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Earliest direct evidence of plant processing in prehistoric Saharan pottery

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Supplementary Table 1 Archaeological phase, P/S ratios, *n*-alkane lipid distributions, stable carbon isotope values, CPI, ACL, weighted mean and P_{aq} values from trimethylsilylated TLEs of potsherds from Takarkori rock shelter and Uan Afuda cave, Tadrart Acacus, Libyan Sahara. The maxima for the *n*-alkane distributions are highlighted in bold.

Lipid distribution - abundance										Stable carbon isotope values ($\delta^{13}\text{C}$)									
Sample no.	Archaeological period	Palmitic/ Stearic ratio	C ₂₃		C ₂₅		C ₂₇		C ₂₉		C ₃₁		C ₃₃		CPI C ₂₃ -C ₃₃	ACL C ₂₃ -C ₃₃	Weighted mean $\delta^{13}\text{C}$	P _{aq}	
			n-alkane	n-alkane	n-alkane	n-alkane	n-alkane	n-alkane	n-alkane	n-alkane	n-alkane	n-alkane	n-alkane	n-alkane					
TAK4	Late Acacus	3.08	47.6	193.0	161.3	114.2	82.5	36.5	n/d	-23.3	-23.2	n/d	n/d	2.1	27.7	-23.2	0.55		
TAK14	Middle Pastoral	1.98	n/d	20.0	20.8	20.4	28.8	13.4	-24.8	-29.4	-29.7	-29.3	-28.8	n/d	n/d	28.9	-29.2	n/d	
TAK23	Late Acacus	2.56	n/d	198.0	368.2	302.2	291.1	142.7	n/d	-18.2	-19.8	-23.0	-23.1	5.7	28.7	-21.4	n/d	n/d	
TAK24	Early Pastoral	5.38	n/d	42.4	101.0	51.3	61.3	29.8	-27.7	-20.1	-20.6	-24.1	-24.8	n/d	28.5	-22.4	n/d	n/d	
TAK82	Middle Pastoral	2.82	n/d	552.6	367.4	368.1	484.2	223.0	n/d	-19.0	-20.5	-21.5	-24.0	8.1	28.5	-21.2	n/d	n/d	
TAK135	Middle Pastoral	4.96	74.0	102.0	89.3	70.7	71.3	39.9	-20.6	-25.0	-21.3	n/d	n/d	n/d	28.2	-23.3	0.55		
TAK159	Middle Pastoral	4.20	31.4	538.5	271.0	180.4	190.6	86.0	-21.0	-22.9	-16.3	n/d	n/d	n/d	27.4	-20.7	0.61		
TAK479	Middle Pastoral	4.55	23.1	221.2	127.6	85.1	92.1	54.0	-27.3	-27.2	n/d	n/d	n/d	5.2	27.7	-27.2	0.58		
TAK709	Middle Pastoral	2.47	n/d	11.2	40.6	40.1	46.1	23.9	-17.7	-22.2	n/d	n/d	n/d	3.9	29.4	n/d	n/d	n/d	
TAK730	Middle Pastoral	4.80	n/d	20.0	20.8	20.4	28.8	13.4	-21.8	-24.0	-27.4	n/d	n/d	5.1	28.9	-25.0	n/d	n/d	
TAK766	Middle Pastoral	2.93	72.1	465.3	117.4	62.7	70.0	n/d	n/d	-18.4	-19.0	-23.6	-24.1	8.1	26.3	-19.5	0.80		
TAK860	Middle Pastoral	4.00	n/d	248.0	164.0	158.0	234.0	125.0	-18.0	-19.0	-19.7	-23.3	-23.7	n/d	28.6	-21.4	n/d	n/d	
TAK873	Middle Pastoral	1.98	n/d	6.6	14.7	16.4	21.4	12.7	-19.9	-17.7	-20.2	-23.9	-24.1	4.6	29.5	-22.4	n/d	n/d	
TAK953	Middle Pastoral	2.10	17.4	96.3	80.2	83.6	131.1	57.7	-21.0	-26.1	-21.4	n/d	n/d	7.6	28.9	-24.0	0.35		
TAK1008	Middle Pastoral	2.46	n/d	93.0	48.0	34.0	45.0	28.0	n/d	-27.4	-24.0	n/d	n/d	n/d	27.9	-26.3	n/d	n/d	
TAK1054	Middle Pastoral	3.13	32.8	38.4	23.6	20.0	31.8	15.0	-27.1	-25.5	-21.0	-24.1	n/d	n/d	28.4	-23.9	0.58		
TAK1072	Middle Pastoral	2.86	24.9	53.3	35.6	24.3	33.7	16.6	-17.6	-18.3	-19.6	-23.8	-23.6	n/d	28.1	-20.7	0.57		
TAK1531	Middle Pastoral	4.00	n/d	44.8	31.7	34.2	37.8	16.1	-21.6	-24.5	-23.4	-26.9	-25.0	n/d	28.4	-24.9	n/d	n/d	
UAF A1	Late Acacus	3.67	n/d	41	20	29	47	35	-29.8	-29.3	-31.2	-32.0	-32.6	n/d	29.2	-24.9	n/d	n/d	
UAF A3	Late Acacus	3.70	n/d	112	55	70	42	n/d	n/d	n/d	n/d	n/d	n/d	n/d	27.3	n/d	0.89		
UAF20	Late Acacus	1.50	n/d	96	78	93	99	56	-26.2	-25.4	-22.2	-24.8	-26.2	n/d	28.7	-21.5	n/d	n/d	
UAF45	Late Acacus	4.69	368	2529	154	224	152	80	n/d	n/d	n/d	n/d	n/d	n/d	26.0	n/d	0.75		
UAF46	Late Acacus	4.66	n/d	216	59	63	71	59	n/d	-27.7	-21.5	n/d	n/d	n/d	27.7	-26.4	n/d	n/d	
UAF50	Late Acacus	4.23	n/d	99	40	19	12	n/d	-27.0	-30.0	n/d	n/d	n/d	n/d	26.3	n/d	n/d	n/d	
UAF84	Late Acacus	14.33	374	1159	359	282	226	99	n/d	n/d	n/d	n/d	n/d	n/d	26.9	n/d	n/d	n/d	

n/d - not determined, signal intensity too low

Late Acacus period 8900-7400 years uncalibrated years BP, 8300-6100 calBC

Early Pastoral 7400-6400 years uncalibrated years BP, 6300-5300 calBC

Middle Pastoral 6100-5000 years uncalibrated years BP, 5200-3900 calBC^{12,13}

P/S ratio - relative abundance ratio $C_{16:0}/C_{18:0}$ fatty acids, values greater than 4 indicate a plant origin

CPI - measures the relative abundance of odd over even carbon chain lengths, e.g. CPI values for

all plant species have strong odd-chain preferences, with CPI values varying between 1.6 and 82.1¹⁷

ACL - weight-averaged number of carbon atoms of the higher plant $C_{25} - C_{33}$ *n*-alkanes¹⁶

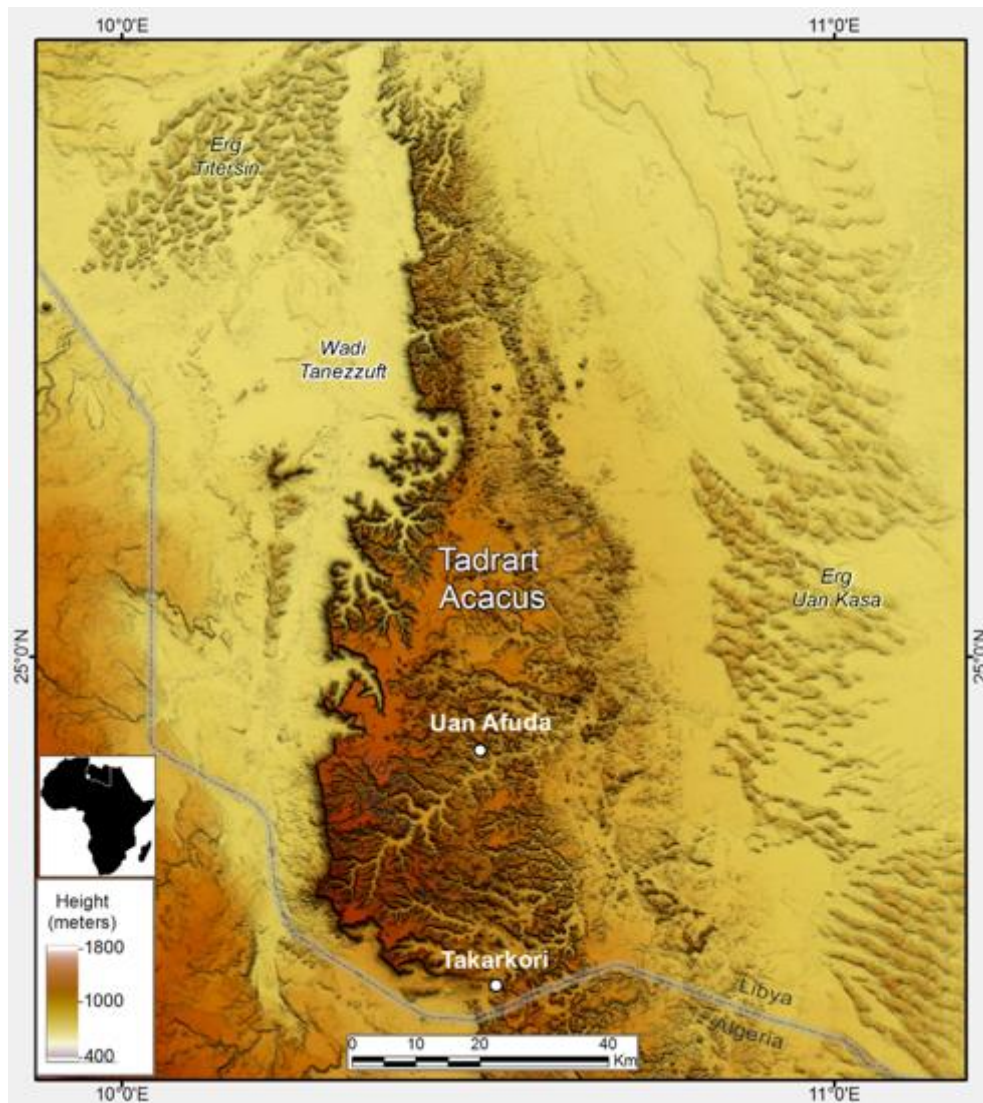
P_{aq} - emergent and non-emergent aquatic macrophyte input, $P_{aq} < 0.1$ corresponds to a terrestrial plant

input, P_{aq} 0.1-0.4 to emergent macrophytes and P_{aq} 0.4-1.0 to submerged or floating macrophytes¹⁸

Supplementary Table 2 Archaeological phase, lipid concentration ($\mu\text{g g}^{-1}$), total lipid in extract (μg) and $\delta^{13}\text{C}_{16:0}$ values from trimethylsilylated TLEs of potsherds from Takarkori rock shelter and Uan Afuda cave, Tadrart Acacus, Libyan Sahara.

Sample no.	Archaeological period	Lipid concentration ($\mu\text{g g}^{-1}$)	Total lipid in extract (μg)	$\delta^{13}\text{C}_{16:0}$
UAFA1	Late Acacus	64.1	122.5	-19.6
UAFA2	Late Acacus	322.4	519.1	-24.2
UAFA3	Late Acacus	95.7	233.5	-21.7
UAF20	Late Acacus	65.3	114.9	-19.2
UAF39	Late Acacus	360.6	548.2	-26.0
UAF45	Late Acacus	154.4	257.8	-22.8
UAF46	Late Acacus	97.0	159.1	-23.4
UAF47	Late Acacus	109.2	304.7	-24.2
UAF48	Late Acacus	288.2	587.9	-19.5
UAF50	Late Acacus	263.0	786.5	-24.2
UAF84	Late Acacus	261.7	554.7	-24.8
UAF85	Late Acacus	189.9	250.6	-18.0
UAF102	Late Acacus	62.5	154.4	-20.8
UAF127	Late Acacus	81.7	116.0	-24.8
UAF153	Late Acacus	47.8	124.7	-23.1
UAF158	Late Acacus	102.2	356.6	-26.5
UAF168	Late Acacus	96.7	184.7	-14.6
UAF169	Late Acacus	508.6	1429.3	-26.1
TAK4	Late Acacus	112.7	331.2	-20.7
TAK5	Late Acacus	41.1	99.0	-23.3
TAK23	Late Acacus	26.8	61.9	-25.7
TAK33	Late Acacus	108.8	118.6	-23.0
TAK13	Early Pastoral	32.9	100.8	-24.5
TAK24	Early Pastoral	1674.6	3583.6	-18.4
TAK2	Middle Pastoral	82.6	284.1	-18.5
TAK7	Middle Pastoral	40.3	93.1	-23.0
TAK12	Middle Pastoral	68.6	172.3	-27.4
TAK14	Middle Pastoral	63.0	116.6	-28.7
TAK17	Middle Pastoral	397.3	925.6	-20.5
TAK38	Middle Pastoral	169.9	373.7	-19.0
TAK42	Middle Pastoral	40.4	76.8	-20.0
TAK47	Middle Pastoral	80.7	203.5	-24.7
TAK82	Middle Pastoral	329.5	520.5	-20.4
TAK128	Middle Pastoral	78.5	64.3	-20.2
TAK135	Middle Pastoral	51.7	113.8	-20.8
TAK159	Middle Pastoral	203.1	396.1	-20.5
TAK188	Middle Pastoral	70.7	41.7	-22.6
TAK475	Middle Pastoral	37.1	61.6	-20.0
TAK479	Middle Pastoral	135.1	89.2	-19.9
TAK709	Middle Pastoral	130.8	227.6	-13.8
TAK730	Middle Pastoral	35.8	66.6	-20.4

TAK732	Middle Pastoral	96.1	230.6	-17.9
TAK766	Middle Pastoral	340.2	1054.7	-19.8
TAK813	Middle Pastoral	27.2	72.2	-20.7
TAK860	Middle Pastoral	45.0	88.6	-19.5
TAK873	Middle Pastoral	107.8	125.0	-18.5
TAK897	Middle Pastoral	13.6	20.3	-20.3
TAK927	Middle Pastoral	55.5	92.6	-20.7
TAK939	Middle Pastoral	60.0	135.6	-21.2
TAK953	Middle Pastoral	169.9	265.0	-21.5
TAK989	Middle Pastoral	90.9	90.0	-25.2
TAK1008	Middle Pastoral	12.3	29.4	-21.0
TAK1054	Middle Pastoral	293.2	504.3	-21.6
TAK1058	Middle Pastoral	205.5	213.8	-16.7
TAK1072	Middle Pastoral	356.2	580.6	-15.4
TAK1531	Middle Pastoral	63.8	107.1	-23.5



Supplementary Figure 1 Map of the Tadrart Acacus massif (SW Libya) with location of studied sites, Takarkori rock shelter and Uan Afuda cave (© The Archaeological Mission in the Sahara, Sapienza University of Rome)



Supplementary Figure 2 (a) view of the cave of Uan Afuda, central Tadrart Acacus (SW Libya), (b) the site stratigraphy consists of Late Pleistocene reddish aeolian sands at the base and the later Early Holocene sequence comprises brownish sediments rich in organic matter, (c) close view of structures including fireplaces from Late Acacus layers, radiocarbon dated to 7500 calBC. (© The Archaeological Mission in the Sahara, Sapienza University of Rome)



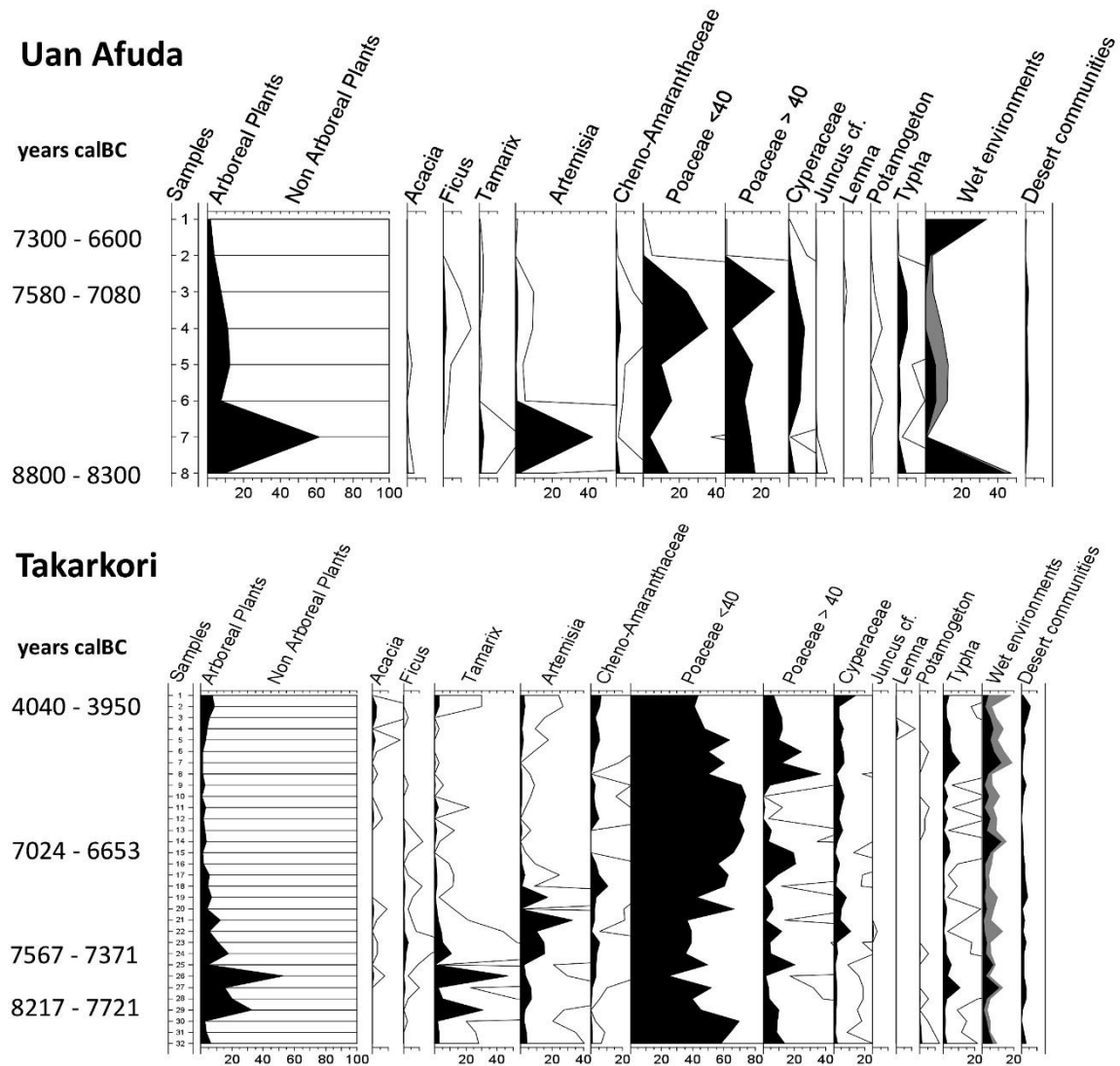
Supplementary Figure 3 (a) view of Takarkori rock shelter, southern Tadrart Acacus (SW Libya), (b) Middle Pastoral layers during excavation, (c) detail of plants remains and Barbary sheep coprolites from Late Acacus 2 stone structure, radiocarbon dated to *ca.* 7600-7300 calBC (© The Archaeological Mission in the Sahara, Sapienza University of Rome)



Supplementary Figure 4 Analysed potsherds from Uan Afuda cave. These originate from Late Acacus hunter-gatherer occupation levels, dated between approximately 8300 and 7300 calBC (© The Archaeological Mission in the Sahara, Sapienza University of Rome)



Supplementary Figure 5 Analysed potsherds from Takarkori rock shelter grouped by archaeological phases from Late Acacus to Late Pastoral periods (8300-3000 calBC). (© The Archaeological Mission in the Sahara, Sapienza University of Rome)



Supplementary Figure 6 The pollen diagrams from the two archaeological sites (top: Uan Afuda; bottom: Takarkori), showing selected taxa. Pollen records demonstrate that wet environments with hygrophilous trees (*Tamarix*, *Ficus*), limno-telmatophytes (*Typha*, *Juncus* cf.) and aquatics (*Lemna*, *Potamogeton*) were locally spread in the Tadrart Acacus Mountains, in the Central Sahara, during the Early and Middle Holocene. Meanwhile, desert communities with *Acacia* and Cheno-Amaranthaceae were less spread than in the current dry phase. High values of Poaceae pollen and *Artemisia* are evidence of human transport (and use) of plants into these cave (top) and rock shelter (bottom) sites⁶

Equation 1 P_{aq} ratio¹⁸

This formula is used to determine the extent of aquatic plant input to lake sediments through expressing the relative proportion of mid-chain length (C_{23} , C_{25}) to long-chain (C_{29} , C_{31}) *n*-alkane homologues¹⁸. A $P_{aq} < 0.1$ corresponds to a terrestrial plant input, P_{aq} 0.1-0.4 to emergent macrophytes and P_{aq} 0.4-1.0 to submerged or floating macrophytes.

$$P_{aq} = (C_{23} + C_{25}) / (C_{23} + C_{25} + C_{29} + C_{31})$$