phytane; CPI – carbon preference index; GI – gammacerane index = gammacerane/[C₃₁ hopane(22S + 22R)/2]; 22R/(22S + 22R) – 22R/(22S + 22R) homohopane (for C₃₂); M/H – moretane/hopane (for C₃₀); C₂₇% – C₂₇ααα(R)/C₂₇–C₂₉ααα(R) steranes; C₂₈% – C₂₈ααα(R)/C₂₇–C₂₉ααα(R) steranes; C₂₉% – C₂₉ααα(R)/C₂₇–C₂₉ααα(R) steranes.

Table 3. The results of elemental analysis of Nangqian oil shale samples

Sample No.	C, %	H, %	O, %	H/C	O/C
NS1	56.18	6.73	11.44	1.44	0.15
NS2	56.58	7.09	10.01	1.50	0.13
NS3	58.43	7.46	10.45	1.53	0.13
NS4	56.51	7.24	9.57	1.54	0.13
NS5	56.91	7.30	9.75	1.54	0.13
NS6	57.03	7.34	10.11	1.55	0.13
NS7	55.51	7.17	9.74	1.55	0.13
NS8	53.60	6.94	9.09	1.55	0.13
NS9	58.01	7.52	9.85	1.56	0.13
N4S10	31.53	3.63	8.96	1.38	0.21
NS11	30.04	3.70	10.83	1.48	0.27

4.3.2. Biomarkers

Gas chromatograms of the saturated hydrocarbons of selected Nangqian oil shale samples are shown in Figure 4, and the parameters of all samples are summarized in Table 2.

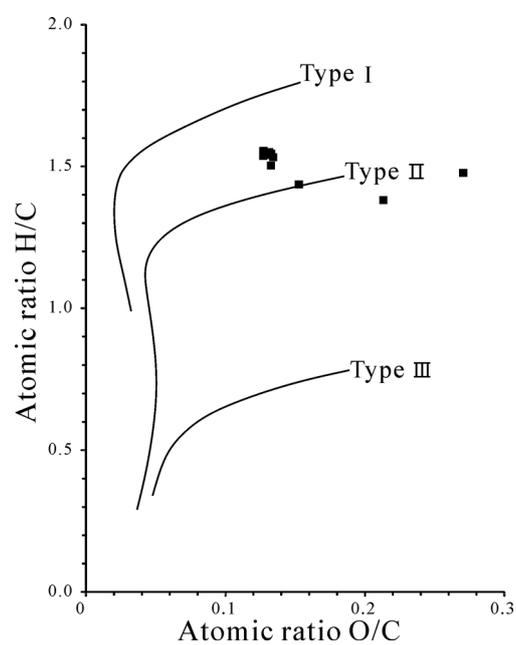


Fig. 3. Cross-plot of H/C vs O/C atomic ratios of the analyzed Nangqian oil shale samples showing the type of kerogen.

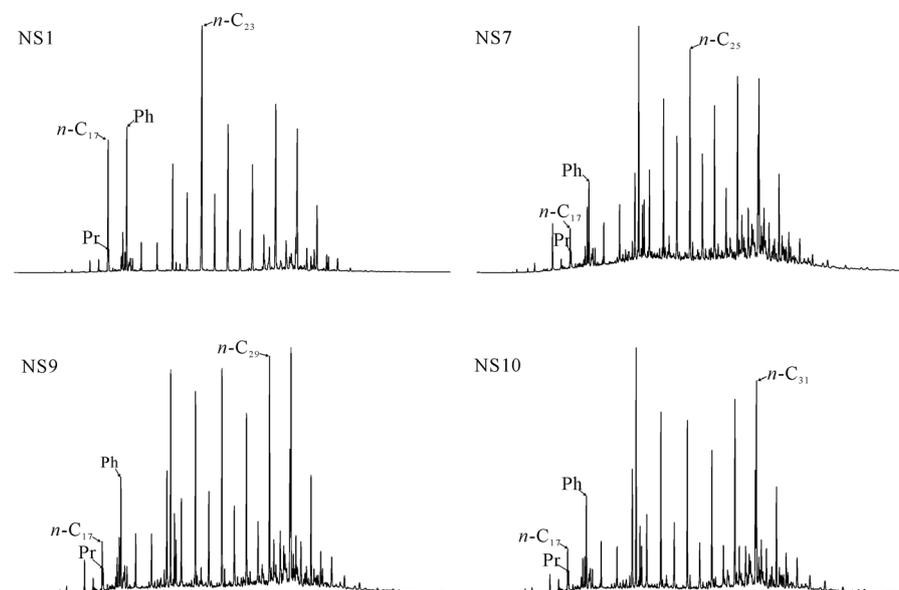


Fig. 4. Gas chromatograms of the saturated fractions of selected Nangqian oil shale samples.

The extracts contain a wide range of n -C₁₅ to n -C₃₈ alkanes. The n -alkane distribution (Fig. 4) is dominated by middle- to long-chain (n -C₂₁₊) alkanes with an odd-over-even preference with high carbon preference index (CPI) values (2.23–4.14). Short-chain n -alkenes (n -C₂₁₋) composed of mixtures of n -C₁₅, n -C₁₇ and n -C₁₉ are derived from algal and zooplanktonic sources, whereas the higher plants are prone to produce long-chain n -alkenes [25]. The much greater concentrations of n -C₂₇, n -C₂₉ and n -C₃₁ alkanes, compared with n -C₁₅, n -C₁₇ and n -C₁₉ homologues, are typical of higher land plants [26].

The mass chromatograms (m/z 217, m/z 191) in Figure 5 depict the distribution of regular steranes and hopanes, respectively, and Table 2 lists the relative proportions of $\alpha\alpha\alpha$ (R) steranes.

The high proportion of C₂₇ steranes indicates the planktonic origin of organic matter, whereas the dominance of C₂₉ points to the input of terrigenous organic material; hence, the composition of steranes could be used to identify the OM source [27, 28]. The samples have a higher proportion of C₂₇ sterane (48–54%) compared to C₂₉ (21–37%) and C₂₈ (15–28%) homologues (Table 2), which gives evidence of a major contribution of aquatic algal-bacterial sources with considerable terrigenous organic matter input, as illustrated by the distribution of steranes (Fig. 6a) and a higher proportion of the short-chain n -alkanes.

Based on the distribution of steranes, the abundance of wax n -alkanes with a marked odd carbon number predominance, as well as high atomic H/C ratios, we propose a mixed source with both aquatic and terrigenous organic input.

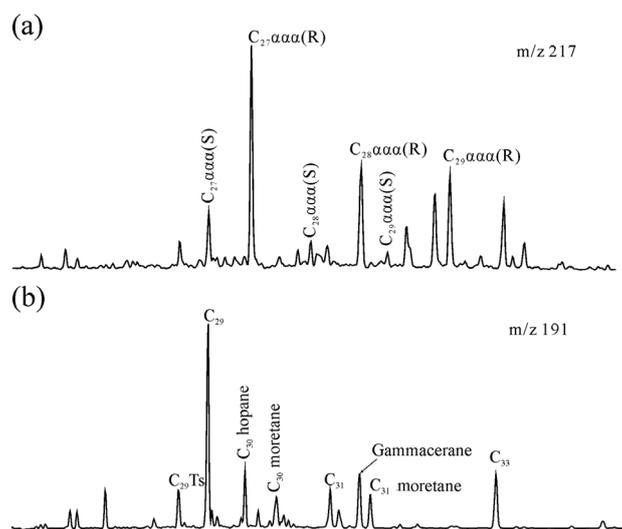


Fig. 5. Mass chromatograms for a representative oil shale sample NS7 showing the distribution of (a) steranes (m/z 217) and (b) hopanoids (m/z 191).

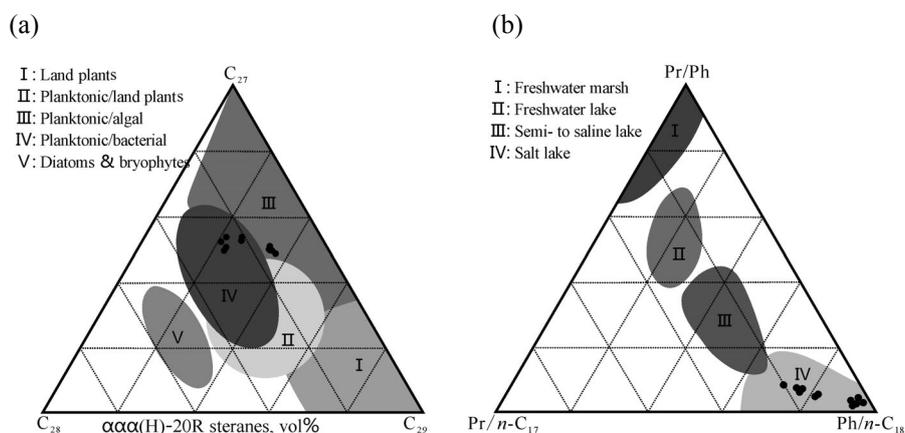


Fig. 6. (a) Ternary diagram of $\alpha\alpha\alpha(H)$ -20R steranes (C_{27} , C_{28} , C_{29}) showing the source of organic matter in the studied oil shale samples (after [28]); (b) ternary diagram of Pr/Ph, Ph/ n - C_{18} and Pr/ n - C_{17} ratios showing the depositional conditions of the samples (after [34]).

4.4. Hydrocarbon potential

Usually, outcrop samples have been somewhat affected by long-term weathering, especially for the rocks from the Tibetan Plateau. In addition, the evaluation criteria of the hydrocarbon source rock are different depending on rock type and the location of the study area [4]. Nevertheless, the oil shale from the lower part of the Gongjue Formation of the Nangqian Basin, with high TOC, PY and HI, is considered to be a high quality source rock, according to the evaluation criteria of the hydrocarbon source rock in the Qiangtang Basin of the Tibetan Plateau [29, 30]. Oil shale samples from the Gongjue Formation are characterized by relatively low maturity and their kerogen type is mostly II. Therefore, if subjected to appropriate burial and heating, the hydrocarbon potential of the Gongjue Formation may be sufficient to generate liquid hydrocarbons. However, due to the current insufficient exploration, poor natural environmental conditions and lack of necessary facilities, the economic utilization of this oil shale is quite impossible. At the same time, Tertiary lacustrine oil shales are widely spread in the interior of the Tibetan Plateau, especially in the basins of central Tibet (e.g., Lunpola and Qiangtang). So, utilization of the Gongjue Formation oil shale might be possible if development and exploitation is carried out concurrently with the respective activities in the adjacent basins.

4.5. Depositional environment

Acyclic isoprenoids occur in significant amounts in all of the studied oil shale samples. The pristane-to-phytane ratios are listed in Table 2. In all the

samples, the Ph content is higher than that of Pr, giving Pr/Ph ratios between 0.10 and 0.23. The Pr/Ph ratio reflects redox conditions during deposition of the source rock [31, 32]. However, Ten Haven et al. [33] argue that the Pr/Ph ratio cannot be used as an indicator of oxygen levels. Peters et al. [20] have suggested that $\text{Pr/Ph} < 0.8$ indicates saline to hypersaline conditions associated with the deposition of evaporite and carbonates. Constructed on the basis of the ratios of isoprenoids and *n*-alkanes, the ternary diagram in Figure 6b implies the deposition of oil shale in a salt lake, further justifying the use of the Pr/Ph ratio as a proof of paleo-lake conditions [34].

Gammacerane, which is considered to be an indicator of reducing and hypersaline conditions [35–36] or associated with a stratified water column [37], is abundant in all of the oil shale samples studied (Fig. 5b). The high gammacerane index, 1.84–3.27 (Table 2), is consistent with the interpretation of low Pr/Ph ratios, reflecting a saline to hypersaline setting associated with evaporate and carbonates deposition.

5. Conclusions

Oil shale samples from the lower part of the Gongjue Formation in the Nangqian Basin were analyzed for geochemical characteristics, paleo-lake deposition conditions and organic matter source. Based on the results of analysis the following conclusions can be drawn:

1. The oil shale samples have high values of TOC (4.17–6.47 wt%), SOM (1193–2923 ppm), PY (28.30–42.43 mg HC/g rock) and HI (449–628 mg HC/g TOC), which gives evidence of a good hydrocarbon generation potential of Nangqian oil shale.
2. The Rock-Eval pyrolysis and biomarker data of the samples confirm maturities in the pre- to early stages of oil generation.
3. Elemental analysis and pyrolysis data show that the organic matter of Nangqian oil shale mostly originates from lacustrine algae. However, based on the odd-over-even carbon number predominance of *n*-alkanes and high proportions of long-chain *n*-alkanes, there may be observed a strong terrestrial source signal.
4. The gammacerane index of 1.84–3.27 and high concentrations of phytane ($\text{Pr/Ph} < 0.23$) reveal that the Nangqian oil shale is associated with a restricted, saline depositional setting and might have had a stratified water column.

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