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# Continental weathering as the source of iron in Jurassic iron oolites from Switzerland



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# Abstract

Iron is extremely insoluble in oxic seawater. The lack of a large aqueous reservoir means that sediments rich in authigenic iron are rare in the modern ocean. In the Middle Jurassic, however, condensed iron-rich sedimentary rocks are widely distributed. Their formation coincides with increased volcanic activity and continental weathering related to the breakup of Pangea, suggesting iron supply through one of these processes. We studied three Swiss shallowmarine iron oolites from Herznach, Windgällen and Erzegg, all from condensed sedimentary sequences of Middle to early Late Jurassic age, to constrain the source of iron to these rocks, combining radiogenic neodymium, strontium and stable iron isotope analyses. Leached authigenic neodymium isotope compositions, which appear to preserve the primary signature, serve as a tracer for the potential involvement of hydrothermal fluids in the formation of the iron oolites. The three iron oolite successions yield crustal Nd isotope compositions ( $\epsilon$ Nd between – 9 and – 7), providing no evidence for the involvement of such fluids. It is, thus, more likely that iron in the sediments derived from detrital fluvial inputs. Strontium isotope compositions, which could potentially support these findings, point to metamorphic overprinting associated with Alpine thrusting. The light iron isotope signatures associated with Middle to early Late Jurassic condensed sequences,  $\delta^{56}$ Fe between – 1.49 and – 0.57‰, suggest that microbially-mediated iron reduction was also involved in generating these sediments.

**Keywords** Middle Jurassic, Iron oolite, Condensed sequence, Continental weathering, Neodymium isotopes, Strontium isotopes, Iron isotopes

# **1** Introduction

The Jurassic Period exhibits high sedimentary facies diversity in the Alpine Tethys, resulting from major changes in plate tectonics, in paleoceanography and in paleoclimate (e.g. Bernoulli & Jenkyns, 1974; Dera et al., 2011; Norris & Hallam, 1995; Rais et al., 2007; Scotese, 1991; Ziegler, 1988). Peculiar oceanographic conditions were responsible for widespread deposition of condensed

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iron-rich sequences and hardgrounds during the Middle Jurassic (e.g. Bernoulli & Jenkyns, 1974; Jenkyns, 1971). A common feature of these deposits is their red or greenish color, originating from abundant iron minerals (e.g. Berner, 1969; Jenkyns, 1971). The source of iron (Fe) in these rocks is particularly enigmatic, given the low solubility of Fe in modern oxic seawater (e.g. Kraemer, 2004; Raiswell & Canfield, 2012; Worsfold et al., 2014). The particular geological setting suggests two possibilities for this source. First, the opening of Tethys was associated with seafloor spreading, leading to the suggestion of a hydrothermal source (e.g. Halliday & Mitchell, 1984; Holz, 2015; Scotese & Schettino, 2017). Second, a tropical climate may have caused enhanced weathering, suggesting continental Fe as another possible source (e.g. Donnadieu et al., 2006; Gehring, 1986a; Holz, 2015). Local enrichment of Fe and its incorporation into sediments is



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often associated with microbial activity, leading to locally reducing conditions within sediment porewaters and the mobilization of Fe in its reduced state (e.g. Dahanayake & Krumbein, 1986; Gehring, 1986b; Préat et al., 2008). Hence, irrespective of the source of Fe, redox related Fe cycling may have been involved in the formation of these iron-rich condensed sequences (e.g. Dahanayake & Krumbein, 1986; Gehring, 1985, 1986b; Glasauer et al., 2013; Kennedy et al., 2003; Salama et al., 2013).

Iron oolites are characteristic Fe-rich condensed sediments of Middle Jurassic age, and are widely distributed across Europe (e.g. Collin et al., 2005; Gehring, 1989). Iron oolites mainly represent condensed sequences, with low detrital sediment supply and low net sedimentation, that have formed in a shallow-marine setting (e.g. Burkhalter, 1995; Clement et al., 2020; Dollfus, 1961; Föllmi, 2016; Heikoop et al., 1996). Iron ooid formation and ooid sedimentation may geographically coincide, but sedimentary redistribution of iron ooids has also been suggested to occur (autochthonous and allochthonous iron oolites, respectively; e.g. Bhattacharyya & Kakimoto, 1982; Brunner, 1999; Harder, 1978; Siehl & Thein, 1989). Iron ooid formation today is restricted to isolated environments, so that it remains unclear how Fe-rich ooids could form pervasively in the Jurassic. Modern iron ooids are known from volcanic settings, in Mahengetang, Indonesia (less than 4.5 ka; Heikoop et al., 1996), and Panarea Island, Italy (actively forming; Di Bella et al., 2019), suggesting a hydrothermal Fe source for both deposits. No evidence for microbially-mediated Fe cycling has been found for these modern iron ooids but it cannot be excluded.

In this study we seek to test the two aforementioned hypotheses for the origin of Fe in Middle to lowermost Upper Jurassic iron-rich sedimentary rocks. We use a variety of geochemical tools, including radiogenic Nd (<sup>143</sup>Nd/<sup>144</sup>Nd), Sr (<sup>87</sup>Sr/<sup>86</sup>Sr) and stable Fe isotope variations ( $\delta^{56}$ Fe), to investigate the origin of Fe in Tethyan iron oolites and their ooid constituents. Contrasting with stable Fe isotope compositions, radiogenic Nd and Sr composition can provide robust information on fluid sources involved in the formation of marine sedimentary phases.

# 2 Studied iron oolites

Three iron oolite dominated condensed successions of Middle to earliest Late Jurassic age were studied, deriving from two different paleo-depositional realms: the iron oolites from Herznach (Herznach Member, Ifenthal Formation) were deposited on the Jura platform, while the Blegi Iron Oolite from Windgällen (Reischiben Formation) and the Planplatte Iron Oolite from Erzegg (Erzegg Formation) formed on the northern Tethyan shelf of the Helvetics (Fig. 1). Detailed information about the sampling locations is given in Table 1.

#### 2.1 Iron oolites from Herznach

The Herznach region lies at the southern margin of the Tabular Jura Mountains in northwestern Switzerland (Diebold et al., 2005, 2006). Post-depositional burial temperatures prevailing in the area barely exceeded 100 °C (Frei, 1952; Gehring & Heller, 1989; Mazurek et al., 2006). Higher burial temperatures of 110-120 °C were recently reported for Late Jurassic to Early Cretaceous dolomite veins (Looser et al., 2019), but whether these temperatures affected the Middle to lowermost Upper Jurassic iron oolites remains unclear. The Middle Jurassic succession in the Jura Mountains consists of several formations containing condensed iron oolite horizons (e.g. Bitterli-Dreher, 2012; Burkhalter, 1996). The studied iron oolite horizons of the Sissach Member (Passwang Formation) and of the Herznach Member (Ifenthal Formation) both represent the top of a shallowing upward succession (e.g. Bitterli-Dreher, 2012; Bläsi, 1987; Burkhalter, 1996; Gygi, 2000; Fig. 2). The deposition of the lower Passwang Formation (Early Aalenian to Early Bajocian) was influenced by sea level fluctuations and subsidence, which resulted in the formation of siliciclastic and carbonate rocks, and iron oolite horizons at the top of most sequences (Burkhalter, 1996). The Sissach Member has a thickness of 5-15 m that accumulated in less than 3 Ma (Early to Middle Aalenian; Burkhalter, 1996; Diebold et al., 2006) and is considered a condensed sequence in the sense of Jenkyns (1971).

The iron oolite of the Herznach Member is a thin succession of iron oolitic marls and limestones of the Ifenthal Formation (e.g. Bitterli-Dreher, 2012; Gygi, 2000; Fig. 2). Syn-sedimentary tectonics resulted in a seafloor



**Fig. 1** Tectonic map of Switzerland with the three studied iron oolite deposits indicated by white stars. H = Herznach, WG = Windgällen and EE = Erzegg. Sample coordinates are given in Table 1. Modified after Federal Office of Topography swisstopo (2005)

Sample No	Coordinates (CH1903 +/ LV95)	Lithostratigraphy	Stage / Age	Age (Ma) Gradstein et al. ( <mark>2012</mark> )	Age (Ma) Gradstein et al. (2020)
H1	2'643'884/1'258'595	Herznach Member (Ifenthal Fm)	E. Cl.–E. Oxf	164.79–160.84	163.67-159.10
H2	2'645'223/1'260'076	Sissach Member (Passwang Fm)	E. Aal.–M. Aal	173.14-170.83	173.70-171.50
WG1	2′699′640/1′183′840	Blegi Iron Oolite (Reischiben Fm)	L. Bj.–E. Cl	169.45-164.63	169.74–163.47
WG3	2'698'953/1'183'704	Blegi Iron Oolite (Reischiben Fm)	L. Bj.–E. Cl	169.45-164.63	169.74–163.47
EE-P4 to EE-P33	2′665′372/1′178′988	Planplatte Iron Oolite (Erzegg Fm)	E. CI.–M. CI	166.07-163.97	165.29–162.65

Table 1 Overview of the investigated iron oolite samples from Herznach (H), Windgällen (WG) and the Erzegg profile (EE-P)

The lithostratigraphy indicates the mappable units given in the geological maps

Fm Formation, E. Early, M. Middle, L. Late, Cl. Callovian, Oxf. Oxfordian, Aal. Aalenian, Bj. Bajocian. All ages used in this study are after Gradstein et al. (2012)



**Fig. 2** Stratigraphic profile of the eastern Jura Mountains and the autochthonous sediment cover of the Aar Massif. Formation names in bold indicate the studied iron oolite deposits. The Herznach area corresponds to the eastern facies type of the Jura Mountains; the Windgällen area to the northern facies type (autochthonous), and the Erzegg area to the intermediate facies type of the Axen nappe. The sediment thickness increases towards more southerly paleopositions in the Aar Massif, which is most likely due to increased subsidence in southern regions (Gisler et al., 2020). Modified after Diebold et al. (2005, 2006), Gisler and Spillmann (2011), Gisler et al. (2020), Wohlwend et al. (2022)

structure characterized by oceanic highs and lows (Bitterli, 1977; Gygi, 2000). The rich ammonite fauna suggests a late Early Callovian to late Early Oxfordian

age for the member (e.g. Bitterli-Dreher, 2012; Gygi, 1981; Gygi & Marchand, 1982; Jeannet, 1951). With a maximum thickness of 5–6 m (Bitterli-Dreher, 2012),

which accumulated in around 4 million years, the Herznach Member is also a condensed sequence. Iron has been suggested to derive from lateritic weathering of adjacent continental areas (Gygi, 1981), and has been suggested to have been mobilized and locally enriched by microbial activity (Gehring, 1985; Glasauer et al., 2013). Remnants of microbial iron enrichment processes within the ooids are documented in cortical layering of ramified µm-sized structures of biological origin (Figs. 3A and B). The iron oolites from Herznach consist of poorly sorted iron ooids, which are often shattered and act as nucleii for other ooids, indicating that this process occurred during the formation of the iron ooids. Ooids with a nucleus made of either tiny quartz crystals or calcite fragments are also found (Bühler, 1986). The various cores are surrounded by concentric layers of limonite or chamosite (Bühler, 1986), goethite and apatite (e.g. Burkhalter, 1995; Gehring, 1985, 1986a) or limonite and hematite (Bodmer, 1978). Only small amounts of pyrite have been found (0.1-0.3%; Bühler, 1986). Other frequently found minerals in the iron oolite deposits from Herznach are, in order of decreasing abundance, calcite, celestine, dolomite, gypsum and sphalerite (Frei, 1952).

#### 2.2 Blegi Iron Oolite from Windgällen

The Blegi Iron Oolite from Windgällen is a subordinate member of the Reischiben Formation (Brückner & Zbinden, 1987; Hantke and Brückner, 2011). It is composed of chamosite ooids embedded in a micritic limestone matrix (Déverin, 1945; Dollfus, 1965; Hänni, 1999). The iron oolite also contains clay detritus with areas of chamosite and pyrite crystals, as well as some fossils such as ammonites, belemnoids, bivalves, small gastropods, foraminifera and bryozoa (Déverin, 1945). The age of the Blegi Iron Oolite has been constrained using the ammonite fauna, which suggests stratigraphic ages of Late Bajocian to Early Callovian (e.g. Dollfus, 1961). The Blegi Iron Oolite has a maximal thickness of 3.5 m, accumulated over about 5 Myr (Dollfus, 1965), and is therefore also a condensed sequence. Despite its small thickness, the Blegi Iron Oolite is prominent in the Windgällen area (Fig. 3E). The deposition of the sediments containing the Blegi Iron Oolite was controlled by syn-sedimentary tectonics (Dollfus, 1965; Trümpy, 1949; Ziegler, 1993). In Early Jurassic times, the Windgällen depositional site was located in a continental setting on the Alemannic Land, the Windgällen Ridge (Trümpy, 1949). Marine transgression affected the Windgällen Ridge only in Late Bajocian times, which is expressed in the absence of the entire Triassic, and Lower Jurassic strata in the region (e.g. Dollfus, 1965; Fig. 2). The iron ooids formed on the flooded Windgällen Ridge, a pelagic rise, at depths greater than the wave base and therefore in a tranquil setting (e.g. Kugler, 1987). Temperature and pressure conditions in the Windgällen region exceeded those at Herznach, with maximal temperatures of 305-410 °C and pressures of  $2.1 \pm 0.7$  kbar (Schenker, 1980, 1986). Intense two-phase folding related to the Windgällen anticline resulted in ooid deformation and new mineral growth, including the decomposition of chamosite ooids and the formation of new plagioclase, quartz and magnetite (Heim, 1878; Déverin, 1945; Baker, 1964; Tan, 1976; Röthlisberger, 1990; Burkhard, 1999; Hantke and Brückner, 2011; Fig. 3C).

## 2.3 Planplatte Iron Oolite from Erzegg

The Upper Bajocian to Lower Oxfordian Erzegg Formation consists of a thick succession of marl and clay schists (Brunner, 1999; Gisler et al., 2020; Staeger et al., 2020; Tröhler, 1966; Fig. 4). The strongly Fe-impregnated marls show an interval with rare, laterally discontinuous intercalations of iron oolites (Brunner, 1999), the informally-named Planplatte Iron Oolite of Early to Middle Callovian age (Gisler et al., 2020; Tröhler, 1966). The Planplatte Iron Oolite is a few meters thick and crops out over a length of about 6 km, extending from the Erzegg in the east, over the Balmeregghorn, to the Planplatte in the west (Brunner, 1999; Tröhler, 1966; Fig. 3F). The iron ooids initially likely formed in a shallow-marine setting, where iron enrichment may have occurred in a reducing milieu at the sediment-water interface (Brunner, 1999). Iron ooids formed in slightly agitated waters in a clay-rich sediment. Occasional storm events shattered the iron ooids and transported them to the south into deeper and more argillaceous environments. Those iron oolitic claystones were later transformed into the schists of the Erzegg Formation (Brunner, 1999). This allochthonous origin of iron ooids of the Planplatte Iron Oolite (Brunner, 1999; Tröhler, 1966) contrasts with the autochthonous iron ooid formation of the Blegi Iron Oolite.

Typical minerals found in the Planplatte Iron Oolite are goethite, chamosite, calcite, clays, hematite, magnetite, siderite, dolomite, quartz, pyrite and muscovite (Tröhler, 1966). The core of the iron ooids are either broken ooids, several ooids (polyooid) or chamositic fragments of echinoderms (Brunner, 1999; Tröhler, 1966; Fig. 3D). The ooids themselves consist of goethite or chamosite (Brunner, 1999; Tröhler, 1966), but apatite has also been reported in the ooid cortex (Brunner, 1999). The clay minerals are believed to be mainly of detrital origin, but diagenetic formation is also possible (Tröhler, 1966). Angular to sub-rounded quartz grains in the matrix support the concept of detrital input and proximal sedimentation. The Erzegg area has been less affected by Alpine



**Fig. 3 A** Principal component map and **B** Fe/C-ratio map of iron oolite sample from Herznach (H1), showing concentric layering of the iron ooid. **C** Magnetite cubes growing at the expense of flattened green chamositic iron ooids related to the Windgällen folding events (WG6). **D** Poorly sorted iron ooids with broken ooid fragments or polyooid as ooid nuclei document ooid transportation processes in the iron oolites from Erzegg (EE-P1). **E** View from Unteres Furggeli to the eastern Windgällen region. The black Blegi Iron Oolite band (marked red) is clearly visible in the field. **F** Sampled profile at Erzegg, with interlayers of iron oolite and marks with clay schists (see profile in Fig. 4)



Fig. 4 Schematic sampled stratigraphic profile at Erzegg, showing the succession of clay schists and iron oolite with marl layers, elemental concentrations, and isotope compositions of the reductive leachates of iron oolites (for data see Tables 2, 4)

metamorphism than the Windgällen, with maximum temperature conditions of 250 °C at pressures of about 2 kbar (Berger et al., 2017; Frey et al., 1980; Herwegh et al., 2017).

# 3 Sampling and methods

We sampled two iron oolite horizons from Herznach (Herznach Member and Sissach Member), one from Windgällen (Blegi Iron Oolite) and several horizons of a detailed stratigraphic section of the Erzegg Formation at Erzegg (Table 1).

Neodymium, strontium and iron isotope compositions were measured on leached iron oolite samples from Herznach, Windgällen and Erzegg. Representative rock samples of each outcrop were collected and sawn into small blocks, avoiding weathered or fractured material and targeting representative portions in terms of ooid abundance. Appropriate rock cubes were hydraulically crushed and subsequently ground in an agate mill. These powders were reductively leached in the clean lab facilities at ETH Zürich, targeting authigenic sedimentary Fe-phases (Blaser et al., 2016). Rock powders (40–60 mg) were reacted with 5 ml of 1.5% acetic acid, 0.005 M hydroxylamine hydrochloride and 0.003 M EDTA (buffered to pH~4 with ammonia) for 30 min on a vortex shaker. After centrifugation, 4 ml of the supernatant leach solution was pipetted off and dried down. The leachates were subsequently fluxed with 1 ml of concentrated HNO<sub>3</sub> for at least 12 h. Sample stock solutions were prepared in 2 M HNO<sub>3</sub> or 6 M HCl.

Elemental concentrations in the leachates were measured using a Thermo-Fisher Element XR ICP-MS at ETH Zürich, as described by Vance et al. (2016). Dilute stock solutions were run in 2% HNO<sub>3</sub> doped with 1 ppb In for internal normalization. Concentrations were calculated relative to an in-house standard. Two secondary standards were run to assess precision and accuracy of the reported concentrations: NRC Canada river standard SLRS-6, and the USGS shale standard SGR-1. Accuracy of the reported concentration data

Sample	Ca (wt.%)	Fe (wt.%)	AI (ppm)	Nd (ppm)	Sr (ppm)	Ca/Fe (molar)
H1	12.9	0.30	201	3.11	72.7	60
H2	28.8	1.02	120	7.37	476	39
WG1	5.04	2.67	146	1.88	55.7	2.6
WG3	15.4	1.20	109	4.53	257	18
EE-P4	0.18	0.19	677	2.18	13.8	1.3
EE-P6	10.9	1.10	340	3.66	460	14
EE-P7	3.52	1.08	353	2.12	278	4.6
EE-P9	3.67	2.01	352	3.11	124	2.5
EE-P12	15.2	1.09	484	5.20	370	19
EE-P18	7.17	1.10	226	3.37	220	9.0
EE-P20	9.87	1.32	515	3.91	283	11
EE-P22	11.6	0.96	369	4.50	350	17
EE-P23	0.95	0.17	1150	1.61	21.1	7.9
EE-P26	10.8	0.85	270	5.82	216	18
EE-P28	14.0	1.60	448	5.53	313	12
EE-P30	14.0	0.29	649	6.02	314	67
EE-P31	16.4	0.56	1264	8.44	388	40
EE-P32	8.34	0.27	954	3.74	203	43
EE-P33	12.0	0.28	847	6.61	276	61

 Table 2
 Elemental concentrations of the reductive leachates of iron oolites

Concentrations are given as leached element per mass of original sample prior to leaching. Concentrations are from Element runs, except for isotope dilution-based Nd concentrations (Sect. 3). Measurement uncertainty corresponds to 5% for Ca, Fe, Al and Sr and to 1% for Nd (1 SD)

(versus certified or literature values) ranges between 2 and 13%, precision is better than 5% (1 SD, Table 2).

Based on Nd concentrations from the Element runs, aliquots of the leachates were spiked with appropriate amounts of a mixed  $^{149}$ Sm/ $^{150}$ Nd spike. Strontium and rare earth elements (REE) were separated from matrix elements and each other on cation resin (AG 50W-X8, 200–400 mesh, 1 ml resin bed). Neodymium and Sm were subsequently isolated from each other and all other REEs on LN spec resin in dilute HCl (50–100 µm, 0.3 ml resin bed; Pin & Zalduegui, 1997). Strontium was further purified on Sr spec resin (50–100 µm, 0.1 ml resin bed; Deniel & Pin, 2001).

Iron isotope compositions were obtained for a selection of seven sample leachates of the three studied iron oolite lithologies (Table 4). A sample aliquot containing 1 µg of Fe was spiked with a  $^{57}$ Fe- $^{58}$ Fe double spike (to achieve a sample/spike ratio of ~ 1) and processed through an anion column containing AG-MP-1M resin. The matrix and Cu were eluted in 7 M HCl with trace peroxide, prior to the elution of Fe in 1 M HCl. For all isotope measurements, the purified elements were fluxed in 14.5 M HNO<sub>3</sub> with 30% hydrogen peroxide (ratio 9:1, 1 ml solution) to oxidize organic compounds deriving from the resins. Iron procedural blanks (< 0.1 ng) were insignificant compared to processed sample Fe. Neodymium, Sr and Fe isotopes, as well as spiked Sm isotope ratios, were measured individually on a Thermo-Fisher Neptune Plus MC-ICP-MS. Instrumental mass bias correction followed Vance and Thirlwall (2002) for Nd and Thirlwall (1991) for Sr, using a  ${}^{86}$ Sr/ ${}^{88}$ Sr ratio of 0.1194. Neodymium and Sr isotope compositions were renormalized to the accepted literature values of La Jolla and NIST SRM 987 (Thirlwall, 1991). Repeated standard measurements during each session yielded external error estimates < 15 ppm for  ${}^{143}$ Nd/ ${}^{144}$ Nd (2 SD) and < 14 ppm for  ${}^{87}$ Sr/ ${}^{88}$ Sr (2 SD). Samples were mostly run at similar concentrations as standards, yielding similar internal errors for both (Tables 3 and 4). Procedural blanks were < 30 pg for Nd and Sr and < 10 pg for Sm and were negligible compared to sample sizes.

Measured radiogenic Nd and Sr isotope compositions were corrected for ingrowth of <sup>143</sup>Nd and <sup>87</sup>Sr, respectively, since their time of formation (e.g. Faure & Mensing, 2005, and references therein) using biostratigraphic age constraints (see Sect. 2). The age uncertainty of the studied iron oolites has little effect on these corrections, less than the uncertainty on the measurements. Therefore, an intermediate age was used for all samples at one location (Tables 3 and 4). Age corrections on <sup>143</sup>Nd used Sm/Nd ratios from isotope dilution, that are associated with uncertainties < 1‰. The uncertainty on Rb/Sr ratios measured on the Element was conservatively

Sample	Age (Ma)	<sup>87</sup> Sr/ <sup>86</sup> Sr (mea	sured) 2 SEM	<sup>87</sup> Rb/ <sup>86</sup> Sr	<sup>87</sup> Sr/ <sup>86</sup> Sr (initial)	2 SD
H1	163	0.707336	0.000010	0.0077	0.707318	0.000013
H2	172	0.707266	0.000011	0.0017	0.707262	0.000013
WG1	167	0.712245	0.000012	0.0036	0.712237	0.000013
WG3	167	0.711407	0.000010	0.0018	0.711402	0.000013
EE-P6	165	0.709685	0.000010	0.0015	0.709682	0.000013
EE-P9	165	0.709657	0.000008	0.0600	0.709518	0.000019
EE-P12	165	0.709360	0.000009	0.0005	0.709359	0.000013
EE-P18	165	0.709722	0.000010	0.0003	0.709721	0.000013
EE-P20	165	0.709486	0.000009	0.0003	0.709486	0.000013
EE-P22	165	0.709700	0.000009	0.0002	0.709700	0.000013
EE-P26	165	0.709159	0.000010	0.0006	0.709158	0.000013
EE-P28	165	0.709521	0.000010	0.0003	0.709520	0.000013
EE-P30	165	0.709093	0.000009	0.0040	0.709084	0.000013
EE-P31	165	0.709558	0.000010	0.0122	0.709530	0.000013
EE-P32	165	0.709320	0.000010	0.0392	0.709229	0.000016
EE-P33	165	0.709914	0.000010	0.0304	0.709844	0.000015

 Table 3
 Strontium isotope compositions for the reductive leachates of iron oolites

Uncertainties on the measured isotope ratios reflect internal errors of the mass spectrometric runs (2 SEM). Uncertainty on the initial ratios combine uncertainties in the age correction with an external error estimate of the measured <sup>87</sup>Sr/<sup>86</sup>Sr ratios (Sect. 3)

Sample	Age (Ma)	<sup>143</sup> Nd/ <sup>144</sup> Nd (measured)	2 SEM	<sup>147</sup> Sm/ <sup>144</sup> Nd	εNd (initial)	2 SEM	δ <sup>56</sup> Fe (‰) (measured)	2 SEM (‰)	δ <sup>56</sup> Fe (‰) (authigenic)
H1	163	0.512222	0.000005	0.1625	- 7.41	0.11	- 1.44	0.03	- 1.49
H2	172	0.512114	0.000005	0.1156	- 8.44	0.09	- 0.94	0.05	- 0.95
WG1	167	0.512139	0.000005	0.1651	- 9.07	0.10	- 0.83	0.04	- 0.83
WG3	167	0.512134	0.000004	0.1343	- 8.50	0.08	- 0.82	0.03	- 0.82
EE-P4	165	0.512226	0.000004	0.1860	- 7.81	0.08	-	-	-
EE-P6	165	0.512179	0.000004	0.1416	- 7.80	0.08	-	-	-
EE-P7	165	0.512181	0.000005	0.1567	- 8.08	0.09	-	-	-
EE-P9	165	0.512186	0.000005	0.1474	- 7.79	0.09	- 0.71	0.04	- 0.72
EE-P12	165	0.512164	0.000004	0.1220	- 7.68	0.08	-	-	-
EE-P18	165	0.512183	0.000004	0.1481	- 7.86	0.08	- 0.65	0.04	- 0.66
EE-P20	165	0.512182	0.000004	0.1350	- 7.59	0.08	-	-	-
EE-P22	165	0.512173	0.000005	0.1511	- 8.11	0.09	-	-	-
EE-P23	165	0.512153	0.000005	0.2267	- 10.10	0.10	-	-	-
EE-P26	165	0.512197	0.000005	0.1311	- 7.23	0.10	-	-	-
EE-P28	165	0.512151	0.000004	0.1131	- 7.75	0.08	- 0.56	0.02	- 0.57
EE-P30	165	0.512147	0.000004	0.1438	- 8.47	0.08	-	_	-
EE-P31	165	0.512142	0.000005	0.1264	- 8.20	0.09	-	_	-
EE-P32	165	0.512117	0.000004	0.1327	- 8.81	0.08	-	-	-
EE-P33	165	0.512109	0.000004	0.1353	- 9.03	0.08	-	_	_

External errors on  $\epsilon$ Nd correspond to 0.15 epsilon-units and to < 1% for <sup>147</sup>Sm/<sup>144</sup>Nd (Sect. 3). Measured Fe isotope compositions are, for most samples, averages of two analyses, with an external uncertainty of  $\pm$  0.08‰ (2 SD, Sect. 3). Authigenic Fe isotope compositions reflect measured compositions corrected for detrital contributions (Sect. 4)

estimated to be 10%. This uncertainty was propagated through the age correction. Samples with elevated Rb/Sr were not processed for Sr isotope compositions. Maximal uncertainties on the age corrected  $^{87}$ Sr/ $^{86}$ Sr ratio correspond to less than 2  $\cdot$  10<sup>-5</sup>. Neodymium isotope compositions are reported in epsilon-notation relative to the Chondritic Uniform Reservoir (CHUR) at the time of sediment formation. The CHUR has a present-day  $^{143}$ Nd/ $^{144}$ Nd ratio of 0.512638 and  $^{147}$ Sm/ $^{144}$ Nd of 0.1967 (Jacobsen & Wasserburg, 1980).

Iron isotopic compositions,  $\delta^{56}$ Fe, are reported in delta notation relative to the standard IRMM-014, with an uncertainty of  $\pm$  0.08‰ (2 SD) based on the long-term reproducibility of secondary standard NIST-3126 (Sun et al., 2021).

# **4** Results

The analyzed iron oolites consist of authigenic iron and carbonate minerals as well as detrital silicates (see Sect. 2). Success in selectively leaching the key phases of interest, authigenic iron minerals, can be assessed using elemental concentrations (Table 2). More specifically, we use Fe to track the leaching of iron minerals, Ca for carbonates, and Al for detritus (see discussion in Sect. 5.1). The iron oolite leachates from Herznach, Windgällen and Erzegg yield molar Ca/Fe ratios between 1 and 67 and Fe/Al ratios between 1 and 88. Al/Nd ratios were usually below 900.

Initial Sr isotope ratios at the time of formation of the iron oolites are more radiogenic than contemporary seawater (Table 3), yielding 0.707290  $\pm$  0.000013 (n=2), 0.711820  $\pm$  0.000013 (n=2) and 0.709486  $\pm$  0.000014 (n=12) for Herznach, Windgällen and Erzegg, respectively. Initial  $\epsilon$ Nd range between - 9 and - 7 except for sample EE-P23 ( $\epsilon$ Nd=- 10.1). Measured  $\delta^{56}$ Fe are between - 1.5 and - 0.6‰ (Table 4). Authigenic Fe isotope compositions, corrected for detrital contributions, are marginally different from measured Fe isotope compositions (<0.05‰; Table 4). These detrital corrections are based on three assumptions: (i) all sample Al is detrital, (ii) detrital contributions have a Fe/Al ratio like the continental crust (mass ratio of ~0.481; Rudnick et al., 2003) and (iii) a crustal Fe isotope composition (0.1‰; Beard et al., 2003).

# 5 Discussion

## 5.1 Limitations of the leaching approach

The iron oolites are composed mainly of a carbonate fraction and of iron ooids. It is therefore possible that the carbonate and the ooids carry different radiogenic isotope signatures if, for instance, the carbonate reflects mostly marine carbonate shells, while the iron ooids formed during mixing of hydrothermal fluids and seawater. Such a formation process has been suggested for modern iron ooids at Panarea in the Tyrrhenian Sea (Di Bella et al., 2019). The reductive leach used here was applied prior to carbonate removal to avoid leaching detrital material (Blaser et al., 2016). Although this resulted in generally low Al/Nd ratios (<900 molar ratio, <170 mass ratio; see Sect. 5.3), it failed to isolate a strongly Fe-enriched phase given Ca/Fe ratios of 1–67 (Table 2; Fig. 5A). However,



**Fig. 5** Leached Nd isotope composition vs. leached **A** Ca/Fe and **B** Al/Nd ratios. The solid blue lines are linear regressions to the data with calculated coefficients of determination ( $r^2$ ). The linear regression model excluded the clay-dominated sample EE-P23. The dashed black line in **B** depicts the Al/Nd threshold of ~ 535 as an indication of authigenic Nd isotope signatures (Huang et al., 2021). The lack of correlation between  $\epsilon$ Nd and Ca/Fe suggests that Fe-rich sediment phases (e.g. goethite, chamosite) are not isotopically distinct from Ca-rich phases (e.g. calcite, dolomite). Similarly, the lack of correlation between  $\epsilon$ Nd and Al/Nd suggests that the authigenic phases extracted by leaching are not contaminated by detrital contributions

there is no correlation between leached Ca/Fe ratios and Nd isotope compositions (Fig. 5A). Thus, though isotope variations between Fe-rich and Ca-rich portions of the iron oolites cannot be completely ruled out, it seems rather unlikely.

# 5.2 Potential for metamorphic overprinting of strontium isotope compositions

Initial Sr isotope ratios at the time of formation of the iron oolites are more radiogenic than seawater at that time (Table 3; Fig. 6) yielding  $0.707290 \pm 0.000013$  (n=2),  $0.711820 \pm 0.000013$  (n=2) and  $0.709486 \pm 0.000014$  (n=12) for Herznach, Windgällen and Erzegg, respectively. These offsets are most likely explained either by: (i) Sr isotope variability in coastal seawater at that time; (ii) allochthonous iron ooid formation on land and later transportation to the depositional basin or; (iii) post-sedimentary overprinting of Sr isotope compositions due to metamorphic fluids.

Due to the long residence time of Sr in seawater of  $\sim 3$  Ma, significant isotopic variability is not expected



**Fig. 6** Reductively leached Sr isotope compositions of the Swiss iron oolites from Herznach, Windgällen and Erzegg compared to the Jurassic seawater Sr isotope curve (with a 95% confidence interval; Wierzbowski et al., 2017). Leached Sr isotopes are more radiogenic than contemporary seawater due to metamorphic overprint (Sect. 5.2). Error bars on ages reflect available age constraints (see Tables 1 and 3). Ages after Gradstein et al. (2012). *Aal.* Aalenian, *Bj.* Bajocian, *Bt.* Bathonian, *Cl.* Callovian, *Oxf.* Oxfordian, *Kimm.* Kimmeridgian, *Tith.* Tithonian, *Berri.* Berriasian

(e.g. Hodell et al., 1990). Previous work has shown that the Sr isotope signature in coastal areas can deviate from the global open marine signature by up to about 0.00025 (e.g. El Meknassi et al., 2018, 2020; Huang et al., 2011; Ingram & Sloan, 1992). Such local offsets may be related to river runoff, which is isotopically highly variable (e.g. Pearce et al., 2015), or, although this seems less likely based on global Sr budgets (e.g. Paytan et al., 2021), to groundwater or diagenetic Sr fluxes. The offsets from contemporary seawater observed at Windgällen and Erzegg are, however, much larger, on the order of  $\sim 0.002$ to ~ 0.005. Such offsets could only be explained if the sediments were formed in highly restricted basins, which is inconsistent with paleogeographic reconstructions (e.g. Scotese & Schettino, 2017; van Hinsbergen et al., 2020; Ziegler, 1993).

A continental origin of iron ooids has previously been proposed for the Paris Basin, where iron ooids may have been derived from latosols (Siehl & Thein, 1989). In Swiss iron oolites, the presence of fragmented iron ooids also imply transportation processes (e.g. Brunner, 1999; Gehring, 1989; Fig. 3D). However, such processes can also occur in marine environments affected by strong ocean currents (e.g. Gehring, 1989; Rais et al., 2007). Although a continental origin of the iron ooids cannot be fully precluded, it seems unlikely. Therefore, the radiogenic Sr isotope compositions of the studied Swiss iron oolites point to a metamorphic, fluid-related overprint, although marine Sr isotope signatures have been measured in some marine sediments in the region (Doldenhorn; Burkhard & Kerrich, 1990).

The studied Swiss iron oolites experienced different metamorphic pressures and temperatures (see Sect. 2). The Sr isotope system is known to be more susceptible to metamorphic alteration than Nd (Hradetzky & Lippolt, 1993; Jenkin et al., 2001; Schaltegger et al., 1994). Metamorphic fluids, usually radiogenic in Sr, are preferentially focused along thrusts where they affect adjacent rocks within a few meters to a few hundred meters (e.g. Burkhard & Kerrich, 1990; Burkhard et al., 1992; Hradetzky & Lippolt, 1993; Huon et al., 1994; Kirschner et al., 1999, 2003). Remnants of metamorphic fluids are documented from fluid inclusion studies for the Erzegg and Windgällen (e.g. Miron et al., 2013; Mullis et al., 1994). Whether the chamosite decomposition in the Windgällen was associated with metamorphic fluids is unclear (Déverin, 1945). Thrusting is documented in the vicinity of the iron oolites from Herznach, Windgällen and Erzegg (Brückner & Zbinden, 1987; Diebold et al., 2005, 2006; Gisler et al., 2020; Hantke and Brückner, 2011; Staeger et al., 2020), so that Sr isotope compositions have likely been altered.



Fig. 7 Authigenic Nd isotope composition of the iron oolites from Herznach, Windgällen and Erzegg compared to the Tethyan Jurassic seawater Nd isotope curve (Dera et al., 2015) and proximal basins. Leached compositions are consistent with contemporary seawater, providing no evidence for the involvement of hydrothermal fluids. Error bars on ages reflect available age constraints (see Tables 1 and 4). Ages after Gradstein et al. (2012). *Hett.* Hettangian, *Sine.* Sinemurian, *Pliensb.* Pliensbachian, *Aal.* Aalenian, *Bj.* Bajocian, *Bt.* Bathonian, *Cl.* Callovian, *Oxf.* Oxfordian, *Kimm.* Kimmeridgian, *Tith.* Tithonian

# 5.3 Neodymium isotope compositions

There is no universally applicable approach for the identification of significant detrital contributions to authigenic Nd, given the diversity of depositional environments and diagenetic processes. A threshold ratio for unaffected authigenic Nd isotope signatures has been suggested for Quaternary marine sediments corresponding to a molar Al/Nd ratio of ~ 535 (mass ratio of 100; Huang et al., 2021). Apart from a few samples (EE-P4, EE-P23 and EE-P32), the leachates show Al/Nd ratios close to this threshold (Fig. 5B). Sample EE-P23 is a clay-dominated sample, which produced a distinctly high Al/Nd ratio and a low  $\varepsilon$ Nd of - 10.1, implying that this signature may not be fully authigenic. All further Nd isotope compositions of the iron oolites from Herznach, Windgällen and Erzegg are similar and range between - 9 and - 7, likely reflecting contemporary seawater (Fig. 7; Dera et al., 2015). In addition, leached Nd isotopes are not correlated with Al/Nd ratios, suggesting that detrital Nd does not affect the authigenic signatures (Fig. 5B).

These low Nd isotope compositions do not argue for the involvement of hydrothermal fluids in the formation of iron oolites. Indeed, they are similar to previous reconstructions of seawater signatures in the Middle Jurassic Tethys (Dera et al., 2015). Slight variations in ɛNd between samples are consistent with generally open depositional basins and can, for instance, be explained by isotopically different fluvial Nd inputs (Dera et al., 2015). Comparable conclusions have already been drawn for the Late Jurassic Swabian epicontinental basin, opening southward into the Helvetic facies belt (e.g. Olivier & Boyet, 2006; Stille & Fischer, 1990), and for Early Jurassic sediments of the Paris Basin (e.g. Dera et al., 2009; Fig. 7). Such an open basin model also suggests that the seawater was oxygenated, as is also documented by the rich fauna in iron oolites (e.g. Dollfus, 1961; Gygi, 2000). Overall, the authigenic neodymium isotope signatures in the studied iron oolites are crustal, and do not provide evidence for significant hydrothermal contributions of Nd. A riverine source of iron, derived from weathering of the adjacent hinterland, has also been proposed by other studies (e.g. Baioumy et al., 2017; Chowns, 1966; Gehring, 1986a; Li et al., 2021).

#### 5.4 Iron isotope compositions

Iron supplied with the sediment load of rivers must be locally enriched to form Fe-rich condensed sequences. Elevated organic matter contents are often associated with iron-rich sediments (e.g. Lalonde et al., 2012), supporting the concept that Fe reduction may occur in restricted environments with oxygen depletion. Gehring (1985) proposed an iron ooid formation model in the vicinity of organic matter. Such an involvement of microbes in iron cycling should be reflected in iron isotope compositions. Microbially-mediated Fe(III) reduction preferentially utilizes the light <sup>54</sup>Fe compared to <sup>56</sup>Fe (Beard et al., 1999; Ellwood et al., 2015), resulting in low  $\delta^{56}$ Fe values, usually lower than average crustal rocks with  $\delta^{56}$ Fe of 0.1‰ (Beard et al., 2003).

The light  $\delta^{56}$ Fe isotope values, between -1.49 and -0.57%, of the iron oolites are consistent with the involvement of microbial processes. Based on Nd isotopes, this Fe is likely supplied by rivers. Detrital Fe transported by rivers is expected to have an Fe isotope signature similar to that of average crustal rocks (0.1‰; Beard et al., 2003). Reductive dissolution of sedimentary Fe involving microbial processes would release isotopically light Fe to pore waters, subsequently incorporated into iron ooids. However, it remains unclear whether a locally reductive milieu is a general feature of the "ooid

factory" or if reduction is restricted to the iron ooids themselves, since remnants of microbial structures inside the ooids are ambiguous (Burkhalter, 1995; Dahanayake & Krumbein, 1986; Gehring, 1986b).

#### 5.5 Iron oolite formation model

Sedimentary condensation tends to occur on topographic highs and in an active hydrodynamic regime, where the accumulation rate is low due to processes such as winnowing, erosion, bypassing and reworking (Föllmi, 2016; Gómez & Fernández-López, 1994; Jenkyns, 1971; Fig. 8). Both conditions are relevant for the three iron oolite deposits in Switzerland. A key factor controlling the formation of iron oolites was likely syn-sedimentary tectonics, which



Fig. 8 Schematic sketch for the formation of ancient and modern iron ooids. The formation of Jurassic iron ooids results from the coupling of sedimentary organic carbon enrichment (due to the delivery of carbon associated with detrital clays), microbially-mediated Fe cycling of sediment-sourced Fe, and sedimentary condensation processes related to agitated hydrodynamics. The variation in authigenic ɛNd between - 9 and - 7 of the studied iron oolites likely reflects variations in the weathered continental source areas. In contrast to ancient iron ooids, modern iron ooids form in a volcanic setting with intense submarine hydrothermal activity (Di Bella et al., 2019). Average continental and hydrothermal  $\delta^{56}$ Fe, the latter including mid-ocean ridge hydrothermal fluids, are from Beard et al. (2003). Continental ENd (- 10 to - 8) represents a range for four modern Swiss rivers (Rickli et al., 2013), which is similar to the average of detrital sedimentary rocks aged 0–200 Ma ( $-10\pm8$ , 1 SD; compiled in Garçon, 2021) and averaged world river suspended loads ( $-11\pm4, 1$  SD; compiled in Garcon, 2021). Hydrothermal ENd represents averaged compositions of mid-ocean ridge basalts (±1 SD; compiled in Gale et al., 2013). Middle Jurassic Tethyan Nd isotope composition cover the temporal variations given in Dera et al. (2015)

resulted in the subsidence of the European Tethyan continental margin, forming submarine plateaus and basins (e.g. Bitterli, 1977; Trümpy, 1949, 1952; Ziegler, 1993). Such plateaus are influenced by strong ocean currents that sweep away sediments, thus decreasing the accumulation rate (e.g. Eberli et al., 2010; Fürsich, 1979; Heezen & Hollister, 1964). This condensation process is likely intensified by presumably little, but not zero, terrigenous sediment input. Temporarily less intense ocean currents favored the deposition of clay minerals at the depositional site (Reineck & Singh, 2012, and references therein), supporting the enrichment of organic carbon, since clay minerals are attractive sites for both iron and organic matter (e.g. Kennedy et al., 2002; Oades, 1988). That clay minerals are relevant for the formation of iron oolites is also supported by their presence in all studied Swiss iron oolites. The same processes are also documented in the Middle to Late Jurassic condensed sequences of the northern continental margin of the Iberian Basin (García-Frank et al., 2012), and in the Late Bajocian to Tithonian condensed Rosso Ammonitico Veronese of the southern continental margin formed on the Trento Plateau (Bernoulli et al., 1979; Martire, 1992, 1996; Préat et al., 2006).

Similarly, the only known place where iron ooids are actively forming today is in a dynamic shelf setting, northeast of Panarea Island in the Tyrrhenian Sea (Di Bella et al., 2019). Several paleoshorelines document sea level changes related to global sea-level fluctuations and long-term uplift at the depositional site (Chappell & Shackleton, 1986; Di Bella et al., 2019; Lucchi, 2009; Lucchi et al., 2007). Fluctuations in sea level shift the zones of deposition and erosion in marine settings, resulting in oscillating sediment accumulation and erosion (e.g. Lewis, 1973; Paulay & McEdward, 1990; Swift & Thorne, 1991). Though rapid sea-level variations can account for low accumulation rates, this seems unlikely to be the sole process for the formation of ancient iron oolites since their formation spans several Myrs (Rais et al., 2007). Our Fe isotope data suggest that microbially-mediated Fe cycling contributed to the formation of iron oolites, whilst Nd isotope compositions provide evidence against hydrothermally sourced Fe.

# 6 Conclusion

Iron-rich condensed sequences are characteristic of the Middle Jurassic Period, and formed during significant changes in plate configuration and climate (e.g. Dera et al., 2011; Rais et al., 2007; Ziegler, 1988). The formation of condensed sequences coincides with increased hydro-thermal activity and increased continental runoff related

to the breakup of Pangea (e.g. Halliday & Mitchell, 1984; Holz, 2015), suggesting that one of these processes could be the source of iron. The breakup of Pangea formed a peculiar submarine relief, with plateaus and basins controlled by syn-sedimentary tectonics (e.g. Trümpy, 1949; Ziegler, 1993). Iron ooids accumulated on the plateaus or highs, where low sedimentation and high erosion rates were influenced by ocean currents and partly by sea level fluctuations, leading to sedimentary condensation.

Leached authigenic Nd isotope compositions yield constraints on the origin of Fe in the studied iron oolite successions in Switzerland. The crustal Nd isotope signature of the iron oolites suggests that the involvement of hydrothermal fluids in their formation is unlikely, and that Fe is instead derived from detrital material supplied by rivers. This contrasts with modern iron ooids, where the source of Fe is presumably hydrothermal (Di Bella et al., 2019; Sturesson et al., 2000). This difference between the ancient and modern Fe source might suggest that either iron ooid formation switched from a crustal setting in the Middle Jurassic to a hydrothermal setting today, or that iron ooids can form in different environments.

Aqueous iron concentrations are generally extremely low in the ocean, but increase in reducing settings due to microbially-mediated Fe reduction, suggesting that microbial mediation is an important process to locally enrich iron. Signs of microbial iron enrichment processes are still preserved in the Middle to earliest Late Jurassic iron oolites and documented in negative Fe isotope signatures.

#### Abbreviations

Fe	Iron
Cu	Copper
Rb	Rubidium
Sr	Strontium
Nd	Neodymium
Sm	Samarium
REE	Rare earth elements
CHUR	Chondritic Uniform Reservoir
HNO3	Nitric acid
HCI	Hydrochloric acid
EDTA	Ethylenediaminetetraacetic acid
SEM	Scanning Electron Microscopy
(MC-)ICP-MS	(Multicollector) inductively coupled plasma mass
	spectroscopy
NRC Canada	National Research Council Canada
USGS	United States Geological Survey
NIST	National Institute of Standards and Technology
IRMM	Institute for Reference Materials and Measurements
Н	Herznach
WG	Windgällen
EE	Erzegg
Gr	Gross Ruchen
USM-I	Lower Freshwater Molasse I
E	Early
M	Middle
L	Late
U	Upper

Hett.	Hettangian
Sine.	Sinemurian
Pliensb.	Pliensbachian
Aal.	Aalenian
Bj.	Bajocian
Bt.	Bathonian
Cl.	Callovian
Oxf.	Oxfordian
Kimm.	Kimmeridgian
Tith.	Tithonian
Berri.	Berriasian
SD	Standard deviation
r <sup>2</sup>	Coefficient of determination

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#### Author contributions

SS, DV, HW, and SW conducted the field work and collected the samples. SS, JR, DV, and SW carried out the analyses. All authors discussed and interpreted the data. SS drafted the initial manuscript, all co-authors provided editorial support. All authors read and approved the final manuscript.

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All discussed new data generated during this study are included in this publication.

#### Declarations

# Ethics approval and consent to participate

Not applicable.

#### **Consent for publication**

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#### **Competing interests**

The authors declare that they have no competing interests.

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