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Climatic and environmental implications from *n*-alkanes in glacially eroded lake sediments in Tibetan Plateau: An example from Ximen Co

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Gas chromatography-mass spectrometry was used to identify a series of *n*-alkanes in the sediments of a typical glacially eroded lake in the eastern Tibetan Plateau. By comparing the distribution patterns of *n*-alkanes in lake sediments, surface soils and cow manure, it was shown that $n-C_{27}$ - $n-C_{33}$ alkanes in the soil ecosystem of Ximen Co are derived from vascular plant species and that the distribution pattern of $n-C_{27}$ - $n-C_{33}$ alkanes remains unchanged during the feeding and digestion processes of herbivores. The relative percentage of C_{27} , C_{29} and C_{31} *n*-alkanes decreased from the bottom to the top of the sediment core showing a trend of degradation of higher plants in the Ximen Co lake region during the formation of the 44 cm core. ²¹⁰Pb dating, combined with pre-existing AMS ¹⁴C dating results showed that the depositional core reflects climatic and environmental variations since about 900 years before present. The *n*-alkane indexes (ACL₂₇₋₃₃, P_{aq} , P_{wax}) are comparable with regional temperature variation, especially recording the Little Ice Age event (LIA). This study highlights that *n*-alkane molecular fossils derived from a typical glacially eroded lake.

Tibetan Plateau, glacially eroded lake, Ximen Co, lake sediment, molecular fossil, *n*-alkane, climatic and environmental change

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Investigation of organic components in lake sediments is considered an effective approach for paleo-climate and paleo-environment reconstruction in the Tibetan Plateau [1–6]. Common approaches used include organic carbon and nitrogen isotopes, total organic carbon and nitrogen contents, C/N ratio, hydrogen index, oxygen index, and pigments, as well as molecular fossils, which have become increasingly popular in recent years. However, there are some difficulties in the application of these lacustrine organic geochemical proxies because the complexity of the organic matter sources in lake sediments leads to uncertainty and multiplicity of proxy interpretation [7] and the physical, chemical and biological effects occurring in the organic matter depositional process may change the environmental information contained within the organic components [8]. Thus it is important to investigate organic components that are relatively stable and have a clear biological source.

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Normal alkanes (*n*-alkanes) are stable lipid molecular fossils, which contain biological source information [7,9] and have been widely used in climatic and environmental variation studies in the Tibetan Plateau in recent years [6,10–14]. Previous studies have shown that the abundance and distribution patterns of *n*-alkanes within the same type of vascular plants exhibit good stability and a unique distribution model [15] whereas *n*-alkanes with different biological sources have very different relative abundance and distribution models [16,17]. Even in different stages of lake evolution, the distribution patterns of *n*-alkanes truly reflect the variation in organic matter from different biological sources in lake sediments and can be used to infer regional climate and environment change.

Ximen Co is an erosional lake, which formed from glacial excavation and erosion of a soft and/or broken rock band in the mountains of a high-latitude area. The lake is characterized by a small size, steep shoreline and great depth. There are abundant ice-scour lakes in the Tibetan Plateau. Previous studies have focused primarily on tectonically formed large lakes, such as Qinghai Lake [5], Nam Co [13,18], and Siling Co [19]. However, research on glacially eroded lakes is relatively lacking. Glacially eroded lakes often exist in cirques or trough valleys and consequently the watershed of glacially eroded lake tends to have significantly steep slopes [20]. Thus, both the glacier water and precipitation have a significant scouring effect on the slope vegetation and soil, transporting large amounts of terrigenous detritus into the lake. Because of the relatively deep water, the sedimentary environment is stable and consequently the organic matter tends to be well preserved. In such a geological setting, the distribution patterns of *n*-alkanes and the extent to which *n*-alkanes reflect the regional climatic and environmental variation are issues worthy of investigation.

Ximen Co, which is a typical glacially eroded lake in the Nianbaoyeze Mountains of the eastern Tibetan Plateau

(Figure 1), was chosen as the area for investigation. We extracted the n-alkane molecular fossils from the lake sediment as well as modern soil samples and cow manure and compared the distribution patterns of these samples. Also, we attempted to reconstruct the climatic and environmental history since about 900 calendar years before present (BP) in the study region using n-alkane indexes.

1 Materials and method

1.1 Sample collection and preparation

Nianbaoyeze (Guoluo Mountain) is located in Jiuzhi in the Guoluo Prefecture within Qinghai Province, which is part of the Bayankala mountain range. The elevation of the prominent peak is 5369 m, which is covered by a modern glacier with a modern snow line altitude of about 5100 m [23]. Ximen Co is a typical glacially eroded lake in the Nianbaoyeze area, which formed by melt water filling the glacial trough in the post-glacial period. The lake's average elevation is about 4020 m and its area is 3.8 km². The average depth of Ximen Co is approximately 40 m, with the maximum depth close to 65 m. The lake water supply is mainly from local precipitation and glacial melt water. Meteorological data show that the Jiuzhi region has the maximum precipitation in Qinghai Province, with an average annual rainfall of 774.3 mm and annual evaporation of less than 1250 mm. The vegetation in the catchment area is dominated by alpine meadow and alpine shrub meadow [24].

Lake sediment, modern soil and cow manure samples were all collected in early July 2009. Figure 1(1–5) shows the lake sediment and soil sampling sites. Vegetation foliage was removed before soil samples were collected below the plants. Fresh cow manure was collected near the lake. A 44 cm long short core (XMC-6) was taken from Ximen Co at 33°22'40.59"N/101°06'21.78"E using a gravity corer. The whole core has a light gray silty clay texture and had no obvious alteration or damage. The core was divided into



Figure 1 Previous studies of *n*-alkanes in lake sediments on the Tibetan Plateau, including Luanhaizi Lake [21], Qarhan Lake [12,22] and Nam Co [13,17] as well as Ximen Co (a). The sampling sites in the Ximen Co lake region which including lake sediments and soil are shown in (b). The dotted lines are elevation contours.

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twenty-two samples of 2 cm increments which were wrapped in foil, put in a sample bag and preserved in a frozen preservation box. The samples were immediately freeze dried upon arrival at the laboratory.

1.2 Analytical methods

Each sample was approximately 2 g and was passed through a number 80 mesh sieve. The soluble organic matter was ultrasonically extracted (3x) with solvent (dichloromethane and methanol, V:V=93:7) for 20 min using an ultrasonic generator. The samples from the three extractions were combined and filtered, and then were concentrated to constant weight. To avoid compound loss, no further fractionation of the lake sediment samples was undertaken. The extracts of lake sediments were air dried and then derivatized by heating with N, O-bis (trimethylsilyl) trifluoroacetamide before GC/MS analysis. Taking into account the high organic matter concentration of modern soil and cow manure samples, the total lipid extracts were fractionated by flash column chromatography into saturated hydrocarbons, aromatics, and non-hydrocarbons. The hydrocarbon component was determined by instrument directly. The glassware was washed with the oxidizers and rinsed with glass-distilled solvents prior to use. Procedural blanks were also analyzed to ensure the absence of possible laboratory contaminants.

GC-MS was performed with a Hewlett Packard 7890A gas chromatograph interfaced with a Hewlett Packard 5975C mass selective detector and equipped with a DB-5 MS column (30 m×0.25 mm ID, film thickness 0.25 μ m). The oven temperature was gradually increased at 3°C/min from 70–300°C and held for 20 min. Helium was used as a carrier gas. The compounds were assigned by comparison with mass spectra and retention times from the literature. The organic carbon isotope composition ($\delta^{13}C_{org}$) was tested on an EA 1112 HT-MAT253 using a standard of V-PDB (Vienna Peedee belemnite). Glycine and collagen standards provided by SIGMA Company were used to test instrument conditions and the total error was less than 0.15‰.

1.3 Dating

The upper 6 cm of XMC-6 was dated using the 210 Pb method and showed that the sedimentation rate was about 0.503 mm/a. Our research group had previously collected a long core, located close to the XMC-6 core, and found that the sedimentation rate was very stable, at least within the upper 265 cm [25]. Thus, the dates of the complete XMC-6 core were calculated from the sedimentation rate of the upper 6 cm. This sedimentation rate is similar to a previous study in this lake, which obtained ages by 210 Pb and 14 C methods [26]. According to this sedimentation rate, the age of XMC-6 is about 875 a BP.

2 Results and discussion

2.1 Distribution pattern of *n*-alkanes in glacially eroded lake sediments

The n-alkanes in Ximen Co lake sediments range from n-C₁₅ to n-C₃₃ and show a unimodal distribution pattern, while the higher-carbon-number n-alkanes have distinct odd carbon predominance, with CPI_h values ranging from 4.68 to 7.66 (Table 1). The high-carbon-number *n*-alkanes ($C_n \ge$ C_{21}) comprise more than 90% of the total *n*-alkane abundance. The low-carbon-number n-alkanes are homologues, without any odd/even predominance, and are present at low to undetectable concentrations in the samples (Figure 2). Generally, the n-alkane distributions of leaf waxes of vascular plants have distinct odd-over-even preference. They usually range from 21 to 35 carbons in chain length, with a maximum at n-C₂₇, n-C₂₉ or n-C₃₁ [27]. However, the *n*-alkane distributions of submerged and floating plants are dominated by n-C₂₁, n-C₂₃ or n-C₂₅ [28]. Thus, the long chain *n*-alkanes in the lake sediment are mainly derived from terrestrial and aquatic vascular plants. The main peak of all 22 samples was $n-C_{31}$, implying that the main allochthonous inputs to Ximen Co are terrestrial herbs [29]. Previous studies showed that plants in the Ximen Co region were dominated by herbs and shrubs, with their pollen concentration comprising more than 90% of the total pollen concentration [30]. Therefore, the higher vegetation characteristics deduced from the n-alkane distribution, are consistent with former pollen data. The relative percentages of $n-C_{27}$, $n-C_{29}$, and $n-C_{31}$ alkanes to total extractable organic matter were calculated by the area normalization method. As shown in Figure 3, the relative percentage of $n-C_{27}$, $n-C_{29}$, and $n-C_{31}$ homologues decreases from bottom to top in XMC-6 indicating a reduction in the contribution of terrestrial organic matter. As the organic matter sources of Ximen Co are mainly terrestrial vascular plants, it is inferred that the total amount of higher plants have reduced during the period of formation of the XMC-6 core. That is to say local higher plants, especially the herbs, degraded gradually in the corresponding period. This phenomenon may be due to climatic variation and the rapid development of local animal husbandry.

It is noteworthy that the abundance of n-C₂₅ alkane is relatively high, indicating aquatic plant inputs, especially a submerged/floating plant contribution [28]. The *n*-alkane distribution pattern in the lake sediments of Nam Co and Ximen Co are similar, with the exception of the n-C₂₁ and n-C₂₃ homologues which showed relatively high abundance in Nam Co [13]. This suggests an obvious *n*-alkane contribution from aquatic plants, but that the types of aquatic plants in these two lakes may be very different. On the other hand, the *n*-alkane distribution patterns of Luanhaizi Lake are very different from Ximen Co and Nam Co, showing the *n*-alkanes as being dominated by n-C₂₃ and n-C₂₅ and some



Figure 2 The *n*-alkane distribution in Ximen Co lake sediments.



Figure 3 Profile trends in the relative percentage of n-C₂₇, n-C₂₉, n-C₃₁ to total extractable organic matter.

samples displaying bimodal distribution with n-C₁₉ as the main peak in low-carbon-number homologues. The carbon preference index in Luanhaizi is lower than in Ximen Co and Nam Co, while in Nam Co P_{aq} values in most samples are greater than 0.4, reflecting the main sources of *n*-alkanes as being aquatic plants, bacteria and algae [21]. The main reason for the *n*-alkane distribution discrepancy between the different lakes might be due to differences in climatic conditions and in the ecosystems.

2.2 Comparison of *n*-alkane distributions in lake sediment, modern soil and cow manure

The *n*-alkane distribution characteristics are similar in all modern soil samples, implying the same vegetation cover and ecological background in the Ximen Co lake region. The *n*-alkanes in all soil samples range from C_{20} to C_{33} , with a maximum at C_{31} , while the distribution range is clearly smaller than the *n*-alkane distribution in the lake sediments (Figure 4(b)). The *n*-alkane homologues $n-C_{25}$ show relatively high abundance with distinct odd-carbon preference. The organic carbon isotope values range from -26.0% to -26.8% with an average of -26.4%. It is believed that the organic carbon isotope in surface soil can distinguish C_3/C_4 plant distribution in *in-situ* vegetation and that this function is identical with the isotopic composition of the long-chain *n*-alkanes [31]. Therefore we conclude that the local vegetation in the Ximen Co lake region is dominated by C₃ species with C₄ species being less abundant. This result is in accord with the observed vegetation and environmental characteristics in this region and is also consistent with previous studies in the Tibetan Plateau [32].

The *n*-alkane distribution of modern soil and lake sediment samples (as shown in Figure 4(a) and (b)) shows clear discrepancies between them. The relative abundance of $n-C_{15}$ -*n*- C_{19} alkanes, which represent the algal and bacterial

 Table 1
 Molecular fossil parameters of lake sediment samples and organic carbon isotope values^{a)}

Depth (cm)	CPI_{h}	ACL ₂₇₋₃₃	$P_{\rm aq}$	$P_{\rm wax}$	$\delta^{13}C_{org}(\%)$
0–2	4.78	29.73	0.36	0.70	-24.4
2–4	5.09	29.67	0.33	0.73	-24.7
4–6	7.04	29.82	0.40	0.67	-24.1
6–8	5.13	29.77	0.33	0.73	-23.6
8-10	5.38	29.80	0.29	0.76	-23.8
10-12	5.31	29.72	0.32	0.74	-24.1
12-14	5.50	29.73	0.39	0.69	-23.7
14–16	5.06	29.68	0.39	0.68	-23.6
16-18	4.81	29.76	0.38	0.69	-23.6
18–20	4.92	29.56	0.39	0.69	-23.8
20-22	4.84	29.57	0.43	0.65	-23.6
22-24	4.68	29.40	0.40	0.68	-23.8
24–26	7.61	29.93	0.34	0.72	-23.7
26–28	5.71	29.70	0.33	0.73	-23.6
28-30	7.03	29.77	0.33	0.74	-23.8
30-32	5.84	29.78	0.36	0.70	-23.9
32–34	6.66	29.91	0.27	0.77	-23.9
34–36	7.66	29.86	0.29	0.75	-23.3
36–38	5.69	29.70	0.36	0.70	-23.6
38-40	6.64	29.75	0.31	0.75	-23.4
40-42	4.90	29.60	0.39	0.69	-23.4
42–44	5.98	29.81	0.32	0.74	-23.6

a) $CPI_h = = od\Delta\Sigma[C_{21-33}]/even\Sigma[C_{22-32}]; ACL_{27-33} = (27 \times C_{27} + 29 \times C_{29} + 31 \times C_{31} + 33 \times C_{33})/(C_{27} + C_{29} + C_{31} + C_{33}); P_{aq} = (C_{23} + C_{25})/(C_{23} + C_{25} + C_{29} + C_{31}), P_{wax} = (C_{27} + C_{29} + C_{31})/(C_{23} + C_{25} + C_{27} + C_{29} + C_{31});$ the organic carbon isotope values versus V-PDB standard, error is less than 0.15%.

contributions and the n-C₂₁-n-C₂₅ alkanes, which represent floating/submerged aquatic plant contributions, are distinctively higher than in the modern soil. However, the n-C₂₇-n-C₃₃ alkanes, which represent the terrestrial higher plants as well as the emergent plant inputs, have a similar distribution in both soil and lake sediment samples. Consequently, the n-alkane distribution in lake sediments not only reflects the signals of higher terrestrial vegetation and emergent plant inputs, but also records the organisms living in Ximen Co, including the floating/submerged plants as well as algae and bacteria.

The *n*-alkane distribution of cow manure in Ximen Co lake region ranges from n-C₂₅ to n-C₃₅, with a maximum at C₃₁, and shows a distinct odd-carbon preference which is indicative of an alpine meadow herb contribution (Figure 4(c)). This distribution pattern is very similar to that for *n*-alkanes in the modern soil. Comparison of the *n*-alkane distribution between the modern soil and cow manure demonstrates that cattle and other herbivores selectively feed on local alpine meadow herbs. However, the distribution pattern of C₂₇ to C₃₃ *n*-alkanes remains unchanged during the feeding and digestion processes of the herbivore.



Figure 4 The *n*-alkane distribution in lake sediments, modern soil and cow manure (*y* error bars represent the standard deviation).

Therefore, varying numbers of grazing animals will not result in the loss of *n*-alkanes as ecosystem and environment indicators.

2.3 The *n*-alkane indexes from Ximen Co are consistent with Tibetan Plateau paleo-temperature records

To maintain a plant's moisture balance and protect its leaf cells, the leaf epicuticular composition of higher plants responds significantly to temperature change. In warmer tropical climates, land plants are postulated to biosynthesize longer chain compounds for their waxy coatings, whereas in cooler temperate regions somewhat shorter chain compounds are produced [27]. The *n*-alkane ACL index, proposed by Poynter (1990), is the concentration weighted mean chain length of the *n*-alkanes present in a geological sample. He studied the ACL index of long-chain *n*-alkanes in marine sediments and suggested that this index can represent the temperature variation in a source area [33]. This result has been supported by observations of *n*-alkanes in

modern leaves. The leaf n-alkane distribution in an individual plant species varies with the seasons, mainly responding to temperature change [34]. This phenomenon is also observed in *n*-alkane studies in loess [35] and peat [36]. However, it should be noted that if climate variation causes the plant species to vary or results in sedimentary source changes, then the ACL value cannot effectively reflect the temperature changes and more accurately reflects changes in the vegetation [12]. The ACL value is greater in higher plants than in lower plants and aquatic algae, with values in angiosperms being higher than in gymnosperms, and C₄ plants higher than in C_3 plants [37]. The foregoing analysis shows that the *n*-alkane distribution is basically constant in XMC-6, indicating that higher plants did not significantly change during the depositional period. Therefore it is suggested that the average chain length of C_{27} to C_{33} *n*-alkane homologues, which are mostly derived from vascular plants, have a potential relationship to regional temperature. As shown in Figure 5(a), the ACL₂₇₋₃₃ of XMC-6 varies between 29.40 and 29.91 with an average value of 29.72 (Table 1). The ACL₂₇₋₃₃ is comparable to standardized temperatures in the southern Tibetan Plateau (Figure 5(a)) [38]. This is especially the case during the LIA phase in the study area (Figure 5(a) shaded part), coinciding with the low-value phase of the ACL₂₇₋₃₃ index. The correlation of standardized temperature and ACL₂₇₋₃₃ index strongly supports the conclusion that ACL₂₇₋₃₃ is sensitive to temperature changes in the study area.

Recently, the P_{wax} and P_{aq} indexes of *n*-alkanes in sediments have been used to estimate the effective moisture changes in wetland environments in the past [11,39]. The P_{aq} index represents non-emergent aquatic plant inputs to lake sediments relative to that from emergent aquatic and terrestrial plants [28]. It is proposed that a high P_{aq} value

corresponds to an increased contribution from aquatic plants, especially submerged and floating plants, indicating high precipitation associated with relatively humid climatic conditions. On the other hand, decreased P_{aq} values correspond to an increase in terrestrial plants and/or emergent plants, indicating decreased precipitation corresponding to a relatively dry climate. This has been confirmed in the Zoigê-Hongyuan peat deposit [11]. P_{wax} reflects the relative abundance of *n*-alkanes derived from terrestrial higher plants as well as emergent vegetation relative to n-alkanes derived from other kinds of plants. Low P_{wax} implies relatively humid climate conditions, whereas high values reflect relatively dry conditions [11]. Paq values in Ximen Co lake sediment samples varied between 0.27 and 0.43, with a mean value of 0.35 (Figure 5(c), Table 1), and P_{wax} values varied between 0.65 and 0.77, with an average value of 0.71 (Figure 5(d), Table 1). A low standardized temperature in the southern Qinghai-Tibet Plateau corresponds to a high $P_{\rm aq}$ index and a significant decrease in the $P_{\rm wax}$ index (shaded part in Figure 5), suggesting a period relatively enriched in submerged/floating plants and a reduction in emergent/terrestrial plants. Moreover, the low ACL₂₇₋₃₃ values just correspond to this phase which is associated with the LIA event. Although the ambient temperature during the LIA was significantly reduced, the effective moisture did not decrease very much. It is suggested that *n*-alkanes derived from submerged and floating plant contribution increased, while the emergent/terrestrial plant contribution decreased during the LIA. The information reflected in the *n*-alkane indexes suggests that the climate characteristics during the LIA in Ximen Co were cold and wet. This inference is consistent with previous results, which were obtained from pollen and peat depositional rates in the study region [40].



Figure 5 Profile trends of δ^{13} Corg, ACL₂₇₋₃₃, P_{aq} and P_{wax} and comparison with the standardized temperature variation of the southern Tibetan Plateau [38]. Ximen Co is located in the eastern Tibetan Plateau, but it is generally believed that the climate conditions are more similar to the southern Tibetan Plateau.

The organic carbon isotope variation in the XMC-6 core is also comparable with the standardized temperature in the southern Tibetan Plateau as well as the n-alkane proxies (Figure 5(e)). However, the $\delta^{I3}C_{org}$ proxy is not in perfect accord with the other proxies, even showing a reverse trend in some parts. This phenomenon was also observed in Qarhan paleo-lake [12]. A possible reason is that the organic carbon isotopes in lake sediments are affected by many factors and the mechanism is complex. Therefore a simple corresponding relationship between the organic carbon isotope and regional temperature is unlikely to exist [41]. However, what is interesting is that the organic carbon isotopic ratio shows a slightly positive excursion in the LIA period. A possible explanation is that the low ambient temperature caused the atmospheric CO₂ concentration to decrease, resulting in the dissolved CO₂ concentration also decreasing. Phytoplankton, floating plants and submerged lacustrine plants use HCO₃⁻ (δ^{13} C=1%) as their organic carbon source and therefore the organic carbon isotope would show a positive excursion during the LIA period [7].

The molecular fossil record in Ximen Co shows that ambient temperature has a significant impact on *n*-alkanes in higher plants, suggested by the strong correlation between *n*-alkane indexes and the paleo-temperature record. Because *n*-alkanes are considered to be chemical or molecular based species, they show very sensitive responses to climatic and environmental variation [42]. Previous studies have shown that *n*-alkanes have an advantage in reflecting environmental changes in peat in the Tibetan Plateau [43]. For instance, dominant plant species may remain unchanged under slight climate variations but their molecular composition may change slightly in response to the variation [44]. Therefore, *n*-alkane indexes can better reflect the climatic and environmental changes than organic carbon isotopes or other organic indexes and this phenomenon is evident in glacially eroded lake sediments.

3 Conclusions

(1) Comparison of the distribution patterns of *n*-alkanes in lake sediments, surface soils and herbivore feces shows that $n-C_{27}-n-C_{33}$ alkanes in lake sediments are derived from higher plants in the soil ecosystem in the Ximen Co lake region. The distribution pattern of $n-C_{27}-n-C_{33}$ alkanes remains unchanged during the feeding and digestion processes of herbivores.

(2) The relative percentages of n-C₂₇, n-C₂₉, and n-C₃₁ alkanes decrease from the bottom to the top of the sediment core showing a trend of degradation of higher plants in the Ximen Co lake region during the formation period captured within the 44 cm core. The reasons for higher plant degradation require further study. The present degradation may be due to climatic factors and the rapid development of local animal husbandry.

(3) The trends of *n*-alkanes proxies (ACL₂₇₋₃₃, P_{aq} , and P_{wax}) are consistent with regional temperature variation, in particularly displaying a clear excursion during the LIA event. It is suggested that the *n*-alkane homologues are sensitive to climatic and environmental variation, and are reliable indicators of paleo-climate and paleo-environment variation in glacier erosion lakes.

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