



Contents lists available at ScienceDirect

Quaternary Science Reviews

journal homepage: www.elsevier.com/locate/quascirev

The stable isotope composition of organic and inorganic fossils in lake sediment records: Current understanding, challenges, and future directions



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ARTICLE INFO

Article history:

Received 15 October 2017

Received in revised form

30 July 2018

Accepted 2 August 2018

Keywords:

Stable isotopes

Lake sediment

Organic remains

Inorganic remains

Diatoms

Invertebrates

Ostracods

ABSTRACT

This paper provides an overview of stable isotope analysis (H, C, N, O, Si) of the macro- and microscopic remains from aquatic organisms found in lake sediment records and their application in (palaeo)environmental science. Aquatic organisms, including diatoms, macrophytes, invertebrates, and fish, can produce sufficiently robust remains that preserve well as fossils and can be identified in lake sediment records. Stable isotope analyses of these remains can then provide valuable insights into habitat-specific biogeochemistry, feeding ecology, but also on climatic and hydrological changes in and around lakes. Since these analyses focus on the remains of known and identified organisms, they can provide more specific and detailed information on past ecosystem, food web and environmental changes affecting different compartments of lake ecosystems than analyses on bulk sedimentary organic matter or carbonate samples. We review applications of these types of analyses in palaeoclimatology, palaeohydrology, and palaeoecology. Interpretation of the environmental 'signal' provided by taxon-specific stable isotope analysis requires a thorough understanding of the ecology and phenology of the organism groups involved. Growth, metabolism, diet, feeding strategy, migration, taphonomy and several other processes can lead to isotope fractionation or otherwise influence the stable isotope signatures of the remains from aquatic organisms. This paper includes a review of the (modern) calibration, culturing and modelling studies used to quantify the extent to which these factors influence stable isotope values and provides an outlook for future research and methodological developments for the different examined fossil groups.

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1. Introduction

Stable isotope analysis provides a versatile tool for investigating lake sediment records based on the link between stable isotope

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ratios and a range of environmental and biological processes, including climate change, hydrology, biogeochemical cycling, and consumer-diet interactions in food webs (Leng and Henderson, 2013). Stable carbon and nitrogen isotope analysis of bulk sedimentary organic matter (SOM) is often used in palaeoenvironmental records as SOM is easy to sample and relatively straightforward to measure (Meyers and Teranes, 2001). Some sediments require chemical pre-treatment to remove carbonates, which in itself can affect the stable isotope composition of SOM (Brodie et al., 2011a; b). The information provided by SOM is always an integrated signal of catchment and in-lake processes, which can make it hard to interpret variations in its isotope composition. For example, an understanding of changes in $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ values of SOM requires detailed information from the catchment as they are dependent on, amongst a range of factors, the composition and amount of input from terrestrial vegetation, anthropogenic nutrient input, lake volume, littoral-to-profundal ratio of the lake basin, productivity, groundwater inputs and stratification (e.g., Meyers and Ishiwatari, 1993).

Stable isotopes of endogenic (bulk) carbonates are also widely used in palaeohydrology and palaeoclimatology. Changes in mineral $\delta^{18}\text{O}$ values are interpreted in terms of changes in precipitation and temperature (Leng and Marshall, 2004; Leng and Barker, 2006). The $\delta^{13}\text{C}$ values of endogenic carbonates are a complex sum of processes taking place in the lake and its catchment, including temperature and productivity-dependent fractionation, dissolved inorganic carbon (DIC) inflow, methanogenesis and methane oxidation, CO_2 dissolution and outgassing and lake stratification (Siegenthaler and Eicher, 1986; Hollander and Smith, 2001; Leng and Marshall, 2004; Schwalb et al., 2013).

To complement stable isotope data derived from analyses of SOM or carbonates, it has become more common to analyse the stable isotope composition of identifiable fossil remains separated and manually picked from lake sediments (e.g. diatoms, invertebrates, plant macrofossils), or to analyse stable isotopes of specific compounds chemically isolated from the sediments (e.g., lipids, amino acids, pigments). These approaches offer the great benefit of targeting specific organism groups or chemical biomarkers, which can reflect particular habitats or locations in a lake, or provide valuable information about their functional roles in an ecosystem. Targeting specific remains and compounds also means that it is easier to understand and test how the stable isotope composition is affected by biogeochemical and taphonomic processes over time.

In this review, we provide an overview of approaches based on stable isotopes measured on taxon-specific samples. We include the isotope systems H, C, N, O, Si, measured on diatoms, calcareous and chitinous invertebrate remains, fish remains, and plant macrofossils. We discuss their palaeolimnological applications and provide an overview of the current understanding of taphonomy and ecology that is required to interpret sedimentary records.

2. Diatoms

Diatoms are unicellular, eukaryotic, micro-organisms, which are ubiquitous in nature. As such, diatom silica is an interesting sediment component for isotope measurements in lake (Leng and Barker, 2006) and marine (Swann and Leng, 2009) environments, where the oxygen ($\delta^{18}\text{O}_{\text{diatom}}$), silicon ($\delta^{30}\text{Si}_{\text{diatom}}$), carbon ($\delta^{13}\text{C}_{\text{diatom}}$) and nitrogen ($\delta^{15}\text{N}_{\text{diatom}}$) isotope compositions can all be used as proxies for environmental change. $\delta^{18}\text{O}_{\text{diatom}}$ tends to be used as a measure of temperature/water composition variation (Fig. 1), $\delta^{30}\text{Si}_{\text{diatom}}$ as a proxy for nutrient availability and utilisation (Fig. 2), and $\delta^{13}\text{C}_{\text{diatom}}$ and $\delta^{15}\text{N}_{\text{diatom}}$ for nutrient cycling/source

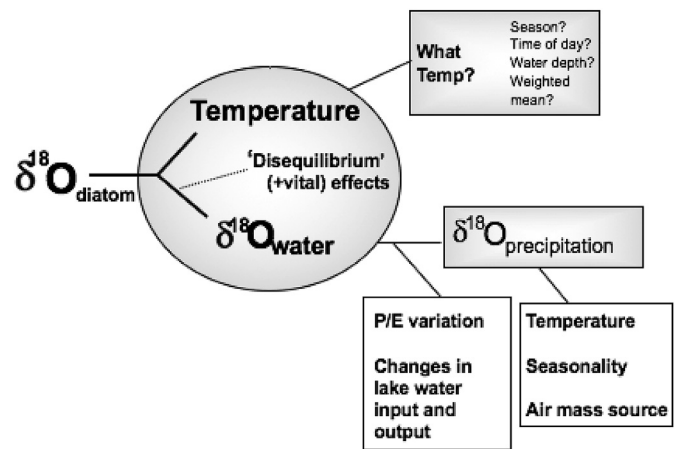


Fig. 1. Controls on the stable oxygen isotope composition of biogenic silica.

investigation (Leng and Henderson, 2013).

Compared to $\delta^{18}\text{O}_{\text{diatom}}$ studies, the application of $\delta^{30}\text{Si}_{\text{diatom}}$, $\delta^{13}\text{C}_{\text{diatom}}$ and $\delta^{15}\text{N}_{\text{diatom}}$ techniques are in their relative infancy, with only a few studies being applied in lacustrine systems (Alleman et al., 2005; Street-Perrott et al., 2008; Swann et al., 2010; Hurrell et al., 2011; Hernández et al., 2011, 2013; Opfergelt et al., 2011; Chen et al., 2012; Barker et al., 2013; Cockerton et al., 2015; Webb et al., 2016; Panizzo et al., 2016, 2018a; b). There are several reviews of the use of isotopes in biogenic (including diatom) silica (including Leng and Barker, 2006; Swann and Leng, 2009; Leng et al., 2009; Leng and Henderson, 2013; Sutton et al., 2018), which highlight, in detail, the many issues associated with such analyses. These will be further elucidated upon here and include issues such as equilibrium fractionation, sample purification, post mortem maturation of diatom silica, and standardisation and inter-laboratory calibrations. Given that more recent advances have been made in $\delta^{30}\text{Si}_{\text{diatom}}$ applications in lacustrine settings, in addition to the body of literature on $\delta^{18}\text{O}_{\text{diatom}}$, we focus this review on these two proxies.

Diatom silica is a structurally complex mineral for $\delta^{18}\text{O}_{\text{diatom}}$ (see section 2.4 below) measurement due to a hydrous component. O isotope extraction generally adopts fluorination (offline) techniques, with measurement via gas-source isotope ratio mass spectrometry (IRMS), a technique which can also be used to measure $\delta^{30}\text{Si}_{\text{diatom}}$ (Leng and Sloane, 2008). However, the use of multi-collector inductively-coupled-plasma mass spectrometry (MC-ICP-MS) is becoming increasingly more dominant for $\delta^{30}\text{Si}_{\text{diatom}}$ analyses due to the reduced sample size needed (~1 mg), while carbon and nitrogen (for $\delta^{13}\text{C}_{\text{diatom}}$ and $\delta^{15}\text{N}_{\text{diatom}}$) are measured on very small quantities of organic material hosted (occluded) within the structure (Webb et al., 2016).

Silicon has three naturally occurring stable isotopes ^{28}Si , ^{29}Si and ^{30}Si with a mean abundance of 92.2%, 4.7% and 3.1% respectively. The isotope composition of any sample (x), for ^{29}Si or ^{30}Si (n), is expressed in delta notation (δ), compared to the reference standard NBS28, using the following equation:

$$\delta^n\text{Si}_x (\text{‰}) = \left(\frac{(^n\text{Si}/^{28}\text{Si})_x - (^n\text{Si}/^{28}\text{Si})_{\text{standard}}}{(^n\text{Si}/^{28}\text{Si})_{\text{standard}}} \right) \times 1000$$

An overview of the key processes affecting $\delta^{30}\text{Si}$ in lakes are shown in Fig. 2. These include processes in a catchment (weathering, soil development, vegetation cover) and in a lake (productivity, water supply, stratification, sedimentation, dissolution) and will be discussed in more detail below.

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