Excavations at Emeelt Tolgoi Site: The third Report on Joint Mongolian-Japanese Excavations in Outer Mongolia

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4 Radiocarbon dating and stable carbon and nitrogen isotopic analyses on human and animal bones from the Emeelt Tolgoi Site

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In order determine the age of tombs at Emeelt Tolgoi, we measured radiocarbon abundance ($^{14}C/^{12}C$) in bone collagen samples extracted from human and animal remains. Additionally, the carbon and nitrogen stable isotope ratios ($\delta^{13}C$ and $\delta^{15}N$) in collagen were measured as well, which reflect their protein source for the last decade prior to their death. The dietary habits at the Emeelt Tolgoi Site is compared with other Mongolian Bronze Age sites.

Materials and Methods

Eleven bone pieces including 3 animals and 8 humans were submitted to Laboratory of Radiocarbon Dating at the University Museum, the University of Tokyo (Table 6). We applied gelatinization to extract collagen from bone (Longin 1971; Yoneda et al. 2002). First, bone surface was cleaned by sandblasting and ultrasonic washing in ultrapure water (Milli-Q water) for 10 min. Cleaned bone pieces was reacted with weak alkali (0.2 M NaOH) for 15-18 hours to remove humic and fluvic acids originated from soil. After washing with ultrapure water to neutral pH, dried bone piece was crushed into fine powder and sealed in a cellulose tube to react with hydrochloric acid (1.2 M) gently for 15-18 hours. After washing with ultrapure water to neutral, remaining portion was recovered by centrifuging. The precipitation was heated at the temperature of 90°C in acidified water (pH 4) for 12 hours to extract gelatin. Dissolved gelatin was purified by Whatman GF/F filter and lyophilized. The weigh of extracted gelatin was recorded, and fraction was applied for the following analyses.

The concentration of carbon and nitrogen in gelatin was measured by Flash 2000 Elemental Analyzer and produced gases were introduced to a ConFlo IV interface and measured for stable isotope ratios in carbon and nitrogen using a Delta V isotope ratio mass spectrometer (Thermo Fisher Scientific, Germany). About 0.4 mg of gelatin were weighed in a tin cup and measured with laboratory standards (e.g. alanine) which can be traced back to international standards (V-PDB for carbon and AIR for nitrogen). Typical uncertainties with carbon and nitrogen isotopic ratios were 0.2‰ in $\delta^{13}C$ and $\delta^{15}N$ notation.

Extracted samples were combusted and reduced into graphite crystal on iron catalyst in two different ways. For the first series, we combusted samples in an evacuated and sealed tube at 850°C for 3 hours in an electric furnace with copper oxide and sulfix (Tube method, according to Minagawa et al. 1984). For the second series of samples, a sample portion containing 1 mg of carbon was combusted into CO_2 using Vario ISOTOPE SELECT elemental analyzer (Omori et al. 2017). Produced CO_2 was introduced to a vacuum line and sealed in a reaction vessel with enough amount of H_2 (2.2 times of CO_2) and 2 mg of iron powder catalysis in an isolated grass vessel with a stop cock. The graphite was reduced on the surface of catalysis at the temperature of 650ºC for 6 hours (Kitagawa et al. 1993).

Reduced graphite was pressed in an aluminum holder for accelerator mass spectrometry (AMS; National Electrostatics Corp. Compact AMS System) at the University Museum, the University of Tokyo. A series of international standards were measured simultaneously, and $\delta^{13}C$ measured by AMS was applied to correct the isotopic fractionations during preparation and measurement to calculate conventional radiocarbon date (CRA, Stuiver and Polach 1977). CRA was shown with one standard deviation error. Calibration was conducted with IntCal13 dataset (Reimer et al. 2013) using a calibration software, OxCal4.3 (Bronk Ramsey 2009).

Results

The results obtained by elemental analyzer was shown in Table 7. Because the gelatin yield less than 1% empirically suggest the degradation of collagen (van Klinken 1999), a human rib from Tomb M30 (above burial pit) might be altered by diagenetic effect. Another criterium, atomic C/N ratio at 4.5 out of biological range between 2.9 and 3.6, also suggested the extracted gelatin from this sample with

C/N at 4.2 could be altered and/or contaminated through diagenetic effect during deposition (DeNiro 1985; van Klinken 1999). Hence, the radiocarbon age on this samples could not be reliable, and we omit this from discussion for the dietary habit at the site. Other ten samples kept collagen in good conditions.

The results of radiocarbon dating is shown in Table 9 and the calibrated dates were illustrated in Figure 90 after removing a significant outlier (208±19 BP on the horse skull from M18). While we expected that the dates on animal remains and human skeletons from the same tombs of M18 and M82 showed similar dates, animal remains generally showed younger dates in both cases. The dates of human showed a significant variability from ca. 1500 cal.BC on skeleton from M18 untill ca. 500 cal.BC on that from M82. Except for a limb fragments found above burial pit of M30 showing younger data round 900 cal.BC, the other 5 skeletons showed general agreement in the early 2nd Millennium cal.BC.

The results of IRMS for $\delta^{13}C$ and $\delta^{15}N$ in comparison with international standards (V-PDB for carbon and AIR for nitrogen) were shown in Table 8 and Figure 91.

The youngest human from M82 showed significantly lower isotopic ratios in both carbon and nitrogen, which is similar to animals identifies as sheep or goat, suggesting the diet based on C3 plants. On the other hands older humans between 900-1500 cal. BP showed significantly higher carbon and nitrogen isotopic ratios. One of possible interpretation for this is consumption of plant using C4 photosynthesis, such as foxtail and broomcorn millets, whose higher valued in $\delta^{15}N$ might affected by manuring or other human activity on cultigens.

In a previous study, we analyzed some human samples from Mongolian Bronze Culture (Yoneda et al. 2016). The Daram uul and the Tevsh uul sites showed radiocarbon ages between 500 to 1000 cal. BC and 1000 to 1500 cal. BC, respectively. Their stable isotopes were compared in Figure 92 with data from Emeelt Tolgoi. Even in the younger period (around 500 cal. BC) the human form Daram uul had C4 based diet, while the diet based on the C3 ecosystem was shown for the diet of M82 of Emeelt Tolgoi, which might not be temporal change in the region. For more detailed discussion, different investigations such a as provenance research using enamel and other morphological features will be required.

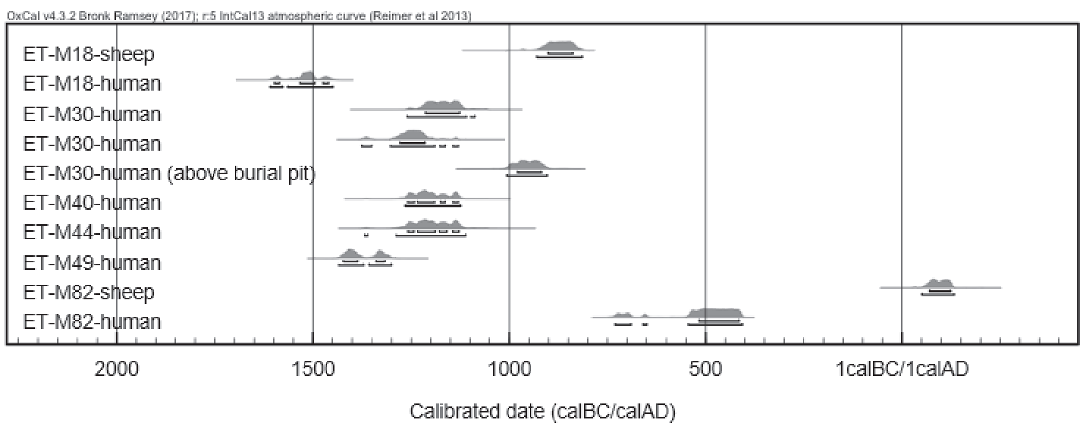

Fig.90 Calibrated radiocarbon ages on the bones from Emeelt Tolgoi.

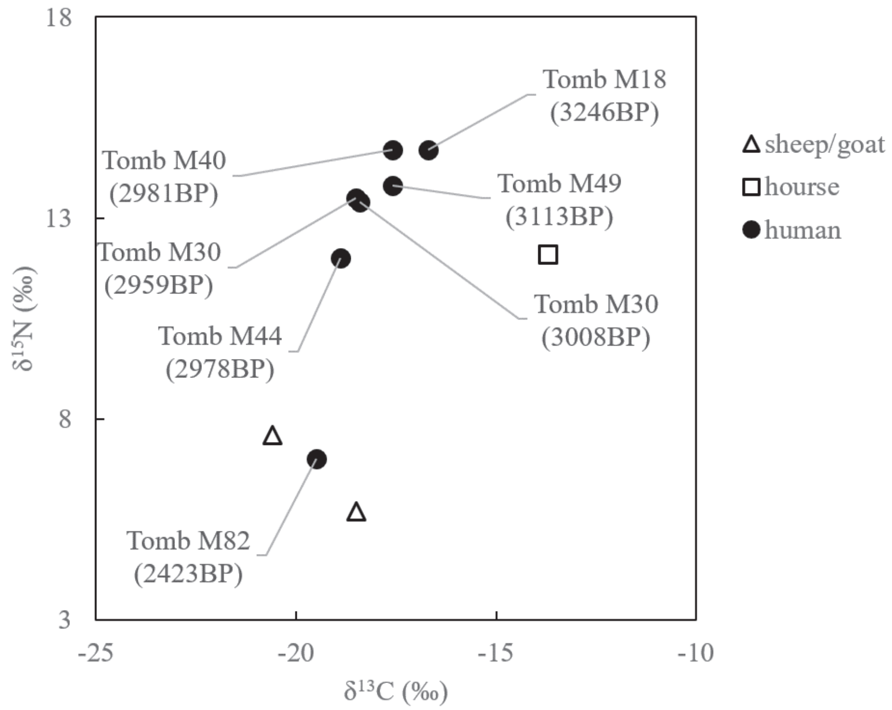

Fig.91 Carbon and nitrogen isotopic ratios in bone collagen. The tomb number and conventional radiocarbon ages (years BP) were shown for human remains.

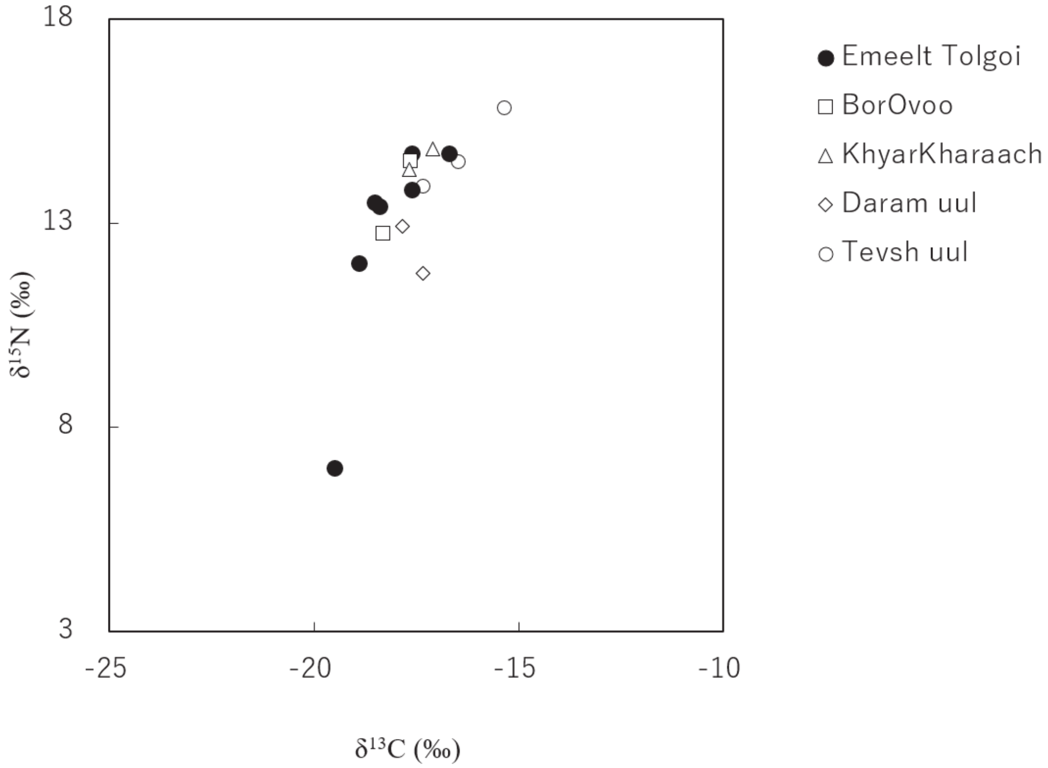

Fig.92 Carbon and nitrogen isotopic rations in Bronze-age human collagen from Mongolia.

Table 6 List o sample analyzed in this study and the yields of extracted gelatin.

Site	Location	Sample	species	skeletal element	Gelatin %
Emeelt Tolgoi	Tomb M18	No.4	horse	skull	9.9%
Emeelt Tolgoi	Tomb M18	No.5	sheep/goat	rib	11.0%
Emeelt Tolgoi	Tomb M18	No.8	human	fragmental piece	9.7%
Emeelt Tolgoi	Tomb M30	No.1	human	phalanx	7.5%
Emeelt Tolgoi	Tomb M30	No.2	human	femur	9.3%
Emeelt Tolgoi	Tomb M30 above burial pit	sample-1	human	limb fragment	**0.8%**
Emeelt Tolgoi	Tomb M40	sample-2	human	rib	3.2%
Emeelt Tolgoi	Tomb M44	No.7	human	rib	5.2%
Emeelt Tolgoi	Tomb M49	No.9	human	fragmental piece	14.0%
Emeelt Tolgoi	Tomb M82	No.6	sheep/goat	rib	7.2%
Emeelt Tolgoi	Tomb M82	No.10	human	fragmental piece	4.0%

Table 7 Carbon and nitrogen contents and atomic C/N rations in extracted gelatin.

Location	Sample	species	%C	%N	atomic C/N ratio	Remarks
Tomb M18	No.4	horse	45.3%	16.6%	3.2	
Tomb M18	No.5	sheep/goat	46.8%	16.7%	3.3	
Tomb M18	No.8	human	45.7%	16.3%	3.3	
Tomb M30	No.1	human	46.0%	16.3%	3.3	
Tomb M30	No.2	human	46.0%	16.5%	3.2	
Tomb M30 above burial pit	sample-1	human	37.6%	9.7%	**4.5**	altered C/N ratios
Tomb M40	sample-2	human	42.0%	14.8%	3.3	
Tomb M44	No.7	human	44.9%	14.7%	3.6	
Tomb M49	No.9	human	46.1%	16.6%	3.2	
Tomb M82	No.6	sheep/goat	46.0%	15.9%	3.4	
Tomb M82	No.10	human	44.7%	14.5%	3.6	

Table 8 Carbon and nitrogen isotopic ratios in gelatin.

Location	Sample	$\delta^{13}C$	$\delta^{15}N$	atomic C/N ratio	Remarks
Tomb M18	No.4	-13.7‰	12.1‰	3.2	
Tomb M18	No.5	-18.5‰	5.7‰	3.3	
Tomb M18	No.8	-16.7‰	14.7‰	3.3	
Tomb M30	No.1	-18.5‰	13.5‰	3.3	
Tomb M30	No.2	-18.4‰	13.4‰	3.2	
Tomb M30 above burial pit	sample-1	-19.2‰	13.8‰	**4.5**	altered C/N ratios
Tomb M40	sample-2	-17.6‰	14.7‰	3.3	
Tomb M44	No.7	-18.9‰	12.0‰	3.6	
Tomb M49	No.9	-17.6‰	13.8‰	3.2	
Tomb M82	No.6	-20.6‰	7.6‰	3.4	
Tomb M82	No.10	-19.5‰	7.0‰	3.6	

Table 9 Conventional and calibrated radiocarbon ages.

Location	Sample	Species	Lab-ID	Conventional radiocarbon age (uncalibrated)	Calibrated age (1 s.d.)	Calibrated age (2 s.d.)	Calibrated age (1 s.d.)	Calibrated age (2 s.d.)	Calibration curve	Remarks
Tomb M18	No.4	horse	TKA-19014	**208 ± 19 BP**	292 cal BP(18.5%)280 cal BP 172 cal BP(38.3%)152 cal BP 8 cal BP(11.4%)-1 cal BP	301 cal BP(30.2%)268 cal BP 212 cal BP(1.7%)205 cal BP 189 cal BP(47.5%)147 cal BP 14 cal BP(16.0%)-5 cal BP	1659AD(18.5%)1670AD 1779AD(38.3%)1799AD 1943AD(11.4%)1951AD	1650AD(30.2%)1682AD 1738AD(1.7%)1745AD 1762AD(47.5%)1803AD 1937AD(16.0%)1955AD	IntCal13, Bomb13 NH2	
Tomb M18	No.5	sheep/goat	TKA-19015	2734 ± 29 BP	2851 cal BP(68.2%)2789 cal BP	2879 cal BP(95.4%)2764 cal BP	902BC(68.2%)840BC	930BC(95.4%)815BC	IntCal13	
Tomb M18	No.8	human	TKA-19018	3246 ± 22 BP	3548 cal BP(8.4%)3536 cal BP 3483 cal BP(50.8%)3445 cal BP 3424 cal BP(9.0%)3410 cal BP	3559 cal BP(15.2%)3526 cal BP 3514 cal BP(80.2%)3399 cal BP	1599BC(8.4%)1587BC 1534BC(50.8%)1496BC 1475BC(9.0%)1461BC	1610BC(15.2%)1577BC 1565BC(80.2%)1450BC	IntCal13	
Tomb M30	No.1	human	TKA-19011	2959 ± 22 BP	3163 cal BP(68.2%)3077 cal BP	3209 cal BP(94.4%)3059 cal BP 3047 cal BP(1.0%)3038 cal BP	1214BC(68.2%)1128BC	1260BC(94.4%)1110BC 1098BC(1.0%)1089BC	IntCal13	
Tomb M30	No.2	human	TKA-19012	3008 ± 22 BP	3227 cal BP(68.2%)3165 cal BP	3325 cal BP(5.9%)3300 cal BP 3252 cal BP(85.2%)3140 cal BP 3126 cal BP(1.7%)3113 cal BP 3093 cal BP(2.6%)3080 cal BP	1278BC(68.2%)1216BC	1376BC(5.9%)1351BC 1303BC(85.2%)1191BC 1177BC(1.7%)1164BC 1144BC(2.6%)1131BC	IntCal13	
Tomb M30 above burial pit	sample-1	human	TKA-17500	2802 ± 21 BP	2928 cal BP(68.2%)2868 cal BP	2956 cal BP(95.4%)2854 cal BP	979BC(68.2%)919BC	1007BC(95.4%)905BC	IntCal13	altered C/N ratios
Tomb M40	sample-2	human	TKA-17501	2981 ± 21 BP	3208 cal BP(11.7%)3192 cal BP 3184 cal BP(39.6%)3141 cal BP 3124 cal BP(6.7%)3113 cal BP 3093 cal BP(10.2%)3080 cal BP	3215 cal BP(95.4%)3074 cal BP	1259BC(11.7%)1243BC 1235BC(39.6%)1192BC 1175BC(6.7%)1164BC 1144BC(10.2%)1131BC	1266BC(95.4%)1125BC	IntCal13	
Tomb M44	No.7	human	TKA-19017	2978 ± 28 BP	3208 cal BP(9.4%)3193 cal BP 3183 cal BP(35.2%)3139 cal BP 3128 cal BP(12.2%)3109 cal BP 3094 cal BP(11.4%)3079 cal BP	3318 cal BP(0.4%)3311 cal BP 3238 cal BP(95.0%)3061 cal BP	1259BC(9.4%)1244BC 1234BC(35.2%)1190BC 1179BC(12.2%)1160BC 1145BC(11.4%)1130BC	1369BC(0.4%)1362BC 1289BC(95.0%)1112BC	IntCal13	
Tomb M49	No.9	human	TKA-19019	3113 ± 22 BP	3373 cal BP(44.8%)3336 cal BP 3288 cal BP(23.4%)3267 cal BP	3384 cal BP(57.4%)3321 cal BP 3307 cal BP(38.0%)3250 cal BP	1424BC(44.8%)1387BC 1339BC(23.4%)1318BC	1435BC(57.4%)1372BC 1358BC(38.0%)1301BC	IntCal13	
Tomb M82	No.6	sheep/goat	TKA-19016	1909 ± 21 BP	1879 cal BP(68.2%)1826 cal BP	1898 cal BP(95.4%)1817 cal BP	71AD(68.2%)125AD	52AD(95.4%)133AD	IntCal13	
Tomb M82	No.10	human	TKA-19020	2423 ± 21 BP	2466 cal BP(68.2%)2364 cal BP	2680 cal BP(11.0%)2640 cal BP 2609 cal BP(2.1%)2599 cal BP 2494 cal BP(82.3%)2356 cal BP	517BC(68.2%)415BC	731BC(11.0%)691BC 660BC(2.1%)650BC 545BC(82.3%)407BC	IntCal13	