



## Research paper

## Clay minerals and isotopes of Pleistocene lacustrine sediments from the western Qaidam Basin, NE Tibetan Plateau

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## ABSTRACT

Since Singer (1984) reviewed clay minerals as they relate to paleoclimates, clay minerals have been used to reconstruct paleoclimates and environments. However, the paleoclimate information recorded by the paleo-clays in sediments will be overprinted or changed if the clay minerals undergo diagenetic transformations or post-depositional diagenetic changes, which are quite common. However, numerous studies still use clay minerals to reconstruct paleoclimates and paleo-environments. Uplift of the Tibetan Plateau since the Late Miocene has significantly impacted the sediments of the Qaidam Basin. However, due to the scarcity of fully dated sediment records, we know little about whether or not and/or how these clay minerals responded to this uplift. In order to understand their response to the uplift, in this study, we present the results of high-resolution profiling of the clay minerals from a 938.5-m borehole core (SG-1) from the Qaidam Basin (NE Tibetan Plateau). These clay minerals are mainly composed of illite, mixed layers of illite/smectite (I/Sm) and chlorite, with minor kaolinite and smectite. The clay minerals may have undergone early diagenesis. The  $\delta^{18}\text{O}$ - $\delta\text{D}$  values of the interlayer water and the clay minerals imply the following. (a) The primary amounts of illite and chlorite were lower than those observed, while the primary amounts of smectite and kaolinite were higher than those observed; the amounts of clay minerals should be careful to be used to reconstruct paleo-environment singly; the changes in the illite “crystallinity” (Kübler Index) and chlorite “crystallinity” (Árkai Index) were related to changes in salinity and temperature. (b) Chemical weathering from 2.77 to 0.1 Ma was stronger than that at present. (c) The  $\delta^{18}\text{O}$  and  $\delta\text{D}$  of the interlayer water ranged from  $-11.8$  to  $82.95\%$  and from  $-114.3$  to  $165.5\%$ , respectively, which was related to pore water and/or lake water undergoing strong evaporation. The isotopic composition of the interlayer water reflects variations in the pore water/lake water or of the reacting solutions, and records changes in environment. (d) The clay minerals recorded climate changes and uplift in the core, with a dramatic shift at 1.2 Ma and two different environment stages at 2.77–1.2 Ma and 1.2–0.1 Ma.

## 1. Introduction

Since Singer (1984) reviewed clay minerals on paleoclimate, clay minerals have been used to reconstruct paleoclimates and paleo-environments (e.g., Singer, 1984; Chamley, 1989; Weaver, 1989; Thiry, 2000; Inoue et al., 2004; Wang and Yang, 2013; Middleton Alexander et al., 2015; Kwak et al., 2016). In previous studies, clay minerals in

lake sediments were of detrital origin and were the by-products of the physical and chemical weathering of rocks, e.g., clay minerals are the typical weathering products of silicate minerals. Chlorite and illite form when physical weathering occurs, and kaolinite and smectite form when chemical weathering predominates. However, the paleoclimate information recorded by the paleo-clays in sediments can be overprinted or changed if the clay minerals are diagenetically transformed.

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Post-depositional diagenetic changes, which alter the original composition of the clay-mineral assemblages, are quite common. Clay minerals often form through the alteration of preexisting minerals, including other clay minerals, in rocks or sediments that are in contact with water, air, or steam, or they can crystallize directly from a solution (Galan, 2006). The likelihood of diagenetic changes occurring increases with increasing burial depth of the sediment. The geochemical environment of clay formation may exert a strong control on the “crystallinity” and degree of order, thermal stability, morphology, and surface characteristics of the clay minerals (Zhou and Keeling, 2013).

For example, temperature is believed to be the most important parameter affecting the illite “crystallinity” (Kübler Index-KI), i.e. the KI value decreases with increasing temperature during sedimentary burial or tectonic overburden (Potel et al., 2016). Many extra-climatic factors affect the stability of the clay minerals, the most important of these being topography, parent-material, lithology, and time (Singer, 1984; Thiry, 2000; Allard and Calas, 2009). Therefore, the relationship between the climate parameters (expressed by the weathering intensity) and clay-mineral formation is not always straightforward.

Although diagenetic changes will overprint the paleoclimate information recorded by the clay minerals, not all of the primary information will be lost. Some information is preserved, e.g., not all of the smectite is transformed into a mixed illite/smectite layer (I/Sm, a thermodynamically metastable intermediate product between smectite and illite) and illite. The crystalline geochemical properties of the clay minerals are very dependent on the physicochemical conditions of the environment they formed in and their post-depositional environment (Singer, 1984; Rusmin et al., 2016). The degree of alteration of the primary minerals and the composition of the secondary products are clearly linked to climate. However, the diagenetic record of the interlayer water in the clay minerals can be altered by exchange between the interlayer water and the ambient water. For example, the hydrogen and oxygen isotopes of the different clays and interlayer water record temperatures ranging from surface temperatures to hydrothermal temperatures of approximately 150 °C–200 °C (e.g., Savin and Lee, 1988). In situ precipitation of I/Sm minerals may take place in chemical and isotopic equilibrium with the reacting solution (Essene and Peacor, 1995; Inoue et al., 2004; Sánchez-Roa et al., 2016). In summary, it is recommendable to use a multi-proxies of clay-minerals for deciphering environmental information.

The Qaidam Basin (Fig. 1), an Asian inland drought basin, was formed as the result of intracontinental deformation and plateau uplift due to the collision of the Indian and Eurasian Plates. Uplift of the Tibetan Plateau since the Late Miocene has had a significant impact on the sediments in the Qaidam Basin. However, due to the scarcity of fully dated sediment records, we know little about whether or not and/or how these clay minerals responded to this uplift and the dry climate. In order to understand their response, in this study, we present the results from high-resolution profiling of the clay minerals from a 938.5-m borehole core (SG-1) from the Qaidam Basin (NE Tibetan Plateau).

In this paper, based on detailed mineralogical and oxygen–hydrogen isotopic analyses of clay minerals from core SG-1, our goal is to identify the following: (a) the primary information recorded by the clay minerals from 2.77 Ma to 0.1 Ma; and (b) the response of the clay minerals to uplift or tectonic activity.

## 2. Geological setting

The Qaidam Basin is a Mesozoic–Cenozoic sedimentary basin located on the northern margin of the Tibetan Plateau in China (Fig. 1). This hyper-arid basin is bordered by the Kunlun Mountains to the south, the Qilian Mountains to the northeast, and the Arjin Mountains to the northwest, which have altitudes ranging from 4000 to 4500 m to over 5000 m asl (Fang et al., 2007). A network of faults exists within the basin (Fang et al., 2007). The Paleogene and Neogene strata are widespread and include intrusive rocks. The exposed rocks in the

drainage basin are variable and contain many intermediate and silicic rocks. The outcrops are mainly grey gneiss, siliciclastic rocks, dolostone, quartzite, phyllite, marble, carbonate, peridotite, serpentinite, augite peridotite, diorite, granite, etc. (Xuan, 1995; Wang et al., 2008). From the Middle Oligocene to the Upper Pliocene, the strata consists of mudstones, calcareous mudstones and marls, intercalating siltstone, very thick gypsum, and rock salt beds in the western basin (Tuo and Philp, 2003). Some secondary or subsidiary basins have become well developed since the Middle Pleistocene as a result of slow uplift (Chen and Bowler, 1986). The lake characteristics of these sub-basins are dramatically different due to their various geological structures.

The core used in this study (38°24′35.3″ N, 92°30′32.7″ E, 2900 m asl) was taken from a playa in the Qahansilatu sub-basin, which is located on the western margin of the Qaidam Basin (Fig. 1). Magnetostatigraphic and optically stimulated luminescence (OSL) results date the entire core (SG-1) at ~2.77–0.1 Ma and show a sedimentary accumulation rate (SAR) of 26.1–51.5 cm/ka (Zhang et al., 2012). The sedimentary sequence is composed of clay, clay-silt, and siltstone intercalated with salt layers (mainly halite), marl beds, and thin and/or scattered gypsum crystals, indicating clay-silt and halite-marl depositional cycles (Wang et al., 2012; Zhang et al., 2012). The fluctuation between evaporate minerals and carbonaceous clay strata indicates a shift between dry and wet climates. Gypsum and halite make up the majority of the evaporate minerals in the core (Li et al., 2010, 2013). Based on sedimentary textures, structures, and diagnostic features, Wang et al. (2012) identified five lithofacies and divided the stratigraphic sequence of the core into nine phases, which exhibit a gradual transition of depositional environments in the last 2.8 Ma: semi-deep fresh lake to semi-brackish lake → shallow brackish lake → perennial saline lake → playa saline lake → saline mudflat. The sedimentary features (Wang et al., 2012), salt minerals (Li et al., 2010, 2013), isotope records (Han et al., 2014; Li et al., 2017) and rare earth elements (Yang et al., 2014) of the study area indicate the long-term persistent aridification of inland Asia since 2.8 Ma.

## 3. Materials and methods

The mineral composition of the Core was examined at 1 m intervals using an X-ray diffractometer (XRD) (Rigaku D/MAX-2000: Cu, Ka1, 1.5406 Å, 40 kV, 40 mA). We used well known accepted methods to analyze the clay mineral. The samples, taken at 1 m intervals, were treated with ultrapure water then diluted with 0.01 mol/L EDTA (Ethylene Diamine Tetraacetic Acid) to remove any soluble ions and calcium carbonate, and organic matter. The clay mineral fraction (< 2 μm) was then separated using a centrifuge. For each sample, this fraction was then scanned sequentially for three distinct XRD patterns, i.e., in natural (air-dried) conditions (N), after saturation with ethylene-glycol for 24 h in desiccators (EG), and after being heated in a muffle furnace at 490 °C for 2 h (H). Diffraction patterns (2θ) were scanned from 3° to 30° with a 0.07° interval. Minerals were identified and semi-quantified based on their (001) reflections. Generally, clay minerals were identified based on their basal reflections at ~17 Å (smectite); 10 Å and 5 Å (illite); 14.2 Å, 7 Å, 4.72 Å, and 3.54 Å (chlorite); and 7 Å and 3.57 Å (kaolinite) (Biscaye, 1964, 1965; Hillenbrand et al., 2009). After being saturated with ethylene-glycol and being heated, some of the overlapping reflections were identified. For example, the 10 Å reflection of illite and the 14 Å reflection of chlorite do not change after saturation with ethylene-glycol, while the 14 Å reflection of smectite shifts to 17 Å.

The illite chemistry is inferred from the position of the 10 Å and the 5 Å/10 Å intensity ratio (Esquevin, 1969; Ehrmann et al., 2005; Zeng et al., 2014). According to Esquevin (1969),  $I_{5\text{Å}}/I_{10\text{Å}}$  ratios > 0.5 correspond to Al-rich illites. The ratio decreases with Mg and Fe substituting the octahedral Al. The higher the ratios, the higher the amount of Al. The  $I_{5\text{Å}}/I_{10\text{Å}}$  ratios of Mg-Fe-rich illites therefore are < 0.5. Similarly, the  $I_{7\text{Å}}/I_{14\text{Å}}$  ratios of chlorite > 4 correspond to Fe-chlorite,

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