Geochemical isotopic composition in the Loess Plateau and corresponding source analyses: A case study of China's Yangjuangou catchment

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A B S T R A C T
Isotopic fractionation technology is widely used in identifying sources and the speciation of geochemical isotopic elements. With the increase in human activity, geochemical element transport by soil erosion has become the most critical environmental problem in the Loess Plateau, so tracing the geochemical element source would help in the identification and management of local soil erosion. In this study, we investigated the spatial distribution of carbon (C), nitrogen (N), oxygen (O), and hydrogen (H) isotopes in water and then further analyzed 13C and 15N in soil and vegetation to better understand the C and N sources and their biogeochemical cycling function in the Loess Plateau. Results showed that mean dual isotopic values of δ13C-DIC in the Yangjuangou Catchment of China's Loess Plateau is mainly controlled by carbonate weathering or soil erosion. The severe erosion in this region has typically occurred in grassy (C4) land-use types devoid of woody vegetation (C3), and this has led to a discrepancy in δ13C between soil and water. We found δ18O and δD in water to be −7.87 ± 0.85‰ and −61.49 ± 3.25‰, respectively, and to show a high positive correlation (r² = 0.81). This suggests that summer rainstorms lead to soil erosion and runoff, which cause a wide range of isotopic values to occur across the Loess Plateau.

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1. Introduction

The Loess Plateau covers an area of 640,000 km². It is located in the upper and middle reaches of China’s Yellow River, where more than 60% of the land is susceptible to soil and water loss (Wang et al., 2014). Owing to this, nutrients are highly susceptible to transport by soil and water during rainfall events. This nutrient export is compounded by anthropogenic activity and industrial and agricultural emissions, which accelerate nutrient deposition and discharge into aquatic ecosystems. Yue et al. (2015), for example, reported significant amounts of carbon (C) and nitrogen (N) in water due to the excessive use of fertilizers from agricultural activities in southwest China. Therefore, identifying the C and N sources and understanding the biogeochemical cycling functions across the Loess Plateau is critical for improving local nutrient management practices as well as water quality.

During N biotransformation processes, biological discrimination between 14N and 15N, two stable isotopes, leads to natural isotopic fractionation. The lighter form of 14N is easily removed at higher rates than the heavier form of 15N, which leads to an increase in 15N concentration values for the remaining product (Miller et al., 2010). Thus, stable isotope ratios of Δ14N—NO3/Δ15N—NO3 have been widely applied in aquatic ecosystems to identify different sources of NO3−, such as wastewater, N deposition, man-made fertilizers, and animal waste (Kendall, 1998; Masetti et al., 2008; Pennino et al., 2014). As it relates to river sediment, N biotransformation starts with N assimilation, followed by N reduction via denitrification. Under rainfall scenarios, 15N values in sediment in polluted areas would eventually be higher compared to unpolluted river areas. Thus, stable N isotope ratios (15N/14N) can be used as a reliable indicator of N sources. However, for watersheds, it is better to identify major N sources via river monitoring (Kendall et al., 2007; Mengis et al., 2001).

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Previous studies have demonstrated that stable isotopes ($\delta^{15}$N and $\delta^{18}$O) of nitrates can be successfully used to elucidate sources of NO$_3^-$ in water based on sources with distinct isotopic signatures (Liu et al., 2006; McMahon and Böhleke, 2006; Panno et al., 2006). Many comprehensive studies have used dual isotope analyses to assess sources and transformations of nitrates in groundwater and rivers (Heaton et al., 2012). Opposing isotope fractionation effects from variations in concentrations and isotopic composition make it possible to distinguish between sorption, nitrification, and denitrification, which are major processes that affect N distribution under field settings. Therefore, the combination of $\delta^{18}$O–NO$_3^-$ can be considered a more reliable indicator of denitrification processes (Kendall, 1998; Wassenaar et al., 2011).

Different mechanisms of C fixation result in different isotope fractionations. $\delta^{13}$C values obtained from plant organic matter reflect the pathways of C fixation in plants (Garcin et al., 2014), whereas $\delta^{13}$C with-in the dissolved inorganic carbon (DIC) reservoir is mainly dominated by exchanges with respiration of plants, atmospheric CO$_2$, the dissolution of carbonate rocks by chemical weathering, oxidation of organic matter, and biological activity in water itself (Brunet et al., 2005; Schulte et al., 2011). Therefore, investigations into C isotopes provide further information related to sources and the evolution of DIC. In addition, elements of $\delta^{18}$O and $\delta^2$D isotopes in stream and lake water are good indicators of sources and transport pathways of incoming precipitation (Wassenaar et al., 2011). $\delta^{18}$O and $\delta^2$D in soil water have also been used to indicate evaporation, infiltration, and mixing processes as well as to quantitatively estimate groundwater recharge and evaporation rates (Cheng and Liu, 2014; Liu, 2011; Liu et al., 2015). Additionally, the analysis of the isotopic composition of $\delta^{18}$O and $\delta^2$D in soil water has been widely used to characterize infiltration profiles and evaporation rates in arid and semi-arid regions (Schachtschneider and February, 2007; Wan and Liu, 2016).

In this study, we investigated spatial variations of geochemical isotopic characteristics in the Yangjuangou Catchment of the Loess Plateau, China. The aim of our study was to: (1) determine the isotopic characteristics of $\delta^{15}$N and $\delta^{18}$O for NO$_3^-$ and of $\delta^{18}$O, $\delta^2$D, and $\delta^{13}$C for DIC in runoff; (2) ascertain $\delta^{15}$N and $\delta^{13}$C for total carbon (TC) and total organic carbon (TOC) in soil and plants; and (3) provide a comprehensive understanding of the origin of the isotopic composition of C and N in the catchment area.

2. Materials and methods

2.1. Study area

Our study area, the Yangjuangou Catchment, is located in the middle region of the Loess Plateau in northern Shaanxi Province, China (36°42′ N, 109°31′ E) (Fig. 1a). This region is characterized by large topographic variations with loess hills and gully land-use types that have maximum altitudinal differences from hill top to gully bottom of 225 m and a gully density of 2.74 km$^2$ (Wang et al., 2010, 2011). The climate is a semi-arid continental type with an average annual rainfall of 535 mm that mainly occurs from July to September and exhibits high inter-annual variation. The soil type is Loess, which is a fine silt to silt in and weakly resistant to erosion. Cultivation in the study area has eradicated natural vegetation,
and after years of rehabilitation, many changes have taken place within the agricultural landscape (Wang et al., 2011).

Grass, shrub, forest, and farmland are the four main land-use types that characterize this watershed. The primary grass species are Artemisia Linn, Elymus repens, and small weeds, while the primary shrubs are Prunus armeniaca and Hippophae rhamnoides in mature forests and Robinia pseudoacacia in young forests. The mountains that surround the catchment act as its boundary. Owing to its natural topography and lower elevation, runoff from each tributary catchment can converge and export water from a single outlet. This makes the watershed a good representation of a closed system (Fig. 1b).

2.2. Sampling and analysis

We collected water, soil, and plant samples from July to October 2015. Twelve locations were selected as water sampling points based on the spatial distribution and land-use types in the catchment (Fig. 1c). Soil samples were collected at a 1-m depth from dam, forest, farm, and grassland land covers; mixed samples were also taken. We selected four typical species for vegetation isotopic analysis: Pterocarya stenoptera, Salix babylonica L., Stipa bungeana Trin., and Agropyron cristatum.

Sampled water was filtered through an organic microporous membrane with a pore size of 0.45 μm. We heated the filtered samples to 80 °C in water and maintained this temperature for 12 h to remove impurities. A segmented continuous flow analyzer (AA3; Germany) was used to measure total nitrogen (TN) and total organic carbon (TOC). Dissolved nitrate (NO3) and dissolved nitrate (NO2) were quantified using the denitrifier method with samples split into two groups depending on NO3, as described in detail by Proemse et al. (2012). Isotope ratios were calculated as δ15N values versus air (atmospheric N2) by comparing them with standards calibrated against International Atomic Energy Agency N-1 and N-2. We also analyzed δ18O/δ13C ratios using thermal conversion to CO gas at 1400 °C in a TC/EA high-temperature conversion elemental analyzer connected online to a Deltaplus XL mass spectrometer (Thermo Finnigan, Bremen, Germany).

We used 0.5% CO2/He or an H2/He gas mix to obtain oxygen (O) and hydrogen (H) isotope ratios, as described in detail by Weynell et al. (2016), and conducted O and H stable isotopic measurements on water samples using the Liquid Water Isotope Analyzer (Picarro, USA). δ13C-DIC was determined by injecting 0.2, 0.5, and 1.0 mL of sample solution into He-rinsed glass vials (Exetainer) (Spötl, 2005) and analyzing the δ13C-DIC with a Deltaplus XL mass spectrometer (Thermo Finnigan). For the isotopic composition of C and N of the collected vegetation, a ball mill was used to ground dry leaves, and then between 2 and 3 mg was weighed into tin capsules, following the methods by Lehmitz and Maranin (2016). δ13C and δ15N of the four species were determined using a mass spectrometer (Thermo Finnigan). Finally, we determined soil C and N content and the associated isotopic composition of C and N using an element analyzer and mass spectrometer (Thermo Fisher Scientific, Bremen, Germany).

C, N, H, and O isotope ratios were expressed as delta values as follows:

\[ \delta^y X = \left( \frac{R_{\text{sample}}}{R_{\text{ref std}}} - 1 \right) \times 1000, \]

where X represents the target isotope; y represents the amount of atoms; and R represents the ratio of heavy to light isotopes, e.g., 13C/12C.

3. Results

3.1. Variations in C and N concentrations in water

Concentrations of TC from different locations in the Yangjuangou Catchment ranged from 33 to 107 mg·L⁻¹, with a mean value of 82.3 ± 17.9 mg·L⁻¹ (Fig. 2a). The majority of these concentrations were above 70 mg·L⁻¹, the exception being that at sample point 11. Concentrations of TIC ranged from 5 to 93 mg·L⁻¹ with a mean value of 57.3 ± 33.5 mg·L⁻¹; TIC showed significant fluctuations and instability in the watershed. We detected relatively low TOC concentrations except at sample points 6 (74.4 mg·L⁻¹) and 7 (69.8 mg·L⁻¹); samples had a mean TOC value of 25.0 ± 25.7 mg·L⁻¹.

In contrast to C concentrations, variations in N concentrations showed a descending trend from sample point 1 to 12 (Fig. 2b). TN concentrations ranged from 2.2 to 22.9 mg·L⁻¹ with a mean value of 9.2 ± 7.9 mg·L⁻¹ but were only 2.2 and 2.9 mg·L⁻¹ at the outlet of the watershed. Similarly, DTN concentrations ranged from 0.5 to 16.3 mg·L⁻¹ (mean 6.9 ± 7.3 mg·L⁻¹).

3.2. Composition of isotopes in water

The isotopic composition of C, O, and H from the upstream point to the outlet of the catchment is shown in Fig. 3a. Dual isotopic values of δ13C ranged from −4.58‰ to −5.58‰, with a mean value of −5.36 ± 0.28‰ and with sample points 3 and 4 having the highest values. δ18O exhibited similar trends to δ13C and showed small fluctuations and a mean value of −8.78 ± 0.85‰. δ2H was higher midstream, and its mean dual isotopic value was −61.49 ± 3.25‰, ranging from −57.10‰ to −67.23‰. We detected distinct spatial distributions in δ15N-N2O and δ18O-NO3 values in the catchment, with δ15N-N2O having a greater variation relative to δ18O-NO3 (Fig. 3b). δ15N-N2O had a mean dual isotopic value of 11.44 ± 6.15‰, ranging from 0.22‰ to 20.52‰ with the lowest value at sample point 8 and the highest at sample point 2. Although δ18O-NO3 showed smaller changes relative to δ15N-N2O, δ18O-NO3 showed higher fluctuations compared to δ18O.
Dual isotopic values of $\delta^{18}O$ ranged from 8.25‰ to 15.11‰ with a mean value of $-11.29 \pm 2.52$‰.

### 3.3. Changes in C and N and associated isotopic characteristics in soil and plants

The four land-use types of the Loess Plateau sampled in this study were dam, grass, forest, and farmland. As Fig. 4 shows, dams had the highest TN and TOC of 5.76 and 190.72 mg·kg$^{-1}$, respectively. Among the four land-use types, soil TN was in the following descending order: dam ($4.52 \pm 1.08$ mg·kg$^{-1}$) > forest ($4.49 \pm 0.16$ mg·kg$^{-1}$) > farm > grassland. In contrast, TOC in soil was in the following descending order: forest > farm > dam > grassland. The average TOC value for dams was 162.76 ± 24.44 mg·kg$^{-1}$, while forests had the highest average TOC value overall (170.39 ± 4.44 mg·kg$^{-1}$).

Dual isotopic values of $\delta^{13}C$ showed notable differences in the different land-use types, whereas dual isotopic values of $\delta^{13}C$-TOC showed little variation (Fig. 5a). Dual isotopic values of $\delta^{13}C$-TOC were $-25.83 \pm 0.52$‰ (dams), $-27.67 \pm 0.72$‰ (grassland), $-26.87 \pm 0.23$‰ (forests), and $-24.22 \pm 0.56$‰ (farmland). In contrast, dual isotopic values of $\delta^{15}N$ also exhibited significant differences: $8.19 \pm 0.15$‰ (farmland), $6.09 \pm 0.67$‰ (forests), $5.77 \pm 0.64$‰ (grassland), and $5.36 \pm 3.8$‰ (dams) (Fig. 5b).

In order to better understand the isotopic composition of the catchment, we also analyzed the composition of $^{13}C$ and $^{15}N$ in the four main species of vegetation in the watershed. Dual isotopic values of $\delta^{13}C$ for P. stenoptera, S. babylonica L., St. bungeana Trin., and A. cristatum were $-27.03 \pm 3.25$‰, $-26.7 \pm 2.78$‰, $-30.63 \pm 1.23$‰, and $-26.742 \pm 1.23$‰, respectively. Dual isotopic values of $\delta^{15}N$ for these species showed no significant differences except for A. cristatum, which had a value of $-2.5 \pm 0.56$‰ (Fig. 6).

### 4. Discussion

#### 4.1. Isotopic relationships and sources in water

Liu et al. (2006) reported that on most occasions $N$ isotopes in water are usually within a few per mil of zero, and this $O$ isotope state roughly reflects local water versus dissolved oxygen at a 2:1 ratio (Kendall et al., 2007).

![Fig. 3. Spatial variation of stable isotopic C, O, and H (a) and $^{18}N$ and $^{18}O$ of nitrate (b) at different locations in the Yangjuangou Catchment.](image)

![Fig. 4. Change in soil C and N under different land uses in the Yangjuangou Catchment.](image)

![Fig. 5. Composition of stable isotope C (a) and N (b) under different land uses in the Yangjuangou Catchment.](image)
Previous studies have reported that dual isotopic values of $\delta^{15}$N in water bodies impacted by manure range from −2‰ to 4‰, for which urea accounts for 0.18 ± 1.27‰ and ammonia fertilizers 0.91 ± 0.88‰ (Pennino et al., 2014). Most $\delta^{15}$N in soil has been found to range from −10% to 15% (Earl et al., 2005; Kendall, 1998), while soil that supports vegetation and soil polluted by domestic sewage range from 4% to 9%. Livestock manure could reach up to 17% (Hale et al., 2014). In addition, Elliott et al. (2007) reported that nitrate originating from both the atmosphere and synthetic nitrate fertilizers is mainly composed of $^{14}$N and $^{18}$O. In our study area, which only contains villages, industrial sewage was considered to be negligible. As Fig. 7a shows, potential NO$_3^-$ sources in the watershed may be derived from synthetic fertilizers, soil organic N, atmospheric deposition, sewage, and manure. Of these, we identified soil N, fertilizers, and manure as the three main pollution sources.

The O atom in nitrate originates from surrounding water (H$_2$O) and atmospheric O$_2$. Kendall et al. (2007) suggested that the contribution of O atoms from local water and atmospheric O$_2$ equates to a ratio of 2:1 during nitrification processes. As Fig. 3a shows, $\delta^{18}$O-NO$_3^-$ values in the watershed all exceeded 8‰, indicating that either atmospheric nitrate was the dominant source or that nitrate from the atmosphere was intensively recycled through the organic N pool, thereby changing its original signature (Mayer et al., 2002; Mengis et al., 2001). The higher levels of both $\delta^{15}$N-NO$_3^-$ and $\delta^{18}$O-NO$_3^-$ in the water of our study area lead us to conclude that rainfall and fertilizers from manmade or livestock sources are the major sources of nitrate in the Yangjuangou Catchment. Telmer and Veizer (1999) and Peng et al. (2014) have shown that river DIC mainly derives from soil CO$_2$, respiration of riverine organic matter, and the dissolution of atmospheric precipitation. Therefore, $\delta^{13}$C is commonly used to identify C sources due to the distinctively different ratios observed in major C reservoirs: −8‰ for atmospheric CO$_2$, 0‰ for marine carbonates, and −27‰ to −12‰ for organic matter, which is based on C$_3$ and C$_4$ assimilation pathways during photosynthesis. On the other hand, other studies have shown C$_4$ mainly ranging from −0.24‰ to −4‰, C$_3$ mainly ranging from −0.19‰ to −1.9‰, and crassulacean acid metabolism mainly ranging from −1‰ to −2.3‰ (Cerling et al., 2011; Gargin et al., 2014; Jin et al., 2010; Singh et al., 2005). In our study, the mean dual isotopic value of $\delta^{13}$C in the watershed was −5.36 ± 0.28‰ (Fig. 7b), indicating that $\delta^{13}$C-DIC in the Yangjuangou Catchment is mainly controlled by carbonate weathering or soil erosion but is also controlled by photosynthesis and respiration. Photosynthesis was the dominant controlling factor in surface water of the watershed. During the photosynthetic process, CO$_2$ is absorbed and $^{12}$C is preferentially taken up, after which residual DIC is enriched with $^{13}$C and $\delta^{13}$C-DIC becomes more positive (Gao et al., 2014, 2016).

The stable isotopic composition of $\delta^{18}$O and $\delta^2$D in water bodies provides insight into the sources and mixing effects of water masses as well as the interaction of water with rocks and minerals (Baskaran et al., 2016; Raida et al., 2011). Average annual $\delta^{18}$O and $\delta^2$D values can be used to track the pathway of water moisture as well as the dominant sources of water (Hren et al., 2009; Tian et al., 2007). However, Gat (1996) believed that atmospheric moisture during tropical storms that have very low $\delta^2$D and $\delta^{18}$O values cannot be fully explained by the usual assumptions related to precipitation and temperature. In our study, $\delta^{18}$O was −7.87 ± 0.85‰ in the watershed (Fig. 7c), which is similar to $\delta^{18}$O values of 6.5‰ during tropical storms and low-pressure systems reported in Puerto Rico (Scholl et al., 2009). This could be explained by the “amount effect,” with lower values associated with higher rainfall amounts and by cloud altitude and atmospheric
condensation temperature (Scholl et al., 2009). In addition, Adame et al. (2016) reported that the characteristic isotopic composition of water from rainfall generates an almost immediate response in the isotopic composition of river flow.

We found a high positive correlation between δ²D and δ¹⁸O (r² = 0.81; Fig. 7c), indicating that δ²D was a function of δ¹⁸O. In our study, the mean dual isotopic value of δ²D was −61.49 ± 3.25‰, which is similar to the δ²D values of rivers and rainfall (40‰–50‰) in the eastern USA during hurricanes (Higgins, 2012). The similarity in our findings of δ²D and δ¹⁸O values to those reported for storm-type systems suggests that the catchment is subjected to variations in water sources and/or precipitation mechanisms (Bowen, 2012), which would lead to soil erosion and runoff effects that subsequently produce a wide range of isotopic values.

4.2. Characteristics of C and N isotopes in the watershed

Ojima et al. (1993) reported that δ¹³C values in soil along with C₂ inputs ranged from 24‰ to 29‰ and that soil along with C₄ inputs ranged from 12‰ to 13‰. In our study, we found that δ¹³C values for rainfall, water, and TC in soil mainly ranged from −10‰ to −5‰, but δ¹³C-TOC values for soil were much less (from −30‰ to −20‰) (Fig. 8). We attribute this discrepancy to the higher levels of δ¹³C-DIC for water, rainfall, and soil. This also provides evidence that DIC in the catchment water mainly derives from precipitation and associated runoff. However, δ³⁴S/δ³²S values for the plants were close to the δ¹³C-TOC values for soil, indicating that the litter from the sampled species is a major source of C in the watershed soil. This is because in the Yangjuangou Catchment, severe erosion typically occurs on grassy (C₄) land-use types devoid of woody vegetation (C₃), whereas channel banks can typically maintain some degree of woody vegetation cover. Accordingly, we consider that δ¹³C could be used to discriminate between sediment derived from these distinct subsoil sources.

We found that the δ¹⁵N values in water corresponded to soil and rainfall but did not correspond to plants, indicating that plants were not the major source of N in water (Fig. 8). Animal manure and manure-derived fertilizers have high δ¹⁵N (Finlay and Kendall, 2007) and landscapes or channel banks may impact soil δ¹⁵N due to their different anaerobic conditions; they may also increase denitrification, which favors the accumulation of ¹⁵N (Mukundan et al., 2010). Therefore, the δ¹⁵N values we obtained showed differences between cultivated sources with waste-derived fertilizer inputs and subsoil in runoff (Yang et al., 2015, 2016).

5. Conclusions

This study demonstrated that land-use types, climate change, and vegetation could be important factors that regulate variations in stable isotopes of C, N, H, and O in stream water and soil in typical watersheds in the Loess Plateau. The spatial distribution of concentrations and isotopes of C, N, H, and O in water bodies, soil, and vegetation reflect the heterogeneity and complexity of the Yangjuangou Catchment. Isotopic values indicated that C and N in most water samples are mainly derived from manure sources, soil erosion, and a mixture of chemical fertilizers and manure during rainfall events. Moreover, volatilization and denitrification processes may affect C, N, O, and H isotopic composition in this catchment. Lastly, the high temperatures and rainfall of the Yangjuangou Catchment during the rainy season may also increase the extent of C and N loss.

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