Article

Chinese Science Bulletin

Geochemistry

June 2012 Vol.57 No.17: 2140–2147
doi: 10.1007/s11434-012-5012-7

Influence of variation in precipitation on the δD values of terrestrial *n*-alkanes on the southern Tibetan Plateau

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Received November 18, 2011; accepted January 6, 2012; published online March 31, 2012

A short lake sedimentary core and ice core were recovered from Lake Chen Co and Noijingangsang Glacier, respectively, in the Lake Yamdrok watershed of the southern Tibetan Plateau. Hydrogen isotope ratios of the ice core (δD_{ice}) and δD values of terrestrial *n*-alkanes (δD_{wax}) in the sediments showed a linear correlation ($R^2 = 0.41$, $P = 0.047$) over the past 80 years, indicating the effect of hydrogen isotope ratios of precipitation (δD_p) on δD_{wax}. However, apparent fractionation ($\varepsilon_{wax\text{-}p}$) values of δD_{wax} relative to δD_{ice} increased with decreasing ice core accumulation (amount of precipitation; $R^2 = 0.65$, $P = 0.0051$), revealing that dry or wet climate variation had a significant impact on δD_{wax}. Thus, precipitation amount and δD_p are both important in determining δD_{wax}.

***n*-alkane, hydrogen isotope, lake sedimentary core, ice core, southern Tibetan Plateau**

Citation: Xie Y, Xu B Q, Wu G J, et al. Influence of variation in precipitation on the δD values of terrestrial *n*-alkanes on the southern Tibetan Plateau. Chin Sci Bull, 2012, 57: 2140–2147, doi: 10.1007/s11434-012-5012-7

Terrestrial *n*-alkanes are important components of leaf epicuticular wax [1,2], which is biosynthesized by plants using mainly meteoric water [3]. Because *n*-alkanes show distinct characteristics such as distribution, carbon chain length, maximum carbon number and others in different plants, they have been widely used in climate change research [3–9]. Generally, leaf water is directly used by plants to synthesize leaf wax *n*-alkanes [3], and the pathway of plant water use that can induce hydrogen isotopic fractionation has been designated "precipitation-soil water-leaf water-leaf wax" [10,11]. Consequently, compound-specific hydrogen isotope ratios of terrestrial *n*-alkanes (δD_{wax}) retrace the hydrogen isotopic composition of precipitation (δD_p) that has been modified during the pathway. Thus, studies of the relationship between δD_{wax} and δD_p, the hydrogen isotopic fractionation in the processes of plant water use and plant biosynthesis, are very important for clear understanding of δD_{wax}. Because the entire process is very complicated, there has been no systematic research on the hydrogen isotopic fractionation of each stage. Therefore, research on the apparent hydrogen isotopic fractionation between δD_{wax} and δD_p ($\varepsilon_{wax\text{-}p}$), and relationships between $\varepsilon_{wax\text{-}p}$ and environmental and biological factors, would be very useful for investigating the meaning of δD_{wax}.

Variations in environmental parameters such as relative humidity (RH), temperature, precipitation, evaporation and others can affect δD_{wax} through changing soil evaporation and leaf evapotranspiration [10–13]. Compared with environmental parameters, the mechanism by which biological factors influence δD_{wax} is more complicated. Although there is no clear understanding of the mechanism, studies of δD_{wax} from different types of higher plants in the same area show that many kinds of physiological characteristics affect δD_{wax}, including life form (e.g. tree/shrub/grass), photosynthetic pathway (C3 vs. C4), leaf anatomy/morphology (stomata/interveinal distance), root-depth (deep-rooted vs. shallow-rooted) and others [10,12,14–18]. For the example of life form, it was found that *n*-alkanes from trees have the highest δD values, whereas grasses have the lowest and shrubs have intermediate values [15]. Nevertheless, these

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factors are intricate and interacting, e.g. climate variation may cause changes in plant cover and adaptive plant physiology [19].

Because δD_{wax} can be influenced by such factors, $\varepsilon_{wax\text{-}p}$ could be variable. In one study, an inverse relationship was obtained between $\varepsilon_{wax\text{-}p}$ and annual rainfall [12]. However, in other studies, δD_{wax} was found to largely reflect δD_p variation, i.e. $\varepsilon_{wax\text{-}p}$ is relatively constant. δD_{wax} of lake surface sediments in a north-south European transect [3] and in North America [11] varied with δD_p; this was also shown by δD_{wax} preserved in soil from eastern China [20]. Therefore, δD_{wax} has been regarded as δD_p for reconstructing paleohydrology [21].

The fact that such distinct arguments are made in these studies is caused by the same research method: lake surface sediments, soils, or plant leaves were sampled with spatial scale [3,11,12,20]. $\varepsilon_{wax\text{-}p}$ was calculated together with δD_p, which was obtained using the Online Isotope Precipitation Calculator (OIPC) [3,11], or was analyzed after collection in the field [14]. However, there are some defects in this method. δD_p shows significant seasonal variation [22], and the hydrogen isotope composition of soil and leaf water also varies because of variable δD_p and environmental factors [23]. Moreover, leaf wax *n*-alkanes can be abraded by precipitation [23], dust [24], and other factors. Furthermore, the rate of biosynthesis and abrasion can be affected by environmental factors and plant physiology [24–27]. Terrestrial *n*-alkanes in lake sediments and soils are preserved during this complicated process. Overall, environmental water hydrogen isotopic composition, hydrogen isotopic fractionation in the plant water-use pathway, and the rate of *n*-alkane "biosynthesis-abrasion" on leaf surfaces and accumulation in media are not constant. Consequently, the (δD values of) "water" and *n*-alkanes used to calculate $\varepsilon_{wax\text{-}p}$ do not share the same temporal range in the "spatial scale" method.

In this study, we determine hydrogen isotope ratios for an ice core and terrestrial *n*-alkanes in lake sediments from the same area of the Tibetan Plateau, to explore relationships between δD_p and δD_{wax}, and between δD_{wax} and climate factors.

1 Materials and methods

1.1 Study area and sampling

The Lake Yamdrok drainage basin (90°08′–91°45′E; 28°27′–29°12′N) is on the southern Tibetan plateau (Figure 1b). Chen Co, which is part of Lake Yamdrok, is a closed lake with 40 km^2 of water surface within a drainage area of 148 km^2. The lake is at an elevation of about 4200 m and is supplied by precipitation and glacial meltwater [28]. Weather records from Langkazi Station in the lake drainage area show a multi-year average annual temperature of 2.6°C. The average annual rainfall is 372.8 mm, with greatest amounts between May and September [29]. Artemisia and Stipa grassland represent the dominant plant cover in the

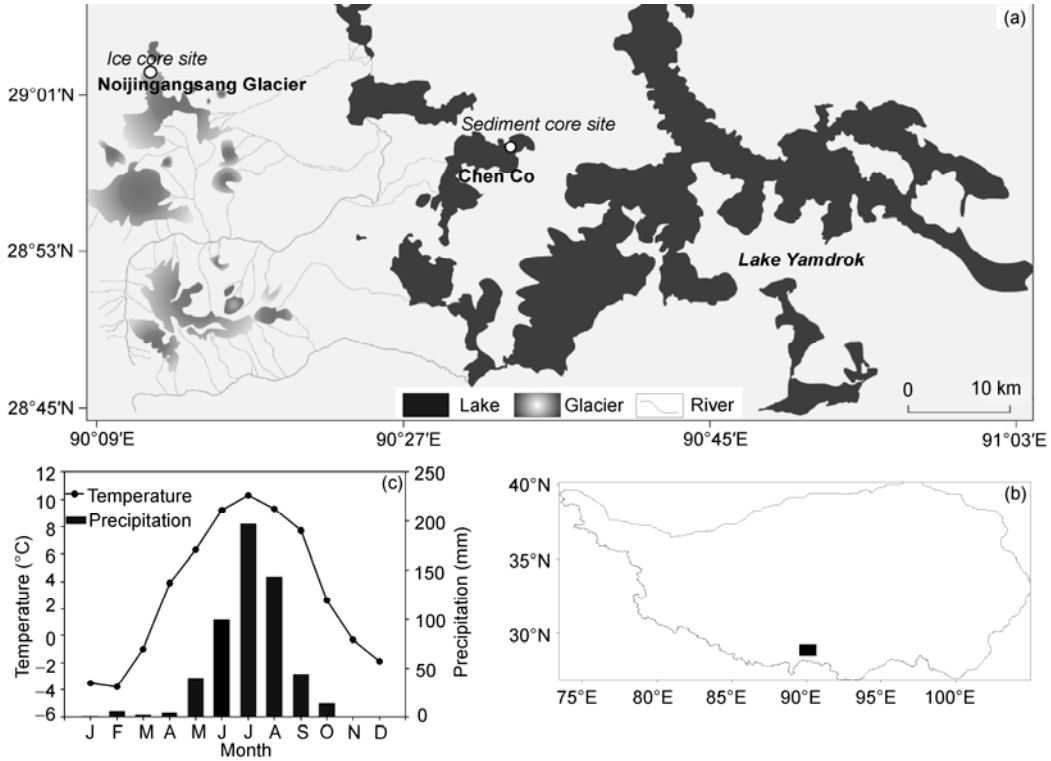

Figure 1 (a) Map of study area, showing sites for recovery of sediment core from Lake Yamdrok, and for recovery of ice core from Noijingangsang Glacier; (b) location of study area on the Tibetan Plateau; (c) monthly precipitation and air temperature for study area.

area [30]. The core (28°58′N, 90°33′E) was recovered in 2005 using a gravity driller in Chen Co at 15 m water depth. It was 33 cm long, and sediments from the top 13 cm were dated and analyzed. The Noijingangsang Glacier, with a maximum elevation of 7206 m a.s.l., lies in the eastern Lhagoi Kangri Mountains and western Lake Yamdrok drainage basin. There are about 50 glaciers around the peak of Noijingangsang, with a total area of about 129 km^2 [31]. The ice core (29°2.4′N, 90°12′E) was 55 m long and was drilled to the bedrock in 2007. It was taken at an elevation of 5950 m from the glacier. The top 36.5 m of the ice core was analyzed.

1.2 Analysis of *n*-alkane concentration and δD values

The experiments, including solvent extraction, *n*-alkane quantification and isotopic analysis, were performed at the Max Planck Institute for Biogeochemistry. The sediment core was subsampled at 1 cm intervals. Samples were freeze-dried and ground to at least 80 meshes. Organic matter was extracted using an accelerated solvent extractor (ASE200, Dionex Corp., Sunnyvale, USA) with CH_2Cl_2/MeOH (10∶1) at 100°C and 13.79 MPa (2000 psi), for 15 min in 2 cycles. Activated copper was added to the samples to remove elemental sulfur. Silica gel chromatography with glass columns was used to further separate total lipid extracts. The hydrocarbon fraction was eluted with 80 mL *n*-hexane. The *n*-alkanes were quantified using gas chromatography (GC; Agilent6890, Agilent, Palo Alto, USA) with atomic emission detection and a DB5ms column (30 m × 0.32 mm × 0.5 μm), by comparison with an external *n*-alkane standard mixture. The injector was operated at 280°C with a split ratio of 1:10. The GC oven was held at 80°C for 2 min, and then raised at 8°C/min to 320°C.

The alkane fraction in *n*-hexane was injected (1 μL) into a HP6890 gas chromatograph. The injector was operated at 280°C in splitless mode. The oven was held for 2 min at 80°C, then heated at 6°C/min to 320°C (held 10 min). The carrier gas flow was held constant at 1.7 mL/min throughout the run. The eluting compounds were transferred to a high-temperature conversion furnace operated at 1425°C [32,33] and quantitatively converted to H_2, which was introduced into an isotope ratio mass spectrometry instrument (Delta^{plus}XL, Finnigan MAT, Bremen, Germany) for compound-specific determination of δD values. Each sample was independently measured three times. All δD values were normalized to the Vienna Standard Mean Ocean Water (VSMOW) scale, using a mixture of *n*-alkanes (C_{10}–C_{32}) of known isotopic composition. After measurement of no more than two samples, the standard mixture was measured independently three times. To ensure stable ion source conditions during measurement, the H_3^+ factor [33] was determined at least once per day. The factor was constant [4.1; standard deviation (sd) = 0.1] over the measurement period. The detailed experimental procedure is found in [3].

1.3 Analysis of the ice core

The ice core was subdivided into 3 cm segments and melted. δ^{18}O and δD of the melted water were measured by MAT253 and Delta V, respectively. The precision was 1‰ for δD and 0.2‰ for δ^{18}O values. The concentration of ions and β activity were measured using ion chromatography (ICS2000, Dionex, USA) and a Mini 20 Alpha-Beta Multidetector, respectively.

1.4 Dating of lake sediments and ice core

The chronology of the sediment core was established with $^{210}Pb_{ex}$ and ^{137}Cs dating methods. Samples from the top 13 cm were analyzed for ^{210}Pb and ^{137}Cs by gamma spectroscopy, using a well-type low background germanium detector (Ortec HPGe GWL, USA). The $^{210}Pb_{ex}$ data from the sediments were interpreted using the constant rate of supply (CRS) model [34]. The distribution of $^{210}Pb_{ex}$ in the sediment profile displayed a significantly exponential relationship with depth (R^2=0.87; Figure 2(a)). A sedimentation rate of 0.161 cm a^{-1} was inferred. The peak of ^{137}Cs at a depth of 8.5 cm corresponded to the beginning of global-scale nuclear tests in AD 1952, and the highest peak at depth 5.5 cm corresponded to larger-scale nuclear explosions in AD 1963 [34] (Figure 2(b)). According to this rate, samples at 7–8 cm and 5–6 cm depths corresponded to 1952–1958 and 1964–1971, respectively, which agree well with the age inferred from ^{137}Cs (Figure 2(b)). Therefore, the inferred dating result is considered robust, and the sediment core dates are from 1927 to 2005.

The chronology of the Noijingangsang ice core is based on seasonal variations of δ^{18}O ratios, the concentration of K^+ and maximum β-activity concentration corresponding to 1963 [35,36] (Figure 3). Based on these measurements, the top 36.5 m of the ice core spans the period 1925 to 2006.

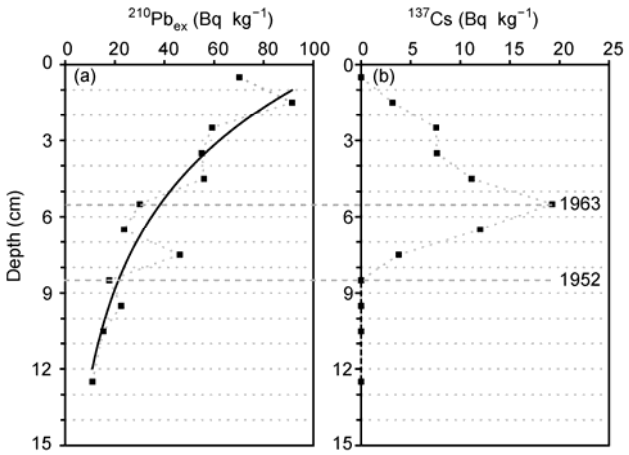

Figure 2 Variations of (a) $^{210}Pb_{ex}$ and (b) ^{137}Cs in lake sediments. Curve shows relationship between $^{210}Pb_{ex}$ and depth.

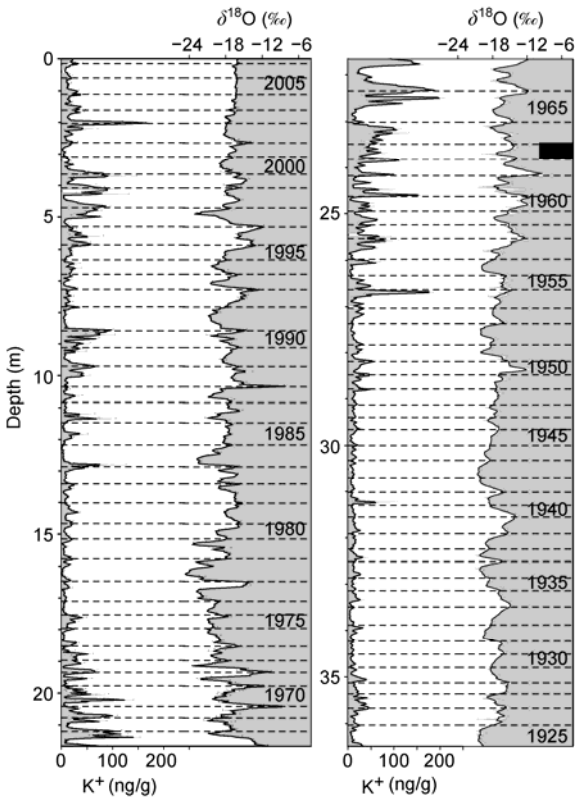

Figure 3 Results of dating Noijingangsang ice core, based on seasonal variation of $\delta^{18}O$ ratio and K^+ concentration. Black bar shows a β activity horizon corresponding to 1963.

2 Results and discussion

2.1 *n*-alkane contents and distributions

The *n*-alkanes in the sediments range from 14 to 32 carbons in chain length, with triple peaks (Figure 4). Among all *n*-alkanes, the short-chain *n*-alkanes that originate from algae and photosynthesis bacteria [37–39] have the lowest concentrations, with weak odd-over-even carbon number predominance. The mid-chain *n*-alkanes, maximizing at *n*-C_{21}, *n*-C_{23} or *n*-C_{25}, have higher concentration than short-chain ones, and reflect the contribution of submerged and floating plants [40,41]. The long-chain *n*-alkanes that are produced by terrestrial higher plants [3,39] have the highest concentrations, and an average Carbon Preference Index (CPI) of 5.5. Moreover, the *n*-C_{31} that comes mainly from grass [39] is always dominant, and this matches the plant cover around Lake Chen Co. These characteristics indicate that the long-chain *n*-alkanes mainly originated from grass around the lake.

2.2 Relationship between δD_{wax} and δD_{ice}

The climate of the study area is mainly controlled by the Indian summer monsoon [22]. Precipitation from May to September accounts for more than 90% of annual rainfall (Figure 1(c)). Thus, the Noijingangsang ice core mainly records summer precipitation signals, including the amount of precipitation and isotopic composition. Another characteristic of the regional climate is that the seasonal variation of temperature follows the same trend as precipitation (Figure 1(c)). Under such climate conditions, the plant growth period [30], including the leaf wax "synthesis-abrasion" process, is from May to September. Thus, the precipitation recorded by the ice core is from the same period as the terrestrial *n*-alkanes in the lake sediments, and the relationship between δD_{wax} and δD_p obtained through comparison of δD_{ice} and δD_{wax} is reliable.

Values of δD_{ice} ranged from −160.55‰ to −87.16‰, with the lowest value in 1978 and highest in 1961. There was a sharp increase from 1975 to 2006 (Figure 5). The δD values of *n*-alkanes are shown in Table 1. We were unable to obtain sufficient material for hydrogen isotopic analysis

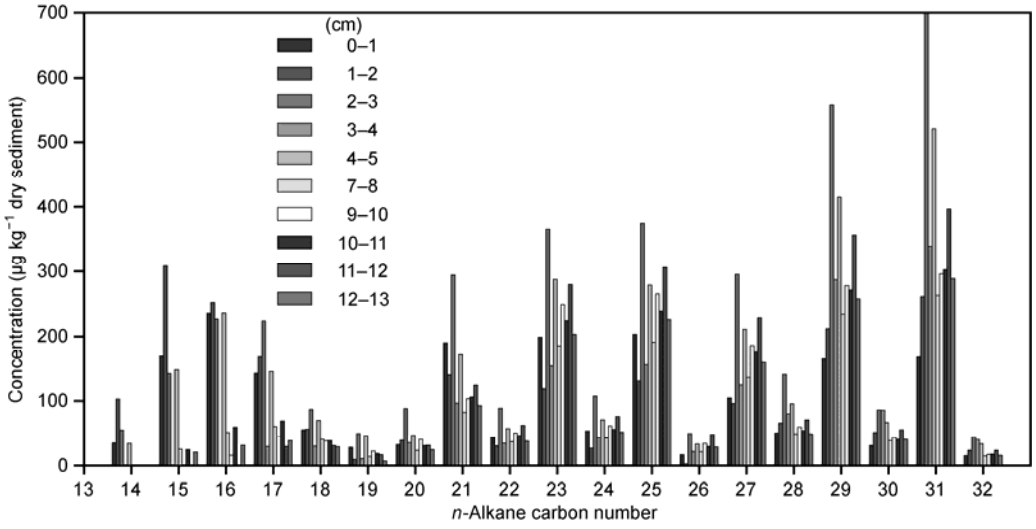

Figure 4 Distribution of *n*-alkanes in the core from Chen Co.

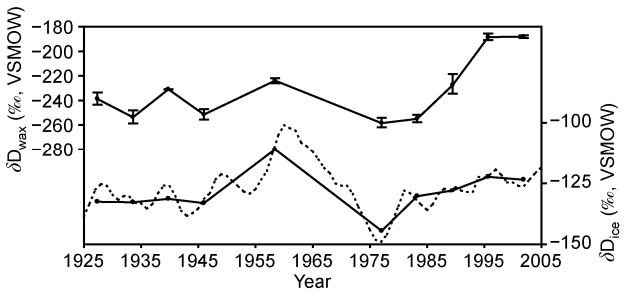

Figure 5 δD_{wax} in Lake Chen Co sediments (top) and δD_{ice} of Noijingangsang ice core (bottom). The dotted line is the 5-year running mean and the solid line (bottom) uses the same chronological scale as sediment dating. Error bars correspond to the δD values for n-C_{29} and n-C_{31}.

of n-C_{27} in some samples. Hydrogen isotope ratios of n-C_{31} and n-C_{29} showed a similar trend and very slight differences in all samples (Table 1). We therefore used weighted-mean δD values of n-C_{29} and n-C_{31} to represent integrated δD_{wax} values of plants around the lake. Values of δD_{wax} were from −258.51‰ to −188.14‰, and rose sharply over the last two decades. The lowest value was in the mid-1970s, and the highest at the beginning of the 21st century (Figure 5). In general, δD_{wax} and δD_{ice} displayed similar trends over the last 80 years (Figure 5), and they showed a reasonably linear correlation relationship ($R^2 = 0.41$, $P = 0.047$). This indicates that δD_{wax} follows δD_p to a certain extent.

2.3 δD_{wax} and ice core accumulation

Although δD_{wax} and δD_{ice} showed similar variations over the last 80 years, there are some obvious differences. For example, δD_{wax} and δD_{ice} had their highest values in 2002 and 1961, respectively; δD_{wax} has increased much more sharply than δD_p since 1975. This may be caused by wet or dry climate variation, and we discuss $\varepsilon_{wax\text{-}p}$ further.

The $\varepsilon_{wax\text{-}ice}$ in this study was calculated by

$$\varepsilon_{wax\text{-}ice} = 1000[(\delta D_{n\text{-}alkane}+1000)/(\delta D_{ice}+1000)-1]. \quad (1)$$

The $\varepsilon_{wax\text{-}ice}$ from our research was not constant. It varied widely, from −143.39‰ to −73.64‰, with an average of −117‰. It varied within a narrow range from 1925 to 1985, and only surpassed the average around 1940. It increased significantly after 1985, greatly exceeding the average (Figure 6).

Ice cores directly record the variation of precipitation [42]. Therefore, wet or dry climate variations may be reflected by ice core accumulation; relatively small accumulation indicates a drier climate [43,44]. The Noijingangsang ice core accumulation (amount of precipitation) was between 292–697 mm, and varied little from 1925–1985. A sharp decrease in its accumulation reflected a very dry climate condition from 1985–2006 (Figure 6). The $\varepsilon_{wax\text{-}ice}$ varied inversely with ice core accumulation over the last 80 years (Figure 6), and an excellent linear correlation ($R^2 = 0.65$, $P = 0.0051$) was observed between the two (Figure 7d). This indicates that wet or dry climate variation strongly influences $\varepsilon_{wax\text{-}ice}$. The reason for this is probably that the precipitation decrease caused lower RH in the area, which can lead to D-enrichment in soil and leaf water through enhancement of soil evaporation and plant evapotranspiration, respectively [12,45,46]. This offsets negative fractionation associated with lipid synthesis [10,11] and produces less negative $\varepsilon_{wax\text{-}ice}$. Therefore, precipitation variation is the essential reason why δD_{wax} variations did not parallel those of δD_{ice}. Statistically, the correlation between precipitation and δD_{wax} is stronger than that between δD_{ice} and δD_{wax} (Figure 7(a), (b)) indicating that the influence of aridity on δD_{wax} is stronger than the effect of δD_p on δD_{wax}.

However, the precipitation "amount effect" [22,47–49] on the southern Tibetan Plateau could cast doubt on the mechanism by which the variation of precipitation affects δD_{wax}. If such an effect exists in the region, one would expect that δD_{ice} would increase with decreasing precipitation, which could cause D-enrichment in δD_{wax}. Surprisingly, δD values from the Noijingangsang ice core and its accumulation did not show significant correlation (Figure 7(c)), which was also discovered by recent research [50]. We conclude that precipitation variation did not influence the isotopic

Table 1 δD values of n-alkanes in Lake Yamdrok sediments

Depth (cm)	Year	n-Alkane δD values (‰ vs. VSMOW)									
		n-C_{23}	SD[a)]	n-C_{25}	SD	n-C_{27}	SD	n-C_{29}	SD	n-C_{31}	SD
0–1	2002	−131	9	−179	5	−160	29	−189	3	−187	3
1–2	1996	−136	6	−180	2	−173	2	−183	1	−191	3
2–3	1989	−165	11	−202	9	−192	15	−218	11	−235	5
3–4	1983	−192	12	−223	10			−252	17	−258	7
4–5	1977	−173	11	−212	10			−254	11	−262	8
7–8	1958	−194	4	−122	16			−226	2	−222	3
9–10	1946	−209	14	−230	6			−247	5	−256	1
10–11	1940							−231	16	−231	5
11–12	1934							−248	4	−259	0
12–13	1927							−234	23	−244	15

a) SD = standard deviation.

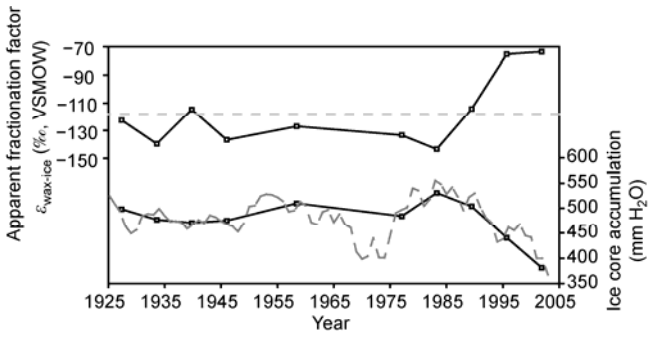

Figure 6 Hydrogen isotope fractionation between terrestrial *n*-alkanes (weighted average of *n*-C_{29} and *n*-C_{31}) and ice core (top); ice core accumulation (amount of precipitation; bottom). Dashed curve is 5-year running mean, and the solid line uses same chronological scale as sediment dating.

composition of the ice core during the last 80 years. This further proves that wet or dry climatic precipitation variations can control δD_{wax}, through changing soil and leaf water hydrogen isotopic composition. The isotopic composition of precipitation depends on a variety of climatic parameters, such as temperature, rainfall amount, humidity and evaporation [51], which could in turn affect δD_{wax} [10–13,45,46]. Thus, we strongly suggest investigating whether δD_p varies with actually observed climate parameters, prior to exploring the relationship between such parameters and $\varepsilon_{wax\text{-}p}$ or δD_{wax}, to find the real relationship between these parameters and δD_{wax}.

2.4 Implications for reconstructing paleoclimate

We show that $\varepsilon_{wax\text{-}p}$ is not constant, and is negatively correlated with precipitation variation. Variations in both δD_p and precipitation could affect δD_{wax}. Thus, both factors should be taken into consideration when using δD_{wax} to explain paleoclimate variation. δD_p is controlled by certain climate factors, and δD_p recorded by ice cores has been used to reconstruct climate, such as temperature [44] variation. If the hydrogen isotope fractionation between terrestrial *n*-alkanes and precipitation is relatively constant as shown by some works [3,11,20], δD_{wax} may be used as δD_p in reconstructing climate variation. However, our result suggests that it is not suitable to regard δD_{wax} as δD_p for reconstructing paleoclimate variation, especially for regions like the Tibetan Plateau where the evapotranspiration effect is very strong [52]. The highest value of δD_{wax} likely indicates a very dry climate, but not a high δD_p value, as in our study. On the other hand, although δD_{wax} is strongly affected by aridity, it may not be well coupled to dry or wet climatic variation. This is also illustrated by research on one loess-paleosol sequence from the Chinese Loess Plateau [45]. The fact that the highest δD_{wax} did not occur during the last glacial maximum, when the climate was very dry, could be

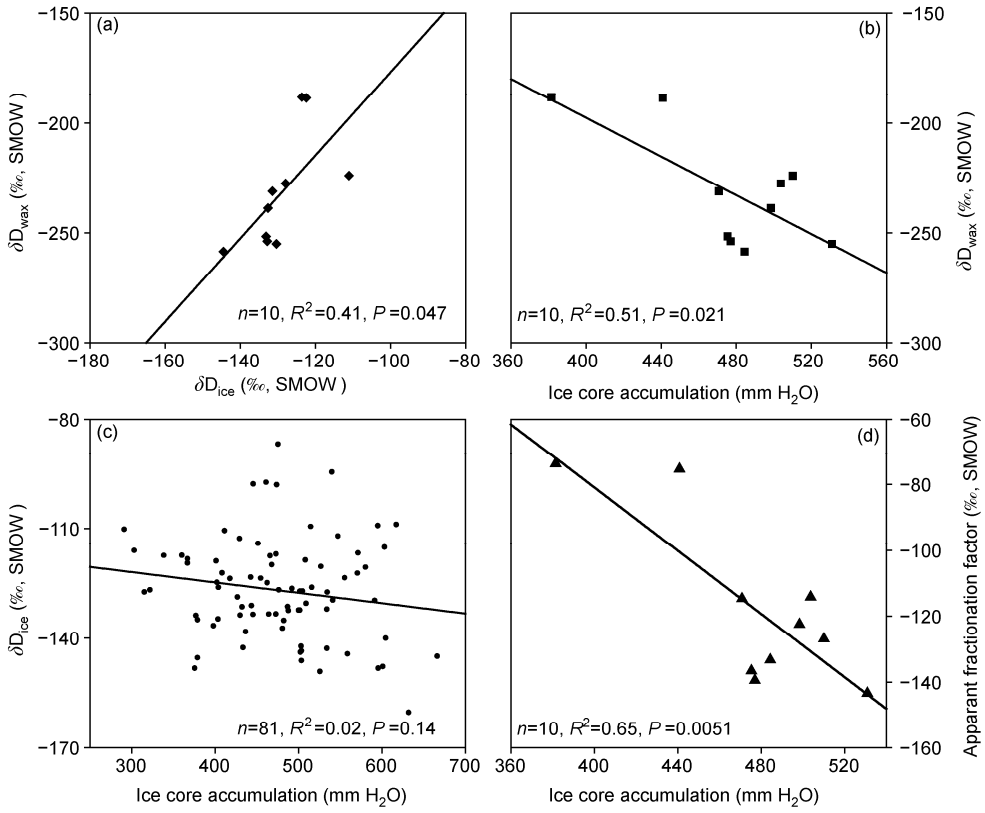

Figure 7 (a) Correlation between δD_{wax} and δD_{ice}; (b) relationship between δD_{wax} and ice core accumulation; (c) relationship between ice core accumulation and δD values; (d) correlation between $\varepsilon_{wax\text{-}ice}$ and ice core accumulation.

caused by the low δD_p. This phenomenon is also found in lake sediments in North America [53]. The δD_{wax} also have this characteristic, which is probably caused by variation of certain climate factors that decreased δD_p. In areas such as central Africa [54] where precipitation and δD_p vary inversely, lower precipitation would induce higher δD_p and D-enrichment in plants. Thus, δD_{wax} would predominantly reflect continental precipitation fluctuations [54].

Undoubtedly, ice cores directly record precipitation variations from the past. However, ice cores can only be obtained in high-latitude and high-altitude regions. The $\varepsilon_{wax\text{-}p}$ is mainly controlled by precipitation, and this provides a new method for reconstructing precipitation variation. Meteoric water δD values can be obtained from δD values of algal biomarkers [3,55,56], such as n-C_{17} from lacustrine sediments. Then, $\varepsilon_{wax\text{-}p}$ can be calculated based on these estimated meteoric δD values and δD_{wax}, for obtaining precipitation variation. However, because the study region here was restricted to the southern Tibetan Plateau, similar research is needed to test whether our findings are applicable to other regions of the world.

3 Conclusions

This study is the first to make a temporal comparison of δD_{wax} and δD_p using a lake sedimentary core and ice core. The δD_{wax} from Lake Chen Co and δD_{ice} from the Noijingangsang ice core showed similar variations over the last 80 years, indicating that δD_{wax} can be affected by precipitation isotopic composition. The hydrogen isotopic fractionation between terrestrial n-alkanes and precipitation was not constant, and had a wide range between −143.39 and −73.64‰. Precipitation variation causes wet or dry climates, which strongly affects δD_{wax} by inducing less/more negative soil and leaf water δD values. Drier climates may lead to higher δD_{wax}. Moreover, the influence of precipitation variation on δD_{wax} is stronger than the effect of δD_p on δD_{wax}. Both δD_p and precipitation should be taken into account during future research on paleoclimate. If δD_p can be characterized, the hydrogen isotopic fractionation between terrestrial n-alkanes and precipitation can be calculated in conjunction with δD_{wax} to reconstruct past annual rainfall fluctuations.

This work was supported by the National Basic Research Program of China (2009CB723901) and the Knowledge Innovation Program of the Chinese Academy of Sciences (KZCX2-YW-Q09-03 and KZCX2-YW-146).

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