Research paper

The Holocene
1–11

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DOI: 10.1177/0959683616646843
hol.sagepub.com
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Climatic or dietary change? Stable isotope analysis of Neolithic–Bronze Age populations from the Upper Ob and Tobol River basins

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Abstract

Dietary changes in the populations inhabiting southwest Siberia and northern Kazakhstan indicate concurrent changes in the economy, at the same time marking the beginnings of East–West interaction across northern Eurasia. The introduction of domestic animal species of Near Eastern origin, such as sheep and goat, dramatically changed the lives of the local population. Past palaeodietary research using stable isotope analysis has mainly focussed on pastoral populations of the Bronze Age period. It is crucial, however, to assess the diets of humans and animals from earlier periods (Neolithic/Chalcolithic) in order to understand the timing and nature of dietary change during the Bronze Age of southwest Siberia and northern Kazakhstan, in particular the possible contribution of environmental change influencing dietary shifts. In this paper, we report the results of stable isotope analysis on 55 human and 45 faunal samples from southwest Siberia (Upper Ob River) and northern Kazakhstan (Tobol River basin), ranging from the Neolithic to the Bronze Age. These data, combined with published human and faunal collagen results from the region as well as new accelerator mass spectrometer (AMS) radiocarbon dating results, indicate little change in animal diet over time, but a notable change in human diet at *ca.* 2500 cal. BC. The data allow us to determine the time when pastoralism came to the fore, with concomitant economic differences to the local population.

Keywords

bone collagen, Bronze Age, Chalcolithic, diet, Neolithic, northern Kazakhstan, pastoralists, southwest Siberia, stable isotope

Received 30 January 2016; revised manuscript accepted 6 April 2016

Introduction

The multi-directional translocation of goods between the western and the eastern fringes of Eurasia reflects the beginning of the global processes of exchange (Boivin et al., 2012; Jones et al., 2011; Kohl, 2007; Linduff and Mei, 2009; Mei, 2003; Sherratt, 1996, 2005). The steppe and the forest-steppe zones of Siberia are an environmental belt that links the outskirts of western Asia and Eastern Europe. This corridor of northern steppe and forest steppe aided the dispersal of animal and crop species, metals, precious stones, as well as new technologies, across wide swathes of Eurasia in prehistory (Kuzmina, 2008; Koryakova and Epimakhov, 2007). Understanding the timing and nature of the transition from hunting and gathering to food production across this zone is of great importance, as the arrival of ovicaprids is linked with a series of concurrent social and economical changes. Based on the studies of zooarchaeological, archaeobotanical data and the study of material culture, it has been postulated that long distance connectivity and interaction began to take place along the southern regions of Central Asia at the end of the 3rd millennium BC (Frachetti, 2012; Spengler III, 2015). At the same time, the processes of long distance interaction for the northern regions of Central Asia and southwest Siberia have been linked to the beginnings of sheep and goat breeding and bronze metallurgy (Grushin et al., 2009). According to Zakh et al. (2010), Kovalev (2011) and others, the aridisation of the climate around 3000 BC in southwest Siberia, which led to the expansion of the steppe ecological zone, has been suggested as a driver of the inflow of pastoralist communities from the southwest to the north and east into former forest-steppe regions. The earliest animal species of Near Eastern origin (sheep and goat) have been found in northern Kazakhstan and southwest Siberia in the sites of the Elunino Culture dated to the second half of the third beginning of the 2nd millennium BC (Kiryushin et al., 2011).

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Figure 1. The location of the sites analysed in this paper: (1) Itkul (Bolshoi Mys); (2) Ust-Isha; (3) Firsovo-XI and XIV; (4) Solontsy-5; (5) Tuzovskie Bugry-I; (6) Teleutsky Vzvoz -I; (7) Kolyvanskoe-I; (8) Berezovaya Luka; (9) Novoilinovka; (10) Lisakovsk; (11) Belkaragai-1; (12) Botai-I; (13) Tavdinsky Grotto; (14) Bozshakol-6; (15) Kirik-Oba-1; (16) Kozai-1.

The introduction of sheep and goat into southwest Siberia and northern Kazakhstan have significantly changed the economy and subsistence of local population. However, the exact timing of this transition and its economical significance is not known. Stable isotope analysis allows the investigation of past human diets and dietary shifts at the individual level and has a potential to reveal when dietary change happened in these regions. To address this question with stable isotope analysis, it is important to simultaneously compare both human and animal remains though time, as both the introduction of pastoralism and the onset of climatic changes can influence the stable isotope values of fauna and human bone collagen (see section 'Discussion'). If a dietary difference was to be found in δ^{13}C and δ^{15}N values between all species of Neolithic/Chalcolithic and Bronze Age herbivorous fauna, including wild species, then this might result from climatic factors and their influence on vegetation. Therefore, the shift in human stable isotope values though time might result from climatic rather than dietary change. On the other hand, if herbivore stable isotope data do not differ between periods, the change in humans though time can be linked with the dietary change, which could be connected with the beginning of pastoralism.

The aim of this paper is to study the dietary change of populations from southwest Siberian and northern Kazakhstan over the sixth to the second millennium BC period. By analysing both human and animal remains using stable isotope analysis, we examine whether there is change in human stable isotope ratios of bone collagen seen over time and whether it correlates with changes in faunal isotope ratios, and thereby pinpointing the timing of the transition to pastoralism in the region.

Background

Archaeological background

The samples for research were collected from various Neolithic to Bronze Age sites located in the region of the Upper Ob River and Tobol River basins in southwest Siberia and northern Kazakhstan (Figure 1). The Neolithic/Chalcolithic sites such as Itkul (Bolshoi Mys), Ust-Isha (Kiryushin, 2002; Kiryushin et al., 2000), Firsovo-XI (Kiryushin et al., 1994), Solontsy-5 (Kungurova, 2005) and Tuzovskie Bugry-I (Abdulganeev et al., 2000; Kiryushin and Kiryushin, 2015) are located in southwest Siberia, in the Upper Ob River basin. The region of the Upper Ob River basin is also referred to as the 'forest-steppe Altai'. The local Neolithic sites are attributed to the so-called Kuznetski-Altaisky Neolithic (Kungurova, 2005) and Bolshemysky Chalcolithic periods (Kiryushin, 2002). The burial grounds of the Neolithic and Chalcolithic periods are small, often just consisting of a single burial; the Neolithic and Chalcolithic settlement sites are mostly located near rivers, steam banks or close to lakes. During the Bronze Age, the size of burial grounds increased dramatically, and organisation and complexity of the sacral space become noticeable (Kiryushin, 2002). The Bronze Age settlements are mainly located close to multiple environmental niches such as hilly slopes, transition zones between steppe and forest steppe, and/or close to rivers, saline and freshwater lakes. The southwest Siberian burial site of Teleutsky Vzvoz-I is attributed to the Early Bronze Age Elunino Culture, and the Firsovo-XIV site is attributed to the Middle Bronze Age Andronovo Culture (Fedorovo variant). The burial and settlement site of

Novoilinovka from northern Kazakhstan is attributed to the Sintashta-Petrovka Culture of the Middle Bronze Age, while the Lisakovsk burial site in Kazakhstan belongs to the Andronovo Culture (Fedorovo and Alakul variant), which in northern Kazakhstan is attributed to the Late Bronze Age.

The Bronze Age period has been associated with significant economical transformations in the societies of the region, from fishing and hunting to breeding domestic animals (Frachetti and Benecke, 2009; Kalieva and Logvin, 2011; Outram et al., 2012). Some researchers relate the changes that occurred in the second half of the third millennium BC with human migrations from the West that not only introduced livestock into the region but also bronze metallurgy (Kovalev, 2011).

The earliest evidence of ovicaprids in the region under study

The earliest animal species of Near Eastern origin (sheep and goat) have been found in northern Kazakhstan and southwest Siberia in the sites of the Elunino Culture dated to the second half of the third to the beginning of the second millennium BC (Kiryushin et al., 2011). The remains of early ovicaprids in northern Kazakhstan were found on the left bank of the Irtysh River at the sites of Shiderty 10 and Shouke 1 dated to the 2500–2300 BC (Merts, 2013). In the Upper Ob River basin of Russia, the earliest ovicaprid bones are coming from the so-called Aleiskaya steppe region, from the settlement of Berezovaya Luka and Kolyvanskoe-I (analysed in this study) and dated from the second half of the third to the beginning of the second millennium BC (Grushin, 2015; Kiryushin et al., 2005, 2011). The bones of ovicaprids from the Early Bronze Age settlement of Berezovaya Luka constitute 99% of all faunal remains (Kiryushin et al., 2005).

In the neighbouring Altai Mountains region, the beginning of pastoralism is related to the Afanasevo Culture (Khazanov, 1994) dating to 2900–2500 cal. BC (Svyatko et al., 2009). The remains of ovicaprids have been mainly found in several stratified settlements with later occupation horizons, and the bones of ovicaprids were not directly dated. Therefore, further research is required to place the ovicaprid remains from the Altai within a reliable timeframe. In central Kazakhstan, the earliest evidence of sheep-/goat-based pastoralism has been found in a Karagash human burial (2920–2712 cal. BC) (Motuzaite Matuzeviciute et al., 2015), which contained both sheep skull and ribs that accompanied the deceased as part of a ritual meal (Evdokimov and Loman, 1989). Finally, recent analysis of *Ovis* sp. from Inner Mongolia in China has demonstrated the earliest dates for Central Asia, estimated to be 4700–4400 cal. BC (Dodson et al., 2014). These data of early sheep in Inner Mongolia could indicate multiple pathways and waves of pastoralism that remain unknown and is a subject for future research.

Previous palaeodietary studies in the region

Previous palaeodietary studies of the populations from southwest Siberia and northern Kazakhstan have mainly focussed on the Bronze Age period. The only stable isotope research on the diet of Neolithic populations included samples from the Preobrazhenka 6 site in the Baraba forest-steppe region, suggesting that the human diet was based on fish and C_3 plants (Marchenko et al., 2015). Archaeological reports describe the Neolithic populations as hunter-gatherer mobile groups that relied heavily on the exploitation of freshwater resources and wild game (Molodin et al., 2012; Motuzaite Matuzeviciute, 2012; Okladnikov, 1959). Attempts to identify domesticated plants in Siberia prior to the Early Iron Age have not been successful to date (e.g. Kislenko and Tatarintseva, 1999; Korobkova, 1987). The Chalcolithic period in northern Kazakhstan in particular is linked with some dietary changes, mainly due to the local domestication of wild cattle in the Tersek Culture (Kalieva and Logvin, 2011) and horses in the Botai Culture (Outram et al., 2009; Zaibert, 1993) belonging to the second half of the fourth millennium BC. Stable isotope analysis of two Chalcolithic individuals from northern and central Kazakhstan (Botai and Karagash) (Motuzaite Matuzeviciute et al., 2015; O'Connell et al., 2003) was inconclusive due to a small sample size.

Studies of the Bronze Age populations drawn on zooarchaeology and stable isotope analysis suggest that fish was exploited as a dietary resource in the Bronze Age period across the steppe and forest-steppe zones (Lightfoot et al., 2014; Molodin et al., 2012; Motuzaite Matuzeviciute, 2012; O'Connell et al., 2003; Ventresca Miller et al., 2014). In combination with published zooarchaeological data (e.g. Frachetti and Benecke, 2009; Kalieva and Logvin, 2011; Outram et al., 2012), stable isotope analysis conducted in the region under study indicates that Bronze Age population diets were mainly based on ovicaprid and cattle meat and milk (Katzenberg et al., 2012; Marchenko et al., 2015; O'Connell et al., 2003; Privat, 2004; Privat et al., 2006; Ventresca Miller et al., 2014), with some contribution of C_4 plants to both fauna and human diets (Lightfoot et al., 2014; Ventresca Miller et al., 2014). The elevated $\delta^{13}C$ values in Late Bronze Age humans in the Minusinsk Basin of Siberia are most likely the result of millet consumption (Svyatko et al., 2013). Archaeobotanical data from the Middle Bronze Age site of Kamennyi Ambar (Sintashta-Petrovka Culture) in the Ural region of Russia report that large quantities of *Chenopodium album* seeds were probably eaten by the local populations, but no evidence of domesticated crops were found (Rühl et al., 2015). As such, to the best of our knowledge, no cereals have been radiocarbon dated to the Bronze Age in the whole of southwest Siberia or northern Kazakhstan to date.

Climate and geographical setting

Southwest Siberia encompasses numerous environmental niches, including forest steppes and steppes with varying proportions of C_4 grasses, hilly slopes, river valleys and both saline and freshwater lakesides which carry a range of unique vegetation varieties (Spengler III et al., 2013).

The distribution of vegetation types, and thus the borders of the forest-steppe and steppe across the region, has changed over time (Krivonogov et al., 2012). These changes have potential isotopic effects on animal and human bone collagen. According to previous palynological research, the Neolithic period in the southwest Siberia coincides with regional aridisation at around *ca.* 5700–4300 BC, when dense boggy vegetation formed in the drying lakes and river banks (Zakh et al., 2010). During the subsequent middle Atlantic period *ca.* 4300–3300 BC, there was an increase in humidity, and the forest-steppe vegetation expanded further south to the Tobol-Ishim River basins. A shift towards aridity was recorded at *ca.* 3100–3000 BC and the southern steppe expanded north (Zakh et al., 2010). During the third millennium BC, the climate became more arid, with periods of abrupt aridisation (from the *ca.* 2800–2600 BC), increased summer temperatures and decreased winter temperatures have been recorded in the Eurasian steppe (Kremenetski, 2003; Kremenetski et al., 1999; Shishlina et al., 2009). Palynological studies in the Tobol-Ishim River and Upper Ob River basins had shown an expansion of grassland at this time, and the forest-steppe was being replaced by semi-arid steppe (Kiryushin, 2002; Zakh et al., 2010). In the floodplains and valleys of the larger rivers, however, the conditions persisted stable and less sensitive to climatic changes (Zakh et al., 2010). This period is believed to roughly coincide with the transition to the Bronze Age and the beginning of pastoralism in the region.

Stable isotope methodology

Past human diets can be assessed by analysing bone chemical composition, as the food that animals and humans eat is incorporated into their body tissues. For dietary analyses, the most important stable isotopes are those of carbon and nitrogen. Stable carbon isotope measurements (expressed as $\delta^{13}C$) can distinguish between diets based on plants using two photosynthetic pathways, C_3 and C_4, and between diets based on marine and terrestrial foods (Lee-Thorp, 2008; Schoeninger and DeNiro, 1984; Vogel and Van der Merwe, 1977). In environments where marine foods are absent, large shifts in carbon isotopic values will be related to the relative proportions of C_3 and C_4 plants in the ecosystem. The proportion of C_4 to C_3 plants very much depends on the aridity of the area, as well as related effects such as salinity, with drier regions having larger proportions of C_4 plants (Collins and Jones, 1986; Tieszen et al., 1979). The faunal carbon isotopic values in southwest Siberia could be elevated as a result of C_4 plants in the animals' diets that today represent a minor component of the flora in the steppe (Iacumin et al., 2004; Makarewicz and Tuross, 2006; Winter, 1981). Studies have also shown smaller but still notable increases in the carbon isotope values of C_3 plants if plants are growing in water-stressed environments (Flohr et al., 2011). Thus, climate change and rising temperatures can affect the $\delta^{13}C$ ratios of plants, as well as consumers (animal and human) that are higher up the food chain.

Nitrogen isotope ratios ($\delta^{15}N$) reflect the trophic level of animals and humans, with higher trophic level consumers having the highest $\delta^{15}N$ values (Hedges and Reynard, 2007). The nitrogen isotope ratios of freshwater (and marine) fish are often elevated compared with terrestrial mammals, as aquatic ecosystems tend to have longer food chains (Richards and Hedges, 1999; Schoeninger and DeNiro, 1984; Schoeninger et al., 1983). Nitrogen isotope ratios can also be affected by various environmental factors. Animals grazing on saline soils (Britton et al., 2008; Heaton, 1987) or living in arid climates (Ambrose and Sikes, 1991; Heaton, 1987; Hollund et al., 2010; Schwarcz et al., 1999) in semi-deserts have higher nitrogen isotope values compared with animals grazing in other environments, such as forest. It has been shown, for example, that aridity increased nitrogen values of the Bronze Age humans of Gansu province in China (Liu et al., 2014). Recent research has also shown higher $\delta^{13}C$ and $\delta^{15}N$ values for animals grazing in marshy areas (Britton et al., 2008). The $\delta^{15}N$ value will be elevated in manured soil resulting in higher $\delta^{15}N$ value in humans consuming fertilised crops (Bogaard et al., 2007; Fraser et al., 2011). These non-dietary factors need to be kept in mind while analysing human and faunal stable isotope data from the Neolithic though Bronze Ages of southwest Siberia and northern Kazakhstan.

Material and methods

Stable isotope analysis

Samples from 17 sites in southwest Siberia and northern Kazakhstan are presented in this paper, combining new data together with published data (Motuzaite Matuzeviciute et al., 2015; Ventresca Miller et al., 2014) (Figure 1, SOM 1a and b, available online). The sites are located in modern steppe and forest-steppe belt which contains a variety of environmental niches. However, humans and animals are not static and would have moved though different environmental zones (Frachetti, 2008; Shishlina et al., 2009); therefore, the isotopic signals in studied individuals of both fauna and humans will reflect an average across more than a single environmental niche.

Bone samples were obtained from four institutions: Altai State University (Russia), Novosibirsk State University (Russia), the Institute of Archaeology in Karaganda (Kazakhstan) and Kostanay State University A. Baitursynov (Kazakhstan) (in total of 55 humans and 45 animals). Animal bones were not available from every site from which human remains were obtained. We tried to collect faunal samples as close as possible to the sampled humans.

Collagen was extracted following the standard method of the Dorothy Garrod Laboratory, at the McDonald Institute for Archaeological Research, University of Cambridge, UK. A volume of 500 mg of bone was taken from each sample and the surfaces of the bone pieces cleaned by sandblasting. Bones were demineralised in 0.5 M aq. hydrochloric acid at 4°C for up to 10 days, changing the acid as necessary, until the mineral phase had dissolved. Then, after rinsing three times with distilled water, the samples were gelatinised in an acidic solution (pH 3) at 75°C for 48 h. The liquid fraction containing the gelatinised protein was filtered off using an Ezee-Filter (Elkay Products) and freeze-dried. Triplicate samples of approximately 0.8 mg were used for each analysis in the Godwin Laboratory, University of Cambridge, using an automated elemental analyser (Costech, Valencia, CA) coupled in continuous flow mode to a Thermo Finnigan Delta V isotope ratio-monitoring mass spectrometer (Bremen, Germany). Stable isotope concentrations are reported relative to the international standards – VPDB for carbon and AIR for nitrogen (Hoefs, 1997). Based on replicate analyses of international and internal standards, measurement errors are <0.1% for $\delta^{13}C$ values and 0.2% for $\delta^{15}N$ values.

Samples were tested for normality using Q-Q plots and Shapiro–Wilk tests. The parametric data were investigated using independent samples Student's *t*-tests. For non-parametric data, the Wilcoxon signed-rank test was used. Statistical analyses were performed using the software R version 3.2.2. Samples were tested for normality using $p < 0.05$ as a statistical significance level.

AMS dating

A total of 10 adult human bone samples were subject to AMS (accelerator mass spectrometry) ^{14}C dating (Table 1). All samples were prepared at the ^{14}CHRONO Centre for Climate, the Environment, and Chronology, Queen's University Belfast. The routine bone pre-treatment procedure of the ^{14}CHRONO laboratory involved a simple ABA treatment followed by gelatinisation (Longin, 1971) and ultrafiltration (Brown et al., 1988) using a Vivaspin® filter cleaning method introduced by Bronk Ramsey et al. (2004). The prepared collagen was sealed under vacuum in quartz tubes with an excess of copper oxide (CuO) and combusted at 850°C to produce carbon dioxide (CO_2). The CO_2 was converted to graphite on an iron catalyst following the zinc reduction method (Slota et al., 1987). The graphite was then pressed to produce a 'target', which was then subject to AMS dating. The ^{14}C age mean and standard deviation were calculated using the Libby half-life (5568 yr), following the conventions of Stuiver and Polach (1977). Calibration of the ^{14}C dates was undertaken using the IntCal013 calibration curve (Reimer et al., 2013). All calibrated ^{14}C ages are given at ±95.4%, that is, $\pm 2\sigma$ probability (OxCal 4.1).

It is important to mention that a reservoir effect could be present in the dated samples, as high $\delta^{15}N$ values, especially among the Neolithic population (e.g. Cook et al., 2002) (see section 'Results'), could suggest possible freshwater fish consumption. Previous studies on the reservoir effect in the Samara region of southwestern Russia, the northern Caucasus and Ukraine regions found *ca*. 400 year reservoir effect in dated individuals linked to fish consumption (Anthony, 2007; Lillie et al., 2009; Shishlina et al., 2012). The studies by Svyatko et al.

Table 1. ^{14}C dates, $\delta^{13}C$ and $\delta^{15}N$ values of humans from southwest Siberia.

Sample ID	Site name	Mean $\delta^{13}C_{VPDB}$ (‰)	Mean δ_{15NAIR}(‰)	^{14}CHRONO Lab code	^{14}C Age	±	AMS $\delta^{13}C$	Cal. BC 95.4 (2σ) OxCal 4.1	Period
Al_01	Itkul (Bolshoi Mys)	−19.68	15.94	UBA-22950	6470	40	−19.1	5510–5342	Neolithic
Al_09	Itkul (Bolshoi Mys)	−22.12	15.60	UBA-22951	6577	35	−20.9	5614–5478	Neolithic
Al_13	Ust-Isha	−21.55	15.94	UBA-22952	5114	47	−20	4035–3792	Neolithic
Al_30	Firsovo-XI	−15.30	11.46	UBA-22953	2044	34	−14.8	166–26	Early Iron Age
Al_38	Firsovo-XI	−23.25	14.24	UBA-22954	6684	39	−19.8	5667–5531	Neolithic
Al_51	Solontsy-5	−18.57	12.48	UBA-22954	6354	41	−16	5469–5224	Neolithic
Al_53	Solontsy-5	−21.56	13.98	UBA-22956	5081	38	−22.4	3965–3792	Neolithic/Chalcolithic
Al_22	Tuzovskie Bugry-I	−23.30	13.75	UBA-22957	5004	36	−19.3	3943–3701	Neolithic/Chalcolithic
Al_40	Firsovo-XIV	−19.90	10.63	UBA-22958	3311	34	−19.4	1684–1512	Middle Bronze Age
Al_45	Teleutsky Vzvoz-I	−19.36	13.13	UBA-22959	3837	33	−18.7	2459–2200	Early Bronze Age

Radiocarbon data calibrated against the IntCal13 calibration curve (Reimer et al., 2013).

Table 2. Summary of $\delta^{13}C$ and δvalues of faunal samples from the Neolithic/Chalcolithic and Bronze Age of the southwest Siberia and northern Kazakhstan.

Species (*n*)	$\delta^{13}C_{VPDB}$ (‰)					δ_{15NAIR} (‰)				
	Max	Min	Mean	SD	Median	Min	Max	Mean	SD	Median
Neolithic/Chalcolithic fauna										
Big mammal (11)	−17.5	−21	−19.3	0.9	−19.3	4.9	10.6	7.3	1.7	6.9
Horse (4)	−18.5	−20.6	−19.6	1.2	−20	3.7	5.9	4.6	1	4.1
Roe deer (3)	−21.4	−19.2	−20.2	1.1	−19.3	4.4	5.5	4.9	0.5	5
Beaver (3)	−19.9	−20.9	−20.4	0.5	−20.3	3.8	4	3.9	0.1	3.8
Dog/wolf/fox (3)	−18.2	−22.3	−19.9	2.2	−19	11.6	9.6	10.5	1	10.5
Bronze Age fauna										
Sheep/goat (28)	−17.6	−19.8	−19	0.5	−19.1	8.5	3.3	6.1	1.1	6
Cattle (23)	−18.4	−20.7	−19.5	0.5	−19.6	5.1	8.8	6.7	1.0	6.6
Horses (15)	−19.3	−20.9	−20.3	0.4	−20.3	6.9	4.2	6	0.8	6.1
Dogs (3)	−17.7	−19	−18.3	0.6	−18.1	12.3	9.3	10.6	1.5	10.3
Saiga antelope (1)	−20.4	−20.4	−20.4			5.3	5.3	5.3		
Sus sp. (1)	−19.6	−19.6				6.1	6.1			

SD: standard deviation.

(2015) in northeastern Kazakhstan suggest a possible reservoir effect of up to 300 years in Chalcolithic samples. On the other hand, Svyatko et al. (2009) and Marchenko et al. (2015) found no evidence for a reservoir effect in Chalcolithic and Bronze Age human samples from the Minusinsk Basin in Siberia and the Baraba forest-steppe in southwest Siberia. As such, the existence of a freshwater reservoir effect on dated individuals from southwest Siberia needs to be assessed on a site-by-site basis.

Results

In total, 49 of the 55 humans (89.1% success rate) and 45 animal (100% success rate) samples produced collagen with atomic C:N values within the acceptable quality range of 2.9–3.6 (DeNiro, 1985). The other human and faunal data used in this publication were taken from already published sources by Motuzaite Matuzeviciute et al. (2015) and Ventresca Miller et al. (2014), making a total of 86 human and 95 faunal data points. For the statistical analysis, we excluded one human outlier Al_30 from the Neolithic Firsovo-XI site with $\delta^{13}C$ values of −15.3‰ and $\delta^{15}N$ values of 11.46‰, as the dating of this sample has shown that the individual belongs to the Early Iron Age period (166–26 cal. BC) (Table 1). The information on faunal data is presented in Table 2 and Figure 2 (all faunal data are described in detail in SOM 1a, available online), on humans in Table 3 and Figure 3 (all human data in detail is described in SOM 1b, available online) and the statistical outputs in Table 4.

Faunal results

All of the faunal data are reported here, but only a subset was examined for change over time – that of the herbivore animals, which included ovicaprids, horses, big mammals (bovines or horses that were not identified to genus due to bone's taphonomic conditions), Saiga antelope and Roe deer.

The faunal data of the herbivore animals comprise 18 individuals attributed to the Neolithic/Chalcolithic period and 67 individuals to the Bronze Age period. The herbivores have carbon isotopic values between −21‰ and −17.5‰ which suggest consumption of mainly C_3 vegetation, with some input of C_4 plants. The nitrogen isotopic values are more variable, a range of 3.7‰ to 10.6‰, but clustering between 5‰ and 7‰, fairly typical of herbivores.

To examine change over time, we first compared the herbivores from the Neolithic (Tavdinsky Grotto and Belkargaly-1) to the Chalcolithic period (Botai-I, Kozhai-1, Belkargaly-1 sites). The statistical results have shown no difference in faunal isotopic results, the $\delta^{13}C$ values ($p = 0.6897$) and $\delta^{15}N$ values ($p = 0.8941$) (Table 4, output 1).

This results allowed us to combine both Neolithic and Chalcolithic herbivore fauna bone collagen results and compare them to the Bronze Age herbivore bone collagen analysis results. A comparison of Neolithic/Chalcolithic herbivores (mean: $\delta^{13}C$ 19.5‰ ± 1 and $\delta^{15}N$ 6.3‰ ± 2) against the Bronze Age herbivores (mean: $\delta^{13}C$ 19.5‰ ± 0.7 and $\delta^{15}N$ values 6.4‰ ± 1.2) shows that statistically there is no difference between periods in both $\delta^{13}C$

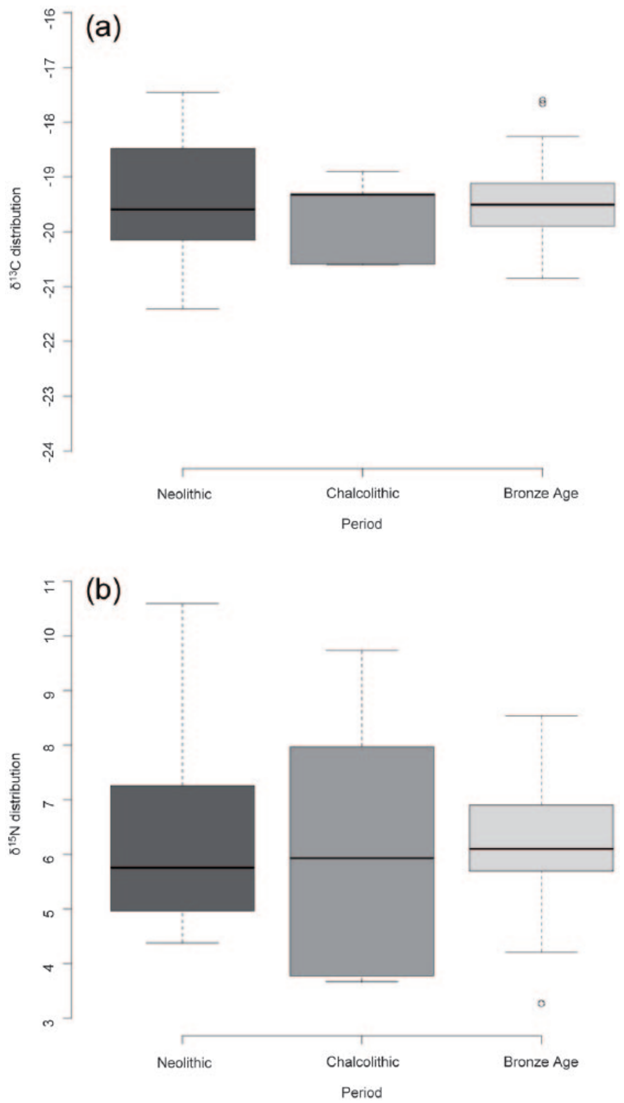

Figure 2. The box plot showing the (a) δ^{13}C and (b) δ^{15}N values of the Neolithic/Chalcolithic and Bronze Age herbivore animals from the southwest Siberia and northern Kazakhstan.

values (p=0.7993) and δ^{15}N values (p=0.798) (see Table 4, output 2, Figure 2).

Of the remaining faunal data, of six canidae (fox, wolves and dogs (Kz31F, Kz08F, Al13F, GM65F, GM64F, GM63F)), δ^{13}C values range between −22.3‰ and −17.3‰, while δ^{15}N values range between 12.3‰ and 9.6‰. The three samples of beavers (Kz30F, Kz32F, Kz33F) have δ^{13}C values of between −21‰ and −19.9‰, while the δ^{15}N values range between 4‰ and 3.8‰. One *Sus*. sp. (Kz_13F) sample was also considered an outlier as it is an omnivore animal that has δ^{13}C value of −19.6‰ and δ^{15}N values of 6.1‰.

Human results

The radiocarbon dates received for this paper from 10 dated individuals range between *ca.* 5700 and 3700 cal. BC for the Neolithic/Chalcolithic periods and between *ca.* 2500 and 2200 cal. BC for the Early Bronze Age period (Table 1). For the Neolithic/Chalcolithic period, the average human–faunal offset values are only 0.1‰ in δ^{13}C and 7.8‰ in δ^{15}N, suggesting that people consumed high amounts of aquatic foods with a long food chain. A similar situation was observed from the Bronze Age period where the average human–faunal offset values are −0.4‰ in δ^{13}C and 5.5‰ in δ^{15}N.

The Neolithic/Chalcolithic humans (n=40) from five sites in southwest Russia have a mean δ^{13}C value of −21.8±1.1‰ and a mean δ^{15}N value of 13.8±0.9‰. The Bronze Age humans (n=45) from four sites in the north of Kazakhstan and southwest Siberia have a mean δ^{13}C value of −19±0.6‰ and a mean δ^{15}N value of 11.8±1‰. First, after comparing the Neolithic versus Chalcolithic humans, no significant differences in both δ^{13}C and δ^{15}N are seen (Table 4; output 3, Figure 3). By comparing Neolithic/Chalcolithic human mean values with the Bronze Age humans, significant differences in both δ^{13}C and δ^{15}N are seen (Table 4; output 4, Figure 3). Despite the small sample size, humans from the Neolithic/Chalcolithic of the Tuzovskie Bugry-I (n=13) are significantly different to the humans from the Early Bronze Age site Teleutsky Vzvoz-I (n=4) (Table 4; output 5), indicating a change in diet.

Discussion

The consistency of δ^{13}C and δ^{15}N values of herbivorous animals across the Neolithic/Chalcolithic and the Bronze Age periods indicates that the documented climatic fluctuation during the Bronze Age did not have any significant influence on the animal isotopic signatures though time, despite potential vegetation change and the possible change in plant δ^{13}C and δ^{15}N values due to aridity and increased salinity (e.g. Flohr et al., 2011; Heaton, 1987).

The significant difference in both δ^{13}C and δ^{15}N values of humans between the Neolithic/Chalcolithic and Bronze Ages indicates a significant shift in human diet between these periods, which cannot be due to isotopic changes in consumed fauna. The humans dating to the Neolithic/Chalcolithic periods have relatively low δ^{13}C and relatively high δ^{15}N which does not suggest a high reliance on terrestrial animals (Figure 4) and rather probably indicates exploitation of aquatic animals. The humans dating to the Bronze Age show higher δ^{13}C values (by up to −2.8‰) which is probably linked with the beginning of livestock breeding in the region and the increased consumption of animals that grazed on mixed C_3/C_4 vegetation, while the lower δ^{15}N values (by up to 2‰) show a drop in the trophic level and a possible reduction of fish consumption. The isotopic data also suggests a reduction in the consumed food variety among humans during the Bronze Age (Figure 4), as they have more narrowly distributed carbon and nitrogen isotopic values than individuals from the Neolithic/Chalcolithic. The isotopic results of the Bronze Age humans cluster closer above the fauna from that period (Figure 4). Noteworthy, the dietary change in the Bronze Age individuals appears to coincide roughly with the earliest evidence for ovicaprids in the region (second half of the 3rd millennium cal. BC; Kiryushin et al., 2005), which might imply a relatively rapid transition to a different diet and economy. Further studies might be able to clarify whether the first pastoralists in the region under study were local populations who adapted pastoralism or people who migrated into these territories with their livestock.

According to the stable isotope data, the dietary shift in the humans of southwest Siberia is visible from the Early Bronze Age period (second half of the third millennium cal. BC). However, looking at the published isotopic data of two humans from Chalcolithic sites in northern Kazakhstan (Botai) and central Kazakhstan (Karagash), their values are very similar to the ones of the Bronze Age periods (Karagash human: δ^{13}C value is −18.2‰ and δ^{15}N is 12.6‰ (Motuzaite Matuzeviciute et al., 2015); Botai human: δ^{13}C=−18.1‰; δ^{15}N=12.4‰ (O'Connell et al., 2003)). As mentioned earlier, the Karagash burial contained the earliest evidence of domesticated sheep in the region, while the Botai site contained the earliest evidence of horse domestication (Outram et al., 2011). Both sites are dated between 3500 and 2700 cal. BC (Motuzaite Matuzeviciute et al., 2015; Outram et al., 2009).

Table 3. Summary of δ^{13}C and δ^{15}N values of human samples from southwest Siberia and Northern Kazakhstan.

Site name (per site-*n*)	Period (total-*n*)	$\delta^{13}C_{VPDB}$ (‰)					δ_{15NAIR}(‰)				
		Min	Max	Mean	SD	Median	Min	Max	Mean	SD	Median
Neolithic/Chalcolithic											
	Neolithic/Chalcolithic (40)	−23.4	−18.6	−21.8	1.1	−21.9	12.5	15.9	13.8	0.9	13.6
Itkul (Bolshoi Mys) (9)	Neolithic	−22.1	−19.7	−21	0.8	−21.1	12.6	15.6	13.4	0.5	13.3
Ust-Isha (3)	Neolithic	−21.7	−21.4	−21.6	0.2	−21.6	15.5	15.9	15.6	0.3	15.5
Firsovo-XI (9)	Neolithic	−23.4	−22.5	−22.8	0.3	−22.8	12.5	14.2	13.3	0.6	13.3
Solontsy-5 (6)	Neolithic	−21.6	−18.6	−20.3	1	−20.6	12.5	15.7	13.6	1	13.6
Tuzovskie Bugry-I (13)	Neolithic/Chalcolithic	−23.4	−20.9	−22.2	0.6	−22.3	13.4	14.6	13.9	0.4	13.6
Bronze Age											
	Bronze Age (45)	−21.3	−17.5	−19	0.6	−18.9	9.9	14.4	11.8	1	11.7
Novoilinovka (8)	Bronze Age	−21.2	−18.6	−18.9	0.2	−18.9	11.2	11.4	10.8	0.2	11.2
Lisakovsk (28)	Bronze Age	−19.7	−17.5	−18.8	0.4	−18.8	9.9	14.4	12.0	1.0	12.0
Teleutsky Zvoz-I (4)	Bronze Age	−21.03	−18.3	−19.7	1.2	−19.7	12.4	13.2	12.8	0.4	12.9
Firsovo-XIV (5)	Bronze Age	−19.9	−19	−19.5	0.3	−19.4	10.5	11.4	11	0.4	10.9

Novoilinovka data were taken from Motuzaite Matuzeviciute et al. (2015) and Lisakovsk from Ventresca Miller et al. (2014).

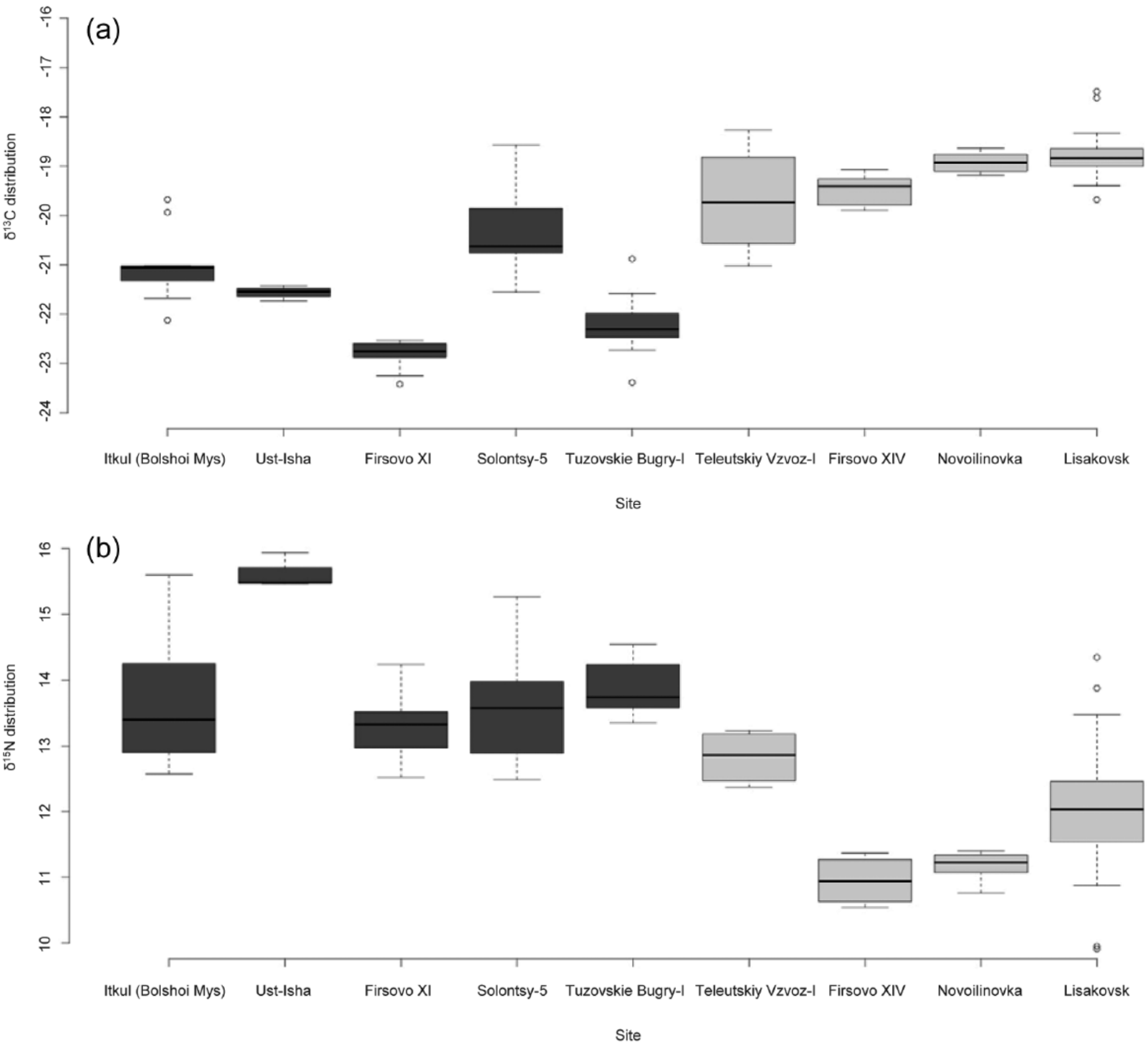

Figure 3. The box plot showing the (a) δ^{13}C and (b) δ^{15}N values of the Bronze Age humans from the southwest Siberia and northern Kazakhstan. Darker colour indicates the Neolithic/Chalcolithic humans, while lighter colour the Bronze Age humans. The human data from the north Kazakhstan are supplemented by Motuzaite Matuzeviciute et al. (2015) and Ventresca Miller et al. (2014).

Table 4. Results of statistical tests.

Comparison	n	$\delta^{13}C$ p value	Test for $\delta^{13}C$ values	$\delta^{15}N$ p value	Test for $\delta^{15}N$ values
Statistical test outputs of fauna					
1. Neolithic versus Chalcolithic herbivores	18	0.6897	*t*-test	0.8941	*t*-test
2. Neolithic/Chalcolithic versus Bronze Age herbivores	85	0.7993	*t*-test	0.798	Wilcoxon signed-rank
Statistical test outputs of human data					
3. Neolithic versus Chalcolithic humans	40	0.12	Wilcoxon signed-rank	0.678	*t*-test
4. Neolithic/Chalcolithic humans versus Bronze Age humans	86	$9.12E^{-14}$	Wilcoxon signed-rank	$3E^{-11}$	Wilcoxon signed-rank
5. Tuzovskie Bugry-I (Neolithic/Chalcolithic) versus Teleutsky Vzvoz-I (Early Bronze Age)	17	$2.78E^{-5}$	*t*-test	0.000294	*t*-test

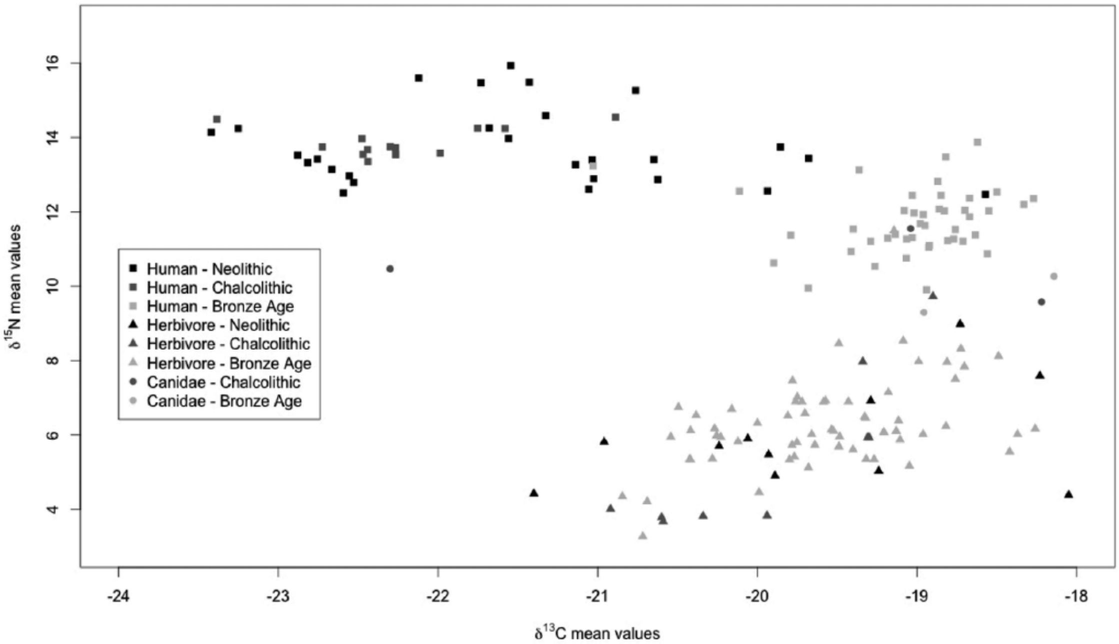

Figure 4. Graph showing $\delta^{13}C$ (*x*) and $\delta^{15}N$ (*y*) values of the Neolithic/Chalcolithic and Bronze Age animals and humans from the southwest Siberia and northern Kazakhstan.

Despite having isotopic data just from two individuals, one can infer that the processes of transition to pastoralism visible in human isotope data started earlier in the south (central/northern Kazakhstan) than in the north (southwest Siberia).

Conclusion

Between the Neolithic/Chalcolithic and the Bronze Age periods of southwest Siberia and northern Kazakhstan, human bone collagen shows an isotopic change in both carbon and nitrogen while that of herbivorous animals remain similar. This suggests that climatic variation did not play a role in the observed change in human isotopic ratios. A change in diet and a reduction in consumed food variety among humans during the Bronze Age are probably the main reasons for the change in isotopic values. The increase in $\delta^{15}N$ values between the Neolithic/Chalcolithic and Bronze Age is probably related to the beginning of animal husbandry in the region and the consumption of animals that grazed on mixed C3/C4 vegetation. The high $\delta^{15}N$ values in both the Neolithic/Chalcolithic as well as Bronze Age humans suggest the consumption of fish, which in turn might result in the presence of a freshwater reservoir effect in the dated individuals. However, this has to be tested further to be confirmed.

The apparent human dietary changes during the second half of the 3rd millennium cal. BC can be linked to the establishment of pastoralism in southwest Siberia and the introduction of domesticated animals into the region. The data show that the processes of pastoralism further south in Kazakhstan, however, may have started slightly earlier. Overall, our results have demonstrated that the processes of globalisation that occurred within the northern regions of Central Asia and southwest Siberia at the third millennium BC coincide with globalisation processes in other regions of Central Asia (discussed in section 'Introduction').

Acknowledgements

We are thankful to C Kneale and J Rolfe for assistance with isotopic analysis, L Barbera Colominas for helping with animal bone identification, S Tur in the Altai State University for anthropological data, I Shevnina from the Kostanai State University A. Baitursynov for providing some faunal samples for this study as well as other researchers who contributed to the archaeological excavations of analysed material: AB Shamshin, N Yu Kungurova, K Yu Kiryushin, AS Fedoruk, SP Grushin and VN Logvin.

Funding

The authors are grateful to the McDonald Institute for Archaeological Research, European Research Council and the Leverhulme Trust for financial support. The archaeological excavations in southwest Siberia were carried out as a part of the Russian

Federation Ministry of Education and Science Project 'The Early Peopling of Siberia: Origins and Evolution of Cultures in Northern Asia' (Resolution No. 220), Altai State University, Grant No 14.Z50.31.0010.

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