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RESEARCH ARTICLE

Reliability of radiocarbon dating on various fractions of loess-soil sequence for Dadiwan section in the western Chinese Loess Plateau

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Abstract The accurate radiocarbon dating of loess-soil sequences plays an essential role in the reconstruction of the environmental and climatic changes in continental settings during the last glaciation and Holocene. However, our knowledge about the reliability of radiocarbon ages of various fractions of soil and loess samples is still insufficient. Here, we present our study results on radiocarbon ages based on bulk organic matter, humin fraction, and carbonate of samples collected from a loess-paleosol section in the western Chinese Loess Plateau. We compare these observations with the optically stimulated luminescence ages and charcoal radiocarbon ages to evaluate the reliability of these fractions. We observed that the radiocarbon ages of humin fraction are very close to those of charcoal and are consistent with the optically stimulated luminescence ages within the experimental errors. We observed a significant deviation in the radiocarbon ages of carbonate and bulk organic matter from those of charcoal and optically stimulated luminescence ages, likely due to the dilution of these fractions during the pedogenetic process. Our results reveal that, except for charcoal, the humin fraction may yield reliable ^{14}C ages for the Chinese loess-soil sequence.

Keywords loess-soil sequence, humin fraction, charcoal, organic matter, radiocarbon dating

1 Introduction

Due to its high accumulation rates, the Chinese loess-paleosol (CLP) sequence appears to be one of the most suitable archives for the reconstruction of Cenozoic continental paleoclimate (Liu, 1985; Ding et al., 1999; Guo et al., 2002). Radiocarbon dating is a useful method that has been widely used to provide a chronological framework for Late Pleistocene–Holocene paleoenvironmental studies (Hatte et al., 2001; Pessenda et al., 2001). However, radiocarbon dating of soils and sediments is also known to be problematic due to the presence of pre-aged carbon (Dodson and Zhou, 2000; Pessenda et al., 2001). Accurate chronologies cannot be easily obtained through radiocarbon dating due to the lack of plant remains and charcoals, which are exceptional materials for reliable ^{14}C dating in loess deposits (Hatte et al., 2001). There is no common agreement on usage of sedimentary fraction or pre-treatment method is best to accurately yield radiocarbon ages in closest agreement with that of plant macrofossils or charcoal.

Several researchers have shown that organic matter in loess is a naturally heterogeneous mixture of multiple fractions, such as folic acid, humic acid, and humin (Dodson and Zhou, 2000; Turney et al., 2000). These fractions have different radiocarbon activities and residence time, which in turn may produce an apparent age that deviates from the actual age of the loess profiles. Therefore, it is generally accepted that these materials should only be dated for chronological purposes in the absence of reliable materials, such as charcoal, wood, or other plant macrofossils (Muhs et al., 2003). In this study, we attempt to examine the applicability of carbonate, bulk organic, and humin fraction for radiocarbon dating through detailed chronological work on a typical loess profile at Dadiwan. The optically stimulated luminescence (OSL) data from the same profile were also used for comparisons.

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2 Stratigraphy

The site studied in this work is located at Dadiwan in the western Chinese Loess Plateau (Fig. 1). It is a well-known Neolithic site characterized by the complex of the Dadiwan (or Laoguantai) cultures (7,800–4,900 yr BP) and the presence of early millet agriculture in China's westernmost interior (Barton et al., 2009; Bettinger et al., 2010; Zhang et al., 2010). The section we sampled is located in Shaodian Village, Qin'an County, Gansu Province, China (35°00′47.3″N, 105°54′53.3″E), which has been reported by Zhang et al. (2010). A geological survey showed that the Malan Loess in this region could reach as deep as 13 m (Zhang et al., 2010). This is a typical loess-paleosol section of the Late Pleistocene and Holocene. The section is composed of early Malan Loess (L1L2), intercalated paleosol (L1S1), late Malan Loess (L1L1), and Holocene soil (S0). This stratigraphic sequence is comparable to other sections in the Chinese Loess Plateau; for example, Yuanbao section in the western part of the CLP (Chen et al., 1997) and Luochuan section in the eastern part of the CLP (Liu, 1985). A detailed lithology of this section is shown in Fig. 2.

The stratigraphy of the uppermost 8.5 m section is described below based on field observations and also evidence from environmental proxies:

0.0–0.8 m: Top soil and A horizon; heavily disturbed by human activities, primarily from modern agricultural activity.

0.8–1.8 m: The top 0.5 m was observed as A/B horizon with abundant krotovina, shown as dark grey, with minimal Holocene loess soil development. The bottom was observed as Bk horizon of a moderately developed friable, tan clayey loess soil and high $CaCO_3$ deposition.

1.8–3.5 m: C horizon of a weakly developed soil on top with some krotovina, and loosely compacted, slightly weathered loess. Massive structure, yellowish loess in the middle section and slight weathered loess with weak soil development at the bottom. This unit was labeled as L1L1 (Fig. 2).

3.5–7.1 m: A moderately developed "up-building'' loessic soil unit (L1S1). The color and structure changed with depth, but is difficult to recognize in the field. Some parts were observed as white carbonate deposit and coarse grain size, whereas others showed minimal strong soil development.

7.1–8.5 m: Yellowish massive-structure loess unit (L1L2 in Fig. 2) with calcrete inclusions and small gravels, showing slight strong leaching of the above soil and fluvial influence.

3 Methods

3.1 Sampling

A 2 m × 3 m trench with a depth of 8.5 m was excavated for archeological survey (Zhang et al., 2010) and sampling. A total of 12 samples were collected at 50 cm intervals with 3 cm thickness along a vertical wall of a trench dug out for ^{14}C analyses. For each sample, up to 3 kg of loess material was taken from the cleaned surface of the section. For OSL dating, eight samples were collected by hammering steel tubes into each section at selected levels.

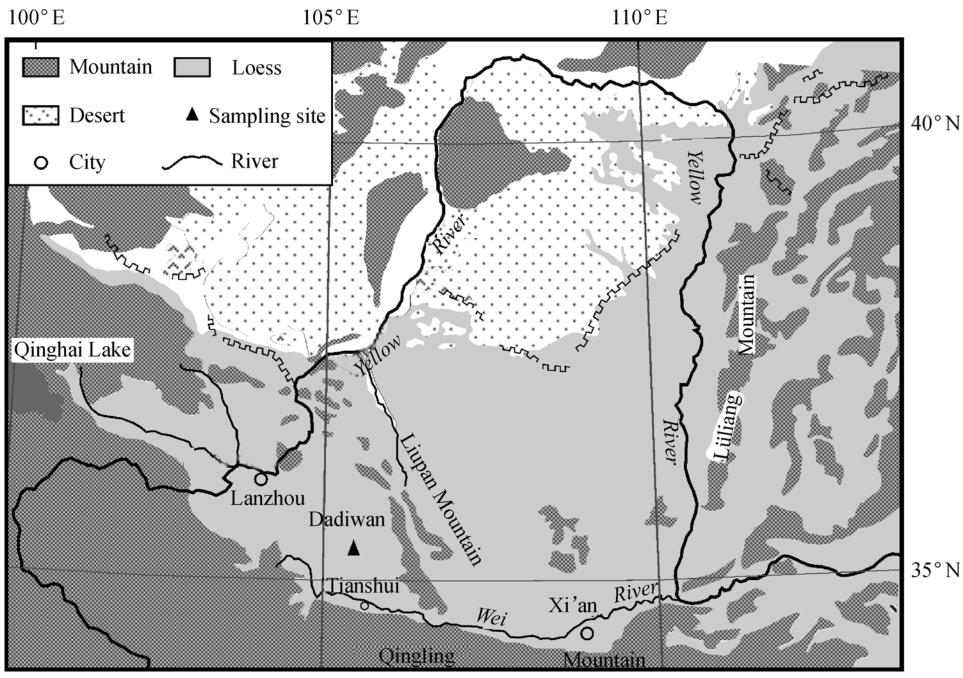

Fig. 1 Location of Dadiwan section in the western Chinese Loess Plateau.

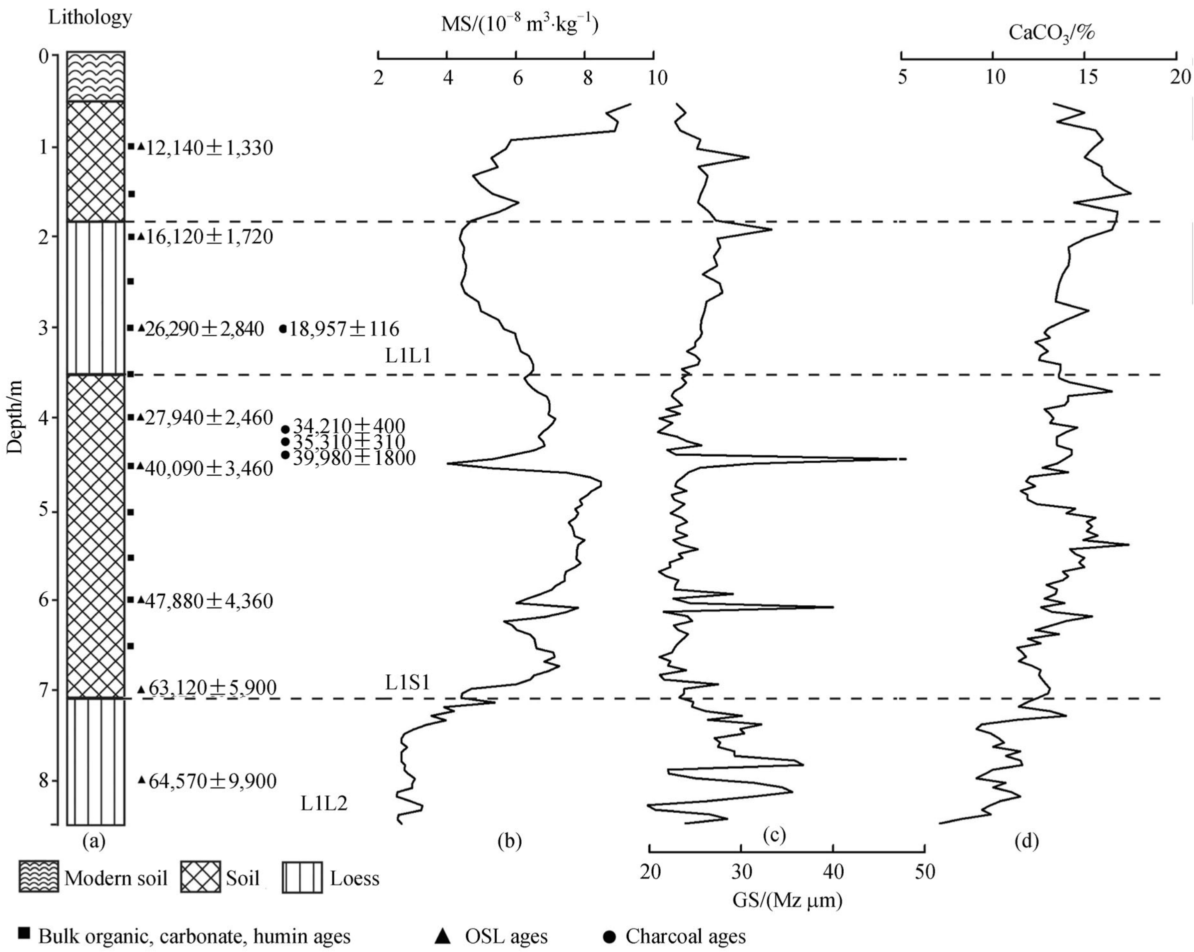

Fig. 2 Lithology of Dadiwan loess-paleosol sequence and variation of magnetic susceptibility, mean grain size and carbonate content with depth (after Zhang et al., 2010). Sample locations, OSL ages (kyr), and charcoal radiocarbon ages (cal BP) are marked beside the lithology. Holocene paleosol S0, loess units (L1L1, L1L2), and weak pedogenic unit (L1S1) of Malan loess are also indicated.

3.2 Radiocarbon analyses

All samples were dried thoroughly at room temperature and then sieved to remove visible plant remains, such as rootlets. The remaining tiny plant detritus were removed via flotation in deionized water. These samples were dried again at 60°C. Four different fractions—carbonate, soil organic matter (SOM), and humin fraction of samples—were used for ^{14}C dating in this study.

The carbonates of loess samples were extracted using 2 mol/L HCl for four hours at room temperature. The deliberated CO_2 gas was then absorbed using hartshorn and benzene. The remaining samples were then dried again at 60°C and combusted to extract SOM for dating.

The humin fraction of pretreated samples was extracted following the conventional acid-alkaline-acid method (Goh and Molloy, 1978; Pessenda et al., 1996; Forbs et al., 2004). To avoid the precipitation of humus products from the solution, a hydroxide treatment (with 0.5% NaOH solution) was applied to extract humus products after a thorough rinsing to remove Ca^{2+} ions after treatment with 2 mol/L HCl solution (Olsson, 2009). The insoluble fraction (humin fraction) of bulk samples was extracted and further treated with 70% HNO_3 solution (Goh and Molloy, 1978). The ^{14}C measurements were carried out at the MOE Key Laboratory of West China's Environmental System, Lanzhou University, using the conventional benzene method and liquid scintillation counting.

Hand-picked charcoals were collected from the same levels as the humic fraction at the sampling site and treated by the conventional acid-alkaline-acid method. The ^{14}C analyses were carried out at the Radiocarbon Laboratories of Lawrence-Livermore (USA) and Peking University (China). The results are representative of the mean age of the charcoal fragments in the layers.

3.3 Optically stimulated luminescence dating

The OSL technique was followed according to the method described in Zhao et al. (2007). All raw samples were treated sequentially with 10% HCl and 20% H_2O_2 solutions to remove carbonate and organic matter. The samples were then wet sieved to isolate particles ranging in size from 90 to 150 μm. This fraction was further separated through the use of heavy liquids with densities of 2.62 and 2.72 g/cm^3 to obtain quartz. After drying, the quartz grains were treated with 40% HF solution for 40 min to remove the outer layer of quartz irradiated with alpha particles and plagioclase remnants. The grains were then treated with 1 mol/L HCl solution for 10 min to remove fluorides created during the HF etching and rinsed with deionized water. The purified quartz grains were mounted on 10 mm diameter aluminum disks with silicone oil as aliquots for measurements. The OSL measurements were conducted in the Luminescence Laboratory of the Cold and Arid Environment and Engineering Research Institute using an automated RisøTL/OSL-DA-15 reader. The OSL signal was detected through two 3.0 mm thick Hoya U-340 filters. Laboratory irradiation was carried out using ^{90}Sr/^{90}Y beta sources mounted within the readers with a dose rate of 0.104 Gy/s. The U, Th, and K contents were determined via neutron activation analysis (NAA) to calculate the dose rate. The dose rate from cosmic rays was calculated on the basis of sample burial depth and the altitude of the section. Water content was calculated as the ratio of wet to dry weights determined from sample weights before and after drying in an oven at 60 °C.

3.4 Environmental proxies

Mass specific magnetic susceptibility, grain size, and carbonate content were used as environmental proxies, which were examined at the MOE Key Laboratory of West China's Environmental System, Lanzhou University. Samples for grain-size analyses were pretreated according to the method proposed by Lu and An (1998). Grain size was measured with a Malven Mastersizer 2000 laser grain-size analyzer in the measurement range of 0.02 to 2,000 μm and 100 size intervals. The concentration of carbonate was measured using the "gas balance method" (Yu, 2007). Mass specific magnetic susceptibility was measured with a Bartington MS2 instrument with a 0.1 range and a dual-frequency (470 and 4,700 Hz) sensor, as described in Chen et al. (1997, 1999).

4 Results and discussion

The resulting ^{14}C and OSL age observation data are listed in Table 1 and shown in Fig. 3. Radiocarbon age results on charcoal from Holocene paleosol during the Neolithic period have been reported by Zhang et al. (2010). These dates are not discussed here due to the strong prehistoric human disturbance caused by agricultural development since ca 8,000 cal yr BP.

Table 1 Radiocarbon and OSL ages of the Dadiwan loess-paleosol sequence in the western Chinese Loess Plateau

Depth/m	Carbonate age/(cal yr BP)	Bulk organic age/(cal yr BP)	Humin fraction age/(cal yr BP)	Charcoal age/(cal yr BP)	OSL age*/(cal yr BP)
1.0	13,471±185	–	12,863±70	–	12,140±1,330
1.5	12,756±126	10,280±89	10,501±203	–	–
2.0	17,779±198	–	16,934±67	–	16,120±1,720
2.5	19,570±228	19,084±159	20,537±163	–	–
3.0	21,915±123	18,611±126	18,699±159	18,957±116 [a)]	26,290±2,840
3.5	22,154±149	–	28,616±419	–	–
4.0	24,424±167	–	31,986±216	–	27,940±2,460
4.2	–	–	–	34,210±400 [b)]	–
4.3	–	–	–	35,310±310 [b)]	–
4.4	–	–	–	39,980±1800 [b)]	–
4.5	25,511±176	33,798±224	39,900±378	–	40,090±3,460
5.0	25,213±162	32,210±218	–	–	–
5.5	26,557±188	34,672±233	–	–	–
6.0	25,520±192	33,701±256	–	–	47,880±4,360
6.5	26,797±201	35,120±242	–	–	–
7.0	–	–	–	–	63,120±5,900
7.5	–	–	–	–	–
8.0	–	–	–	–	64,570±9,900

Notes: The radiocarbon age is expressed as calendar year (cal yr BP), calculated using Calib 6.0.1 and the INTCAL09 tree-ring dataset (Reimer et al., 2009). Analytical uncertainties are reported as 1σ. The calendar dates were calculated. a) Analyses at Peking University (China). b) Analyses at Lawrence-Livermore (USA). *: data from Zhang et al. (2010).

Terrestrial plant macrofossils and charcoal are considered as the most reliable materials for ^{14}C dating (Törnqvist et al., 1992; Björck et al., 1998; Turney et al., 2000; Fontana, 2007) given biological inert and physically stable regarding isotopic changes in the sedimentary environment. Therefore, charcoal in loess stratigraphy is thought to be one of the most suitable materials for ^{14}C dating. Even if organic compounds are absorbed from the neighboring soil layers, an acid-alkaline-acid treatment can be used to remove any contaminations (Pessenda et al., 2001). The ^{14}C ages obtained from the humin fraction are in agreement with those from charcoal during the period of 18–40 cal kyr BP (Fig. 3). They are also in accord with the OSL ages within the analytical uncertainties for the same layer. Therefore, it is possible to construct a firm chronological framework for the loess-soil sequences through ^{14}C dating of the humin fraction of samples if other, more dependable datable materials are not available. We propose that the humin fraction of loess is a more reliable dating material than the SOM fraction. Our results support previous observations on radiocarbon dating of various soil profiles at other sites (e.g., Orlova and Panychev, 1993; Cook et al., 1998; Pessenda et al., 2001).

The results also indicate that both the OSL and ^{14}C age results obtained from various fractions are generally consistent between 1.0 and 2.5 m in depth along the section (Fig. 3). We observed that the ages of carbonate and bulk organic matter are systematically younger than those of charcoal, humin fraction, and the OSL ages below the depth of 3 m. It is noteworthy that the ages of carbonate are much younger than those of other fractions. Previous studies revealed that climatic conditions in the western part of the CLP and across North China were generally humid during MIS 3 (Pachur et al., 1995; Yang et al., 2004). Therefore, we inferred that the much younger ages of carbonate might have resulted from its strong eluviation and from the overlying soil in the pedogenic processes during the humid MIS 3 stage (59–29 cal kyr BP). In contrast, during cold and arid periods, steppe vegetation dominated the landscape, the accumulation rate of loess was relatively high, and the leaching process was weak (Hatte et al., 2001). Therefore, the ages of carbonate are only slightly older than those of SOM during the Last Glacial Maximum (LGM) and Early Holocene, corresponding to the depths of 1.0, 1.5, 2.0, and 2.5 m, whereas they are much older than those of SOM during the humid MIS 3 stage (Fig. 3).

Previous work has revealed that younger materials might have been incorporated into the samples through bioturbation, penetration, and subsequent decomposition of rootlets or translocation of soluble organic matter (Geyh et al., 1983; Wang et al., 1996; McGeehin et al., 2001; Mayer et al., 2008). This appears to be the case in our study. For example, the radiocarbon ages of carbonate tend to be much younger than those of other fractions, particularly during the humid MIS3 stage (Fig. 3). A closer look at the radiocarbon ages during this period reveals a systematic deviation of the ages of bulk organic matter and carbonate (Fig. 4). The radiocarbon ages of bulk organic matter are also much younger than the OSL ages (Fig. 3). It should be noted that most ages of L1S1 paleosol formation (ca. 28–60 cal kyr BP) were close to the upper limit of radiocarbon dating. This may be a result from two different. One is inevitable pollution of modern carbon during sample treatment in the laboratory for dating samples older than the radiocarbon limit, which was observed by Pigati et al. (2007). Another may be due to pedogenesis. During the paleosol formation, carbonate leached easier than SOM,

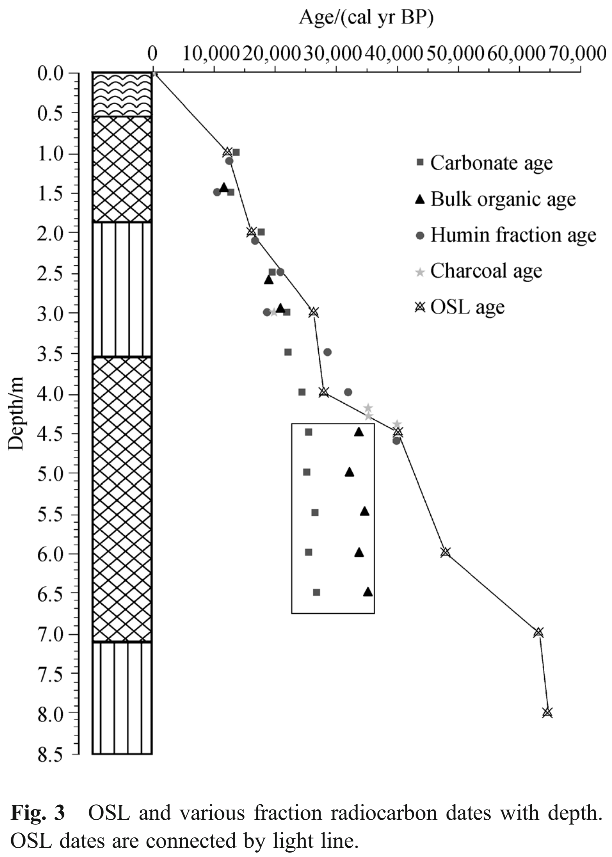

Fig. 3 OSL and various fraction radiocarbon dates with depth. OSL dates are connected by light line.

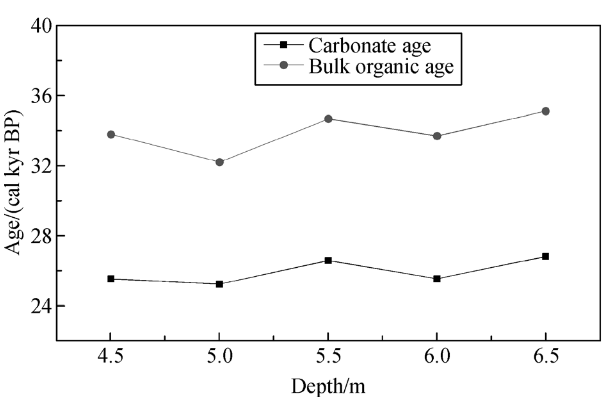

Fig. 4 Comparison of radiocarbon ages on bulk organic matter and carbonate.

although both SOM and carbonate moved downward. This observation can reasonably explain why radiocarbon age of SOM was older than that of carbonate, but both were much younger than OSL date. Therefore, we propose that radiocarbon age should typically be younger than soil formation age because of the rejuvenescence of SOM due to its eluviation and redeposition in the upper layers. Thus, great care should be taken when the stable isotopic composition of bulk organic carbon is used to reconstruct palaeoenvironment.

5 Conclusions

Radiocarbon dating on various fractions in conjunction with OSL dating of a loess-soil sequence in the western Chinese Loess Plateau reveals the promise of humin fraction as a reliable material towards an accurate chronology in this region. Specifically, the consistency of radiocarbon ages on humin fraction with those on charcoal and OSL ages suggests that the humin fraction is one of the most useful materials for building up a relatively reliable radiocarbon chronology for the loess-soil sequences. Further improvements in understanding the molecular structure and isotopic fractionation of this fraction may allow us to obtain more accurate chronological information. Our study also suggests that more attention should be paid when the stable isotopic composition of bulk organic matter is used to reconstruct past environmental changes.

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