

## Supplementary Information

### The potential of stable carbon and nitrogen isotope analysis of foxtail and broomcorn millets for investigating ancient farming systems

Yu Dong<sup>1,2,\*</sup>, Xiaoguang Bi<sup>1</sup>, Rubi Wu<sup>2</sup>, Eric J. Belfield<sup>3</sup>, Nicholas P. Harberd<sup>3</sup>, Bent T. Christensen<sup>4</sup>, Mike Charles<sup>2</sup>, Amy Bogaard<sup>2</sup>

1. Institute of Cultural Heritage, Shandong University, Qingdao, Shandong 266237, P. R. China
2. School of Archaeology, University of Oxford, Oxford OX1 3TG, United Kingdom
3. Department of Plant Sciences, University of Oxford, Oxford OX1 3RB, United Kingdom
4. Department of Agroecology, Aarhus University, AU-Foulum, Blichers Allé 20, DK-8830 Tjele, Denmark

\*Corresponding author. E-mail address: [yudong@sdu.edu.cn](mailto:yudong@sdu.edu.cn)

**Table S1.** The carbon and nitrogen isotope values and contents of foxtail millet (*Setaria*), broomcorn millet (*Panicum*), and wheat (*Triticum*) grains grown under different manuring and watering levels.

Taxon	Manuring Levels	Watering Levels	Sample ID	$\delta^{13}\text{C}$ (‰)	C%	$\delta^{15}\text{N}$ (‰)	N%	
<i>Setaria</i>	1%	low	SET030	-12.1	42.1	14.1	1.7	
			SET033	-11.9	41.3	13.6	1.7	
			SET033_rep	-11.9	40.5			
		medium	SET038	-12.7	40.1	10.3	2.3	
			SET055	-12.3	41.8	10.4	1.8	
			SET064	-11.7	39.7	11.2	1.9	
			SET064_rep	-11.7	41.5			
			SET065	-12.2	41.3	8.2	2.7	
			SET065_rep			8.5	2.7	
	high	SET045	-11.8	40.5	11.3	2.0		
		SET047	-11.8	46.0	10.4	1.9		
		SET051	-11.7	44.9	11.8	1.7		
	5%	medium	SET052	-12.4	44.5	16.4	1.9	
			SET053	-12.4	43.7	15.2	2.0	
			SET054	-12.4	41.3	15.9	1.7	
		high	SET007	-12.0	45.4	14.3	2.8	
			SET012	-13.6	43.1	16.1	2.4	
			SET024	-12.2	41.0	12.8	1.7	
<i>Panicum</i>		1%	low	PAN001	-13.5	40.5	14.4	1.6
				PAN008	-13.6	44.8	15.4	1.6
				PAN009	-13.6	46.5	14.3	2.1
	medium	PAN017	-14.0	39.5	13.4	1.9		
		PAN018	-13.9	37.1	13.3	1.8		
		high	PAN010	-14.2	43.8	10.9	1.8	

			PAN010_rep	-14.1	44.9	11.1	1.7
			PAN016	-14.2	40.4	12.1	2.5
	5%	medium	PAN019	-13.7	43.0	15.7	1.6
			PAN020	-13.7	38.3	14.6	1.5
		high	PAN026	-13.9	41.2	16.0	1.8
			PAN027	-14.0	39.9	13.5	1.6
			PAN027_rep	-14.0	39.4		
			PAN028	-13.8	38.8	14.8	1.7
<i>Triticum</i>	1%	low	WHE001	-28.6	46.4	12.5	3.3
			WHE002	-29.2	42.5	13.2	3.2
			WHE003	-29.3	44.2	13.8	3.7
		medium	WHE007	-28.7	42.9	10.0	3.5
			WHE008	-29.0	42.7	11.2	3.7
			WHE009	-29.2	43.8	11.3	3.3
			WHE009_rep			11.8	3.3
		high	WHE004	-30.8	42.3	11.9	3.4
			WHE005	-30.0	40.0	11.3	3.4
			WHE006	-30.4	42.5	10.9	3.1
			WHE006_rep	-30.3	43.9		
	5%	medium	WHE010	-30.7	41.5	15.2	3.9
			WHE011	-29.7	40.9	18.2	4.1
		high	WHE012	-30.2	42.2	15.4	3.9
			WHE013	-31.4	42.2	16.4	3.8

**Table S2.** The  $\delta^{13}\text{C}$ , %C,  $\delta^{15}\text{N}$  and %N values of foxtail (*Setaria*) and broomcorn millet (*Panicum*) grains heated at different temperatures and durations.

Taxon	Temp (°C)	Time (hrs)	Sample ID	$\delta^{13}\text{C}$ (‰)	C%	$\delta^{15}\text{N}$ (‰)	N%	
<i>Setaria</i>	50	48	UNFM01	-11.7	38.0	0.1	1.6	
			UNFM02	-11.5	38.3	0.2	1.5	
			UNFM03	-11.6	38.3	0.1	1.5	
	215	4	F215-4A	-11.5	39.6	0.7	1.6	
			F215-4B	-11.5	39.4	0.4	1.7	
			F215-4B_rep	-11.5	39.4	0.3	1.6	
			F215-4C	-11.6	39.3	0.5	1.8	
			8	F215-8A	-11.4	40.6	0.9	1.6
				F215-8B	-11.3	40.8	0.3	1.5
				F215-8C	-11.5	40.4	0.9	1.8
			24	F215-24A	-11.4	42.7	0.7	1.8
				F215-24B	-11.4	44.2	0.9	1.9
	F215-24C	-11.3		45.6	1.2	1.9		
	230	4	F230-4A	-11.3	40.3	0.5	1.6	
			F230-4B	-11.6	40.2	0.7	1.7	

		8	F230-4C	-11.6	39.8	0.2	2.0
			F230-8A	-11.4	41.2	1.0	1.5
			F230-8A_rep	-11.4	41.4	1.0	1.5
			F230-8B	-11.4	41.3	1.1	1.7
			F230-8C	-11.4	41.5	0.7	1.8
		24	F230-24A	-11.5	53.6	1.3	2.8
			F230-24B	-11.3	53.7	1.2	2.5
			F230-24C	-11.1	51.0	0.7	2.4
245		4	F245-4A	-11.4	47.9	0.9	2.1
			F245-4B	-11.3	46.9	0.8	2.1
			F245-4C	-11.4	46.2	1.3	2.1
		8	F245-8A	-11.2	53.3	1.1	2.5
			F245-8B	-11.3	53.5	1.5	2.5
			F254-8C	-11.3	53.0	1.4	2.5
			F245-8C_rep	-11.3	52.9	1.5	2.5
		24	F245-24A	-11.2	51.6	1.4	3.2
			F245-24B	-11.0	51.3	1.7	3.1
			F245-24C	-11.2	50.5	1.6	3.0
260		4	F260-4A	-11.5	55.4	1.3	2.7
			F260-4B	-11.4	52.5	1.6	2.4
			F260-4C	-11.4	53.0	1.7	2.4
		8	F260-8A	-11.1	54.7	1.4	2.8
			F260-8B	-11.3	54.7	1.6	2.7
			F260-8C	-11.3	54.3	1.3	2.7
		24	F260-24A	-11.0	50.4	1.7	3.9
			F260-24B	-11.2	49.6	2.0	3.8
			F260-24C	-11.0	49.6	1.4	3.9
			F260-24C_rep	-11.0	47.9	1.5	3.7
<i>Panicum</i>	50	48	UNCM01	-12.5	37.7	0.4	1.9
			UNCM02	-12.7	37.8	0.0	1.9
			UNCM03	-12.4	38.2	0.3	2.3
	215	4	C215-4A	-12.5	39.1	0.5	2.1
			C215-4B	-12.5	39.1	0.7	2.2
			C215-4C	-12.8	38.6	0.7	2.3
		8	C215-8A	-12.5	39.8	1.0	2.2
			C215-8B	-12.5	39.4	0.9	2.1
			C215-8C	-12.5	39.1	1.2	2.3
		24	C215-24A	-12.4	43.0	1.4	2.6
			C215-24B	-12.4	43.8	1.1	2.6
			C215-24B_rep	-12.4	43.9	1.1	2.7
			C215-24C	-12.5	43.2	1.5	2.6
	230	4	C230-4A	-12.3	39.9	0.9	2.2
			C230-4B	-12.4	40.5	0.9	2.2
			C230-4C	-12.5	38.5	0.9	2.2

	8	C230-8A	-12.4	48.1	1.3	2.9
		C230-8B	-12.3	44.2	1.3	2.5
		C230-8C	-12.5	46.5	1.2	2.7
	24	C230-24A	-12.3	50.3	1.8	3.5
		C230-24B	-12.3	52.3	1.2	3.2
		C230-24C	-12.3	51.9	1.6	3.3
245	4	C245-4A	-12.4	46.3	1.5	2.8
		C245-4A_rep	-12.4	46.0	1.5	2.8
		C245-4B	-12.4	46.2	1.5	2.7
	8	C245-4C	-12.3	46.8	1.3	2.7
		C245-8A	-12.3	52.0	2.0	3.2
		C245-8B	-12.3	51.2	2.0	3.4
	24	C245-8C	-12.4	52.2	1.6	3.4
		C245-24A	-12.2	52.3	1.7	4.0
		C245-24B	-12.1	51.0	2.1	4.4
	4	C245-24C	-12.1	50.5	1.9	4.1
		C260-4A	-12.3	53.3	1.5	3.3
		C260-4B	-12.4	52.4	1.7	3.2
260	4	C260-4C	-12.3	52.6	1.7	3.1
		C260-4C_rep	-12.3	52.3	1.6	3.1
		C260-8A	-12.3	53.7	1.8	4.0
	8	C260-8B	-12.3	54.1	1.7	3.5
		C260-8C	-12.0	49.6	1.9	4.9
		C260-24A	-12.0	50.0	2.1	5.2
	24	C260-24B	-12.1	50.2	1.9	4.9
		C260-24C	-12.3	49.6	2.0	3.6

**Table S3.** Standard deviations for the carbon and nitrogen isotopic compositions of the calibration standards used in each analytical session associated with the data presented in this paper.

Standard	Run ID <sup>i</sup>	<i>n</i>	$\delta^{13}\text{C}$ ( $\pm 1\sigma$ , ‰)	$\delta^{15}\text{N}$ ( $\pm 1\sigma$ , ‰)
IRM-1 <sup>ii</sup>	RLAHA_181217	14	0.05	–
USGS41	RLAHA_181217	7	0.19	–
IRM-1	RLAHA_190116	10	0.05	
USGS41	RLAHA_190116	5	0.03	
IRM-1	RLAHA_190121	9		0.25
IRM-2 <sup>iii</sup>	RLAHA_190121	6		0.30
IRM-1	RLAHA_190123	4		0.68
IRM-2	RLAHA_190123	2		0.49
USGS40	RLESA_20220118	3	0.03	0.03
USGS41a	RLESA_20220118	3	0.05	0.18
USGS62	RLESA_20220118	3	0.04	0.02

- i. Runs with an ID starting with RLAHA were analyzed at the Research Laboratory for Archaeology and the History of Art (RLAHA), University of Oxford, Runs with an ID starting with RLESA were analyzed at the Joint International Research Laboratory of Environmental and Social Archaeology (JoInRLESA), Shandong University.
- ii. IRM-1 is an internal standard, the material is alanine.
- iii. IRM-2 is an internal standard, the material is seal collagen.

**Table S4.** Standard reference materials used to monitor internal accuracy and precision.

Standard	Material	Mean $\delta^{13}\text{C}$ (‰, VPDB)	Mean $\delta^{15}\text{N}$ (‰, AIR)
IAEA-CH-6	Sucrose	-10.449±0.033	–
EMA B2159	Sorghum	-13.68±0.18	1.58±0.17

VPDB stands for Vienna Pee Dee Belemnite; AIR stands for Ambient Inhalable Reservoir.

**Table S5.** Mean and standard deviations of all the check (QA) standards analyzed in the analytical sessions associated with data presented in this paper.

			$\delta^{13}\text{C}$ (‰, VPDB)	$\delta^{15}\text{N}$ (‰, AIR)
Standard	Run ID *	<i>n</i>	Mean ± 1σ	Mean ± 1σ
IAEA-CH-6	RLAHA_181217	7	-10.15 ± 0.03	
IAEA-CH-6	RLAHA_190116	5	-10.20 ± 0.04	
EMA B2159	RLAHA_190121	6		1.65 ± 0.46
EMA B2159	RLAHA_190123	2		0.73 ± 0.34
EMA B2159	RLESA_20220118	12	-13.44 ± 0.09	1.67 ± 0.12

\* Runs with an ID starting with RLAHA were analyzed at the Research Laboratory for Archaeology and the History of Art, University of Oxford, Runs with an ID starting with RLESA were analyzed at the Joint International Research Laboratory of Environmental and Social Archaeology, Shandong University.

**Table S6.** Carbon and nitrogen isotope compositions of samples analyzed in duplicate.

Sample ID	$\delta^{13}\text{C}$ (‰)		$\delta^{15}\text{N}$ (‰)		Analysis Lab*
	A	B	A	B	
PAN010	-14.09	-14.20			RLAHA, University of Oxford
PAN018	-13.93	-13.94			RLAHA, University of Oxford
PAN027	-14.04	-14.01			RLAHA, University of Oxford
WHE006	-30.40	-30.34			RLAHA, University of Oxford
WHE014	-25.54	-25.54			RLAHA, University of Oxford
SET033	-11.85	-11.85			RLAHA, University of Oxford
SET064	-11.73	-11.72			RLAHA, University of Oxford
PAN010			10.89	11.15	RLAHA, University of Oxford
SET038			9.80	10.11	RLAHA, University of Oxford

SET065			8.22	8.52	RLAHA, University of Oxford
WHE009			11.25	11.26	RLAHA, University of Oxford
C215-24B	-12.39	-12.39	1.12	1.12	JoInRLESA, Shandong University
C245-4A	-12.39	-12.40	1.52	1.45	JoInRLESA, Shandong University
C260-4C	-12.32	-12.34	1.68	1.58	JoInRLESA, Shandong University
F215-4B	-11.54	-11.54	0.37	0.33	JoInRLESA, Shandong University
F230-8A	-11.36	-11.38	0.95	0.97	JoInRLESA, Shandong University
F245-8C	-11.30	-11.30	1.42	1.46	JoInRLESA, Shandong University
F260-24C	-10.96	-11.01	1.39	1.48	JoInRLESA, Shandong University
UNCM03	-12.39	-12.47	0.27	0.25	JoInRLESA, Shandong University

\* RLAHA stands for the Research Laboratory for Archaeology and the History of Art, and JoInRLESA stands for the Joint International Research Laboratory of Environmental and Social Archaeology.

### Standard Uncertainty

Standard uncertainty for the  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  measurements of the samples was estimated following Szpak et al. (2017).

For analysis done at RLAHA, University of Oxford, the systematic errors ( $u_{(bias)}$ ) were calculated to be  $\pm 0.28\text{‰}$  for  $\delta^{13}\text{C}$  and  $\pm 0.18\text{‰}$  for  $\delta^{15}\text{N}$ . Random errors ( $uR_{(w)}$ ) were calculated to be  $\pm 0.09\text{‰}$  for  $\delta^{13}\text{C}$  and  $\pm 0.36\text{‰}$  for  $\delta^{15}\text{N}$  based on the pooled standard deviations of the check standards and sample replicates. Standard uncertainty was determined to be  $\pm 0.29\text{‰}$  for  $\delta^{13}\text{C}$  and  $\pm 0.41\text{‰}$  for  $\delta^{15}\text{N}$ .

However, one run (RLAHA\_190123 which includes the measurement of nitrogen isotope values of wheat) done at RLAHA, University of Oxford, has relatively large systematic errors ( $u_{(bias)}=0.87\text{‰}$ ) and slightly big random errors ( $uR_{(w)}=0.59\text{‰}$ ). We need to be cautious in interpreting the nitrogen isotope values of wheat in current study.

For analysis done at JoInRLESA, Shandong University, the systematic errors ( $u_{(bias)}$ ) were calculated to be  $\pm 0.30\text{‰}$  for  $\delta^{13}\text{C}$  and  $\pm 0.19\text{‰}$  for  $\delta^{15}\text{N}$ . Random errors ( $uR_{(w)}$ ) were calculated to be  $\pm 0.08\text{‰}$  for  $\delta^{13}\text{C}$  and  $\pm 0.12\text{‰}$  for  $\delta^{15}\text{N}$  based on the pooled standard deviations of the check standards and sample replicates. Standard uncertainty was determined to be  $\pm 0.31\text{‰}$  for  $\delta^{13}\text{C}$  and  $\pm 0.22\text{‰}$  for  $\delta^{15}\text{N}$ .

### References

Szpak, P., Metcalfe, J. Z., and Macdonald, R. A. (2017). Best practices for calibrating and reporting stable isotope measurements in archaeology. *J. Archaeol. Sci. Reports* 13, 609–616. doi: 10.1016/j.jasrep.2017.05.007.