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The freshwater reservoir effect in northern West Siberia: ^{14}C and stable isotope data for fish from the late medieval town of Mangazeya

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ABSTRACT

The freshwater reservoir effect (FRE) was estimated for the northern part of West Siberia for the first time, based on securely dated samples from the late medieval town of Mangazeya existed mainly in AD 1601–1650. Twelve specimens of six species of freshwater fish were selected for ^{14}C dating, and C and N stable isotopes analysis. The FRE varies from ca. 310 to ca. 1970 ^{14}C years, and there is no consistency in terms of relationship to feeding habits or trophic level of fish; the FRE fluctuations for Mangazeya, as well as for other regions in northern Eurasia, are noteworthy. This information should be taken into account when ^{14}C dating of bones of humans and domestic animals, who consume significant amount of freshwater fish, is conducted. This also applies to ^{14}C dating of food crusts on the surface of pottery.

1. Introduction

The radiocarbon (^{14}C) age offsets for aquatic organisms *versus* the terrestrial ones were initially recognized in the 1950s–1960s (e.g. Deevey et al., 1954; Berger et al., 1966; see also Stuiver and Polach, 1977; Stuiver and Braziunas, 1993). While the marine reservoir effect is now relatively well studied worldwide (Reimer and Reimer, 2001; Ascough et al., 2005; Cook et al., 2015; Alves et al., 2018), the investigations of freshwater reservoir effect (FRE) in rivers and lakes are still not numerous (e.g. Lanting and van der Plicht, 1998; Geyh et al., 1998; Dufour et al., 1999; Cook et al., 2001; Keaveney and Reimer, 2012; Philippsen, 2013, 2015; Shishlina et al., 2014; Fernandes et al., 2016). For the vast territory of Siberia and the Russian Far East, covers ca. 14, 000,000 km^2, only a handful of studies—based mainly on archaeological materials with unknown calendar age—are published until today (Nomokonova et al., 2013; Schulting et al., 2014, 2015; Svyatko et al., 2015, 2017a, 2017b; Losey et al., 2018). The importance of FRE for Siberia is now clear due to increasing number of ^{14}C dates produced using food residues on the surfaces of pottery vessels from West Siberia and the Trans-Urals (e.g. Zaretskaya et al., 2012; Pieznoka et al., 2020), and the reliability of these ^{14}C values may be affected by the FRE. This is why the research on samples of freshwater fish from Siberia with known calendar age is very timely.

Unfortunately, it is quite difficult to acquire such specimens due to the lack of pre-bomb materials in museums and a few recent excavations of both late Middle Ages and historic sites in Siberia with faunal remains. In addition, there is a reluctance of museum curators to break the sealed glass vessels with fish and mollusks from the late nineteenth – early twentieth centuries preserved in regional museums. All these circumstances hamper the investigations of the FRE in Siberia. Here we present new data on different species of fish recovered from the late medieval town of Mangazeya in West Siberian Arctic (another English spelling is Mangazeia; see Fisher, 1944; Naumov, 2006; Hartley, 2014) (Fig. 1).

2. Material and methods

We were able to obtain bones of several species of freshwater fish during the latest excavations in 2001–2004 of the abandoned town of Mangazeya (Vizgalov and Parkhimovich, 2008). In total, 12 samples of adult fish were acquired (Table 1). Fish bones were collected during the excavations, and exact position of samples was recorded using common archaeological practice (grid, number of cultural layer, depth from the surface). The fish sizes were determined, with perches and carps of 15–25 cm long, nelma and pike – 40–80 cm long, and sturgeon – 60–100

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cm long (Vizgalov et al., 2013: 335–336). The sizes for whitefish were not measured, although the adult individuals were selected for this study.

For taxonomic identification of fish from Mangazeya, subfossil fish remains from the reference collections housed in the Museum of the Institute of Plant and Animal Ecology (Yekaterinburg), were used. Identification was conducted according to standard zooarchaeological procedures (e.g. Reitz and Wing, 2008).

Our samples belong to the following species: perch (*Perca fluviatilis* L., 1758), carp (*Leuciscus* sp.), nelma (*Stenodus leucichthys* Guldenstadt, 1772), whitefish (*Coregonus* sp.), pike (*Esox lucius* L., 1758), and sturgeon (*Acipenser baeri* Brandt, 1869). We added to this collection four bones of adult modern fish caught from the Taz River during the excavations in 2001–2004, including burbot (*Lota* L., 1758), perch, nelma, and pike (Table 1). For comparison, we used data from Losey et al. (2018) on carbon and nitrogen stable isotopes of fish from the Ust'-Polui site, located in the modern city of Salekhard (Fig. 1) and dated to ca. 250 BCE – AD 150.

The town of Mangazeya (66°41′36″ N, 82°15′16″ E; Fig. 1) is the first Russian settlement in Siberian Arctic established by Cossacks in AD 1601 on the bank of the Taz River, ca. 190 km from its mouth (e.g. Forsyth, 1992; Bobrick, 2014). Since this time, it was heavily involved in trade of furs, mammoth tusks, and walrus ivory, the most lucrative business in Siberia in the seventeenth century. Mangazeya existed as a major trade and administrative center of the northern part of West Siberia until the 1670s, although its importance declined since the late 1640s–1650s (Fisher, 1944; Belov et al., 1980). In 1677, all town's functions were transferred to Novaya [New] Mangazeya (today's Turukhansk) in the Yenisei River basin (Fig. 1).

First large-scale excavations of the Mangazeya were initiated in 1968 and continued for four years (1968–1970, 1973) (Belov, 1977; Belov et al., 1980, 1981); the latest campaigns were undertaken in 2001–2004 (Vizgalov and Parkhimovich, 2008). Throughout all these years, a plethora of artifacts was recovered from well-survived cultural layer in

Fig. 1. Northern part of West Siberia with major rivers, towns and cities, and the studied site.

permafrost conditions, including faunal remains (Vizgalov and Parkhimovich, 2008). Fish bones were collected in the part of town occupied in AD 1601–1650, based on the finds of Russian coins. The majority of them is dated to 1605–1645, but only 13% of coins correspond to 1630–1645 (Vizgalov and Parkhimovich, 2008: 133) which may reflect the gradual decrease in economic activities since the 1640s.

The selected samples were the subject to stable isotope analysis (C and N), and the ^{14}C dating. Collagen extraction followed Longin's (1971) method. Around 100–500 mg of fragmented bone was demineralized in 10 ml 8% HCl for 20 min at room temperature, and rinsed with milliQ-water. After that, the sample was immersed for 15 min in 1% NaOH and rinsed again with milliQ-water. Subsequently, 1% HCl was added for neutralization of alkali, and the sample was washed with milliQ-water. For all these steps, Ezee-filters were used. Gelatinization of the extract was done in a solution with pH = 3, at 90 °C for 12 h. The gelatin was filtered with a Millipore 7 μm glass filter and freeze-dried. Stable isotopes ($\delta^{13}C$ and $\delta^{15}N$) values, % C, % N, the atomic C:N ratio, and the ^{14}C age were run on the gelatinized fish collagen.

Carbon and nitrogen stable isotope compositions were measured as the ratios of the heavy isotope to the light one (i.e. $^{13}C/^{12}C$ or $^{15}N/^{14}N$), and are reported in delta (δ) notation as parts per thousand (per mill, ‰):

$$\delta^{13}C(or\ \delta^{15}N) = \left(\left[R_{sample}/R_{standard}\right] - 1\right) \times 1000$$

where R is $^{13}C/^{12}C$ or $^{15}N/^{14}N$, relative to internationally defined standards for carbon (Vienna Pee Dee Belemnite, VPDB) and nitrogen (Ambient Inhalable Reservoir, AIR).

Stable isotope analyses for archaeological fish from Mangazeya (Table 1) were performed in duplicate on a Thermo Flash EA/HT elemental analyzer, coupled to a Thermo Delta V Advantage Isotope Ratio Mass Spectrometer via ConfloIV interface (ThermoFisher Scientific, Bremen, Germany) at the Department of Earth and Environmental Sciences, University of Leuven (Leuven, Belgium). Standards used were IAEA-N1, IAEA-C6, and internally calibrated acetanilide. Analytical precision was 0.25‰ for both $\delta^{13}C$ and $\delta^{15}N$ based on multiple measurements of the standard acetanilide.

Samples of modern fish were first cooked, and vertebrae were collected for analysis. Before the extraction of collagen, the fat was deleted from the surface using the mixture of chloroform and methanol.

Stable isotope measurements for modern fish from the Taz River (Table 1) were conducted on a Isoprime PrecisION IRMS coupled on an Elementar varioIsotope Cube (Elementar, Germany and UK), at the Laboratory of Radiocarbon Dating and Electronic Microscopy, Institute of Geography RAS (Moscow, Russia), against IAEA-600, B2155, and B2159 (Elemental Microanalysis Ltd) standards. The analytical precision was 0.2‰ for both $\delta^{13}C$ and $\delta^{15}N$.

Carbon and nitrogen concentrations in bone gelatin in relation to the bulk weight were also determined for all samples included into this study; these are referred to as weight percentage of carbon and nitrogen (% C and % N). These quality indicators provide information on protein degradation. The atomic C:N ratio of the bone collagen samples was used to classify the samples as uncontaminated or contaminated (e.g. De Niro, 1985; Ambrose, 1990). Samples with values outside the 2.9–3.6 range were regarded as less reliable.

All archaeological samples were transformed into graphite using the automatic graphitization system AGE (Ervynck et al., 2018), and ^{14}C concentrations were measured with Accelerator Mass Spectrometry (AMS) at the Royal Institute for Cultural Heritage (Brussels, Belgium) (Boudin et al., 2015). The OxCal 4.3 program (Bronk Ramsey, 2009) and atmospheric ^{14}C data from Reimer et al. (2009) were used to transform historical calendar date into radiocarbon data (BP). The FRE offsets (in ^{14}C years) were calculated as this:

$$FRE = {}^{14}C\ age\ of\ freshwater\ fish - {}^{14}C\ equivalent\ of\ historic\ age$$

Because the fish from the Mangazeya town can be dated using historical information to ca. AD 1625 (median value of AD 1600–1650, see above), the R-simulate converted AD 1625 into ^{14}C date of ca. 340 BP. The standard deviation (1σ) is accepted as ± 25 years. The uncertainty of a FRE offset was calculated using this formula:

$$\sigma_{FRE} = \sqrt{\sigma_a^2 + \sigma_b^2}$$

where σ_{FRE} is the RFE offset uncertainty (1σ), σ_a is ^{14}C date uncertainty (1 σ), and σ_b is the uncertainty of the ^{14}C age corresponding to historical date (1σ).

3. Results and discussion

There are two types of fish in the studied collection in terms of feeding patterns, carnivorous and non-carnivorous (Pavlov and Mochek, 2006; Kizhevatov and Kizhevatova, 2015). As for the former, these are nelma, pike, and burbot. They are migrating species, and inhabit most of the year the Taz River, with less time in the estuary of the Taz. Pike and burbot are fed on fish, and nelma – mainly on small fish, crustaceans, and insect larvae. Perch can be considered as semi-piscivorous species,

Table 1
Stable isotope data, apparent ^{14}C ages and FRE offsets for fish bone collagen from the Mangazeya town.

Species	Sample No.	Atomic C:N ratio	$\delta^{13}C$, ‰	$\delta^{15}N$, ‰	^{14}C date (BP)	^{14}C date uncertainty, (1 σ, BP)	Lab No. RICH-	FRE offset, BP	FRE offset uncertainty (1 σ), BP
Fish from excavations of the Mangazeya town									
Perch	1009/01	3.1	−26.9	+9.3	1010	25	26469.1.1	670	35
Perch	1009/02	3.2	−27.7	+9.5	650	25	26470.1.1	310	35
Carp	1009/03	3.2	−28.9	+9.6	975	25	26471.1.1	635	35
Carp	1009/04	3.1	−28.7	+9.6	800	25	26472.1.1	460	35
Nelma	1009/05	3.1	−22.4	+12.8	1160	25	26473.1.1	820	35
Nelma	1009/010	3.1	−24.7	+12.7	960	25	26474.1.1	620	35
Whitefish	1009/06	3.2	−24.7	+9.5	1200	25	26476.1.1	860	35
Whitefish	1009/07	3.1	−28.0	+11.9	1205	25	26478.1.1	865	35
Pike	1009/08	3.1	−27.6	+10.4	720	25	26480.1.1	380	35
Pike	1009/09	3.0	−23.9	+12.0	1350	25	26481.1.1	1010	35
Sturgeon	1009/011	3.1	−28.9	+9.5	2310	25	26482.1.1	1970	35
Sturgeon	1009/012	3.1	−27.0	+11.7	1580	25	26484.1.1	1240	35
Modern fish from the Taz River near the Mangazeya town									
Perch	4802	3.2	−24.7	+16.6	–	–	–	–	–
Nelma	4803	3.3	−29.0	+11.1	–	–	–	–	–
Pike	4804	3.3	−30.0	+9.7	–	–	–	–	–
Burbot	4805	3.7[a]	−27.7	+12.7	–	–	–	–	–

[a] This can be less reliable sample due to C:N ratio more than 3.6 (Ambrose, 1990).

and it is fed mainly on crustaceans, worms, and insect larvae, and to lesser extent on small fish. It inhabit the Taz River all the year round.

As for the non-piscivorous species, they are represented by sturgeon, whitefish, and carp (cyprinids). Sturgeon spend most of time or the entire life in the estuary of Taz River (also called Taz Bay, with freshwater conditions), and only rare individuals enter the Taz River. Residents of Mangazeya either procured sturgeon in the estuary of Taz or brought it from the Ob River, several hundred kilometers west of Mangazeya although it was easy to transport fish by watercraft. It feeds on small invertebrate animals: crustaceans, worms, mollusks, and insect larvae. Whitefish feed on zoobenthos, and it is a migrating species like nelma, pike, and burbot. Carp inhabits the Taz River all the year round, and it is fed on benthos and zooplankton.

In Table 1 and on Figs. 2–4, the analytical results for fish from the town of Mangazeya are presented. As for C and N stable isotopes, nelma and burbot have the highest δ^{15}N values – ca. 12.7‰ (Fig. 2). The rest of fish has the δ^{15}N in the range of ca. 9–12‰; very high δ^{15}N value of 16.6‰ for perch (sample 4802, Table 1) seems to be an outlier. As for the δ^{13}C values for all species, they are in the range from −22.5‰ to −30‰ (Fig. 2). We do not observe any clear patterns in both δ^{13}C and δ^{15}N

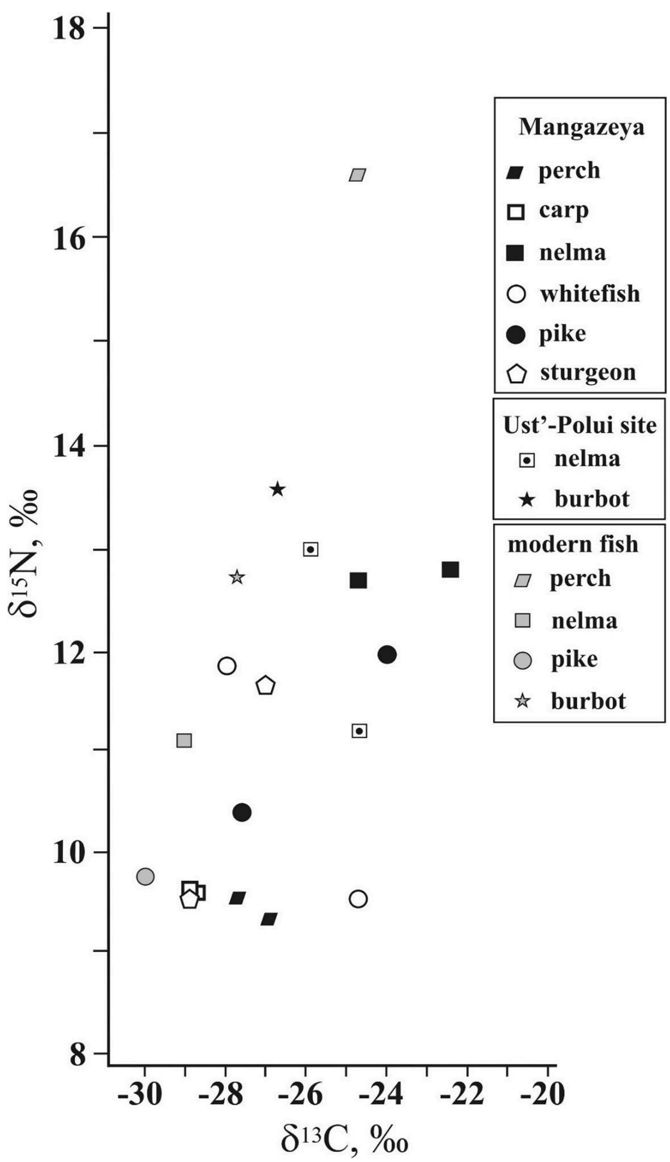

Fig. 2. Carbon and nitrogen stable isotope values for fish from northern West Siberia (data for Ust'-Polui are from Losey et al., 2018).

ratios related to feeding habits and trophic levels of the analyzed species of freshwater fish from the Taz River.

The FRE offsets *versus* δ^{13}C and δ^{15}N values are presented on Figs. 3 and 4. Using the data obtained, it is clear that the FRE for the northern West Siberia is quite large, up to ca. 1970 years (Table 1). This is an important observation not available previously for the entire Western Siberia (cf. Svyatko et al., 2017a, 2017b). For the upper course of Lena River in Eastern Siberia, similar FRE values were detected, ca. 260–1000 years (Schulting et al., 2015). In the Altai Mountains of Southern Siberia, quite large FRE values were established for the Katun River (ca. 1100 years) and Chuya River (ca. 580 years) (Svyatko et al., 2017a). For some other regions of Siberia, such as Edarma, Yenisei, and Karasuk rivers in Eastern Siberia, smaller FRE values were measured – ca. 160–750 years. For northern part of Kazakhstan, neighboring to southern West Siberia, the FRE values are in the range of ca. 300–670 years (Svyatko et al., 2015, Svyatko et al., 2017a).

Despite the limited amount of samples used in this study, it is clear that the FRE for freshwater fish from the Mangazeya varies greatly. For example, the FRE values for two specimens of sturgeon (1009/011 and 1009/012) are almost 750 years apart. For pike, large FRE variation—ca. 630 years—can be also observed. For other species, the FRE fluctuates within several hundred years: carp – 185 years; nelma – 200 years; and perch – 360 years (Table 1; Figs. 3 and 4). The large FRE and stable isotope variations between different species of fish and other aquatic organisms (e.g. Baikal seal, *Pusa sibirica* Gmelin, 1788) were detected in northern Eurasia previously (e.g. Dufour et al., 1999; Katzenberg et al., 2010; Philippsen and Heinemeier, 2013; Fernandes et al., 2013; Schulting et al., 2015; Svyatko et al., 2017; Ervynck et al., 2018).

As for the Siberian lakes, the picture could also be complicate. For example, in Lake Baikal the FRE may be estimated as ca. 700–1000 years (Nomokonova et al., 2013; Schulting et al., 2014). This is in accord with other regions in northern Eurasia, such as Germany where the FRE are in the order of ca. 640–1400 years (Olsen et al., 2010; Fernandes et al., 2016), and in extreme case of Iceland—with influence of ^{14}C-depleted water from springs in the areas of active volcanism such as Lake Mývatn—ca. 3500 years and even more (Ascough et al., 2011).

It is therefore clear that consumption of large amount of fish by prehistoric and Medieval populations may result in distortion of the ^{14}C dates of human bones due to FRE. For example, in the Lake Baikal region people in the Neolithic and Bronze Age relied heavily on aquatic food sources (Katzenberg et al., 2010). Fish was important component of food for prehistoric people in Siberia and the neighboring Kazakhstan (e.g. O'Connell et al., 2003; Privat, 2004; Privat et al., 2005; Lobanova and Gimranov, 2016).

Another important issue is the ^{14}C dating of food crust on the surface of pottery vessels. Even though sometimes this is the only way to get ^{14}C date for destroyed or totally excavated site where terrestrial materials were either not available or not collected, scholars should be aware that charred food remains can be easily affected by FRE. This conclusion was initially put forward by Fischer and Heinemeier (2003), and was highlighted again (Boudin et al., 2010; Philippsen, 2013, 2015; Hart et al., 2013; Heron and Craig, 2015; Piezonka et al., 2020).

As it was correctly mentioned by Ervynck et al. (2018:413) who studied the FRE in the Scheldt River basin (Belgium and northern France), "The observations in this study serve as a warning against assessing FREs for a particular aquatic system on the basis of only a limited sample size comprising only a limited number of species and size classes, as this may lead to the variability in FRE being significantly underestimated." We are still in the beginning of understanding the FRE for Siberia and the Russian Far East, and new research will bring more data. In our opinion, the highest priority should be given to samples with known age, obtained from either pre-AD 1950s museum collections (with exact date of acquisition) or excavations of short-lived historical towns and other settlements with unequivocal records of their age in chronicles and other documents (e.g. Vernadsky, 1969; Soloviev, 1988; Perrie, 2006; Witzenrath, 2007).

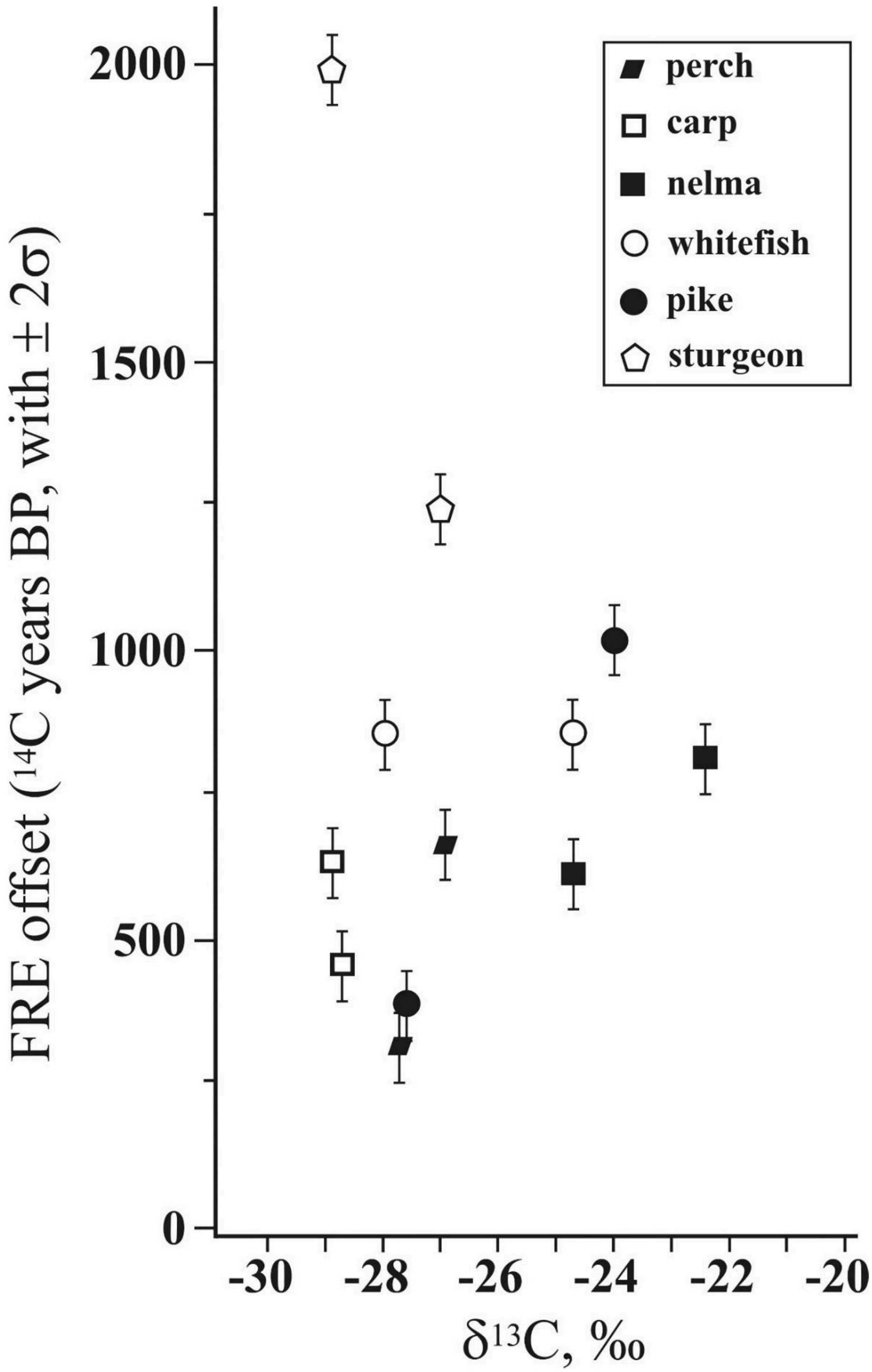

Fig. 3. FRE offset (with ± 2σ) *versus* $\delta^{13}C$ ratio for fish from the Mangazeya site.

As for possible cause of FRE in archaeological fish from Siberia and elsewhere, several explanations were suggested: 'old' carbonates from the Proterozoic, Paleozoic and Mesozoic rocks like limestone and chalk (i.e. 'hard water') (e.g. Philippsen, 2013; Philippsen and Heinemeier, 2013; Nomokonova et al., 2013; Schulting et al., 2014, 2015); feeding patterns for fish (e.g. Fernandes et al., 2013); high alkalinity of water (e.g. Keaveney and Reimer, 2012); long residence time for water in some large lakes like Baikal (Schulting et al., 2014); or combination of all or some of these factors (e.g. Philippsen, 2013). It is not clear to us at this stage why the ^{14}C age for archaeological fish from Mangazeya is much older than calendar time, and to understand it is the task for the future research.

Another important issue is a high degree of variability of FRE even for a single body of water or the same species of aquatic organisms, as it was repeatedly observed in Europe and Asia (e.g. Fernandes et al., 2013; Philippsen, 2013; Schulting et al., 2014, 2015). As a result, large datasets are required to investigate the patterns of FRE for Eurasia in general, and for Siberia in particular.

4. Conclusions

Based on the analysis of freshwater fish from the town of Mangazeya

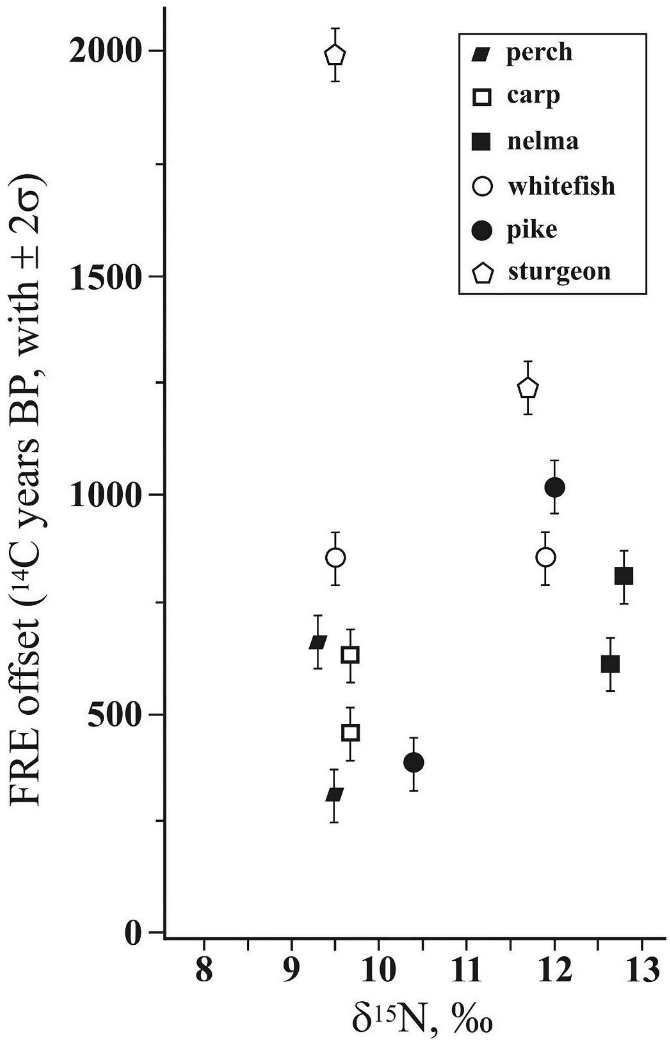

Fig. 4. FRE offset (with ± 2σ) *versus* $\delta^{15}N$ ratio for fish from the Mangazeya site.

with relatively well-known historic age, we were able to securely estimate the FRE for northern West Siberia for the first time. It turned out that it varies from ca. 310 years to ca. 1970 years. Large FRE fluctuations are detected for samples of the same species of fish, and it is currently impossible to find out the nature of this phenomenon although it is commonly observed in other parts of northern Eurasia. As it was suggested before, it is extremely difficult to estimate the exact contribution of FRE to the ^{14}C age of humans and domestic animals (like dogs and pigs) who consumed large amount of freshwater fish, and to adjust their ^{14}C dates to the contemporaneous atmospheric ^{14}C activity in order to calibrate these ^{14}C values. It is therefore insecure to conduct the ^{14}C dating of these materials in West Siberia, as well as food crust on the surface of pottery. More work is still necessary to investigate the FRE in Siberia, giving priority to the samples with well-established historical age.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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