



Relationships between Source Inputs and Lipid Geochemistry of Lake Sediments on the Northern Tibetan Plateau, China

Chenjun Wu, Jincai Tuo, Ru Chen, Mingfeng Zhang, and Yan Liu

Key Laboratory of Petroleum Resources, Northwest Institute of Eco-Environment and Resources, Chinese Academy of Sciences, Lanzhou, Gansu, P. R. China

ABSTRACT

Surface sediment samples were collected along the margins of Lakes Dachaidan, Xiaochaidan, Gahai (Delinha), Chaka, Gahai, and Qinghai, and from the Jinyingtan peat deposit, to study the lipid geochemistry of sediments on the Tibetan Plateau, China. TOC contents of the lake sediments varied in different saline lakes, with higher values in Lakes Qinghai and Gahai, which may be related to low salinity and abundant aquatic organisms. Fatty acids and *n*-alkanes were the two most abundant lipid constituents. Fatty acids were dominated by short-chain, indicating they were mainly from aquatic organisms, algae and bacteria organic matter. *n*-Alkanes were dominated by mid-chain and long-chain *n*-alkanes, and were derived mainly from aquatic macrophytes and terrestrial plants. The carbon isotopic compositions of short-chain, branched, unsaturated and cyclic fatty acids, and short-chain *n*-alkanes show an increasing trend in lake sediment samples from less saline lakes to more saline lakes. In less saline lakes, $\delta^{13}\text{C}$ values of algae-sourced compounds show relatively enriched ^{12}C , because of higher primary production and CO_2 assimilation. The $\delta^{13}\text{C}$ values of long-chain fatty acids and mid-chain/long-chain *n*-alkanes are positively correlated and show no obvious change from west to east, indicating similar macrophytes and terrestrial higher plants input in the studied samples.

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Introduction

Many organic geochemical studies in paleolimnology are carried out to determine the sources and relative proportions of organic matter (OM) types in the sediment (Volkman et al. 2008). Tracking OM sources in soils and sediments are important for understanding OM cycling in aquatic environments (Berner 1989; Hedges et al. 1997). Lake sediments are important archives of past environmental changes. These deposits contain organic and inorganic material of autochthonous and allochthonous origin (Pearson et al. 2007). Lake sediments typically accumulate at a rate of 1–10 mm/a (Smol et al. 2001a, 2001b) and can provide high-resolution records of local and regional climate and environmental changes. Lake sediments contain diverse lipid compounds derived primarily from organisms that live in and around the lake. Hence, differences in lipid composition among lakes reflect differences in lake and catchment biota (Pearson et al. 2007). Recently, new lipid compounds have been discovered in marine and lacustrine sediments, and terrestrial higher plants and microalgae have been identified as sources for these lipids (Sikes et al. 2009; Volkman et al. 1998, 2008). Some compounds degrade slowly or are transformed into more stable chemical structures and can be used as biomarkers to investigate sources of organic matter in the sediment (Sauer et al. 2001; Schouten et al. 2003; Volkman et al. 1998).

The lipids biomarkers in sediments such as *n*-alkanes, phospholipids fatty acids, and *n*-alkan-2-one correspond to certain types of organisms. The distribution patterns of the lipids biomarkers can reflect their biological source, according to different chain length lipid biomarkers derived from different biological sources. Compared with chain length signatures of the lipids biomarkers, specific carbon isotopic composition signature can be used to better distinguish among organic sources, providing unique insights that may not be revealed when using biomarkers (Fang et al. 2014; Sikes et al. 2009). The specific carbon isotopic composition of fatty acids and *n*-alkanes can be used to identify the different primary producers, and enable reconstruction of the local vegetation history and past aquatic environmental conditions (Ficken et al. 2000, 2002; Fuhrmann et al. 2003; Huang et al. 1999; Sikes et al. 2009; Volkman et al. 2008; Zhang et al. 2004). Studies that use multiple biogeochemical techniques, such as biomarkers in combination with carbon isotope analyses, have been able to elucidate the sources of organic matter in sediments. Whereas measurement of bulk organic matter variables can be equivocal with respect to OM source, use of multiple compound-specific measures has proven to be a powerful tool for identifying such sources (Drenzek et al. 2007; Goñi et al. 1997; Gordon and Goñi 2003; Hedges et al. 1997; Kao et al. 2006; Mead et al. 2005;

Naraoka and Ishiwatari 1999; Pearson and Eglinton 2000; Sikes et al. 2009).

The Tibetan Plateau is >3000 m above sea level and represents one of the most unique tectonic features on earth, attracting wide interest in paleoenvironmental studies. The water budgets of most lakes in this elevated region feature higher levels of evaporation than water input, and thus, these lakes become increasingly saline over time (Tuo et al. 2005). Such hydrological changes are expected to impact microbial and biogeochemical processes in these lake environments. Vegetation is predominantly C_3 plants, varying from swampy meadows to mountain shrubs (Lu et al. 2001; Xu et al. 2006). Lipid biomarkers have been successfully used to provide information about sources, preservation, and historical changes in organic matter of lake sediments from the Tibetan Plateau (He et al. 2014; Pu et al. 2013; Tuo et al. 2011; Wang et al. 2012). Duan et al. (2011, 2016) studied the potential biologic sources of sedimentary *n*-alkanes based on the distributions and hydrogen isotopic compositions of *n*-alkanes in surface sediments from Lake Qinghai and Lake Gahai on the northern Tibetan Plateau. Carbon isotope compositions of *n*-alkanes of lake sediments from the Tibetan Plateau have been previously studied to interpret sedimentary OM sources and environmental conditions (Aichner, Herzschuh, et al. 2010; Aichner, Wilkes, et al. 2010). Source characterization of sedimentary organic matter using stable carbon isotopic composition of *n*-alkanes and fatty acids has been reported in freshwater Lake Dianchi, China (Fang et al. 2014). Liu et al. (2011) studied salinity control on long-chain alkenone distributions in lake surface waters and sediments of the northern Qinghai–Tibetan Plateau. However, the application of carbon isotope compositions of fatty acid for OM source identification in saline lake sediments of the Plateau has relatively seldom been reported.

Saline lakes on the northern Tibetan Plateau are representative zones of peat development areas with different sedimentary environments, salinities, and types of sediments. The saline lakes generally have shallow water depths, similar temperatures, but large salinity differences. Lakes can respond quickly to environmental perturbations with more pronounced geochemical signals due to the smaller size of the reservoir. Lipids biomarkers distribution and carbon isotope compositions of lake sediments are sensitive to aquatic environments. In order to study OM sources and salinity control on lipids geochemistry characteristics of lake sediments, recent surface sediment samples were collected from multiple saline lakes on the northern Tibetan Plateau. The main objectives of this study were: (1) to obtain detailed information about organic matter sources of lake sediments on the northern Tibetan Plateau by evaluating the distribution patterns of the lipids biomarkers, including *n*-alkanes, fatty acids, and *n*-alkan-2-ones. (2) To evaluate the reliability of specific carbon isotopic composition of fatty acids and *n*-alkanes as potential proxies for reconstructing changes in organic matter sources and for future palaeolimnological investigation on the sediment cores in this climate-sensitive region.

Study site

The Qinghai Lakes (Lake Qinghai and its surrounding satellite lakes) have abundant geochemical features because their salinities range from freshwater to brackish water (Li et al. 2006). Lake Qinghai is in the northeastern part of the Qinghai-Xizang (Tibet) Plateau and it is a typical continental brackish-water lake. To the northeast and northwest of Lake Qinghai, a few small satellite lakes with varying salinities have formed because the lake level has decreased in recent centuries (Figure 1). Most of the satellite lakes are more brackish than Lake Qinghai because of their very limited freshwater inputs. Lake Qinghai is a 26.5 m deep perennial lake with an area of 4400 km² and an elevation of 3198 m above sea level. The lake water is brackish and alkaline with a salinity of 14 g/l, and it is rich in sodium sulfate. Lake Dachaidan, ~400 km northwest of Lake Qinghai, has a salinity of 275 g/l and is rich in magnesium sulfate. Lake Xiaochaidan, ~30 km southeast of Lake Dachaidan, has a salinity of 333 g/l and is rich in magnesium sodium sulfate. The salinity of Lake Gahai (Delinha), 91 g/l, is intermediate between those of Lake Xiaochaidan (333 g/l) and Lake Qinghai (15 g/l) and is rich in magnesium-sodium sulfate. Lake Chaka, located ~100 km west of Lake Qinghai, is the most saline satellite lake around Lake Qinghai, with a salinity of 324 g/l and is rich in magnesium sulfate. The satellite Lake Gahai is a small independent lake northeast of Lake Qinghai and has a salinity of 33 g/l and an abundance of sodium-sulfate (Table 1).

Materials and methods

Sampling information

To obtain a representative lipids biomarkers and carbon isotope composition record of different depositional lake environments on the northern Tibetan Plateau, 25 surface sediment samples, including mixed samples of vegetation and algae from lakeshore surface and mud sediments, were collected along the margin of Lakes Dachaidan, Xiaochaidan, Gahai (Delinha), Chaka, Gahai and Qinghai and from the Jinyingtan peat deposit (Figure 1; Table 2). Samples were stored in sealed clean glass bottles after collected and kept frozen at −25 °C until the analysis. Lake sediments samples were characterized into following several types according to properties: mixed samples of vegetation and algae from lakeshore surface (chl), root soil mud (soil mud containing grassroots, rM), surface mud (mud collected from lakeshore surface, fM), bottom mud (mud collected with water depth about 30 cm, bM), and peat (P).

Geochemical analysis

All samples were freeze-dried and extracted by a modified Bligh-Dyer procedure. Briefly, 1–5 g (depending on the TOC content in the samples) of dried sediment samples was suspended in a methanol-dichloromethane-phosphate buffer, treated for 2–3 min with an ultrasonic probe, and then kept overnight at room temperature. The samples were

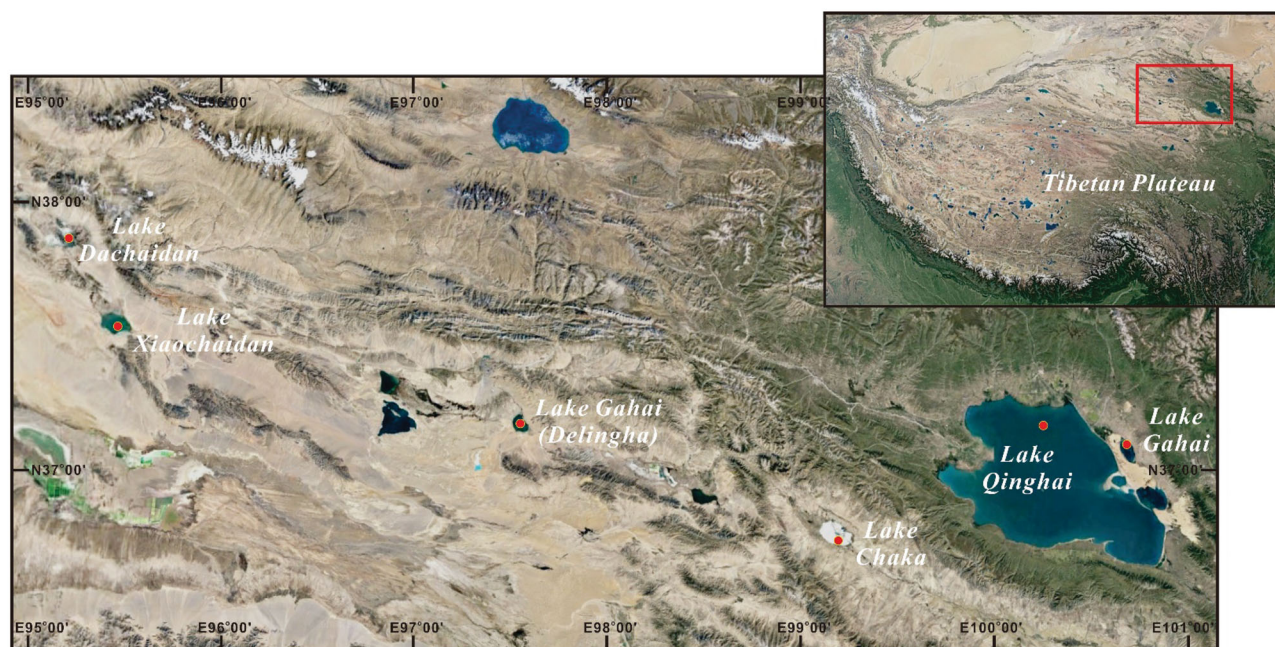


Figure 1. Map showing the location of sampling and studied salt lakes (Lake Qinghai and its surrounding satellite lakes) on the northern Tibet Plateau.

Table 1. Water chemistry of studied salt lakes.

Sampling location	Altitude (m)	pH	Salinity (g/l)	Chemical compositions (g/l)					
				Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	Cl ⁻	SO ₄ ²⁻
Lake Dachaidan	3153	7.3	275	88.39	3.22	9.7	0.453	155.89	16.57
Lake Xiaochaidan	3208	7.9	333	104.91	3.47	0.43	12.91	181.22	29.55
Lake Gahai (Delingha)	2852	8.3	91	27.33	0.25	0.31	4.18	45.26	13.09
Lake Chaka	3065	7.3	324	80.23	4.47	0.12	26.51	187.71	23.63
Lake Qinghai	3198	9.2	14	3.95	0.15	0.85	0.01	5.99	2.33
Lake Gahai	3225	9.1	33	9.68	0.45	0.01	1.38	13.69	6.19

centrifuged to separate the phases, and the dichloromethane phase was dried. This total lipid fraction was fractionated by column chromatography on silicic acid and by sequential elution with dichloromethane, acetone, and methanol, which resulted in three fractions with different polarities: neutral lipids, glycolipids, and phospholipids. The phospholipid fractions were separately dissolved in 1 ml of dichloromethane-methanol and subjected to mild-alkali hydrolysis (1 M KOH-methanol); thus, the free fatty acids were methylated to fatty acid methyl esters.

The contents and distribution patterns of lipid biomarkers and their individual carbon isotopic compositions were determined by gas chromatography-mass spectrometry (GC-MS) and gas chromatography-isotope ratio mass spectrometry (GC-IRMS) to identify the variations in the sources of the OM deposited in lake sediments and alpine meadow soil. The standard C₂₄D₅₀ was used as an internal reference for comparative purposes. For each analysis, the same weight of the sample was dissolved in the same volume of solvent and the same amount of internal standard was added. GC-MS analyses of the neutral and alkali hydrolyzed phospholipid fractions were performed on a Hewlett Packard 6890 N gas chromatograph connected to a Hewlett Packard 5973 N mass spectrometer. The gas chromatograph was equipped with a DB-5 MS fused silica capillary column (30 m × 0.25 mm), and helium with a flow rate of 1 ml/min

was used as the carrier gas. The mass spectrometer was operated with an electron energy of 70 eV, and the ion source temperature was 230 °C. The oven temperature was held for 1 min at 80 °C, increased from 80 to 280 °C at 3 °C/min and then was held for 20 min at 280 °C. The GC-MS data were acquired and processed with a Hewlett Packard Chemstation data system.

The carbon isotope analyses of individual compounds were performed on a Delta Plus XP gas chromatography combustion isotope ratio mass spectrometer. The gas chromatography was performed using a Thermo Finnigan GC COMBUSTION III system equipped with a DB-5 fused silica capillary column (30 m × 0.25 mm), and helium with a flow rate of 1 ml/min was used as the carrier gas. The oven temperature was held for 1 min at 80 °C, increased from 80 to 280 °C at 3 °C/min, and held for 20 min at 280 °C. Isotopic values were calculated by integrating the *m/z* 44, 45, and 46 ion currents of the chromatographically separated compound combustion peaks and those of the CO₂ standard spikes produced at regular intervals. The reproducibility and accuracy of the analysis were evaluated routinely using laboratory standards of known $\delta^{13}\text{C}$ values (C₁₆–C₃₂ *n*-alkanes). Typically, one injection of a laboratory standard was performed for every eight sample analyses. The isotope values are reported relative to the Pee Dee Belemnite (PDB)

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