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ARTICLE

New results of stable isotope and petrographic studies of Jurassic glendonites from Siberia

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ABSTRACT
We present the results of an optical microscopy, cathodoluminoscopy and isotopic study on nine glendonite concretions (calcite pseudomorphs replacing metastable hexahydrate ikaite) from Lower-Middle Jurassic sediments of Northeast Russia (Anabar Bay and Lena River region). Glendonite concretions are mainly found within Late Pliensbachian, Toarcian, Aalenian, Bajocian and Lower Bathonian clastic sediments, correlating to episodes of global climatic cooling as determined by independent paleoclimate proxy data. Stable carbon and oxygen isotopic values of glendonite concretions suggest that the primary source of carbon was derived from diagenetically altered organic matter, and the source of oxygen was from seawater. The secondary diagenetic cement is characterized by a significantly lighter $\delta^{18}O$ and significantly heavier $\delta^{13}C$ signature than the isotopic characteristics of the bulk rock glendonite concretion. This secondary diagenetic cement is thought to have precipitated rapidly during burial diagenesis and since it occupies a significant volume of the glendonite concretion, it has the potential to significantly influence the isotopic composition of bulk rock glendonites.

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Glendonites; stable isotopes; paleoclimate; diagenesis

Introduction

Glendonites, representing calcite pseudomorphs replacing metastable calcium carbonate hexahydrate (ikaite), typically form in cold-water marine and non-marine environments, ranging from Precambrian to recent in age. Some aspects concerning glendonite formation are still poorly understood: ionic composition of seawater (Stockmann et al. 2018), role of bacterial sulfate reduction during ikaite-glendonite transformations (Qu et al. 2017) or additional factors and their combinations (Hu et al. 2012; Zhou et al. 2015; Morales et al. 2017; Tollefsen et al. 2018), but their ubiquitous association with cold-water environments makes them useful as paleoclimate indicators. Irrespective of geological age and their host rocks, glendonites are characterized by specific petrographic features recognizable in thin section, including a number of successive calcite generations. Since the early 1970s, carbon and oxygen stable isotope values of glendonites from different localities and different ages were measured. The results of these studies have revealed a relatively wide range of $\delta^{13}C$ values in glendonites, ranging from +30 to +50 ‰ VPDB (Last et al. 2013; Vickers et al. 2018). Although it is often acknowledged that glendonite concretions crystallized in stages over a protracted period of time, technical difficulties in extracting samples from different calcite generations often make it difficult to analyze the isotopic compositions of successive generations separately. There are now a few research papers (e.g., Frank et al. 2008; Vickers et al. 2018) discussing the isotopic composition of calcite from both bulk rock concretions (i.e., the homogenized composition of all constituent calcite generations) and different generations of their constituent cement. In this study, we aim to further identify variations in carbon and oxygen isotopic composition between bulk rock glendonites and their secondary diagenetic cement.

Geological setting

Our study is based on material collected from Lower to Middle Jurassic sediments of Anabar Bay and Cape Kystatym in the northwestern part of the Siberia platform (Fig. 1). Lower-Middle Jurassic deposits of Anabar Bay consist of a relatively thick shallow marine succession (up to ~900–1000 m) of interbedded clay, silt and sand, with occasional carbonate bands (mainly concentrated in the Upper Pliensbachian and occasionally within the Bathonian) as well as carbonate concretions scattered throughout the succession (Fig. 2). All glendonite-bearing host rocks are characterized by low organic carbon contents, with glendonites absent from the organic-rich (Total organic carbon (TOC) up to ~3%) Lower Toarcian strata (Suan et al. 2011), both here and elsewhere. The studied succession is characterized by diverse assemblages of macro- and microfossils (Nikitenko et al. 2013), providing robust biostratigraphic control on the age of all glendonite-bearing units. In contrast with the Anabar Bay succession, the Jurassic succession exposed at Cape Kystatym is relatively (~200 m thick) and exposed along the banks of the Lena River (Kirina & Meledina 1974). A single glendonite-bearing sandstone bed has been identified here and dated as Late Bajocian (Kirina & Meledina 1974; see also Morales et al. 2017). In addition, glendonites of both Cape Kystatym and nearby Cape Khorongho (located on the opposite bank of the Lena River) are accompanied by the presence of numerous

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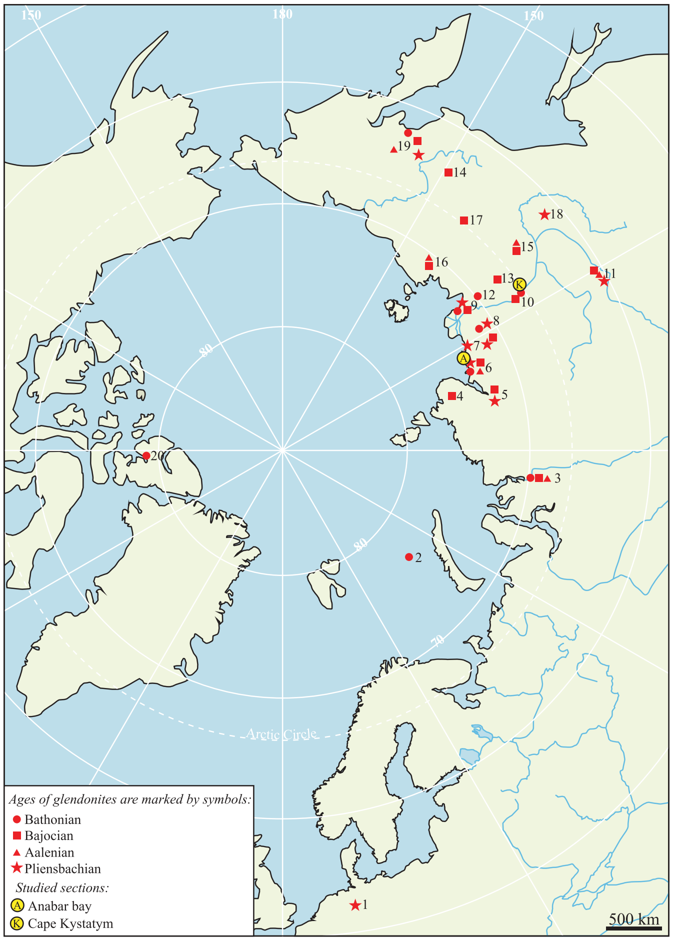

Figure 1. Map showing studied sections and distribution of glendonite concretions in the Northern Hemisphere. For locations and references see Attachment.

dropstones (Fig. 3). The sections along the banks of the Lena River are characterized by relatively rare occurrences of stratigraphically significant macrofossils (particularly ammonites and retroceramid bivalves), and a biostratigraphic zonation based on microfossils has not been developed from these sites to date.

Brief review of early and middle Jurassic climates of Siberia and glendonite distribution

As ikaite precipitation in modern-day natural environments occurs ubiquitously under relatively cold-water conditions, there is a general agreement that the occurrence of glendonites in the geological record is indicative of a cooling event. This is especially true for the Mesozoic, as deep-water glendonite occurrences (from abyssal paleodepths) of this age are unknown and all described Mesozoic glendonites are found in host rocks characterized by typical shallow marine shelf macrofaunal assemblages (Kaplan 1980). Based on paleobotanical and palynological evidence, northern Siberia was characterized by a humid and warm-temperate climate during the earliest Jurassic (Ilyina 1985). This generally warm climate was interrupted by a pronounced cooling event during the Late Pliensbachian, extensively studied in the European basins (Dera et al. 2011; Gómez et al. 2016), but also recognizable in the north of Siberia based on changes in macrofossil assemblages. These assemblage changes include the disappearance of Submediterranean and Subboreal benthonic bivalve, foraminiferal and ostracod taxa (Zakharov et al. 2006). Glendonites are widely distributed (Attachment, Fig. 1) in Late Pliensbachian shallow marine sediments across northern Siberia and Northeast Russia (Kaplan 1978; Morales et al. 2017; authors' data). Calcite pseudomorphs appear in outcrops along the banks of the Lena River (Sach 1976; Ivanovskaya 1967), Olenek River (Gusev 1950), Aldan River (Tuchkov 1962), Viluy River (Kirina 1966) and Viliga River (Sach 1976), as well as in outcrops in Anabar Bay (Suan et al. 2011) and Khatanga Bay (Voronov 1961). Stable oxygen isotope values from Late Pliensbachian bivalves also support relatively low seafloor paleotemperatures during this time.

By contrast, the beginning of the Toarcian is marked by a rapid warming event and marine transgression in northern Siberia (Zakharov et al. 2006). This warming can be clearly identified by changes in marine macrofossil and terrestrial floral assemblages, including the appearance of Subboreal and Submediterranean molluscan and foraminiferal taxa, and the appearance of thermophilic plant taxa (Ilyina 1985). Glendonites and dropstones have not been identified within Lower Toarcian sediments, not only from Siberia but anywhere in the World. The Early Toarcian warming event was relatively short, and the Late Toarcian is marked by the beginning of the next cooling event, initiated by isolation of the Arctic basin from the NW European basin due to North Sea lithospheric uplift (Korte et al. 2015). Faunal and floral assemblages of the Late Toarcian are dominated by Boreal taxa, and occasional glendonites reappear in this time interval (Nikitenko 2009; Rogov & Zakharov 2018).

During the Middle Jurassic, the climate of northern Siberia was relatively cold (Ilyina 1985; Zakharov 1994; Rogov & Zakharov 2018). Faunal and floral assemblages of this time were dominated by endemic taxa, although the Early Bajocian was characterized by the brief influx of warm temperate flora and fauna (Ilyina 1985; Meledina 1991), suggestive of a transient warming event. Climatic cooling during the Late Bajocian was marked by radical changes in fossil assemblages and a peak in glendonite abundance (Voronov 1961; Baibarodskih et al. 1968; Nikitenko 2009; Rogov & Zakharov 2010, 2018; Suan et al. 2011; Nikitenko et al. 2013 amongst others). The presence of ice-rafted dropstones of this age in the Lena River basin has been suggested by Tuchkov (1973), and numerous dropstones of this age were also found in the same region during this study (Fig. 3). Since the beginning of the Bathonian, gradual warming and an increase in aridity, marked by a significant increase in abundance of *Classopollis* pollen, occurred in southern Siberia (Vakhrameyev 1982) and also recognized in other areas (Hu et al. 2017). In northern Siberia, climatic warming was less significant or had a smaller impact on terrestrial flora, although the pollen species *Marattisporites, Lophotriletes torosus* Sach. et Iljina and *Classopollis* (Ilyina 1985) are also recorded there. The Bathonian is also characterized by a decrease in both glendonite abundance and pseudomorph size compared to the Bajocian (Kaplan 1978).

Boreal transgression occurred during the Late Bajocian and Callovian, but whilst the climate of southern Siberia remained warm and arid, there is evidence that the climate

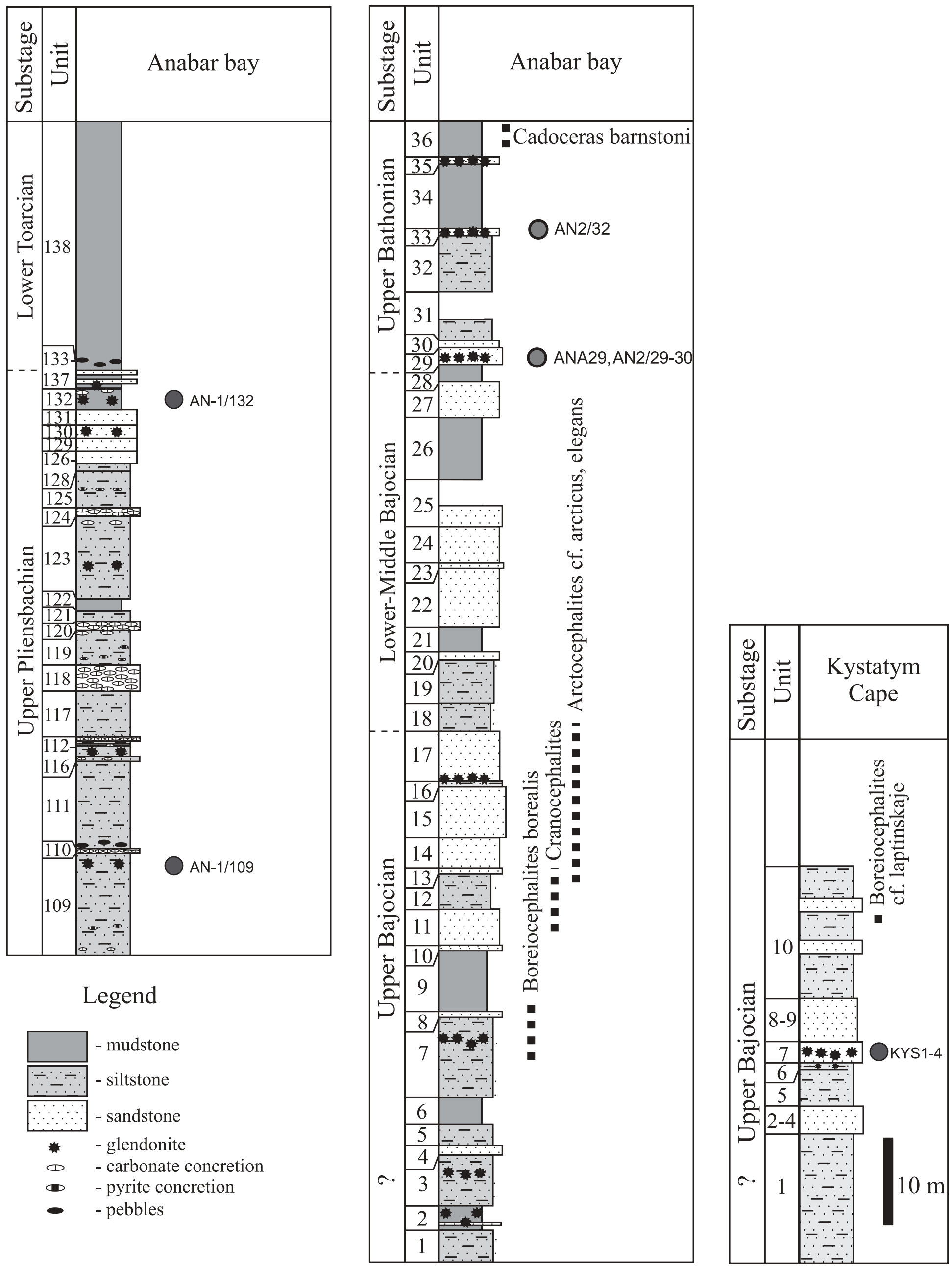

Figure 2. Stratigraphic logs of studied sections and samples settings.

Figure 3. Photos of dropstones in sandstones Cape Khorongho (Bathonian) section. Coin diameter is 20,5 mm.

of northern Siberia was relatively humid. This time period is characterized by the widespread abundance and dominance of *Classopollis* pollen across southern Siberia (Ilyina 1985). Glendonites become rare during the Callovian before disappearing from northern Siberia in the Oxfordian. The occurrence of rare glendonites despite climatic warming during the

Callovian has been attributed to increasing water depths associated with marine transgression in northern Siberia (Ilyina 1985; Levchuk 1985).

Materials and methods

Optical microscopy and cathodoluminoscopy were carried out on 8 polished uncovered thin sections of glendonite concretions on an Olympus BX-53 microscope. A cold cathodoluminescence (CL) apparatus CITL model Mk5-2 (Cambridge Instrument Technology Ltd., Cambridge, UK) was used in automatic regime at 10–15 k V, 354 µA, and residual vacuum of 0.003 mBar. 8 glendonite concretions were analyzed to find out their isotopical characteristics. To evaluate differences in isotopic composition of bulk rock concretions and their constituent cement (see Petrology of glendonite concretions), samples from host concretions (KYS-4a, AN2/29-30a) and infilling cement (KYS-4b, AN2/29-30b) were collected. Carbon and oxygen isotope analyses on carbonates were performed on the Thermoelectron system, including a Delta V Advantage Mass Spectrometer with Gas-Bench-II, in the Geological Institute of the Russian Academy of Sciences, Moscow. Isotopic results were calibrated against C-O-1 and NBS-19 standards reacted with H_3PO_4 at 50°C, with analytical precision for both $\delta^{18}O$ and $\delta^{13}C$ of ±0.2‰.

Results

Morphology and petrology of glendonite concretions

Upper pliensbachian (samples AN-1/132 and AN-1/109)

The glendonite concretion (Fig. 4A) occurs in clayey siltstone (see Fig. 3A in Suan et al. 2011), with an almost regular round shape and size up to 7 cm. The concretion consists of two generations of calcite. The first forms about 30% of the concretion, with an idiomorphic isometric shape and crystals up to 0.5 mm in diameter (C1, Fig. 5 A, B). Calcite crystals C1 are dark brown in color with concentric zonation in cross polarized light and are non-luminescent. The rest of the concretion comprises calcite cement with crystals up to 0.2 mm in diameter (C2 on Fig. 5 A, B), which are colorless in cross polarized light and exhibit a weak red luminescence. Thin wavy fractures cross-cut both C1 and C2 and are infilled by non-luminescent calcite (green arrow on Fig. 5 A, B).

Upper bajocian (samples KYS-1, KYS-2, KYS-3, KYS-4)

Glendonite concretions are up to 6–7 cm in diameter and have a slightly elongated shape (Fig. 4 B, C, D, E). The concretions are also composed of two generations of calcite. Calcite of the first generation (C1, Fig. 5 C, D, E) forms about 30% of the concretion and has almost irregular elongated colorless crystals that may be corroded (white arrows on Fig. 5 D) or transected (white arrows on Fig. 5 E) by calcite C2. The first generation of calcite exhibits

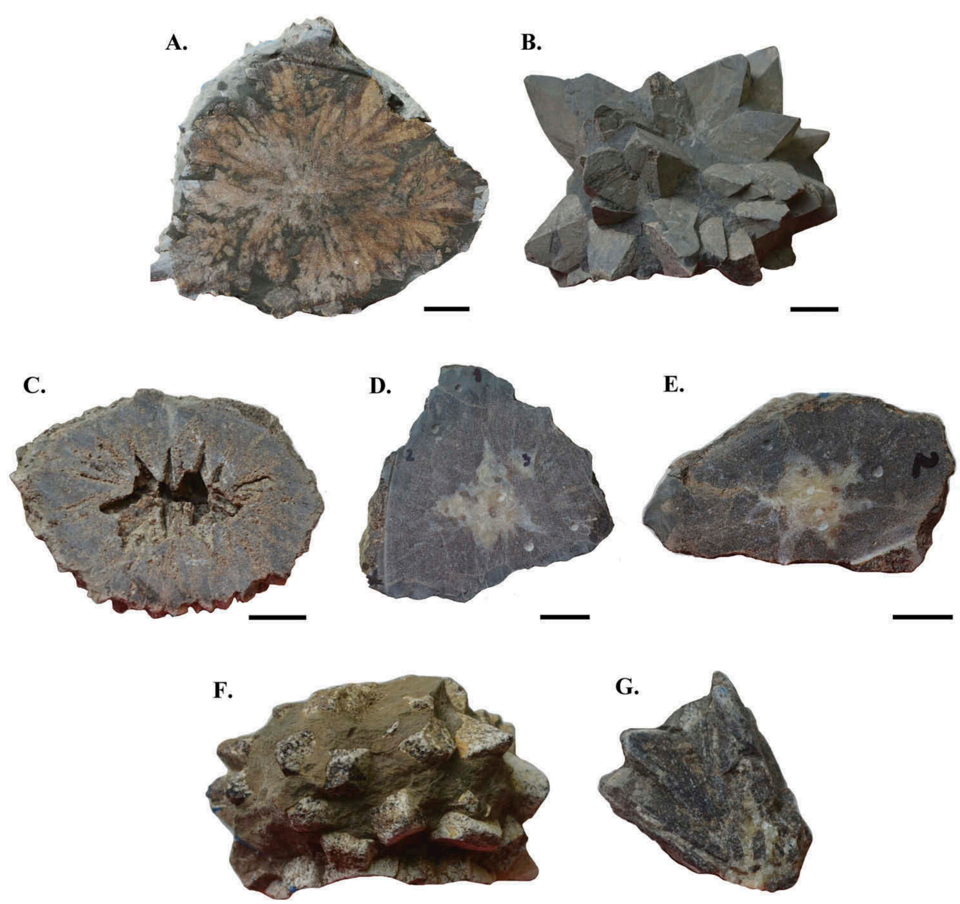

Figure 4. Photos of glendonite concretions: **A**. sample AN-1/109 (Upper Pleinsbachian), **B**. sample KYS-1 (Upper Bajocian), **C**. sample KYS-2 (Upper Bajocian), **D**. sample KYS-3 (Upper Bajocian), **E**. sample KYS-4 (Upper Bajocian), **F**. sample AN2/32 (Upper Bathonian), **G**. sample AN-2/29–30 (Bathonian). Length bar is 1 cm.

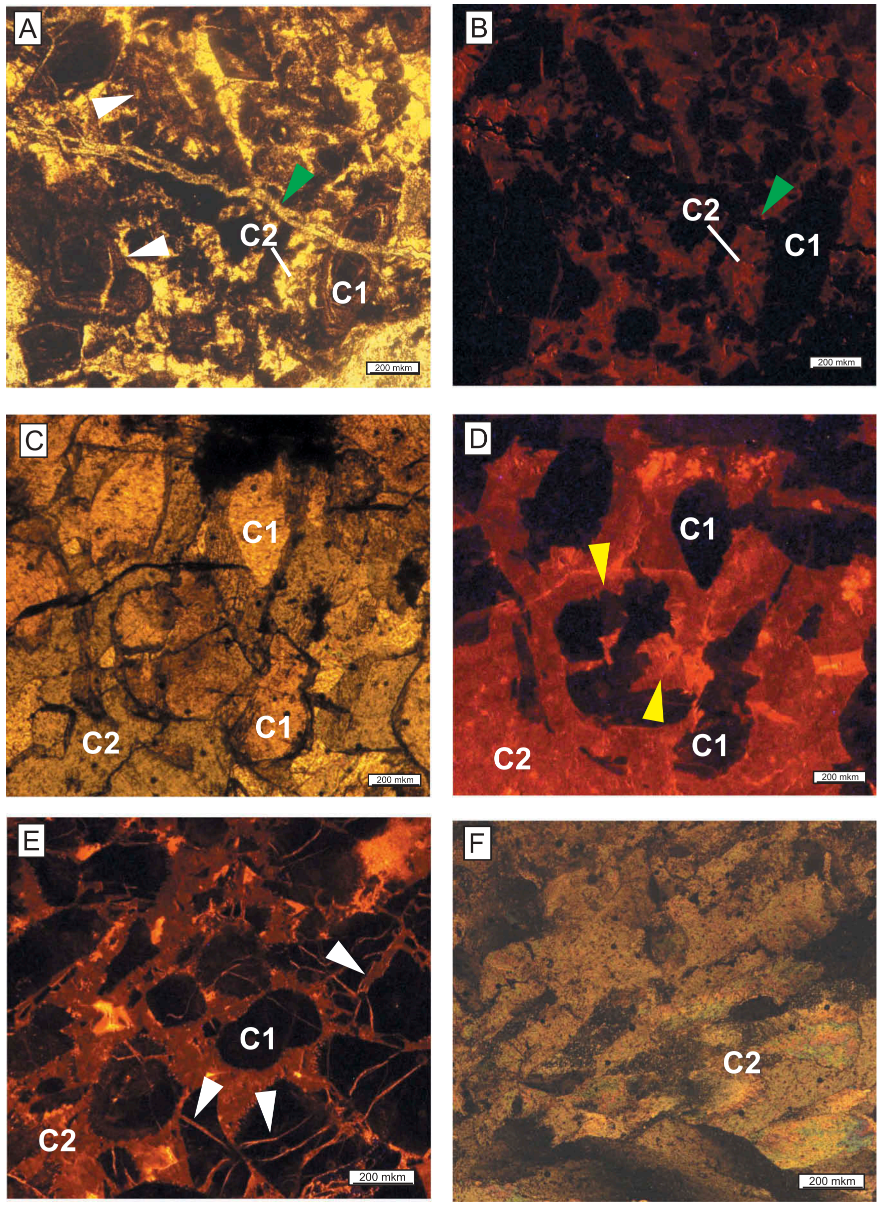

Figure 5. Petrology and cathodoluminescence characteristics of the studied glendonite concretions: **A** – plane-polarized microphotograph of the sample AN-1/109 (Upper Pleinsbachian), **B** – same in CL, **C**. plane-polarized microphotograph of the sample KYS-3 (Upper Bajocian), **D** – same in CL, **E**. CL microphotograph of the sample KYS-1 (Upper Bajocian), **F**. cross-polarized microphotograph of the sample KYS-4 (Upper Bajocian). Abbreviations are: C1 – ikaite-derive phase of calcite, C2 – calcite cement, white arrows point at idiotopic calcite crystals, green arrow points at late fracture filled with calcite, yellow arrows point at corrosion and replacing of C1 calcite with C2 calcite.

a weak brownish or no luminescence. A significant volume of the glendonite concretions is formed by the calcite C2 (C2 on Fig. 5 C, D, E, F), composed of colorless crystals with undulose extinction in cross polarized light. Calcite C2 fills pores between C1 calcite crystals and can be seen macroscopically as it occupies the central part of the concretion (Fig. 4 D, E). The calcite cement has elongated crystals up to several mm in diameter and exhibits a dull orange luminescence.

Bathonian (samples AN2/32, AN2/29-30, ANA29)

Petrography and cathodoluminescence of the studied glendonites that came from Bathonian sediments have a lot in common with pseudomorphs from the Upper Bajocian sediments. The concretions have an elongated shape and are up to 6 cm in diameter (Fig. 4G, H). They are composed of 2 calcite generations of which are a brownish color in cross polarized light. Calcite of the first generation (C1) forms about 30% of the concretions and has an irregular distribution throughout the concretion. Under cathodoluminescence, C1 is patchy dull brown, reddish or non-luminescent. Calcite of the C1 generation may be overgrown or even replaced by calcite C2 with dull orange to red luminescence. Calcite C2 fills remaining pore space within the concretions and forms about 70% of the total volume of the glendonite pseudomorphs.

Isotopic composition

Isotopic compositions of the bulk rock glendonite concretions (i.e., the homogenized isotopic value of all calcite generations) was studied. For two concretions (KYS-4 and AN2/29-30, Fig. 4E, G), additional samples were collected from the central part of the concretion filled with only calcite C2.

For bulk rock glendonite concretions, $\delta^{18}O$ values range from – 18.1 to – 3.5 ‰ VPDB and $\delta^{13}C$ values range from – 30.2 to – 14.3 ‰ VPDB. The calcite C2 is characterized by $\delta^{18}O$ of – 13.5 to −7.5 ‰ VPDB and $\delta^{13}C$ of −14.8 to −9.6 ‰ VPDB (Table 1, Fig. 6).

Discussion

Generalized data on spatial and stratigraphical distribution of glendonite findings in Siberia and Northeastern Russia (see Attachment for references) shows wide distribution of glendonites across the studied area. Distribution of glendonite findings (see section *Brief review of Early and Middle Jurassic climates of Siberia and glendonite distribution)* is generally in a good agreement with cooling events (Upper Pliensbachian, Aalenian, Bajocian and Bathonian) registered by changes in faunal and floral assemblages described from the studied region.

Microscopical observations of the studied glendonite samples reveals common features between glendonites of different stratigraphic intervals within the Jurassic succession. Glendonite concretions do not show any macrozonation that is thought to be formed during ikaite-calcite transformations and latter diagenesis (Vickers et al. 2018).

The studied glendonites are formed by two calcite phases – C1 and C2. Crystals of calcite phase C1 are idiotopic or anhedral that is usually caused by corrosion and replacing of C1 calcite by C2. The first phase of calcite crystals (C1) is interpreted as ikaite-derived calcite according to its optical characteristics – brown crystals, sometimes zoned in plane light, isometric or slightly elongated, non-luminescent (for detailed descriptions of calcite generations in glendonites see Huggett et al. 2005; Teichert & Luppold 2013; Vickers et al. 2018). Remnants of primary ikaite-calcite are often transected, corroded or replaced by calcite C2. C1 calcite occupies about 30% of the glendonite.

Calcite C2 has elongated crystals up to 1 mm in diameter. Crystals are radiaxial and show undulose extinction. Radiaxial calcite is thought to fill pore space in a number of different environments, including within the (a) meteoric vadose zone (Richter et al. 2011), (b) marine-meteoric water mixing zone (Kim & Lee 2003), or (c) burial diagenesis (Flugel 2010). Richter et al. (2011) observed that crystals of radiaxial calcite can precipitate from normal seawater, brackish water, or freshwater, and typically exhibit rapid precipitation rates.

Petrographical observations revealed that isotopic composition of the studied glendonite concretions is determined by initial composition of two components – ikaite-derived calcite (C1 phase) and calcite cement C2. For two samples we managed to extract C2 phase and analyze its isotopic composition. Comparison of isotopic composition of bulk glendonites and calcite C2 shows that radiaxial calcite cements are depleted in ^{13}C and enriched in ^{18}O isotopes (at least for 5 ‰ PDB for both isotopic ratios, Fig. 6). Comparison of the isotopic composition of the studied samples with those given in (Campbell 2006) let us conclude that isotopic ratios of the C2 calcite show similarities to carbonate cements precipitating during burial diagenesis from porous waters that were affected by presence of inorganic carbon and decomposition of organic matter.

We made an estimation of isotopic composition of ikaite-derived calcite (C1 phase) in two samples AN2/29–30 and KYS-4. Calculation showed that $\delta^{13}C$ = −29 ‰ VPDB and $\delta^{18}O$ in samples AN2/29–30 and KYS-4 is +4.5 and +5.8 ‰ VPDB respectively. Isotopic values of AN2/29–30 and KYS-4 samples correspond well with isotopic composition of recent ikaite reported from numerous localities (Stein & Smith 1986; Zabel & Schultz 2001; Greinert & Derkachev 2004; Lu et al 2012). Estimated isotopic values of AN2/29-30 and KYS-4 samples reveal that the main source of carbon isotopes decomposed marine organic matter. Calculations show that oxygen isotopic composition of ikaite-derived calcite was influenced by seawater.

Isotopical composition of glendonites from the Lower-Middle Jurassic sediments were reported by several researchers (Teichert & Luppold 2013; Morales et al. 2017). Glendonites with relatively low isotopic values of $\delta^{13}C$ were published by Teichert and Luppold (2013) and van de Schootbrugge et al. (2019) (for Pliensbachian glendonites of the Northern Germany) and Morales et al. (2017) (for Upper Pliensbachian, Upper Bajocian and Bathonian glendonites from Anabar Bay and Lower Lena river basin). Referring to the low $\delta^{13}C$ values (up to −48 ‰ VPDB) aforementioned authors assume that source of carbon might be various including marine organic matter, oil fractions or thermogenic methane. However, values of $\delta^{13}C$ for ikaite-derived calcite obtained in current research (−29 ‰ VPDB) and its congruence with values of $\delta^{13}C$ in recent ikaite crystals observed in near-freezing environments (Zabel & Schultz 2001; Greinert & Derkachev 2004) along with wide distribution of glendonites in shallow deposits during cool-

Table 1. Isotopic composition of bulk concretions and cement calcite from glendonite concretions of north-western part of the Siberian platform.

Sample	Stratigraphic unit	Concretion section	$\delta^{18}O$ (‰ V-PDB)	$\delta^{13}C$ (‰ V-PDB)
AN2/32	Upper Bathonian	Bulk concretion	−7.3	−21.7
ANA-29	Bathonian	Bulk concretion	−7.9	−30.2
		Bulk concretion	−8.7	−27.9
AN2/29-30a		Bulk concretion	−8.1	−15.4
AN2/29-30b		Calcite cement	−13.5	−9.6
KYS-1	Upper Bajocian	Bulk concretion	−6.3	−14.3
KYS-3		Bulk concretion	−4.6	−19.2
KYS-4a		Bulk concretion	−3.5	−19.1
KYS-4b		Calcite cement	−7.5	−14.8
AN-1/109	Upper Pliensbachian	Bulk concretion	−5.4	−16.6
1/132		Bulk concretion	−10.6	−18.0
		Bulk concretion	−18.1	−14.9

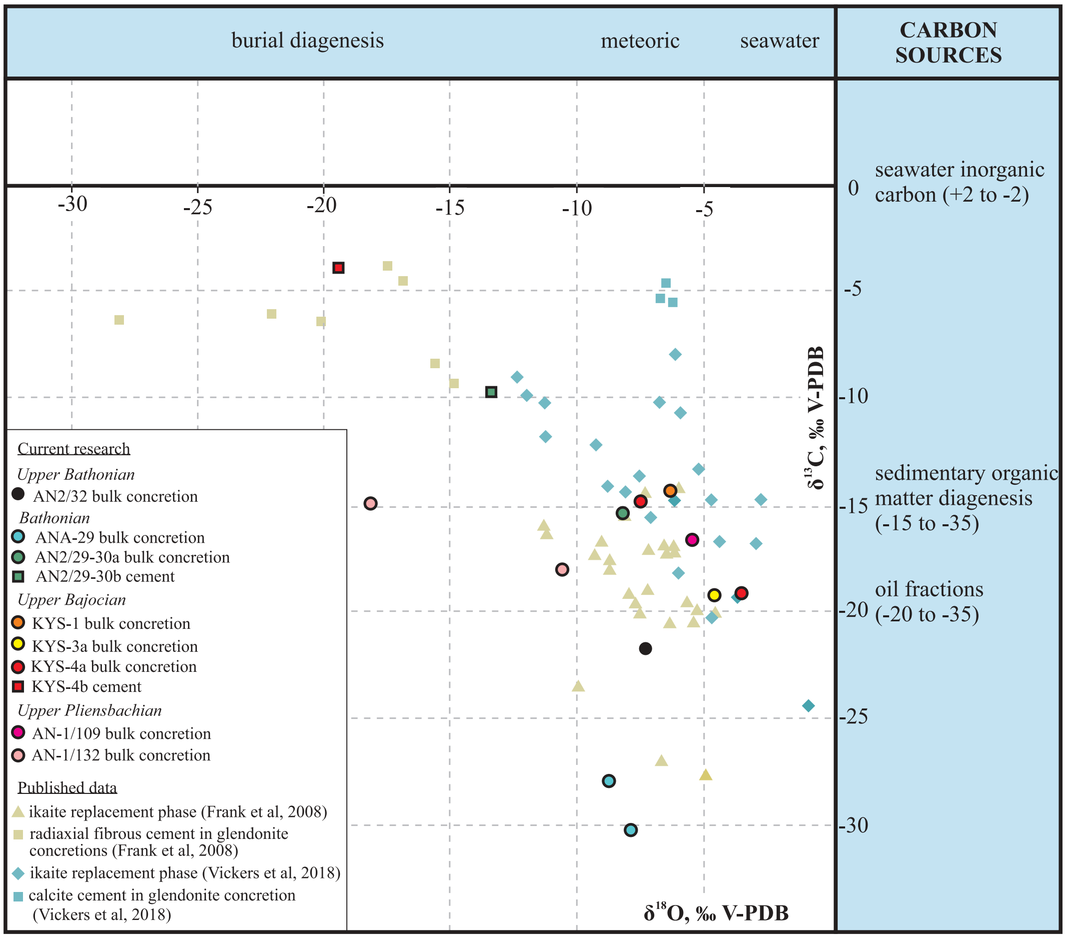

Figure 6. Isotopic composition of studied glendonite concretions and comparison with isotopic composition of glendonite concretions and calcite cement from published data. Sources of oxygen and carbon stable isotopes are given from Campbell (2006).

climate time intervals in the studied area support the use of glendonites as indicators of cold climate.

Conclusions

Our data on petrographic, cathodoluminescence and isotopic characteristics of the studied samples along with records of glendonite distribution in Siberia and Northeastern Russia let us draw the following conclusions:

(1) Ikaite precipitation occurred in a shallow cold-water basin that confirms that the sedimentation of the studied strata occurred in a cold climate.
(2) Geochemical environment of ikaite precipitation was determined by presence of marine organic matter resulted in low carbon isotopic values (up to −30 ‰ PDB) estimated for ikaite-derived calcite.
(3) During diagenesis a significant volume of early ikaite-derived calcite was corroded or replaced by cement calcite that influenced greatly on the isotopic composition of the bulk glendonite concretion.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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