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Pulse of intense oxidative weathering during the latest Paleoproterozoic

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ABSTRACT

The mid-Proterozoic (ca. 1.8–0.8 Ga) is broadly characterized by low atmospheric oxygen and pervasive oceanic anoxia punctuated by pulses of oxygenation. However, the causes underpinning these transient events remain elusive. Here, we report on 1.64 billion-year-old black shales and dolostone from the Chuanlinggou Formation in the North China Craton that represent sediment deposited during the break-up of the supercontinent Columbia. The rocks are characterized by high initial $^{187}Os/^{188}Os$ values, comparable to modern seawater values, alongside positive isotopic compositions of copper similar to those found in shales deposited during the Great Oxidation Event between 2.5 and 2.2 billion years ago. Additionally, we note high Th/U ratios and Chemical Index of Alteration values. Collectively, our data suggest a positive feedback initiated by continental fragmentation, with increased oxidative weathering on land, greater nutrient supply to the oceans, enhanced primary production of marine cyanobacteria, and ultimately, higher rates of oxygen production, perpetuating the feedback loop.

INTRODUCTION

Atmospheric oxygen has influenced the ecology and biogeochemical functioning of marine ecosystems through time (e.g., Planavsky et al., 2021). The Great Oxidation Event (GOE), occurring between 2.5 and 2.2 billion years ago (Ga), marked a significant rise in atmospheric oxygen (O_2) levels from trace amounts to over 10^{-5} of the present atmospheric level (e.g., Konhauser et al., 2011; Poulton et al., 2021). Subsequently, it has been proposed that atmospheric O_2 levels declined to a low, albeit uncertain, level over the next ~1 billion years (e.g., Planavsky et al., 2014). However, a growing body of research indicates that the redox landscape over this period was much more complex than previously understood, with evidence suggesting the deepening of the chemocline and pulsed oxygenation events (e.g., Zhang et al., 2016, 2018; Yang et al., 2024). Nonetheless, the cause of these oxygenation events remains uncertain, as does their extent—whether they were locally limited to shallow seawater (referred to as oxygen oases) or indicative of a global increase in atmospheric oxygen.

Both rhenium (Re) and osmium (Os) exist in various redox states at the Earth's surface (Kendall et al., 2015). Under oxidizing atmospheric conditions, Re is transported to the oceans in the form of ReO_4^- (e.g., Lu et al., 2017), and the β^--decay of ^{187}Re to ^{187}Os has a half-life of 41.6 b.y. (Smoliar et al., 1996). The $^{187}Os/^{188}Os$ ratio of seawater reflects a time-varying balance between radiogenic and unradiogenic Os fluxes to the ocean (Kendall et al., 2015). Modern seawater has a high $^{187}Os/^{188}Os$ ratio of ~1.06 due to riverine runoff of radiogenic Os derived from oxidative weathering (~1.5) (Peucker-Ehrenbrink and Ravizza, 2000). A subordinate flux of unradiogenic Os originates from the dissolution of cosmic dust and hydrothermal alteration of ultramafic oceanic rocks (~0.13) (Peucker-Ehrenbrink and Ravizza, 2000). Thus, temporal shifts in the $^{187}Os/^{188}Os$ ratio of seawater reflect changes in Earth's surface redox processes, and past seawater $^{187}Os/^{188}Os$ ratios calculated from sedimentary samples are expressed as initial $^{187}Os/^{188}Os$ (e.g., Kendall et al., 2015).

Copper (Cu) isotopes have also been utilized as a potential proxy for paleo-redox conditions in both marine and terrestrial environments on early Earth (e.g., Chi Fru et al., 2016). The weathering of the continents constitutes a significant source of Cu input into the modern oceans (Little et al., 2017). Consequently, the Cu isotope composition of rivers will reflect the Cu supplied by weathering, although subsequent biogeochemical processes within rivers may modify this primary signature. Previous studies have argued that oxidative weathering of continental sulfides, such as chalcocite (Cu_2S) and chalcopyrite ($CuFeS_2$), should have increased the release of dissolved Cu(II) and delivered more ^{65}Cu-enriched runoff to the oceans (e.g., Chi Fru et al., 2016, and references therein).

In this study, we conducted an analysis of elemental concentrations, Re–Os, and Cu isotopic compositions in sedimentary rocks, including black shales and dolostone, from the Chuanlinggou Formation (dated to ca. 1.64 Ga) within the North China Craton (NCC). Our aim was to evaluate terrestrial weathering processes and the evolution of atmospheric O_2 levels during the latest Paleoproterozoic. This geological unit holds significance because it contains some of the oldest, most well-preserved, and extensive deposits of ca. 1.8–0.8 Ga black shale within the NCC. Its deposition has been correlated with the initial break-up of the Columbia supercontinent (Kusky and Li, 2003; Lin et al., 2019; Ernst et al., 2021).

GEOLOGICAL SETTING

In the late Paleoproterozoic (ca. 1.85 Ga), the tectonic system of the NCC underwent a fundamental change due to the collision of two continental blocks. The opening of Yanshan Basin was situated at the margin of the

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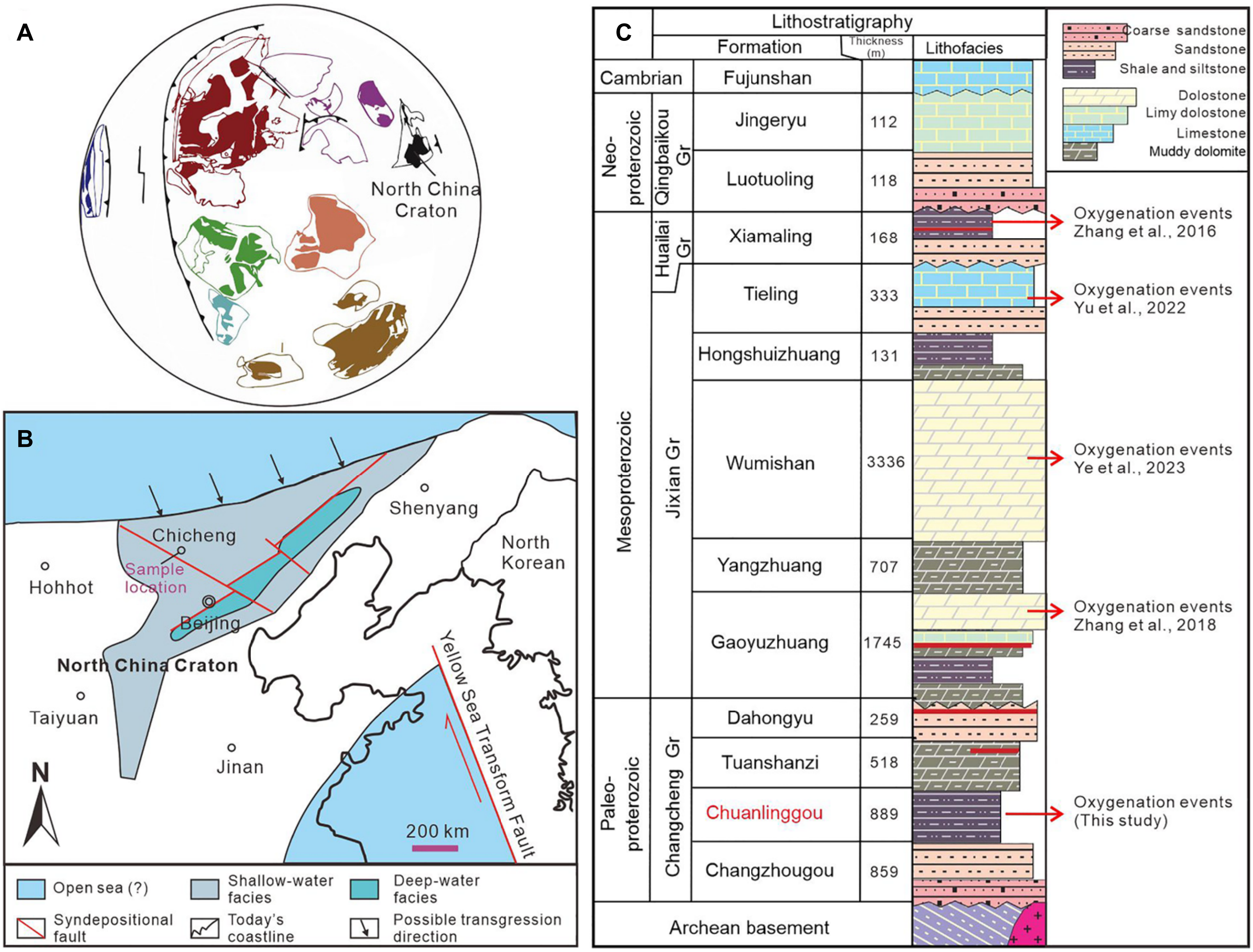

Figure 1. (A) Latest Paleoproterozoic global paleogeographic reconstruction (after Pisarevsky et al., 2014) and the location of the North China Craton. (B) Paleogeographic context for the Yanshan Basin during the deposition of Chuanlinggou Formation (after Lin et al., 2019, and references therein). (C) Ca. 1.8–0.8 Ga stratigraphic profile of the Yanliao Basin (after Yang et al., 2024).

NCC between 1.8 and 1.6 Ga (e.g., Kusky and Li, 2003; Figs. 1A and 1B). Paleoproterozoic strata are well exposed in the Yanliao Basin, exhibiting metamorphism limited to prehnite–pumpellyite facies. Within this context, the Changcheng Group (containing the Chuanlinggou Formation), dating from 1.65 to 1.60 Ga, represents the initial suite of sedimentary cover rocks that unconformably overlie the Archean basement (Fig. 1C). During the latest Paleoproterozoic, the Yanliao Basin was likely connected to the open ocean in the north (Fig. 1B; Lin et al., 2019). Black shales, ironstones, dolostone, and interbedded sandstones of the Chuanlinggou Formation record a transition from a shallow marine to subtidal low-energy environment. Abundant geochronological studies have constrained the depositional age of the Chuanlinggou Formation to ca. 1.64 Ga (see Lin et al., 2019, and references therein), an age comparable to the Barney Creek in the McArthur basin, Northern Australia (Ernst et al., 2021, and references therein).

MATERIALS AND METHODS

Chuanlinggou Formation samples (black shale and dolostone) were obtained from drill core ZK83-6 within the Zengjiagou iron deposit, located in Chicheng City, Hebei Province, China. Following a detailed petrographic examination, we proceeded to sample for total organic carbon (TOC), total sulfur (TS), and major and trace elements, as well as Re–Os and Cu isotopic analysis. Detailed information regarding the sampling procedure and analytical methods can be found in Texts S1 and S2 in the Supplemental Material[1], respectively.

[1]Supplemental Material. Rock description, methods, supplemental notes, Figures S1–S4, and Tables S1–S2. Please visit https://doi.org/10.1130/GEOL.S.26948314 to access the supplemental material; contact editing@geosociety.org with any questions.

RESULTS

The black shale samples exhibit higher TOC and TS contents, ranging from 0.13 wt% to 4.68 wt% and 0.03 wt% to 2.13 wt%, respectively, compared to the dolostone samples, which range from 0.05 wt% to 0.64 wt% for TOC and 0.02 wt% to 0.62 wt% for TS. Thorium (Th)/uranium (U) ratios in the black shale ranges from 0.94 to 7.21, with an average value of 4.53 (Table S1; see footnote 1). The Cu/Aluminum (Al) ratios display a wide range, from 0.4 to 22.31, spanning the Post-Archean Average Shale (PAAS) value of 5 (Taylor and McLennan, 1985). The entire section has high Chemical Index of Alteration values minus potassium (CIA-K) of 84–99.

The Re–Os isotope results for 13 samples are detailed in Table S2. Among them, eight black shale samples show $^{187}Re/^{188}Os$ ratios ranging from 2.80 to 136.40, with corresponding $^{187}Os/^{188}Os$ ratios of 1.227–5.118. The initial

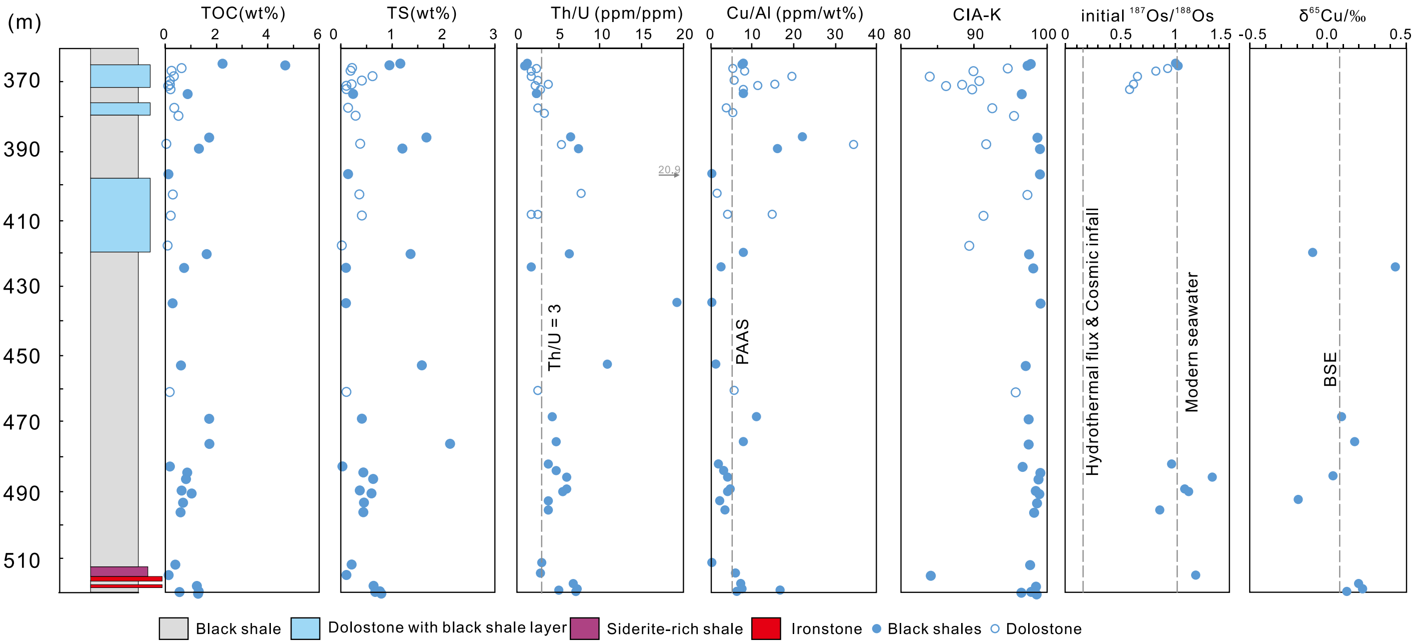

Figure 2. Stratigraphy and geochemistry of the studied drill cores. TOC—total organic carbon; TS—total sulfur; CIA-K—Chemical Index of Alteration minus potassium; PAAS—Post-Archean Average Shale; BSE—bulk silicate earth.

$^{187}Os/^{188}Os$ ratio, calculated from the deposition age of 1.64 Ga, ranges from 0.84 to 1.34, with an average of 1.06 ± 0.14. The $^{187}Re/^{188}Os$ ratios of five dolostone samples vary from 77.24 to 157.30, while $^{187}Os/^{188}Os$ ratios ranged from 2.785 to 5.184. The calculated initial $^{187}Os/^{188}Os$ ratio for dolostone was 0.58–0.93, with an average of 0.72 ± 0.13. Detailed Cu isotopic compositions are listed in Table S1. The black shale samples show $\delta^{65}Cu$ values ranging from −0.19‰ to 0.43‰, with an average of +0.12‰ ± 0.17‰ (n = 10).

DISCUSSION

Evidence of Oxidative Weathering

Recent studies have used chromium-sulfuric acid as the dissolution medium for Re–Os analyses (e.g., Selby and Creaser, 2003; Rooney et al., 2011). This choice is made because CrO_3-H_2SO_4 dissolves detrital components much less efficiently than does inverse aqua regia, and it successfully demonstrated more precise and accurate Re–Os isochron ages (Selby and Creaser, 2003). However, here we chose instead to use the inverse aqua regia method because it reduces the blank levels of Re introduced via the more utilized CrO_3-H_2SO_4 method. Moreover, both Re and Os in our black shale samples show no correlation with Al (which serves as a proxy of the detrital component) and initial $^{187}Os/^{188}Os$, suggesting that dissolved detrital components are insignificant. However, they do display a significant positive correlation with TOC and TS, strongly indicating their authigenic nature within the studied samples. Moreover, our samples include dolostone, which is a pure chemical sedimentary rock with extremely low clay mineral content but a high initial $^{187}Os/^{188}Os$ value. Collectively, we believe this method confirms that the initial $^{187}Os/^{188}Os$ obtained from our samples can record the Os isotopic composition of the contemporaneous ocean during its deposition (e.g., Kendall et al., 2009) (Text S3 and S4).

The initial $^{187}Os/^{188}Os$ ratio of the black shale (1.06 ± 0.14) is similar to that of modern seawater (Figs. 2 and 3A), while the average initial $^{187}Os/^{188}Os$ ratio of the dolostone is 0.70 ± 0.12. Therefore, the initial $^{187}Os/^{188}Os$ ratio, reflecting that of contemporaneous seawater, is similar to present-day seawater (~1.06), which is significantly higher than mantle values (~0.13) (Peucker-Ehrenbrink and Ravizza, 2000). Consequently, we conclude that the riverine influx of radiogenic ^{187}Os from weathering and erosion of the upper continental crust predominated over the influx of non-radiogenic Os from hydrothermal alteration of ultramafic oceanic rocks (Kendall et al., 2009, and references therein).

In the modern ocean, Cu has a relatively short residence time of a few thousand years (Hayes et al., 2018). Thus, it is possible that changes in riverine input could influence global ocean Cu budgets. This influence can occur in two ways: (1) changes in the isotopic composition of the predominant weathered lithology, and/or (2) variations in the intensity of oxidative weathering and any associated isotope fraction-

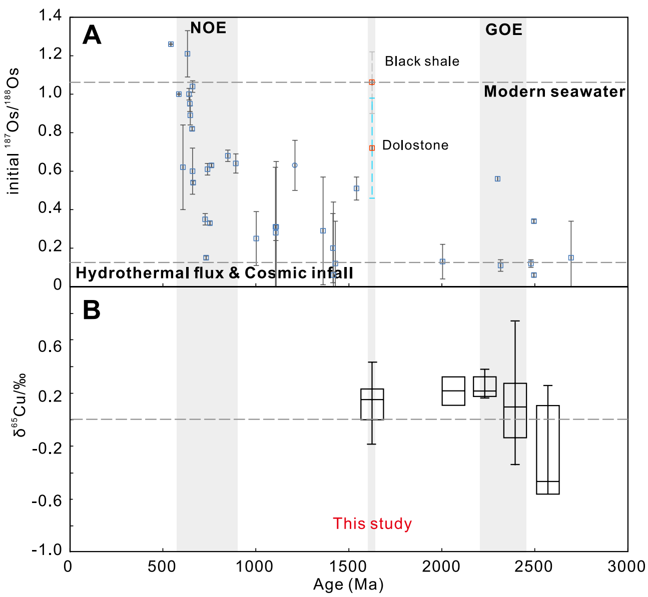

Figure 3. Diagram showing the evolution of initial $^{187}Os/^{188}Os$ (A) and $\delta^{65}Cu$ values (B) of Precambrian sedimentary rocks. The data of initial $^{187}Os/^{188}Os$ are from Sekine et al. (2011), Kendall et al. (2015), Lu et al. (2017) and references therein, and the bar represents the deviation. The rare data of $\delta^{65}Cu$ values are from Chi Fru et al. (2016). NOE—Neoproterozoic Oxygenation Event; GOE—Great Oxidation Event.

ations (Chi Fru et al., 2016; Little et al., 2017). The former is unlikely to exert a major control because clastic sediments and igneous rocks possess constrained Cu isotope compositions (0.08 ± 0.17‰; Moynier et al., 2017). The second scenario is more likely because oxidative weathering of all lithologies appears to correlate with the preferential retention of isotopically light Cu in the source area, consistent with the riverine supply of waters being enriched in ^{65}Cu due to continental sulfide oxidation (Chi Fru et al., 2016). In contrast to pre-GOE shales with negative $\delta^{65}Cu$ values, our black shale samples show slightly positive values (up to 0.43‰) (Fig. 2), which is similar to those observed in GOE and post-GOE shales (Chi Fru et al., 2016; Fig. 3B). These findings can be explained by oxidative weathering of continental sulfides delivering dissolved riverine Cu with a positive $\delta^{65}Cu$ signature (Chi Fru et al., 2016).

The Th/U ratios also provide an indication of surface weathering because it produces elevated ratios due to the oxidation of U(IV) to soluble U(VI) (McLennan and Taylor, 1991). Although U can be re-enriched during sedimentation under reducing conditions, leading to lower Th/U ratios, a Th/U ratio greater than 3 suggests significant weathering (McLennan and Taylor, 1991). In addition, some black shale samples have U concentrations higher than that of PAAS (3 ppm, Taylor and McLennan, 1985), which is unlikely, due to a small U oceanic reservoir causing a high Th/U ratio. Hence, samples with high Th/U ratios likely indicate intense oxidative weathering in the source area (Fig. 2). Moreover, CIA has been widely used to quantitatively evaluate the intensity of continental chemical weathering (Nesbitt and Young, 1982). The entire section has high CIA-K values, which also show a positive correlation with initial $^{187}Os/^{188}Os$, further indicating intense continental weathering (Text S5).

In support of our findings, the ca. 1.64 Ga Cuizhuang Formation shale and ca. 1.70 Ga Yunmenshan Formation ironstone in the south margin of the NCC, both exhibiting high CIA-K values and elevated initial $^{187}Os/^{188}Os$ values, provide further evidence of continental weathering (Zuo et al., 2022; Chu et al., 2023). Collectively, these formations, along with our own findings, point to a period of intense oxidative weathering during late Paleoproterozoic.

The Origin and Cause of Intense Continental Weathering

Throughout much of the Archean and Paleo- and Mesoproterozoic, seawater $^{187}Os/^{188}Os$ ratios typically resemble those of the unradiogenic mantle-extraterrestrial end-member, with values of 0.13 (Fig. 3A). It was not until the late Neoproterozoic that seawater $^{187}Os/^{188}Os$ ratios reached values up to $\sim$1.2 (Fig. 3A). Most ca. 1.8–0.8 Ga seawater samples show extremely low $^{187}Os/^{188}Os$ values (Fig. 3A), corresponding to periods of low atmospheric O_2 (e.g., Planavsky et al., 2014). Interestingly, our samples display high initial $^{187}Os/^{188}Os$ ratios, which are closer to that of the Neoproterozoic, but significantly higher than those of the earlier Precambrian (Fig. 3A), suggesting a surge in continental weathering at ca. 1.64 Ga.

Enhanced erosion of the rocks, as recorded by high initial $^{187}Os/^{188}Os$ ratios, may be driven by increases in atmospheric O_2 or elevated levels of CO_2 (e.g., Yin et al., 2023). In addition to high initial $^{187}Os/^{188}Os$ ratios, our samples also exhibit positive $\delta^{65}Cu$ signature, high Th/U and CIA-K values with minimal variations throughout the section. These findings suggest that persistent oxidative weathering is most likely responsible for the high initial $^{187}Os/^{188}Os$. However, this does not necessarily discount the possibility of increased chemical weathering due to elevated CO_2 levels, particularly given the expected higher CO_2 levels associated with Large Igneous Province (LIP) activity (Ernst et al., 2021). Therefore, a simpler explanation for our observations points toward increased oxygenation, akin to the period following the GOE, as suggested by studies such as those by Konhauser et al. (2011) and Poulton et al. (2021). Similar to some intervals of 2.5 Ga and 2.3 Ga sedimentary rocks (Fig. 3A; Sekine et al., 2011; Kendall et al., 2015), as well as the Ediacaran–Phanerozoic period, which exhibit more radiogenic $^{187}Os/^{188}Os$ ratios (Lu et al., 2017), the intense continental weathering at 1.64 Ga likely coincided with a transient rise in atmospheric O_2 levels. Indeed, this is further supported by Yang et al. (2024), who used iron speciation to demonstrate that during the deposition of Chuanlinggou Formation there was a deepening of the marine chemocline.

The ca. 1.64 Ga Chuanlinggou Formation was deposited in the Yanliao Basin, a continental rift associated with the early break-up of the Columbia supercontinent on the northern margin of the NCC (Fig. 1A) (Kusky and Li, 2003; Chu et al., 2007). It has been proposed that periods of high O_2 production are most likely to occur during tectonically active periods (such as continental break-up or the opening and closing of oceans), when rapid weathering and erosion deliver more nutrients to the oceans, thereby boosting rates of cyanobacterial production of photosynthetic O_2 (Campbell and Allen, 2008). We support such a model. Firstly, Columbia's break-up was accompanied by the emplacement of ca. 1.65–1.62 Ga LIPs in the NCC, Baltica, Laurentia, and West Africa (Ernst et al., 2021, and references therein). The weathering of freshly erupted mafic rocks from these LIPs would have significantly increased the supply of phosphorous (P) to the ocean, as mafic rocks have much higher P contents than those of other igneous rocks (e.g., Horton, 2015). This, in turn, would have stimulated increased planktonic cyanobacterial activity and subsequent O_2 production. Secondly, we argue that the break-up of the Columbia supercontinent also expanded the amount of coastline (Fig. 1A), providing more shallow, habitable environments for cyanobacterial mats. Chu et al. (2007) have provided evidence of enhanced rates of organic carbon and pyrite burial in the NCC during the onset of break-up of the Columbia supercontinent, both of which are efficient mechanisms for increasing O_2 levels in both the atmosphere and ocean (e.g., Berner et al., 2007). Taken together, we propose that these indicate a plausible link between the break-up of the Columbia supercontinent and the intense continental weathering caused by an oxygenation event at ca. 1.64 Ga.

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