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The origin of objects of invertebrate descent from the Khvalynsk Eneolithic cemeteries (Northern Caspian region)

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A B S T R A C T

The summary of the study of invertebrates used for decorative items from Khvalynsk Eneolithic Cemeteries is presented. AMS radiocarbon dating confirmed that freshwater *Unio* mother-of-pearl shells and nacre discs, manufactured from them, are coeval with the site and most likely local in origin. Tubes of the sea worms and marine shells are fossil and some may originate from local deposits. Other are probably related to the ancient Paratethys basin sediments and can have diverse origin spanning from Caucasus and Caspian shores to Carpathian and Mediterranean area. *Glycymeris* L. clams are most likely originated in the latter area. The discussion of the obtained AMS ^{14}C results agrees with a possibility of a noticeable ingress of younger carbon, irremovable by standard methods and affecting the determinations, probably related to a bacterial activity in deposits.

1. Introduction and regional setting

The Copper Age burial grounds known as Khvalynsk Eneolithic Cemetries (KEC), are located in the Khvalynsk district of the Saratov region on the right bank of the Volga River (Fig. 1), occupying an area of over 1000 m^2. Individual, multi-tiered and single-tiered burials (about 160 skeletons) were discovered and excavated in 1977–1979 (further down marked as KEC-I – first stage excavation) and in 1987–1988 (KEC-II – second stage excavation) (Agapov et al., 1990; Pestrikova and Agapov, 2010). Most buried men, women and children were lying in a supine position; in some rare cases the posture of the deceased was a flexed position on the right or the left side. Grave offerings included ochre, clothes decoration, ceramics, stone tools, metal objects. High-status burials contained scepters, axes and head bands with copper rings.

KEC are situated at an ideal habitat for various birds and mammals: the wide Volga River floodplain with numerous channels, islands and lakes; forests; high and steep river banks; steppe areas. The large number of items and decorations from animal bones and teeth of different taxa was excavated. Vertebrates (birds and mammals) are represented by species still living in the area: Swan (*Cygnus* sp.), large predatory bird from Accipitridae family, White-tailed eagle (*Haliaeetus albicilla* L., 1758), Great bustard (*Otis tarda* L., 1758), also Beaver (*Castor fiber* L., 1758), Elk (*Alces* L., 1758), Red deer (*Cervus elaphus* L., 1758) and Wild boar (*Sus scrofa ferus* L., 1758).Besides the mentioned species, the cattle skeletal elements and "rings" from the tubular bones of a middle sizes ungulates (Kirillova, 2010) were also present. Multidisciplinary investigation of obtained materials together with anthropological and zooarchaeological studies of KEC allowed researchers to define the specific Khvalynsk pastoralist culture of the Volga basin area. It was formed on the base of Samara culture and occupied wide area – from Middle Volga in the north down to the Sea of Azov in the south-west, and Ural River in the south-east. Culturally it was close to the Sredny Stog Eneolithic culture, so that even one of the hypothesized origins of Khvalynsk culture was a suggested migration of Sredny Stog population eastward because of progressive aridization (Klein, 2007).

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Fig. 1. Location of the Khvalynsk Eneolithic cemeteries.

The isotope composition of collagen of bones of the deceased buried in the Khvalynsk burial ground demonstrates that river fish was an important component on their diet system (Shishlina et al., 2009). The assemblage of the grave offerings (scrapers, knives, perforators) indicates that people treated hides and skins and made such items as embroidered cloths ornamented with shell and bone beads. Artifacts indicating rank such as copper rings, ocher, stone scepters were, probably, acquired through trade exchange (Agapov et al., 1990; Pestrikova and Agapov, 2010).

A series of ^{14}C dates of the samples from the Khvalynsk burial ground produced an age range of 5500–4500 cal BC (Agapov et al., 1990; Chernykh et al., 2000). Calibrated radiocarbon dates and chronology of the Khvalynsk burial ground was based on human and animal bones and shells. However, ^{14}C dates of human bones obtained for cultures whose representatives consumed significant share of aquatic food can be older due to the reservoir effect (Lanting and van der Plicht, 1998; van der Plicht et al., 2016).

Paired ^{14}C dating of human, sheep, and cow bones from the Eneolithic Khvalynsk graves was used in order to quantify the size of the reservoir effect. The analyzed human bone from KEC-II grave 10 is 220 ± 95 years older than a cow bone from the same grave. The date of a ring made of an ungulate bone from KEC-I, grave 147, is the same as for the cow bone. For this grave, the reservoir effect appears to be about 275 ± 60 years (Shishlina et al., 2009).

This indicates that the chronology of Khvalynsk burials may be actually younger, possibly in the range of 4900–4200 cal yrs BC.

A great number of decorations made from invertebrate material, dominantly from various mollusc shells were discovered (Figs. 2 and 3). Especially numerous were rows of disk shaped beads, made of the freshwater Unionidae shells. Nacre beads were found to appear older than bone items (Chernykh et al., 2000). Their apparent radiocarbon age was in the range of 6500–7100 years, which corresponds to approximately 6400–5200 cal BC. The reason for this most probably is the local reservoir effect, which is discussed later.

Decorations made from marine organisms on the other hand were rare and represented commonly by rows of Scaphopoda and *Glycymeris* shells and fragments of calcareous tubes produced by polychaete worms of the family Serpulidae. The latter was only rarely noted as source material for decorations (Einwögerer, 2000; Ippolitov, 2010).

Studies to establish allochthonous or autochtonous character of invertebrate materials from KEC, as well as to correspond its geological age with the period of necropolises formation were done previously (Goncharova, 2010; Ippolitov, 2010; Kirillova, 2010; Popov, 2010). The earlier suggestion (Agapov et al., 1990), on the freshwater molluscs (Unionidae) origin of numerous nacre discs was confirmed. Also, taxonomic affiliation of sea worm tubes and marine bivalves used for decorations was determined, as well as origins of some materials. The local origin of freshwater shells and worm tubes was proposed. However, the question of sources for other invertebrates' materials in KEC, and whether they are coeval remained open. In the current study we are trying to extend the range of studied materials, produce new and combine the available data, and so shed more light on the origin of invertebrates used to make decorations from Khvalynsk burials, narrowing the possibilities.

2. Materials and methods

Samples of invertebrate materials from KEC-I, deposited at the Archaeological Department collection of the State Historical Museum, Moscow (SHM), and from KEC-II, deposited in Samara Regional Museum, Samara (SRM) were used in this study.

2.1. Bio-morphological study

Mollusc shells were studied by a malacofaunistic method including the analyses of taxonomical structure, taphonomy, biostratigraphical position, historical development, and biogeography of molluscs.

Serpulid tubes were provisionally identified among beads macroscopically, using principal definitive characters, and afterwards these beads were checked with SEM in longitudinal fractures. Serpulid tube structural diversity, which counts up to 13 types of crystal matter arrangement combined within 1–4 layers (Vinn et al., 2008; Ippolitov et al., 2014), supports both confident

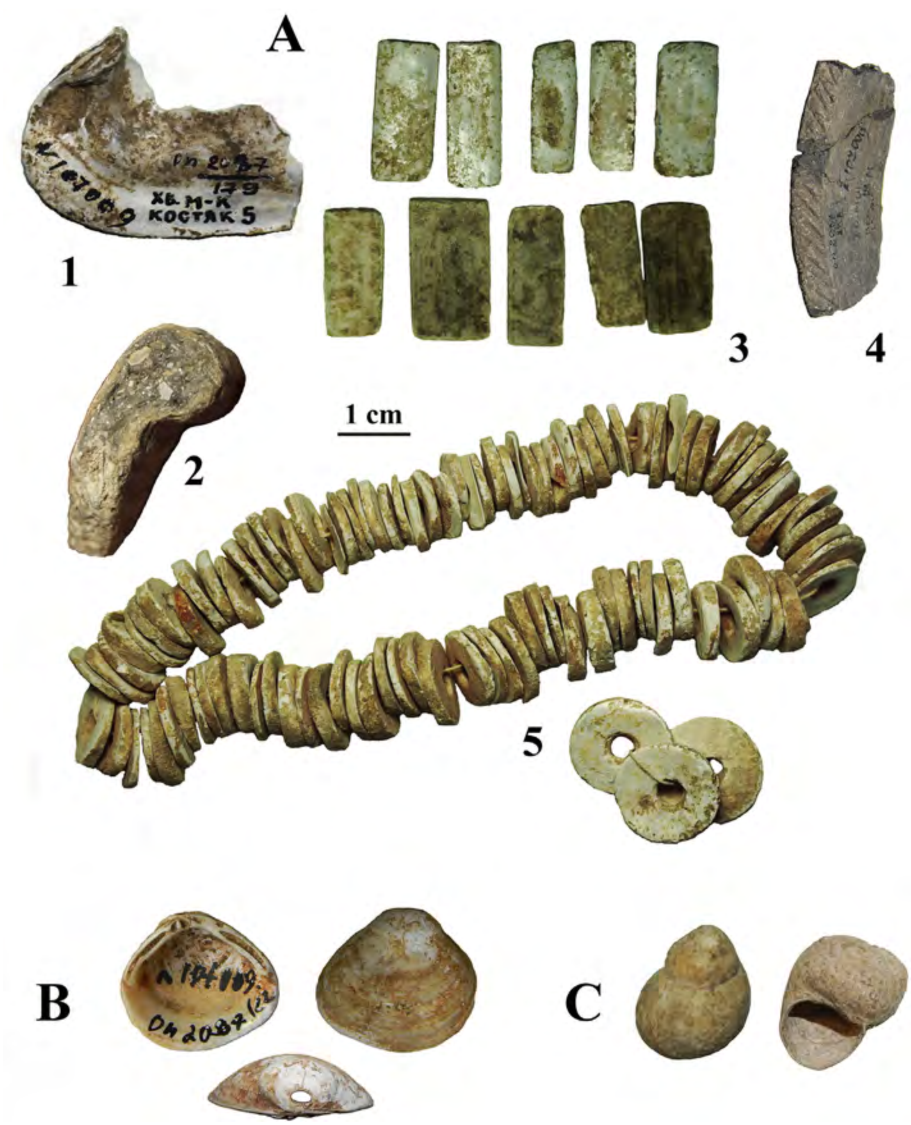

Fig. 2. Freshwater mollusc shells in use by Khvalynian population (KEC-I materials from SHM collection): A Unionidae shells: 1. fragment of *Unio* cf. *pictorum* from internment 5; 2. Unionidae shells fragment in the clay of the pottery from the burial; 3. Nacre squares (stamps or overlays); 4. Ornament on a clay pot, probably made with a nacre stamp; 5. Mother-of-pearl discs. **B** *Corbicula fluminalis* shell with the artificial hole at the umbo. **C** *Viviparus* cf. *viviparus* shells.

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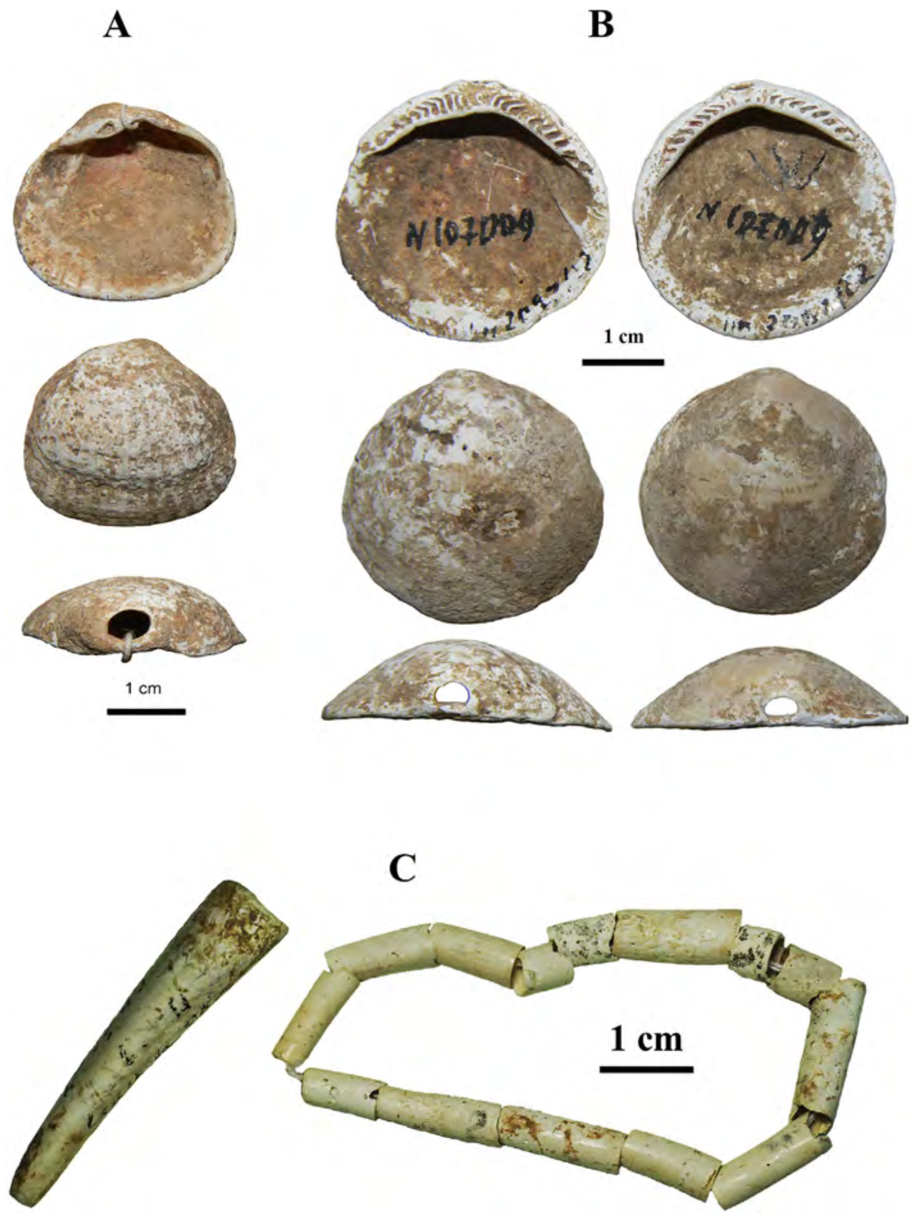

Fig. 3. Decorations made from marine shells in KEC-I: **A** *Didacnoides* cf. *caucasicus* shells with a man-made hole at the umbo. **B** *Glycymeris* clams with holes at umbos. **C** – Scaphopoda (tusk shell) samples.

recognition of serpulid nature of the beads and further possibility of more detailed identification up to genus level.

2.2. Isotopic study

Three samples of different shells (*Glycymeris*, pearl disk and *Unio* shell) were tested earlier for stable isotopes ^{13}C and ^{18}O to determine their marine or freshwater origin. Measurements were performed at the Laboratory of isotope geochemistry and geochronology of the Geological Institute of Russian Academy of Sciences, Moscow with a Delta V Advantage mass-spectrometer and Gas Bench II device from the Thermoelectron Corporation. The samples and standards IAEA C-O 1 were decomposed using H_3PO_4 at the 50 °C. The $\delta^{13}C$ values are given in per mil (‰) relative to the PDB and $\delta^{18}O$ - relative to the SMOW standard. Accuracy (reproducibility) of $\delta^{13}C$ and $\delta^{18}O$ measurements was ±0.2‰.

Five samples of shell material were tested for radiocarbon by AMS analysis at Australian Nuclear Science and Technology Organisation (ANSTO). After visual inspection for the absence or for the presence of any powdery, potentially extraneous, calcite deposition the samples went through the rigorous cleaning (Hua et al., 2001), namely shell surfaces were physically cleaned by abrasion of 10–25 percent of thickness with a Dremel® tool followed by chemical etching of another 10% with 0.5 M HCl for 1–5 min under sonication at room temperature. Aragonite shell species (OZT370, 371, 374) were then tested for the presence of calcite by a Feigl staining test (Kato et al., 2003). No non-uniformity or signs of white patches were observed allowing concluding that shells were continuous aragonite and no noticeable calcite diagenesis took place. Following the staining test, samples were cleaned using 0.25 M HCl, rinsed in Milli-Q, oven dried. Hydrolysis was performed with concentrated H_3PO_4 acid in acid pre-cleaned glassware. First ~25% of evolving CO_2 were also discarded to eliminate any possible surface diagenesis contamination. The remaining evolving CO_2 was collected, converted to graphite (Hua et al., 2004), and measured for ^{14}C on STAR AMS at ANSTO (Fink et al., 2004). Results were corrected for laboratory blanks, determined on a number of IAEA-C1 reference material (Marble) samples processed the same way as studied shells, and for $\delta^{13}C$ isotopic fractionation, which was determined on residual graphite material after AMS analyses. Stable isotope measurements were performed on a separate elemental analyser Elementar vario MICRO cube coupled to a Micromass Isoprime IRMS.

3. Results of study

3.1. Stable isotopes and radiocarbon measurements

Shells of *Glycymeris, Unio* and pearl disk were tested for stable isotopes ^{13}C and ^{18}O. Results of measurements are presented in Table 1.

The results confirmed the expected the freshwater origin of mother-of-pearl discs and *Unio* shells. Also, *Glycimeris* shells were found to be of marine origin as expected.

Shell materials were tested for radiocarbon by AMS analysis. Radiocarbon results as well as calibrated calendar age ranges are presented in Table 2.

Radiocarbon age of the studied *Unio* sample (Table 2) came out in agreement with previously determined for these specimens (Chernykh and Orlovskaya, 2010). The situation is more complex for the other studied shell items as they return ages not just by far preceding Khvalynsk culture but even Holocene. The possible causes of this are discussed later.

3.2. Fresh water shells and items made from them

3.2.1. Unio shells

The abundance of mother-of-pearl discs (Fig. 2 A5) and their good preservation advocate for local material contemporary with necropolis period (Agapov et al., 1990), which is supported by their stable isotope and radiocarbon determinations. Disk diameters are from 3 to 15–20 mm, thickness from 2 to 5 mm. Small discs are usually flat, while large ones are bent and have varying thickness, following the shape of the parent clam. Mother-of-pearl squares were also found in Khvalynsk I burials. Possibly, they were used as stamps to make ornaments or as overlays for pottery (Fig. 2 A).

Nacre items were usually produced from the live clams since the dried down shell produces too much waste. Also the mollusc body can be used as food. Consequently, the workshop should not be far from the settlement and the source of raw material. Judging from the distribution of the members of Unionidae family, most plausible species were the Painter's mussel (*Unio pictorum* (L., 1758)) and the Swollen River mussel (*U. tumidus* Philipsson, 1788). The Thick Shelled river mussel (*U. crassus* Philipsson, 1788) is also probable, but not the thin and fragile river mussels of *Anodonta* genus. All of the named species are widely spread in rivers and lakes of Eastern Europe. A fragment of mollusc shell of the Unionidae family, defined by us as *U.* cf. *pictorum* (Fig. 2A–1) of the suggested source species was found near the skeleton 5 in KEC-I burials. Same shells were discovered near the skeleton 2 in KEC-II. They exhibited signs of artificial wear and holes at umbos (Goncharova, 2010). These finds suggest that *U.* cf. *pictorum* was the main local raw material for manufacturing mother-of-pearl items.

Nacre discs are numerous in Neolithic and Bronze Age archaeological sites (Skelya, Mariupol, Khvalynsk and other cultures) in the southern parts of Russian Plain. They are less common to the east (Glazkov culture of the Baikal area) and to the west (cultures of the Balkans and Danube area), and are very rare for the more ancient cultures.

3.2.2. Corbicula shells

Other bivalve clams used for Khvalynsk decorations were identified as *Corbicula fluminalis* (O.F. Müller, 1774) (Fig. 2 B). It is a heat-loving freshwater clam species currently inhabiting rivers, lakes and brackish water bodies of Central and Southeast Asia, Middle East, Transcaucasia, and North Africa. Grave finds usually exhibited an artificial hole in the umbo section of the valve, and edges were sometimes ground flat. In the Khvalynsk period this clam geographic range did not include Lower and Middle Volga regions. However, they did populate rivers, deltas and estuaries of the Northwest coast of Black Sea, Transcaucasian and Caspian Southeast. Also, they are very common for Late Pleistocene sediments in Lower Volga area (Astrakhan' region) and Caspian shores, with excellent preservation. Those layers were deposited along the late Khazarian (MIS 5) transgression of Caspian Sea, which was warmer and more salty than in present (Yanina, 2012a,b, 2014). This species is also present in interglacial (Middle and Late Pleistocene) deposits along the Black Sea coast, southern Aral area, in Pliocene deposits up till Middle Volga and Belaya river (Bashkortostan), in Eopleistocene layers in Volga River basin up till Kama River, and in Late Pleistocene deposits in Bashkortostan (Goncharova, 2010). All that provided Khvalynsk people with ample sources of *Corbicula*, local and distant.

3.2.3. Viviparus shells

Gastropods from the family Viviparidae were encountered among the shell material in KEC, and were identified as the river snails *Viviparus* L., 1758 (Fig. 2 C). This species mainly inhabits major, slow-moving, lowland rivers and lakes all over Europe and prefers calcareous (alkaline) waters. For the period of Khvalynsk Eneolite the snails of this genus populated similar area as nowadays; and most probably the source of shell material from KEC was local.

3.3. Marine molluscs in Khvalynian burials

Three major groups of marine molluscs were found in Khvalynsk burials. These are *Didacnoides*, Scaphopoda and *Glycymeris* clams.

3.3.1. Didacnoides shells

Some shells among KEC-II decorations were described (Popov, 2010; Goncharova, 2010) as *Didacnoides* cf. *caucasicus* (Andrusov, 1923), a currently extinct genus and species. We found this species among KEC-I materials (Fig. 3 A). The discovered clams exhibited poor preservation, and demonstrated signs of natural roundness as well as man-made grounding and holes in umbos.

Table 1
Carbon and oxygen isotope composition of shells from KEC-I burials (from Kirillova, 2010).

Sample code	$\delta^{13}C$ ‰ PDB	$\delta^{18}O$ ‰ SMOW	Shell origin	Sampled specimen
K-2	1,9	29,0	Marine	*Glycymeris* clam
K-3	−12,0	18,3	Fresh water	Mother-of-pearl disk
K-5	−11,7	18,1	Fresh water	*Unio* shell

Table 2
Results of AMS ^{14}C analyses.

Shell sample	ANSTO	$\delta(^{13}C)$ per mil		percent Modern Carbon		Conventional ^{14}C Age		Cal years, BC	
	code	$\delta(^{13}C)$	1σ error	pMC	1σ error	yrs BP	1σ error	Median	2σ range
Glycymeris 2087/252, 15 k	OZT370	1.7	±0.1	0.51	±0.02	42380	±380	43400	44100–42700
Scaphopoda 2087/265	OZT371	0.3	±0.1	0.45	±0.02	43390	±450	44250	45200–43400
Serpulidae 2037/66	OZT372	−0.6	±0.1	0.58	±0.03	41410	±430	42600	43400–41700
Serpulidae 2087/70	OZT373	−5.3	±0.1	0.15	±0.02	52200	±1200	54300[a]	49900–55500[a]
Unio 2087/192, 107009	OZT374	−12.2	±0.1	40.45	±0.2	7270	±40	6150	6200–6050

[a] Calendar date for OZT373 was obtained by extrapolation beyond the IntCal13 calibration curve range.

This species is known from the Lower Pleistocene deposits on Caucasian and (seldom) on Mangyshlak shores of Caspian Sea (Nevesskaya et al., 1997). Consequently, only these two routes to Khvalynian burials can be considered.

3.3.2. Scaphopoda shells

Scaphopoda shells are rather numerous in Khvalynian burials and belong to three genera of the family Dentaliidae: *Coccodentalium* (Sacco, 1896) (*C.* cf. *trautscholdi* (Koenen, 1868)), *Antalis* Sacco (Adams and Adams, 1854) and *Fissidentalium* Fischer, 1885. Genus *Fissidentalium* is known since Cretaceous till modern times, widely spread and numerous in the seas of Atlantic, Pacific and Indian oceans. In the Miocene, this species populated Paratethys Sea, and survived till Pliocene in the Mediterranean. Species belonging to genus *Antalis* are also known from the Mesozoic. They are widespread in modern seas, and populated seas of Western and Eastern Europe in Palaeogene and Neogene, including Paratethys up till Middle Miocene. *Antalis* shells from Kvalynsk appear closer to Paratethys Neogene species and younger Mediterranean forms.

Coccodentalium cf. *trautscholdi* (Koenen, 1868) is known mainly from the Upper Eocene. It had a wide geographic spread in warm subtropical seas, in Late Eocene from Western Europe to Turkmenistan. Shells of this genus are known for Mediterranean from Late Oligocene till Pliocene only. In modern times just two species survived in Indian and Pacific oceans. Large fossilised shells were used to make decorations. Collections sites were probably to the East from Caspian Sea (*C.* cf. *trautscholdi*) or in the Eastern part of Paratethys basin in Middle Miocene deposits (*Fissidentalium*). Also the latter could originate from the Neogene deposits in Mediterranean.

Scaphopoda shells do not require much processing, since they have a natural through-hole opening at posterior end. Whole shells as well as fragments grouped by size were used for necklaces. Only large Scaphopoda of genus *Coccodentalium* were cut into fragments, though the use of broken bits with its later processing cannot be excluded. *Fissidentalium* and *Antalis* shells were not cut into fragments as a rule, being too fragile.

3.3.3. Glycymeris shells

Shells identified as *Glycymeris* L., 1758 were noted among the decorations unearthed in studied burials. They were usually of fair to good preservation, often with edges ground flat and man-made holes at umbos. *G. glycymeris* is marine species currently common for Atlantic shelf zone from Gibraltar to North Sea, and in Western and Eastern Mediterranean to Aegean Sea. In Europe for the period of Khvalynsk Eneolite (5000–4500 yrs BC) the bivalve clams of this genus had similar extent. It is reasonable to accept these as a source of shell material for KEC.

Glycymeris clams were noted in archaeological sites of various ages from Mediterranean, British Isles, southern reaches of Russian Plain, Northern Caucasus, Kazakhstan, Southern Siberia (Gryaznov, 1927; Kruglov et al., 1941; Vasiliev and Matveeva, 1986; Agapov et al., 1990; Light, 2003; Kiryushin et al., 2011). The description of Nalchik Eneolithic burials presents pictures of shells, defined as *Glycymeris* by A.G. Eberzin (Kruglov et al., 1941). Eberzin also claimed that the noted samples of clams at the Northern Caucasus at the period of Nalchik burials formation could be only of fossil origin, and judging from their good preservation proposed their Mediterranean origin (Kruglov et al., 1941). Determining the age of the *G. glycymeris* shell from the KEC-I hopefully could provide a clue to its origin.

However, the age of these clams from KEC burials, used by Eneolithic humans for decorations, determined by radiocarbon came out in 43960–42550 years range. That would take these shells to MIS 3 interstadial, and they require sea water with salinity not less than modern Mediterranean.

The nearby Caspian could not serve as a source of shells of this genus, since in MIS 3 it was a brackish water body (the end of Atelian regression and the early phase of Khvalynian transgression (Badyukova, 2007)) with mollusc fauna consisting mainly of Cardiidae (*Didacna, Monodacna, Hypanis* genera) as well as *Dreissena*. The Black Sea of the time was going through the Surozh transgression, less saline then modern state. There are not much data on its malacofauna. However, even in Karangatian transgression of the Black Sea with the highest known salinity for the basin, its well-studied malacofauna had no *Glycymeris* (Nevesskaya, 1965; Yanina, 2012a,b; Büyükmeriç and Yıldırım, 2016; Büyükmeriç et al., 2016). Hence one should not expect it in the Surozh basin, either.

The only possible source of these clams from the Mediterranean direction is the Mediterranean itself. *Glycymeris* shells are widespread in Eneolithic cultures of the Balkan-Carpathian region. Decorations made of *Glycymeris* shells are found in burials in all the area from the Balkans to Khvalynsk and even further East. It appears logical that they reached Volga region by barter and trade with neighbors. In MIS 3 Mediterranean was a warm basin with sea level below modern but not less than −30 m (Selivanov, 1996). We are not aware of publications covering the malacofauna of the period. However, since in the previous Tyrrhenian transgression (MIS 5) *Glycymeris* clams were widely spread, it is reasonable to expect it in MIS 3 Mediterranean basin. The problem is that those deposits were submerged by Holocene sea rise in Khvalynsk times. The only possibility then was to collect shells, which were dislodged and redeposited in Holocene sediments or preserved in Late Pleistocene middens well above the Holocene water level. Current consensus is that level of −30 m was reached 10-9 Kyrs ago. Transgression reached maximum at about 4–6 Kyrs BP with level exceeding modern by a few meters. Modern level was established not earlier than 3–4 Kyrs BP (Kaplin and Selivanov, 1999).

3.4. Sea worms tubes

Serpulid beads are rare. Tubes of serpulids are relatively long and have a wide hole in their central part, so producing the beads would require just cutting into short fragments. Among artifacts from KEC two types of serpulid beads are present, which can be interpreted as belonging to two different sources.

3.4.1. *Type I*

The first type was found in burials 23–25, skeleton №24 KEC-I. This type counts 8 elongated larger beads, up to 5–6 mm in outer diameter and 5–13 mm in length (Fig. 4A). Their serpulid nature is acknowledged by remarkably preserved morphological characters - longitudinal irregular sulci on the surface (Fig. 4 A2), typical for serpulids (Fig. 4 C), and a tooth-like bend of transverse growth lines (Fig. 4 A).

Ultrastructural study revealed only relicts of the primary structure. The combination of thick wall with 5–6 mm tube diameter is atypical for known recent serpulids, so we hypothesized that this group of beads derived from local finds of fossil tubes (Ippolitov, 2010). Morphologically comparable species is *Mucroserpula tricarinata* (J. de C. Sowerby, 1829), common in the Callovian–Oxfordian deposits (Jurassic, ~157 Ma; Ippolitov, 2007) of the Saratov region. The beads are likely to be produced from anterior, uncoiled part of the tubes (Fig. 4 B, D).

3.4.2. *Type II*

The second type of beads was found within two chaplets from KEC–II. This type includes elongated smaller beads (up to 7 mm length and up to 3 mm in diameter), with wall thickness 1/5–1/6 of diameter and having somewhat rounded hexagonal cross-shape. Three of them were subjected to SEM study.

A single hexagonal bead revealed two-layered structure (Fig. 4C). The tube wall consists of isometric or slightly elongated crystals 0.5–1 μm, but near the outer part of the wall there are inclined interbands, consisting of spherulites (Fig. 4C 2, 3). Longitudinal section also revealed parabolic growth lamellae (Fig. 4C–3), characteristic for serpulids. Two more beads from another chaplet (1 with hexagonal and 1 with rounded cross-section) revealed only isometric crystals, similar to described above, but having no spherulites near the outer side. Isometric crystals composing the walls in all cases can be interpreted as diagenetic derivate from elongated crystals, typical for most recent serpulids (Kupriyanova and Ippolitov, 2015). The combination of such crystals with thin spherulitic coverage along the outer side is the commonest architecture of serpulid tubes (Ippolitov and Rzhavsky, 2008; Vinn et al., 2008). Remarkably, in our samples spherulites were not found observed immediately at the outer surface, probably due to attrition by the hands of the Khvalynians.

Hexagonal shape of the tube and the ultrastructure of its wall are compatible with a single known serpulid species, *Serpula crenata* (Ehlers, 1908; non Costa, 1861; Vinn et al., 2008). However occurrences of this species include mostly deep environments of Indian and Pacific oceans (ten Hove and Kupriyanova, 2009),

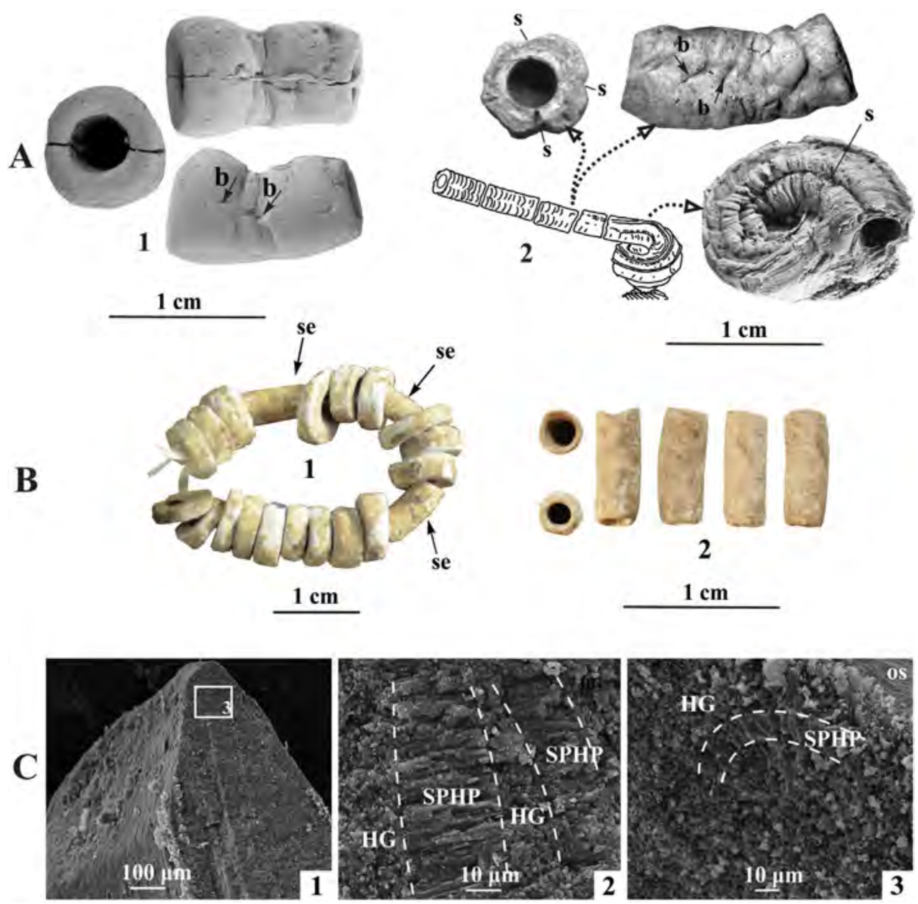

Fig. 4. Beads made of serpulid tubes from KEC and comparative material. **A** Serpulid bead of the type I and its possible source: 1. Bead from burial № 23–25, skeleton №24, KEC-II, coated with ammonium chloride; 2. *Mucroserpula tricarinata*, a fossil serpulid from the Jurassic deposits of European Russia and its reconstruction (after Ippolitov, 2010). **B** Serpulid bead of the type II: 1. Chaplet made of scaphopod and few serpulid fragments; SHM 107009 (KEC-I, burial № 21); 2. Isolated serpulid bead from the same chaplet, note barely perceptible hexagonal cross-section. **C** Ultrastructure of the bead of the second type: 1. Longitudinal section across the margin; 2. Spherulitic layers near the outer surface; 3. Parabolic growth lamellae near the outer surface. Abbreviations: b –bend of growth lines, s – sulci; se – serpulid beads; os – outer surface; ic – structure of isometric crystals; sph – spherulitic layers.

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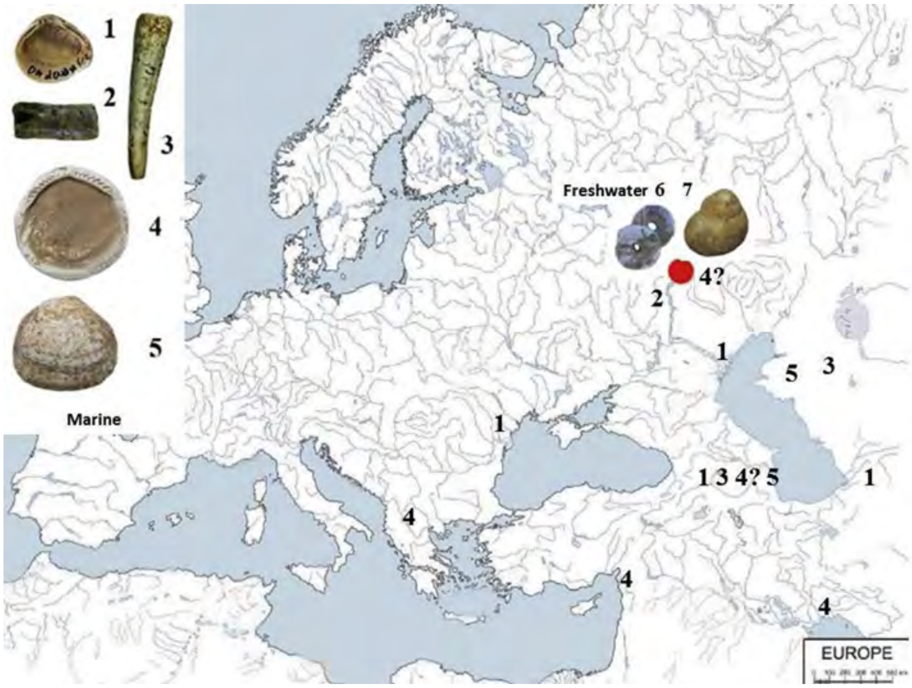

Fig. 5. Locations of possible sources for shells and sea worm tubes to KEC area (marked red). Marine shells (fossils) – various deposits: 1 - *Corbicula fluminalis*; 2 - Serpulid tubes of the first type; 3 - Scaphopoda (tusk shell); 4 - *Glycymeris*; 5 - *Didacnoides*; Freshwater shells (live) - local. 6 Mother-of-pearl discs and *Unio*; 7 *Viviparus* cf. *viviparus*.

making the affinity of beads with this species questionable. Radiocarbon AMS analysis performed for two of hexagonal beads, provided the age of 41550–55500 years making them not coeval with Khvalynian culture (Table 2) and obviously coming from some deposits. Fossil serpulids with comparable morphology are unknown at the moment, so the question about geographic origin of this second type remains open. Alternatively, available data about *S. crenata* distribution both in time and space can be incomplete.

4. Discussion

4.1. Ages and origin of freshwater material

We have performed radiocarbon analyses of the shell material from Khvalynsk burials with the aim of resolving the question of whether the live contemporary specimen were used as a source of raw material for decorations and other items, or fossils from various deposits. The calibrated age of *Unio* shells noticeably predates (Table 2) the expected period for Khvalynian culture of about 4500 BCE. The most obvious explanation is the significant local fresh water reservoir effect. We are not aware of any direct determination of reservoir effect for aquatic organism of these reaches of Volga River. However, the studies of shells and terrestrial samples from archaeological sites in the Lower Volga region (Zaitseva et al., 2009) demonstrated the age offset of aquatic material in the range of 500–1500 years. Studies of sites in between Volga and Don Rivers (Shishlina et al., 2009) also indicated reservoir age offset for aquatic organisms of about 1000 years. This agrees well with determinations for other European rives Danube and Rhine tributaries where reservoir offsets from 1000 to 1700 years were noted. Assuming such an offset for *Unio* material from Khvalynsk graves allows reconciling their radiocarbon determinations with expected necropolis age range, supporting the local contemporary production of the goods.

4.2. Ages and origin of marine shells

Marine shells were found not coeval with KEC, and originate from sediments. The apparent ages of all the studied specimens pointed to the MIS 3 period deposits, most of which are currently submerged by postglacial sea level rise. It is theoretically possible to invent some complex scenario to explain the appearance of shells of this age in Khvalynsk burials. There is however an alternative and simpler explanation. It requires the presence of some undetectable diagenesis of younger material into shells in deposits or in burials, which was not removed even by vigorous cleaning procedures. It is easier to suggest this for the studied Serpulidae tubes, since they are often built from calcite or calcite-aragonite mixture. Because of that they were not tested for the presence of calcite diagenesis by Feigl staining test prior to dating. In the second type of serpulid tubes, which were studied for radiocarbon by AMS, the principal layer is represented by homogenous granular (HG) structure, while in recent forms crystals of this layer normally have more or less elongated shape forming IOP structure (Vinn et al., 2008). This indicates at least some degree of diagenetic re-crystallization within serpulid beads. If we indeed agree to the presence of some unremoved diagenesis and more modern material in these supposedly fossil and radiocarbon dead shells we may use them as an indicator of this process, and subtract from the obtained results the percent Modern Carbon value averaged between two serpulid tubes as a blank correction.

In this case all ^{14}C results for marine shells in Table 2 would become either non-distinguishable from background, i.e. of infinite age in radiocarbon sense, apart from one case (OZT372), which would have > 50,000 years age limit, also agreeing with fossil origin from deposits predating MIS3. The result for *Unio* shell would not be noticeably affected by such a small close to background change and would still have the range of 6200–6100 cal yrs BC (2 sigma).

Such an explanation of the apparent age is especially welcome for Scaphopoda shells since it is difficult to come with any plausible scenario for the origin of the material under the assumption of correct radiocarbon ages; on the other hand Paratethyan deposits provide ample material. But the difficulty is that no signs of diagenetic calcite deposits were noticed on aragonitic Scaphopoda shells, tested by staining prior to dating. However, it was demonstrated (e.g. Busschers et al., 2014) that marine shells in deposits can be affected by diagenetic processes, possibly caused by bacteria, adding more recent carbonates in the same polymorph as the original shell structure (i.e. aragonite to aragonite and calcite to calcite). As a consequence, it is not possible to notice any diagenesis in such shells with staining tests. Also, even the very rigorous surface cleaning and large mass removal by etching would not be able to guarantee complete contamination avoidance.

For studied *Glycymeris* shells it would mean the fossil pre-MIS3 origin as well. Numerous deposits with well-preserved malacofauna and *Glycymeris* genus finds are noted in the Volga region (Geology of the USSR, 1967). But *G. glycymeris* is not noted there as well as in other Tertiary marine sediments of the Volga, Caspian and Ciscaucasia regions (Kirillova and Popov, 2005). It is worth noting that all fossil *Glycymeris* of Volga region mentioned above are different from those found in KEC. Lower Miocene deposits corresponding to Paratethys and very rich in *G. pilosa* (L., 1767) present in Central Georgia of the Caucasus (Geology of the USSR, 1964) demonstrating the suitability of conditions for this genus. Hence, the fossil origin of *Glycymeris* clams in Khvalynsk burials is plausible with a number of sources with shells from *Glycymeris* taxon situated closer than Mediterranean material. At the same time this does not negate the possibility of Mediterranean region to be the source of the studied shells (Büyükmeriç and Ilgar, 2016). Tyrrhenian transgression terraces (MIS 5) are widely present in Mediterranean, and often contain shells of *G. glycymeris*. There are even isolated instances of uplifted MIS 3 terraces (Goy and Zazo, 1986; Bruckner, 1986; Brancaccio et al., 1986; Dumas et al., 1988). Therefore, Khvalynian shells could be sourced from many possible locations. Currently we are unable to point exactly to a particular deposit as a source of the clams used.

5. Conclusions

Mollusk shells found as grave goods in Khvalynsk Eneolithic cemeteries excavations I and II were dated with high precision radiocarbon AMS determinations. Also, the stable isotope test on some items confirmed a freshwater origin of mother-of-pearl and Painter's mussel Unionidae shells. Radiocarbon dating of *Unio* shell confirmed its contemporary origin with Khvalynsk culture, if expected significant reservoir effect is taken into account. The required reservoir age correction of about 1500 years agrees well with values known for East European rivers and water bodies from the region adjacent to Khvalynsk settlements.

Marine shells, on the contrary, returned the apparent ages falling into the MIS 3 period. While this clearly rules out the use of contemporary live shells collected at their current habitats (Mediterranean Sea and Persian Gulf are closest to Khvalynsk suitable bodies of water), it poses difficult boundary conditions on scenarios of their extraction and collection since most of the MIS3 deposits are currently submerged by postglacial sea level rise.

We are suggesting as a plausible explanation – an undetectable by standard test methods post depositional diagenesis of younger carbonates into the shell structure in the same polymorph form producing the determined apparent age of shells, while their true age is anywhere beyond the radiocarbon limit. Summing up all the discussed data we may suggest the current logistics for invertebrates' material, delivered to Khvalynsk territory (Fig. 5):

- Freshwater bivalves (*Unio* cf. *pictorum*) are local and coeval with KEC.
- For fossil *Didacnoides* cf. *caucasicus* and *Corbicula fluminalis* the East Caucasian or East Caspian routes are suggested.
- Scaphopoda fossil shells could come from a wide area of ancient Paratethys Sea; the closest locations with good deposits are on the Ustyurt plateau and in Azerbaijan area of the Caucasus.
- One out of the two types of serpulid tubes from KEC derives from the local Jurassic deposits, while the origin of another type remains questionable.
- *Glycymeris* shells could potentially come from yet unknown local ancient deposits or together with *Corbicula fluminalis* fossils from the East Caucasian area. However, the Mediterranean/Balkan route is very plausible.

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