



# Biomarker Records From Eocene Lacustrine Sequence in the Eastern Tibet Plateau and Its Implication for Organic Matter Sources

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The Eocene is the initial stage of the Cenozoic global cooling. Compared with the abundant marine records, the continental records of Eocene are scarce. Throughout the Eocene, a series of continuous deposition of gypsum and volcanic tuff-bearing red clastic sediments have developed in the Nangqian Basin (NB). In this work, representative sediments were collected from the NB, and lipid biomarkers and compound-specific carbon isotopes of *n*-alkanes were analyzed. Based on the robust paleomagnetic age–depth model, from the early to the late Eocene, the compound-specific carbon isotopic compositions ( $\delta^{13}\text{C}_{23}$  and  $\delta^{13}\text{C}_{25}$ ) increased with the sedimentary facies changed. At the same time, the relative proportion of mid-chain length to the long-chain length homologs (P<sub>aq</sub>) decreased, and the peak carbon number ( $C_{\text{max}}$ ) shifted from  $n\text{C}_{21}$ ,  $n\text{C}_{22}$  or  $n\text{C}_{23}$  to  $n\text{C}_{16}$ ,  $n\text{C}_{25}$ ,  $n\text{C}_{27}$ , or  $n\text{C}_{31}$ . We ascribed these variations to the climate drying and water level turning high as indicated by the lithology change from fluvial to lacustrine facies and the terrestrial inputs from neighboring mountain belts in the middle–late Eocene. Moreover, we compared our *n*-alkane results with other records from the TP and the global sea level and marine benthic  $\delta^{18}\text{O}$ . We found that a nearly synchronous deformation and drying of the eastern TP caused by the India–Asia collision in the early Eocene was closely related to the arid conditions and topographically changed in the northern TP. The climate variations in the Eocene in the NB were mainly controlled by the global climate change and the uplift of the TP and affected by the Paratethys Sea on a long scale.

**Keywords:** Nangqian Basin, Eastern Tibet Plateau, organic geochemistry, Eocene, lacustrine sediments

## INTRODUCTION

The Eocene, which is the initial stage of the Cenozoic global climate from a “greenhouse” state to an Oligocene “icehouse” state, is considered as a “doubthouse” of global climate conditions (Zachos et al., 2008; Westerhold et al., 2020). Most of the continuous long-scale records for the Eocene paleoclimate are from ocean boreholes (Ogg and Bardot, 2001; Florindo and Roberts, 2005; Suganuma and Ogg, 2006; Edgar et al., 2010) and marine outcrops (Dallanave et al., 2009; Hollis et al., 2013; Agnini et al., 2016). In comparison with various marine records, the climate change from the evidence of lacustrine records during the Eocene, especially in the Tibetan Plateau

(TP), is little known. Recently, various geochemical records from the TP have been reported. In the northeastern TP, the Eocene strata and related paleoclimate of the Xining (Dai et al., 2006; Fang et al., 2019), Linxia (Feng et al., 2021; Feng et al., 2022), and Qaidam basins (Fang et al., 2019; Wu et al., 2021) have been reported. Based on these paleoenvironmental records, several mechanisms were formulated to interpret the environmental changes (Bosboom et al., 2011; Bosboom et al., 2014; Ding et al., 2014; Kaya et al., 2018; Westerhold et al., 2020). Furthermore, the Cenozoic sedimentary basins, especially the basin in the eastern Qiangtang Terrane, can provide an excellent record of the early India–Asia collision history during the Paleogene (Tang et al., 2017; Li et al., 2019; Li et al., 2020a; Xiong et al., 2020).

The Nangqian Basin (NB) (~32–33°N, ~96°E), in the eastern TP currently, contains a long and continuous sequence of the Eocene sediments (Horton et al., 2002; Li et al., 2019; Zhang et al., 2020). Most importantly, the NB is the intersection of the East Asian Monsoon (EAM), the Indian Monsoon (IM), and the westerlies but also the junction of the humid monsoon region in Eastern China, the arid inland region in Northwest China, and the alpine region of the TP, which is very sensitive to the changes of the environment and climate (Horton et al., 2002; Li et al., 2019). The early Cenozoic successions of gypsum and volcanic tuff-bearing red clastic sediments in the NB provide precious climatic and environmental archives (QBGMR, 1991; Horton et al., 2002; Zhang et al., 2020). Many works on the Cenozoic paleoenvironment and paleoclimate of the TP have been carried out in the NB, including the lithofacies, evaporate minerals, magnetostratigraphy, pollen-spores, clay minerals, major and trace elements, clumped isotope, carbonate oxygen, and carbon isotopes, which suggest that, with the rising altitude, the climate gradually became arid and cold in the eastern TP during the Eocene (Du et al., 2017; Yuan et al., 2017; Li et al., 2019; Fang et al., 2020; Yuan et al., 2020a; Zhang et al., 2020; Zhao et al., 2020). However, due to the lack of long sedimentary sequence and robust age constraints, the driving mechanism of the climate change is not clear.

In addition to the proxies mentioned before, lipid biomarkers originate from organisms directly, which can respond to the environment rapidly, and can be preserved in sediments stably, indicating that they preserved reliable climate records (Moldowan et al., 2005; Peters et al., 2007; Liu et al., 2015; He et al., 2020; Wang et al., 2021a, Wang et al., 2021b). *n*-Alkanes have high abundance in organisms, which can be preserved in lacustrine sediments for millions of years, and their original isotopic signals can also be well preserved (Chikaraishi and Naraoka, 2003; Aichner et al., 2010; Castañeda and Schouten, 2011; Sachse et al., 2012; Ouyang et al., 2015). For example, Liu et al. (2018) used Paq, ACL, and  $\delta D$  to determine the source of sedimentary *n*-alkanes and study the degree of water mass restriction (open or closed) of an ancient lake basin. Rao et al. (2016) demonstrated a complex relationship between the isotopic composition of precipitation and the precipitation amount or East Asian summer monsoon intensity by applying the compound-

specific carbon and hydrogen isotopes of terrestrial long-chain *n*-alkanes. Lin et al. (2020) applied detrital zircon U–Pb geochronology and leaf wax *n*-alkane  $\delta D$  to analyze the surface uplift history of the Hoh Xil Basin. The *n*-alkanes and the corresponding compound-specific isotopic compositions display the application of reconstructing the paleoclimate and paleotopography in lacustrine sediments in the TP.

In the present study, the age–depth framework of the NB has been constrained by high-resolution palaeogeomagnetic records with absolute ages (Horton et al., 2002; Spurlin et al., 2005; Zhang et al., 2020). We applied the lipid biomarker (*n*-alkanes) and the corresponding compound-specific carbon isotopic compositions to reflect the variations of organic matter sources and their corresponding paleoclimate conditions. Moreover, we compared our results with other records from the TP during the Eocene to discuss the possible driving mechanisms from the perspective of biological variations.

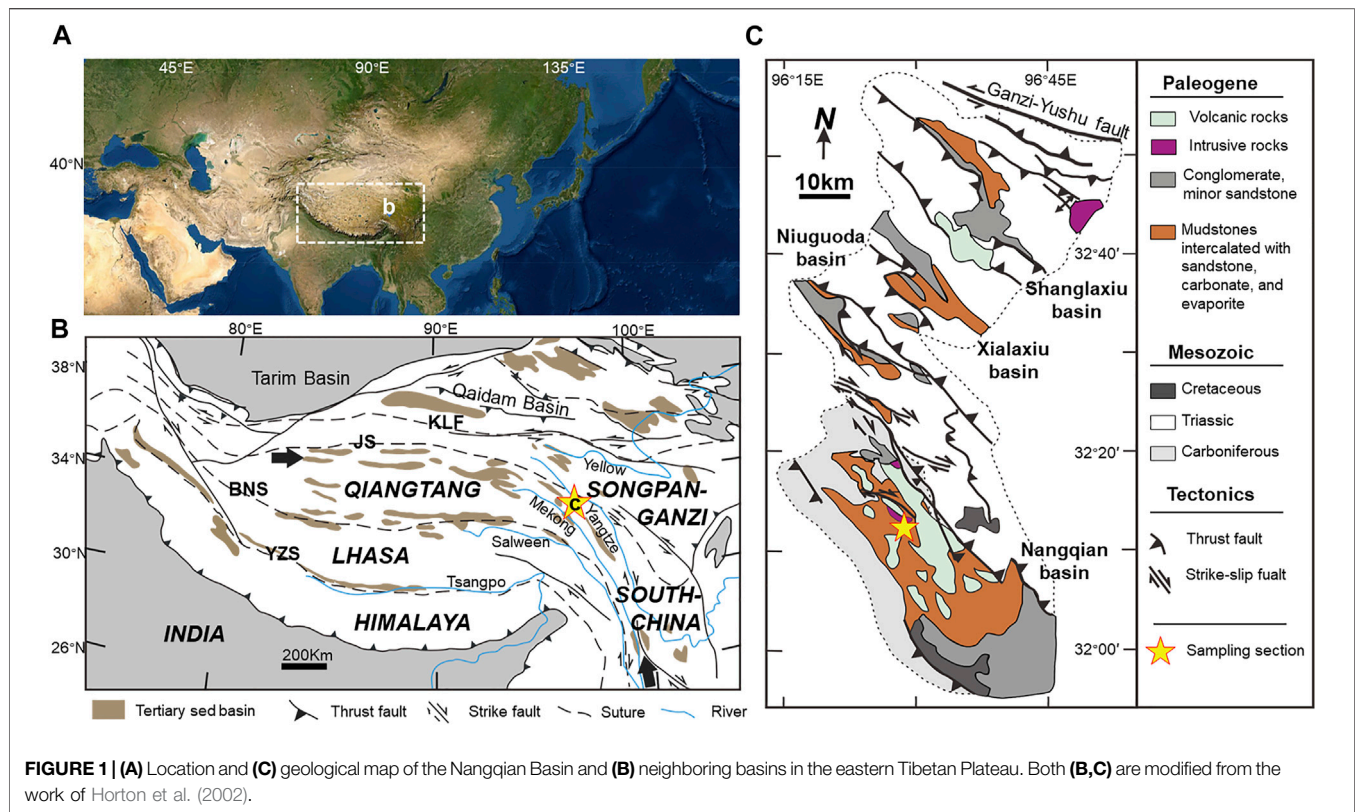
## GEOGRAPHIC AND STRATIGRAPHIC SETTING

### Geological Setting

The current NB, located on the border between the Qinghai Province and the Tibet Autonomous Region, is about 4,500–5,000 m (Figure 1A). The India–Asia collision formed north-eastward extrusion, which facilitated a series of contraction deformation and strike-slip faults in the eastern Tibet, including the Yushu–Nangqian thrust belt and the Jinshajiang strike-slip fault system (Hou et al., 2003; Yin and Harrison, 2003; Spurlin et al., 2005). The NB is one of the series of narrow, elongated Paleogene basins in the Yushu–Nangqian region between the Songpan–Ganzi and Qiangtang terranes (Figures 1B,C). It is ~80 km long in the south–north direction and ~15 km wide in the east–west direction (QBGMR, 1991; Horton et al., 2002; Yuan et al., 2020).

The NB is characterized by a continental seasonal monsoon climate today and impacted by the Asian Monsoon (mainly the Indian Monsoon) (Li et al., 2019). The average annual temperature is 4.1°C, and the average annual precipitation is 538.1 mm, based on the observation from the nearest meteorological station from 1961 to 2013 (Hou et al., 2003; Wang et al., 2012; Wei and Han, 2015). The modern soils of the surrounding mountains and the basin surface are dark brown soils and steppe soils, respectively (CAS-TPET, 1985; Xiong and Li, 1990).

The Paleogene strata in the NB are characterized by prominent red beds, which contain limestone/marlite, gypsum, volcanic rock, and tuff (Horton et al., 2002; Li et al., 2019). The Neogene–Quaternary only appears sporadically in or near Paleogene basins along the Jinshajiang suture belt and is dominated by light-colored (yellow–green–gray) fluvial-dominated sediments with coals (QBGMR, 1991; Horton et al., 2002; Yuan et al., 2020a). A clear regional unconformity exists between the Paleogene and the Neogene–Quaternary (QBGMR, 1991; Horton et al., 2002; Zhang et al., 2020).



## Chronology

The NB contains three parts of the Paleogene deposits (QBGMR, 1991; Horton et al., 2002). Zhang et al. (2020) provided well-dated palaeogeomagnetic age of the section and the absolute age was tested using the method of zircon U–Pb dating. The lower section is dominated by brownish-red fine conglomerates, sandstones, and siltstones–mudstones, with a depth of 1,050–690 m and age of 52.5–48.0 Ma. Reddish luvic paleosols cover fine sandstone and siltstone–mudstone, with thin limestones occasionally observed (QBGMR, 1991; Horton et al., 2002; Zhang et al., 2020). A 20 m-thick andesite with an Ar–Ar age of  $51.2 \pm 0.2$  Ma appears near the basement (Horton et al., 2002; Spurlin et al., 2005). The middle part is mainly composed of marl and limestones intercalated with reddish siltstone–mudstone, with a depth of 690–400 m and age of 48.0–43.3 Ma (Horton et al., 2002; Spurlin et al., 2005; Zhang et al., 2020). The upper part is mainly purplish-red gypsum mudstone, intercalated with a large number of thin siltstone and gypsum layers and occasionally with thin dolomite layers (QBGMR, 1991; Horton et al., 2002). The depth of the upper part is 400–0 m and the age of that is 43.3–35 Ma (Zhang et al., 2020). Volcanic tuff rocks were found in this part, with an Ar–Ar age of  $38.2 \pm 0.1$  Ma (Horton et al., 2002; Spurlin et al., 2005) and a zircon U–Pb age of  $37.3 \pm 0.56$  Ma (Zhang et al., 2020). The lithological change is interpreted as a gradual transition drought from a braided river, alluvial fan, and ephemeral shallow pond/lake environment to a semi-brackish lake in a distal floodplain and finally to playa mudflat and saline lake environments (Figure 2B).

## SAMPLING AND METHODS

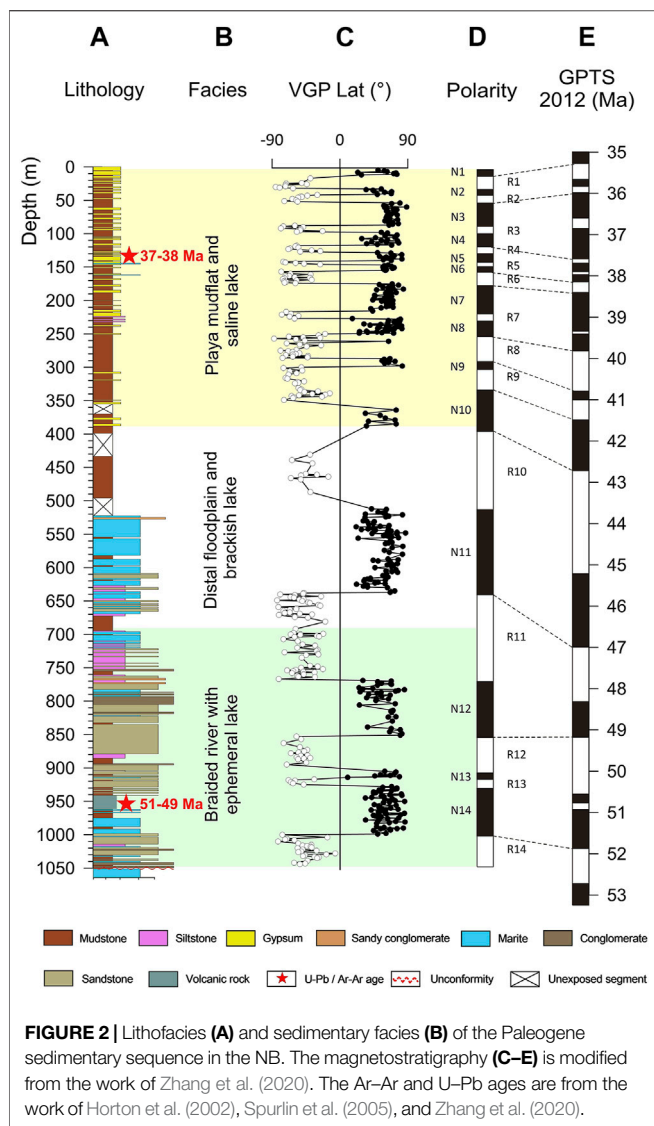
The Nangqian section is located along the Zhaqu River with a total thickness of 1050 m (Figure 1). According to lithology, a total of 67 bulk samples were selected. The 67 sediment samples were collected from 52.5 to 35 Ma in a depth interval from 0 to 1,050 m. Before collecting samples, the weathering denudation surface was knocked out with a geological hammer. Then, fresh samples were obtained and put into degreasing cloth bags for storage.

## Biomarker Analyses

In this study, 22 samples were tested for biomarker analyses (Supplementary Table S1). The 22 samples are divided into two sections. There are 12 samples in the lower section (age 51.8–46.4 Ma) and 10 samples in the upper section (age 42.3–35.5 Ma).

The extraction process of biomarkers is as follows: the bulk samples were extensively cleaned to remove any possible contamination from recent organic material and crushed to fine powder larger than 100 mesh. Powdered samples (~400 g) were weighed into pre-extracted filter paper thimbles (the filter paper was extracted using a Soxhlet extractor with DCM for 72 h previously) and Soxhlet extracted at 50°C continuously for 72 h using a Soxhlet extractor (DCM: MeOH = 9:1, v:v). The glassware used in this experiment, such as injection bottle, weighing bottle, and conical bottle, had been washed with acetone, rinsed with clean water and ultrapure water, dried in an oven, sealed with clean tinfoil, and then, burned (400°C for 5 h) in a muffle furnace





previously. Tweezers and other tools were washed with DCM 2–3 times before used.

Total lipid extracts were loaded onto a solid phase extraction column (ANPEL, silica gel 500 mg). The *n*-alkanes were eluted with *n*-hexane (5–10 ml). After drying *n*-alkanes, 1  $\mu$ L of the sample in hexane was injected into a GC–MS-QP2020NX (Shimadzu, Japan). This instrument was equipped with an Rtx-5 MS gas chromatograph which was fitted with a 30 m  $\times$  0.25 mm i. d. fused silica capillary column, coated with a film (0.25  $\mu$ m) of 5% phenyl-methyl-DB-5. The carrier gas was 99.995% high purity helium, and the control mode was linear velocity. The heating program was set as follows: initially at 80°C for 2 min, rapidly increased to 210°C at a rate of 4°C/min, then increased from 210 to 295°C at a rate of 3°C/min, and held at 295°C for 20 min. The column flow rate was 1.0 ml/min, and the injection volume was 1  $\mu$ L. The diversion mode showed no diversion. The MS was operated with ionization energy of 70 eV, and the temperature of ion source was 250°C. The

GC–MS was tuned using perfluorotributylamine (PFTBA) and blank samples were analyzed to check the background. The interface temperature of GC–MS was 300°C. The solvent delay was set to 2 min. Both GC–MS scan and GC–MS single ion monitoring mode (SIM) were used for acquisition.

## Compound-Specific $\delta^{13}\text{C}$ Analyses

According to the preliminary results of 22 biomarkers, 23 samples were selected for the test of compound-specific carbon isotopic compositions (Supplementary Table S1). There were 12 samples in the lower section and 11 samples in the upper section. The extraction process of *n*-alkanes for compound-specific carbon isotopic compositions was the same as that described in Biomarker Analyses.

The test of carbon isotopic values of individual *n*-alkanes was finished on a GC (Agilent 6890) coupled to an isotope ratio mass spectrometer (IRMS, Thermo Scientific MAT 253) via a combustion interface (GC Combustion III) (Key Laboratory of Petroleum Resources Research, Chinese Academy of Sciences). High-purity helium was used as a carrier gas at 2 ml min<sup>-1</sup>. The gas chromatograph was fitted with a 30 m  $\times$  0.32 mm i. d. fused silica capillary column, coated with a film (0.25  $\mu$ m). The oven temperature was programmed to be initially held at 80°C for 3 min, increased to 300°C at a rate of 3°C/min, and held for another 30 min. Individual compounds were oxidized at 940°C when flowing through an oxidation ceramic micro-reactor filled with twisted wires (NiO/CuO/Pt). Three pulses of standard pure CO<sub>2</sub> gas, pre-calibrated against a commercial reference CO<sub>2</sub>, were injected via the GC–C III interface to the IRMS for the computation of  $\delta^{13}\text{C}$  values of sample compounds. A set of *n*-alkanes (*n*-alkane mixture type A7) with known  $\delta^{13}\text{C}$  values acquired from Indiana University were measured daily to ensure the accuracy of the machine. The standard deviation for duplicate analyses of this standard was  $<\pm 0.3\%$ . The  $\delta^{13}\text{C}$  values were reported with reference to the PDB standard. The resolution of hydrocarbon peaks obtained during GC–IRMS was similar to that obtained in GC–MS analysis.

## Calculation of Biomarker Proxies

As for the biomarker proxies, the index of proportion of aquatic plants (Paq, Ficken et al., 2000) values was calculated as follows:

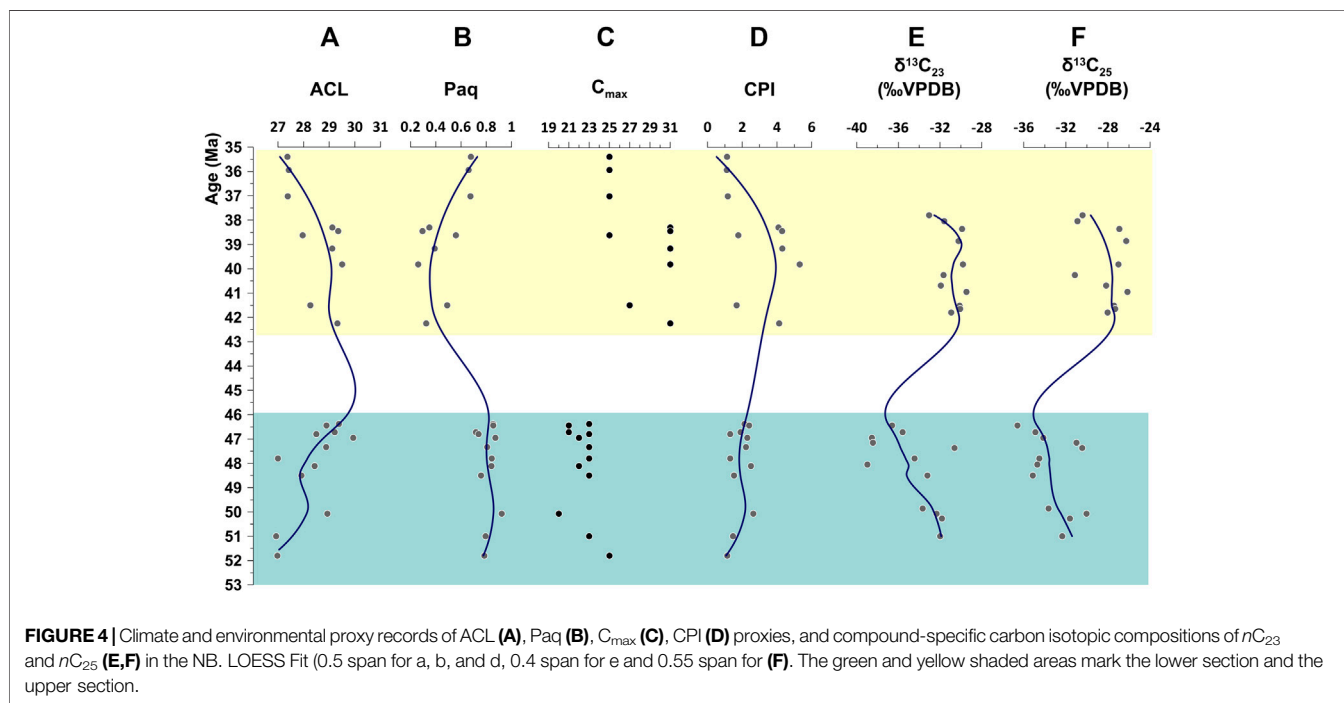
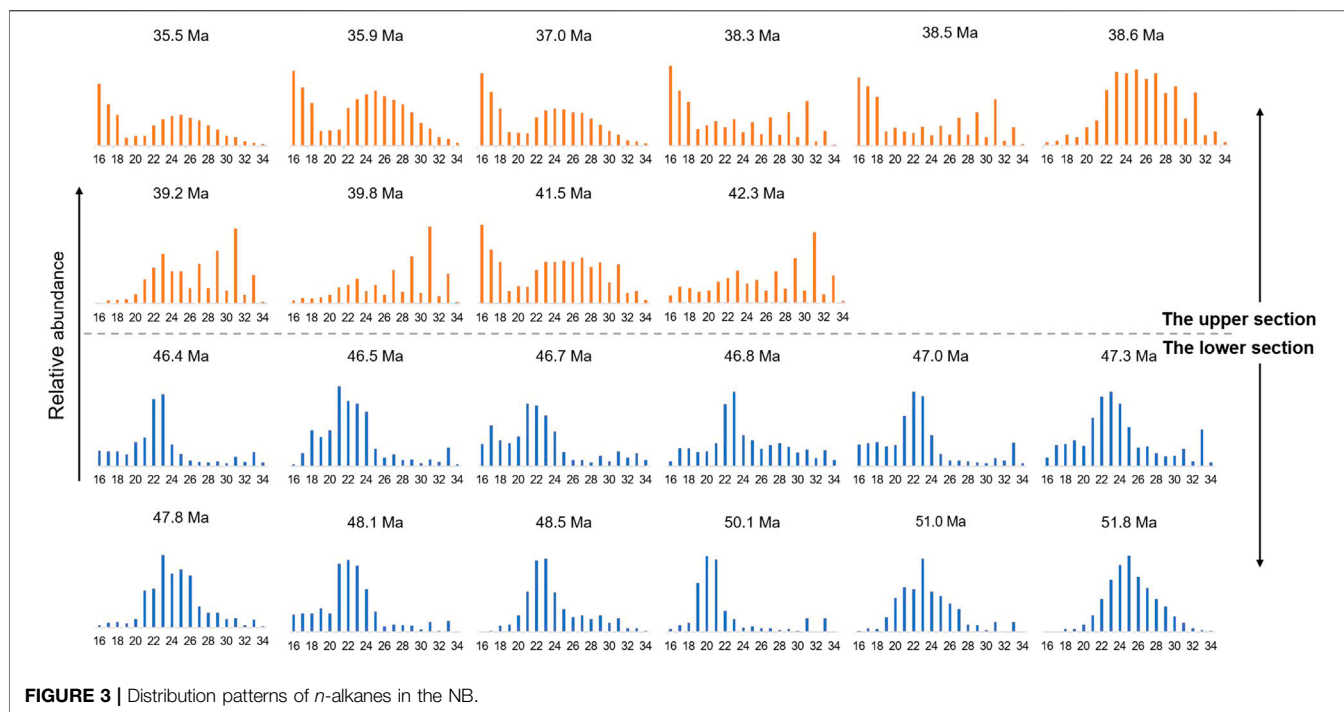
$$\text{Paq} = (nC_{23} + nC_{25}) / (nC_{23} + nC_{25} + nC_{29} + nC_{31}).$$

The carbon preference index (CPI) values of the extracted long-chain *n*-alkanes ( $nC_{26}$  to  $nC_{34}$ ) were calculated using a modified formula of Cranwell (1984) and Ratnayake et al. (2006) as follows:

$$\text{CPI} = 1/2 \left[ \frac{(nC_{27} + nC_{29} + nC_{31} + nC_{33}) / (nC_{26} + nC_{28} + nC_{30} + nC_{32})}{+(nC_{27} + nC_{29} + nC_{31} + nC_{33}) / (nC_{28} + nC_{30} + nC_{32} + nC_{34})} \right].$$

The average chain length (ACL) values were calculated using a modified formula of Poynter et al. (1989) as follows:

$$\text{ACL}_{27-33} = (27 \times nC_{27} + 29 \times nC_{29} + 31 \times nC_{31} + 33 \times nC_{33}) / \times (nC_{27} + nC_{29} + nC_{31} + nC_{33}).$$



## RESULTS

The distribution patterns of *n*-alkanes in the NB sediments are shown in Figure 3. The distribution of *n*-alkanes in the 12 samples of the lower section is mainly unimodal and maximized at  $nC_{21}$ ,  $nC_{22}$ , or  $nC_{23}$ . The distribution of

*n*-alkanes in the other 10 samples of the upper section is mainly bimodal and mainly maximized at  $nC_{16}$ ,  $nC_{25}$ ,  $nC_{27}$ , or  $nC_{31}$ .

The Paq and CPI values of the two sections changed significantly (Figures 4B,D). In the lower section, the Paq values were between 0.72 and 0.92, with an average of 0.82. In

the upper section, the Paq values were between 0.26 and 0.68, and the average value was 0.47. Meanwhile, the average value of CPI increased from the lower section to the upper section, with an average of 1.90 to an average of 2.90.

In **Figures 4E,F**, compound-specific carbon isotopic compositions ( $\delta^{13}\text{C}_{23}$  and  $\delta^{13}\text{C}_{25}$ ) were heavier in the upper section than those in the lower section. From the lower to the upper section, the average of  $\delta^{13}\text{C}_{23}$  changed from  $-34.69\text{‰}$  to  $-30.79\text{‰}$ , and  $\delta^{13}\text{C}_{25}$  showed a similar increase from an average of  $-33.26\text{‰}$  to an average of  $-28.16\text{‰}$ .

## DISCUSSION

### Degradation Degree of *n*-Alkanes in the Nangqian Basin

For *n*-alkanes, their typical odd-to-even preference in the long-chain part suggests a low degree of degradation (Luo et al., 2012; Duan et al., 2020). In sediments, CPI > 1 means a predominance of odd over even chain lengths (Eglinton and Hamilton, 1967). Sediments from the upper section of the NB are characterized with CPI > 1, except for the top three samples (age of 35.5, 35.9, and 37.0 Ma), indicating a low degree of degradation. In the lower section, although the CPI > 1 was not remarkable, the odd-to-even preference is still observed in the low proportion of the long-chain *n*-alkanes (according to the distribution patterns in **Figure 3**). Combined with the analysis of organic sources reflected by the distribution patterns of *n*-alkanes, the low CPI values in the lower section was the result of the small input of terrestrial plants, which made the odd-to-even preference in the long-chain *n*-alkanes difficult to observe. Therefore, it can be concluded that the degradation of *n*-alkanes in this study would not affect the restoration of paleoenvironment.

### Variations in Biomarker Proxies and $\delta^{13}\text{C}_{\text{alk}}$

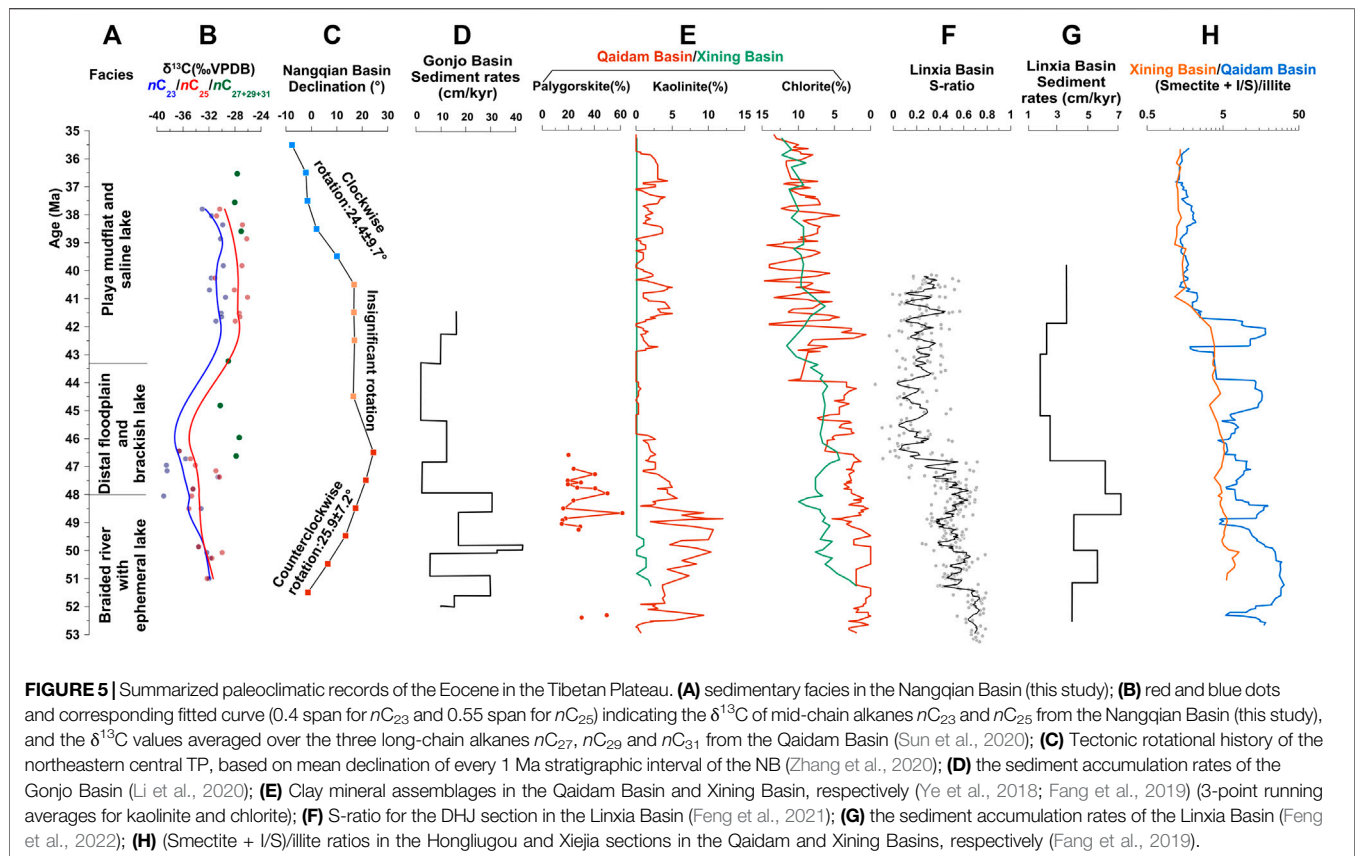
Previous studies have shown that the mid-chain length *n*-alkanes are mainly from aquatic plants, such as submerged and floating plants (Meyers and Ishiwatari, 1993; Ficken et al., 2000; Yu et al., 2021). However, the contribution of terrestrial plants to the mid-chain *n*-alkanes is inevitable, especially in the upper section (**Figure 3**). Nevertheless, the carbon source of terrestrial plants is atmospheric  $\text{CO}_2$  and that of aquatic organisms is  $\text{HCO}_3^-$  (Meyers and Ishiwatari, 1993; Meyers, 2003; Wang et al., 2013), possibly resulting in the different composition of  $\delta^{13}\text{C}$  values. The  $\delta^{13}\text{C}$  value of  $\text{HCO}_3^-$  is heavier than that of atmospheric  $\text{CO}_2$ , leading to more negative  $\delta^{13}\text{C}$  values of terrestrial plants, indicating that if the  $\delta^{13}\text{C}$  value of mid-chain *n*-alkanes ( $n\text{C}_{23}$  and  $n\text{C}_{25}$ ) was seriously influenced by terrestrial plants, more negative  $\delta^{13}\text{C}$  values were likely to be observed in the upper section. However, the  $\delta^{13}\text{C}$  value of mid-chain *n*-alkanes ( $n\text{C}_{23}$  and  $n\text{C}_{25}$ ) increased in the upper section, displaying an opposite trend, which indicates that the influence of terrestrial plants on the compound-specific carbon isotopic compositions of the mid-chain *n*-alkanes was limited. Therefore, in this study, we proposed that the  $n\text{C}_{23}$  and  $n\text{C}_{25}$  were contributed by the aquatic plants.

Jiang et al. (2021) investigated the  $\delta^{13}\text{C}_{\text{org}}$  values of surface sediment samples from 55 lakes in the mid-latitude Asia to

establish the relationship between the  $\delta^{13}\text{C}$  value of aquatic organisms and water depth in lacustrine settings. In freshwater and brackish lakes,  $\delta^{13}\text{C}_{\text{org}}$  variation resembles an arched pattern with depth, with relatively positive  $\delta^{13}\text{C}_{\text{org}}$  values corresponding to the depth of ~1–10 m. According to the sedimentary facies in the NB (**Figure 2B**), it transitioned from the braided river, alluvial fan, and ephemeral shallow pond/lake environment to a semi-brackish lake in a distal floodplain predominates, suggesting that the lake level in the NB in the mid–late Eocene was higher than that in the early Eocene. The lake water level recorded by  $\delta^{13}\text{C}_{23}$  and  $\delta^{13}\text{C}_{25}$  is consistent with the variations of sedimentary facies (**Figures 4E,F**), showing a relative higher water level in the mid–late Eocene.

In addition, *n*-alkanes can truly reflect the input characteristics of parent material sources (Meyers, 2003). Ficken et al. (2000) proposed the Paq index to indicate the non-emergent (submerged and floating-leaved) aquatic plants input to lake sediments relative to that from the emergent aquatic and terrestrial plants, which expresses the relative proportion of mid-chain length ( $n\text{C}_{23}$ ,  $n\text{C}_{25}$ ) *n*-alkanes to long-chain length ( $n\text{C}_{29}$ ,  $n\text{C}_{31}$ ) homologs. Further studies have indicated that the Paq value can be used to represent the effective moisture and water level, and basically, the larger the Paq, the more hydrocarbon input from aquatic organisms, the more precipitation and the wetter climate (Nichols et al., 2006; Pu et al., 2011; Zheng et al., 2007). In the Linggo Lake, the high Paq with the heavy  $\delta\text{D}_{\text{alk}}$  implied abundant precipitation brought by the westerlies, indicating that the Linggo Lake was at a high water level under a humid environment (Hou et al., 2003). In the present study, the average value of Paq decreased from 0.82 (ranging from 0.72 to 0.92) to 0.47 (ranging from 0.26 to 0.68) (**Figure 4B**). The decrease of Paq in the NB is contrary to the high water level as recorded by sedimentary facies and compound-specific carbon isotopic compositions, which implied that the Paq values were affected by other factors (Liu et al., 2015).

Judging from the provenance analysis of sediments in the NB (Zhang et al., 2019), the initially accumulated sediments of the NB has changed from relatively small internal drainage networks and short main-stem rivers in this region in the Paleocene to the nearby thrust belts in the Eocene (Horton et al., 2002; Spurlin et al., 2005). Based on the stable and clumped isotopic evidence of carbonates, the NB was 2.7 (+0.6/−0.4) km in elevation while the hypsometric mean elevation of surrounding mountains was  $3.0 \pm 1.1$  km above sea level during the late Eocene (Li et al., 2020b). In addition, palynology records indicated that the high-elevation genus *Tsugaepollenites* presented in the Ria Zhong section in the NB in the late Eocene (Yuan et al., 2020a; Yuan et al., 2020b). In addition, the crustal deformation and thickening induced by intracontinental subduction between the Lhasa and Qiangtang terranes caused a rapid uplift of the Central Watershed Mountain, associated with the regional climate change transforming the landscape from desert to forest in the Gonjo Basin (Xiong et al., 2020). In view of the previous studies, the specific tectonic settings can further explain this topographic difference in the NB. Spurlin et al. (2005) proposed that a nearly 40% upper-crustal shortening, occurring in the early Cenozoic, was observed over the area,



which accounted for most of the surface uplift of the Nangqian region. Horton et al. (2002) proposed that the fold-thrust belts were the consequence of contraction triggered by the India–Asia collision. Based on the previous studies, in the mid–late Eocene, the Nangqian region appeared as small intermontane sub-basins and received plentiful terrestrial sediments from the neighboring mountain belts. The Paq, ACL, and  $C_{max}$  contain the common information of terrestrial plants from nearby mountains and aquatic plants from the playa lake/mudflat, so the water level was no longer the sole factor affecting Paq. This explained why Paq values showed the opposite result of sedimentary facies when interpreting the water level.

Evaporite minerals are also sensitive to environmental and climatic changes (Warren, 2000, 2006; Meister et al., 2011; Zhu et al., 2021; Zhang et al., 2021; Zhang et al., 2022). In the mid–late Eocene, gypsum, a typical evaporite mineral, indicated a dry climate in the NB, which was absent in the early Eocene (Yuan et al., 2020b; Zhang et al., 2020). The adjacent areas, such as the Gonjo Basin, Qaidam Basin, and Tarim Basin, showed a succession of gypsiferous red beds with an upward increase in evaporites (Wang et al., 2008; Wang et al., 2016; Tang et al., 2017; Li et al., 2020a; Xiong et al., 2020), further suggesting an increasingly arid climate.

To sum up, the climate in the early and mid–late Eocene was completely different. In the early Eocene, the terrain was generally low and flat, with a humid climate and low water level. After the tectonic movements caused by the India–Asia collision, the NB

and surrounding areas appeared as small intermontane sub-basins, characterized by an arid climate.

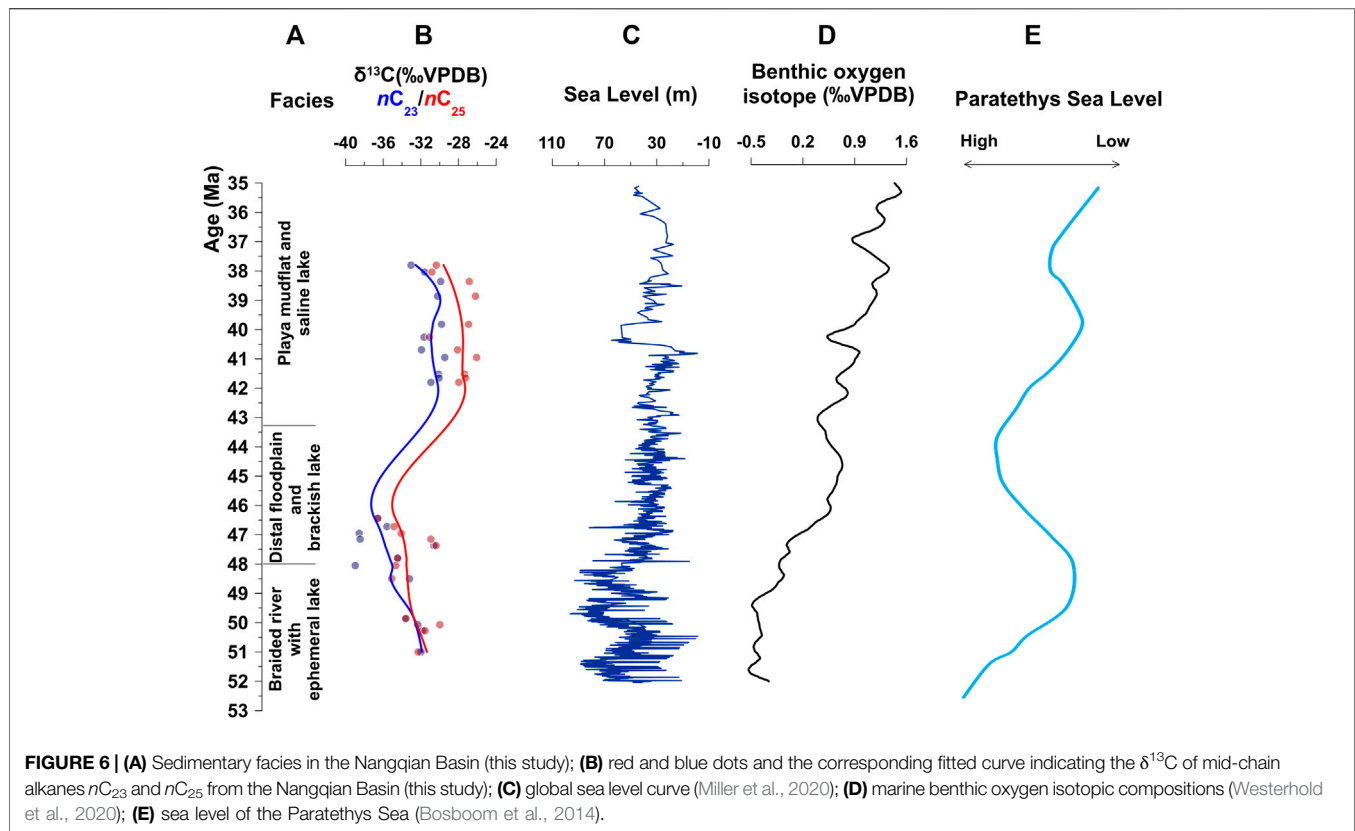
## Driving Mechanisms of Eocene Climate Variations in the Eastern Tibetan Plateau

From the early to the late Eocene, the sedimentary facies and the water level recorded by compound-specific  $\delta^{13}C$  in the present study implied a notable climate transition from humid to arid conditions and a topographical change in the eastern TP.

Previous studies have indicated that three main forcing mechanisms, including the uplift of the Tibetan Plateau, the retreat or incursion of the Paratethys Sea, and global cooling, may be responsible for the change (Bosboom et al., 2011; Bosboom et al., 2014; Ding et al., 2014; Kaya et al., 2018; Westerhold et al., 2020).

The global cooling could be a first-order control in the NB beyond the impact of the India–Asia collision and the retreat or incursion of the Paratethys Sea. The global cooling reduced evaporation from the sea and thus the water vapor flux to the air (Fang et al., 2019; Westerhold et al., 2020). The rapid global sea level fall accompanied by the shrinking of the sea area increased the distance from the study area to the sea, which reduced the marine-derived water vapor supply (Miller et al., 2020). Via the modulation of the water vapor supply by the westerlies, the long-term global cooling in the Eocene reduced the water vapor supply to the continental and, thus, caused further





drying in the NB. Furthermore, the good correlation between the water level and climate in the NB, the northeastern TP, and the global climate proxies implied that the global climate system served as the main driver of climate variations in the study area (Westerhold et al., 2020; **Figure 5H, Figure 6A–D**).

Exploring the past topography is essential for disentangling the complex interactions between orography and climate (Fang et al., 2019; Xiong et al., 2020; Wu et al., 2021). Our records reflected a significant early Eocene deformation and climate change in response to the India–Asia collision. From the paleomagnetic evidence, a counterclockwise rotation of  $25.9 \pm 7.2^\circ$  during 52–46 Ma supported the basin deposition, rotation, and volcanism by northward compression and eastward extrusion of the eastern Lhasa and Qiangtang Blocks as early response to the India–Asia collision (**Figure 5C**). During ~52–48 Ma, a relatively high sedimentation rate in the Gonjo Basin suggested an active tectonic setting in the eastern TP, which coincided with the clockwise rotation (**Figure 5D**, Li et al., 2020b). Interestingly, a relatively low sedimentation rate appeared in the Gonjo Basin at ~48–41 Ma, which coexisted with a rapid sedimentation rate decrease in the Linxia Basin at ~47–40 Ma (**Figure 5G**). The paleomagnetic results in the Xining Basin revealed a stable tectonic setting with no significant rotation at ~48–41 Ma (Dupont-Nivet et al., 2008). In addition, an arid environment appeared at ~49.5–47 Ma in the Qaidam Basin and Xining Basin recorded by palygorskite, kaolinite, and chlorite (**Figure 5E**, Ye et al., 2018; Fang et al., 2019) and at 47.6 Ma in the Linxia Basin recorded by the S-ratio (**Figure 5F**, Feng et al., 2021),

which coincided with arid climate conditions recorded by transitions of sedimentary facies in the NB (**Figure 5A**), suggesting a nearly synchronous deformation and climate change across the eastern and northern Tibetan Plateau in the early Eocene. These analyses show that the India–Asia collision could be the most direct factor of climate change in the NB, and the eastern TP.

A vast shallow epicontinental sea extended across Eurasia and was well-connected to the Western Tethys before it retreated westward and became isolated as the Paratethys Sea during the Paleogene (Bosboom et al., 2014). It regulated the hydrological cycle in Central Asia by the westerlies, so the westward retreat of this sea was as important as the Tibetan Plateau uplift in forcing aridification in the Asian continental interior (Dupont-Nivet et al., 2007). Previous studies on the Tarim and Tajik basins indicate that there were three sea-level cycles during 52–35 Ma (**Figure 6E**) (Bosboom et al., 2014; Kaya et al., 2018). However, due to the lack of data in our section between 46 and 43 Ma, it is difficult to assess the impact of Paratethys Sea on the aridity of the eastern TP. For all that, the influence of Paratethys Sea would be long-term and gradual, and the stepwise sea retreat from Central Asia amplified the aridification of the Asian interior (Bosboom et al., 2014; Carrapa et al., 2015; Sun et al., 2016; Kaya et al., 2018). From **Figure 2A**, the trend of gradual drought in the NB has been well recorded by the lithology change. Although the retreat or incursion of the Paratethys Sea failed to compare with the organic records in NB, it is still an important factor worthy of further research.



In general, our observations recorded a significant change in sedimentary facies from braided river facies to brackish lake facies in the early-mid Eocene in the eastern TP. At the same time, the compound-specific carbon isotopic compositions of *n*-alkane ( $\delta^{13}\text{C}_{23}$  and  $\delta^{13}\text{C}_{25}$ ) recorded the rise of the lake level. In the mid-late Eocene in the NB, the terrestrial input in the sediments increased, which corresponded to the climate drying and topographic change at this time. In comparison with three main driving mechanisms, our records were consistent with global climate changes, related closely with the India-Asia collision in the early Eocene, and the relevance with Paratethys Sea needs further study. We claim that the climate variations in the Eocene in the study area were mainly controlled by the global climate change and the uplift of the Tibetan Plateau, but the impact of the Tethys Sea cannot be ignored.

## CONCLUSION

We applied the *n*-alkanes and the compound-specific carbon isotopic compositions in the NB to reflect the paleoclimate conditions in the eastern Tibet Plateau. From our multi-proxy records, we came to the conclusion that the NB underwent rapid drying from the early to the mid-late Eocene. The compound-specific carbon isotopic compositions ( $\delta^{13}\text{C}_{23}$  and  $\delta^{13}\text{C}_{25}$ ) were affected by the water level deepening and showed a rapid positive. From the *n*-alkene records in the NB, the type of aquatic organism in the Eocene lacustrine sequence had an evident change from submerged and floating plants to emergent and terrestrial plants, responding to climate drying and orographic uplifting. Meanwhile, the lithofacies changed from the braided river, alluvial fan, and ephemeral shallow pond/lake environment to a semi-brackish lake in a distal floodplain and finally to playa mudflat and saline lake environments. We regard the change of multi-proxy records by *n*-alkenes consistent with the sedimentary facies change and aridity in the NB and responded synchronously in the northern TP, which was mainly controlled by the global

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cooling and the uplift of the Tibetan Plateau and affected by the Paratethys Sea on a long scale.

## DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the **Supplementary Material**, further inquiries can be directed to the corresponding authors.

## AUTHOR CONTRIBUTIONS

JW: participation in the whole work and drafting of the article; YW, GW, and ZW: perception and design and final approval of the version to be published; and WH, TZ, XM, PZ, HM, XY, SL, and LL: data analysis.

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## SUPPLEMENTARY MATERIAL

The Supplementary Material for this article can be found online at: <https://www.frontiersin.org/articles/10.3389/feart.2022.849041/full#supplementary-material>

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