



Review article

Interpretation of the nitrogen isotopic composition of Precambrian sedimentary rocks: Assumptions and perspectives



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ABSTRACT

Nitrogen isotope compositions in sedimentary rocks ($d^{15}N_{sed}$) are routinely used for reconstructing Cenozoic N-biogeochemical cycling and are also being increasingly applied to understanding the evolution of ancient environments. Here we review the existing knowledge and rationale behind the use of $d^{15}N_{sed}$ as a proxy for the Precambrian N-biogeochemical cycle with the aims of (i) identifying the major uncertainties that affect analyses and interpretation of nitrogen isotopes in ancient sedimentary rocks, (ii) developing a framework for interpreting the Precambrian $d^{15}N_{sed}$ record, and (iii) testing this framework against a database of Precambrian $d^{15}N_{sed}$ values compiled from the literature, and (iv) identifying avenues of focused research that should increase confidence in interpreting Precambrian $d^{15}N_{sed}$ data. This review highlights the intrinsic complexity of the $d^{15}N_{sed}$ proxy and the significant effort that remains to realize its potential. Specifically, it is crucial to gain a better understanding of how and when diagenesis and metamorphism affect the $d^{15}N$ of bulk and kerogen-bound nitrogen. Ultimately, more data are required to apply statistics to interpreting $d^{15}N_{sed}$ variability within given geological time intervals. Finally, numerical modeling of the $d^{15}N_{sed}$ variability expected in different environments under varying redox scenarios is necessary to establish a predictive template for interpreting the ancient nitrogen isotope record. In spite of the challenges facing the application of this proxy to the Precambrian, the existing $d^{15}N_{sed}$ record shows several features possibly related to the stepwise oxygenation of the surface environment, underlining the potential for nitrogen isotopes to reveal clues about the evolution of early Earth.

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1. Introduction

Characterizing the evolution of the marine nitrogen biogeochemical cycle through time and identifying its driving factors is critical to fully understanding the evolution of Earth's surface environments for at least two interconnected reasons. First, nitrogen (N) is present in the ocean mostly as the bioavailable species nitrate (NO_3^-), nitrite (NO_2^-), ammonium (NH_4^+) and dissolved organic nitrogen (DON). Because N is one of the major constituents of life (with C/N ratio between 4 and 10 for the oceanic phytoplankton), these bioavailable species exert a strong control on primary productivity, and hence on the biogeochemical C cycle (e.g., Tyrrell, 1999; Falkowski, 1997; Gruber and Galloway, 2008; Canfield et al., 2010). Second, N is also present in Earth's atmosphere, mostly as di-nitrogen (N_2), which is by far the most abundant gas in the atmosphere (78.06% by volume today), but also in trace amounts as N_2O , NH_3 and other NO_x species, whose abundances are in part controlled by the marine N-biogeochemical cycle. Both the N_2 partial pressure and the abundance of these trace greenhouse gas species in the atmosphere influence the global climate, linking it to the N-cycle (Buick, 2007; Goldblatt et al., 2009; Roberson et al., 2011; Thunell and Kepple, 2004).

An obvious approach to reconstructing the evolution of the marine N-biogeochemical cycle, and hence its role in shaping ancient surface environments, is to interrogate the isotope composition of N preserved in sedimentary rocks ($\delta^{15}\text{N}_{\text{sed}}$). As for any geochemical tracer, the ability to extract meaningful paleoenvironmental information from N isotopes requires several conditions be met. First, reliable (precise and reproducible) $\delta^{15}\text{N}_{\text{sed}}$ measurements are necessary. Second, post-depositional processes must not have significantly modified $\delta^{15}\text{N}_{\text{sed}}$ values. Finally, and perhaps most challengingly, $\delta^{15}\text{N}_{\text{sed}}$ values must be related to processes in the biogeochemical nitrogen cycle.

In the last 50 years, major progress has been made in understanding controls on N isotope signatures of nitrate, particulate organic matter, surface sediments (e.g. Somes et al., 2010; Tesdal et al., 2013; Thunell et al., 2004) and early diagenetic processes (e.g. Robinson et al., 2012) in modern environments. Although many uncertainties remain, these studies have yielded several guidelines to the interpretation of N isotopes in modern sediments: (i) in anoxic basins and continental platforms, $\delta^{15}\text{N}$ of primary producers is transmitted to the sediment, (ii) the $\delta^{15}\text{N}$ value of primary producers typically reflects the $\delta^{15}\text{N}$ of assimilated N, and (iii) the speciation and $\delta^{15}\text{N}$ values of bio-available N in the surface ocean mostly depend on the dominant processes in the N cycle in the ocean (and hence on ocean redox structure). Available data indicate that $\delta^{15}\text{N}_{\text{sed}}$ signatures are faithful proxies for biogeochemical processes in the marine realm at least in the recent past (e.g. Galbraith et al., 2008).

Nitrogen isotope analyses of ancient sedimentary rocks began in earnest in the 1980's (e.g. Hayes et al., 1983; Zhang, 1988). In the case of Precambrian rocks, the field developed slowly for the subsequent 30 years mostly due to the technical difficulty of making measurements in N-poor samples. Two other issues have also historically impeded the application of N isotopes to the Precambrian: uncertainties about the preservation of a primary $\delta^{15}\text{N}$ signal in often metamorphosed samples and limited understanding of how the modern N cycle and its isotopic transcription into sedimentary organic matter can be applied to interpreting Precambrian datasets. However, recent technical advances have opened the door to rapid and reliable analyses of minute quantities of N, sometimes coupled with simultaneous analyses of organic carbon (e.g. Polissar et al., 2008). Application of the proxy has thus accelerated

in the past five years. Numerous N isotope studies in Precambrian sedimentary rocks have been performed with two broad objectives: (i) reconstructing the N-biogeochemical cycle and its relations to water column redox structure of Precambrian marine environments (e.g. Papineau et al., 2009; Busigny et al., 2013; Stüeken, 2013; Ader et al., 2014) and (ii) documenting the Precambrian evolution and radiation of organisms performing the main metabolic pathways of the N cycle, such as NH_4^+ oxidation (Thomazo et al., 2011), denitrification (Beaumont and Robert, 1999; Garvin et al., 2009; Godfrey and Falkowski, 2009) and N_2 -fixation (Stüeken et al., 2015a).

The application of N isotope systematics is bound to increase with a surge in interest in the coevolution of Precambrian environments and life, which is driving multi-proxy approaches to extract increasingly nuanced information about ancient nutrient cycling and metabolic pathways. In light of significant recent progress, it is timely to revisit and fortify the underpinnings of the nitrogen isotope interpretational framework as applied to the Precambrian. To this end, rather than reviewing how $\delta^{15}\text{N}_{\text{sed}}$ data have been interpreted in terms of the Precambrian marine N-biogeochemical cycle (e.g., Pinti and Hashizume, 2011; Thomazo et al., 2009; Thomazo and Papineau, 2013), the objectives are (i) to evaluate our confidence in applying the N isotope system to very old rocks and (ii) to identify future directions of research to progress the application of the N isotope system to unraveling Precambrian environments and biogeochemical processes.

We have subdivided this paper into six sections. The first four sections are dedicated to reviewing the main conditions required to extract meaningful information from N isotopes and to discuss their applicability to the Precambrian (Fig. 1): (i) that analytically reliable $\delta^{15}\text{N}_{\text{sed}}$ measurements can be made; (ii) that absence of post-depositional modification of $\delta^{15}\text{N}$ value of surface sediments after deposition can be demonstrated or inferred; (iii) that $\delta^{15}\text{N}$ values of surface sediments can be assumed to be systematically related to the composition of primary producers; and (iv) that $\delta^{15}\text{N}$ values of primary producers reflect speciation and isotope composition of the N assimilated in the photic zone. The fifth section explores the extent to which different possible modes of global marine N-cycling should translate into distinct and recognizable $\delta^{15}\text{N}$ signatures based on conceptual models of N-cycling in oceans with different redox structure. The last section uses an updated compilation of published Precambrian $\delta^{15}\text{N}_{\text{sed}}$ data as a platform to identify and interpret the main features of the Precambrian N isotope record.

2. $\delta^{15}\text{N}$ measurements of Precambrian sedimentary rocks

Precambrian sedimentary rocks commonly contain little total N due to their typically low organic matter content and their long and often complex post-depositional history, (Supplementary Table 1 and references cited therein). These low N-contents present an analytical challenge, which until recently strongly limited the acquisition of N isotope data. Isotopic measurements on Precambrian rocks are mostly bulk analyses ($\delta^{15}\text{N}_{\text{bulk}}$), in which the measured N includes both organically bound N and nitrogen incorporated in minerals, typically in the form of NH_4^+ bound within clays and other silicate minerals. But N isotopes have also been measured in kerogens ($\delta^{15}\text{N}_{\text{ker}}$), graphite, mineral separates ($\delta^{15}\text{N}_{\text{NH}_4}$) and fluid inclusions ($\delta^{15}\text{N}_{\text{N}_2}$). All measurements are made on gaseous N_2 via gas-source isotope-ratio mass spectrometry (IRMS), but several methods and apparatuses have been used to oxidize organic-N and/or silicate bound NH_4^+ from ancient sedimentary rocks into N_2 gas, which can then be measured by IRMS.

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