DIRECT ^{14}C DATING OF EARLY AND MID-HOLOCENE SAHARAN POTTERY

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ABSTRACT. The aim of this study is to directly radiocarbon date pottery from prehistoric rock-art shelters in the Tassili n'Ajjer (central Sahara). We used a combined geochemical and microscopic approach to determine plant material in the pottery prior to direct ^{14}C dating. The ages obtained range from 5270 ± 35 BP (6276–5948 cal BP) to 8160 ± 45 BP (9190–9015 cal BP), and correlate with the chronology derived from pottery typology. Our results document the transition from pre-Pastoral to Pastoral contexts, dated to the early-mid Holocene transition, and confirm that vegetal temper in pottery can provide reliable ^{14}C ages within Saharan contexts.

INTRODUCTION

Current literature generally views the central Saharan highlands as refuge zones that favored protracted settlement during the Holocene (e.g. Camps 1969; Aumassip and Tauveron 1993; Aumassip et al. 1996; Cremaschi and di Lernia 1998; Ozenda 2004; Le Houérou 2009; Migliore 2011). As such, these are key areas to understand how human communities resettled after the Late Glacial Maximum, and how the return of drought and subsequent desertification impacted their way of life after the Holocene climate optimum. In the Tassili n'Ajjer-Tadrart Acacus, a cross-border zone between SE Algeria and SW Libya (Figure 1), a broad range of data provides evidence of a gradual shift from a hunter-gatherer and forager way of life (pre-Pastoral), to the adoption of a semi-nomadic herder status (Pastoral) (Aumassip and Tauveron 1993; Cremaschi and di Lernia 1998, 1999; di Lernia 1999; Cremaschi 2001; Garcea 2001; Biagetti et al. 2004; Hachid et al. 2010). According to the available chronometric data, the shift from pre-Pastoral to an early Pastoral context dates to the early-mid Holocene, around 7500 BP, and follows a rise in aridity recorded between 8000–7500 BP (Cremaschi and di Lernia 1998; Cremaschi 2001); it is documented both in the Tadrart Acacus (cf. Cremaschi and di Lernia 1998; di Lernia and Manzi 1998; Biagetti et al. 2004; Dunne et al. 2012) and in the southernmost Tassili n'Ajjer (Aumassip and Delibrias 1982–1983; Hamoudi 2002). However, postdepositional mixing and compression (deflation) often affect archaeological sites (e.g. rockshelters) and indicate that alternative means are needed to achieve a fuller understanding of the physiognomy of transitional contexts, and fill the gaps that might exist and hint at discrete depositional sets.

There is a need to establish an absolute chronology at an artifact level in the central Saharan highlands. Pottery, from an extensive Holocene cultural sequence, is available across the eastern to western Sahara, yet some typological patterns still remain unclear (Mohammed-Ali and Khabir 2003) and we lack a timetable of individual motif changes. During initial trials in the 1960s, various prob-

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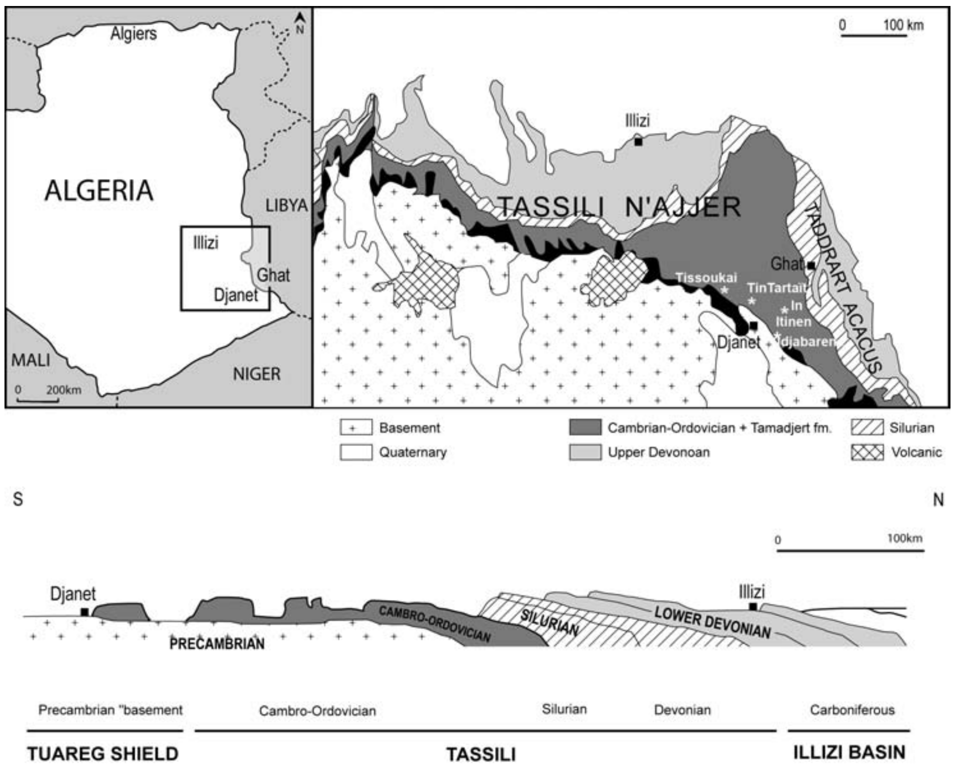

Figure 1 Location and simplified geological map of the Tassili n'Ajjer (modified from Eschard et al. 2005)

lems regarding pottery ^{14}C dating, due to complex organic components, were well documented (Ralph 1959; Evans and Meggers 1962; Stuckenrath 1963). In addition to the carbon present in clay, which may not be directly linked to the age of the pottery, other sources of carbon are often present: either included as temper (plants, limestone, shells, grog-chamotte, and bones) or from fuel when the pottery is fired. Organic coatings and food deposits can also be added after manufacture or during pottery use, and earthenware can subsequently acquire contamination from carbonates and organic acids in the soil. Different protocols have been published (Gabasio et al. 1986; Johnson et al. 1986; Evin et al. 1989; Hedges et al. 1992; Delqué-Količ 1995), and more recently the ^{14}C dating of lipids present in organic residues provided good results (Stott et al. 2003; Berstan et al. 2008). In Saharan-Sahelian areas, where a good match was found between the ^{14}C dates obtained from plant-tempered pottery and associated material (charcoal, bone, or enamel apatite), results indicated that most of the problems associated with pottery ^{14}C dating in temperate environments do not apply in arid environments (Saliège and Person 1991a,b, 1994; Bernus et al. 1999; Sereno et al. 2008; Hatté et al. 2010).

The aim of this study is to directly date the prehistoric pottery from the central Saharan highlands north of the Tropic of Cancer. Prior to ^{14}C dating, we performed a microscopy study to select the appropriate sherds to be dated, in order to maximize the obtainable information from this geochemical and chronological investigation. The full description of archaeological sites and contexts and pottery analysis will be published in a separate paper.

MATERIAL

The study area constitutes the sandstone tabular massif N-NE of the Tuareg shield (Figure 1). The plateau (~1800 m), its pediment, catchment area, and Admer erg (~900 m) are part of the hyperarid zone comprising a desert steppe landscape and contracted vegetation around wadis and water pools (Ozenda 2004; Baumhauer and Runge 2009). Within the desert zone, the Tassili n'Ajjer reflects a specific historical reality that to date is marked by Mediterranean influences and a high-altitude-dependent endemism. The area is a biosphere reserve of the MAB program and a listed World Heritage rock-art park by UNESCO (http://www.unesco.org/new/en/natural-sciences/environment/ecological-sciences/biosphere-reserves/africa/algeria/tassili-najjer/; http://whc.unesco.org/en/list/179).

Samples were obtained from both pre-Pastoral and Pastoral contexts. The pre-Pastoral potsherds ($n = 4$) come from the Tin Tartaït rockshelter in the Meddak (Alimen et al. 1968; MNHN series, Paris). In this site, the majority of the potsherds have pre-Pastoral patterns, only a few being time-specific to the subsequent Pastoral period (Figure 2, Table 1). The pottery is rather thick, coarse, and low-fired, with a very dark ground mass. This site also has pre-Pastoral paintings, including a pictorial shape that does not fit within a Neolithic herders' context (e.g. Mori 1998; di Lernia 1999; Garcea 2001). Potsherds from 3 well-known Pastoral contexts in the Meddak were also sampled: In Itinen ($n = 2$), Tissoukaï ($n = 2$), and Idjabarene rock-art shelters ($n = 3$) (Figure 2).

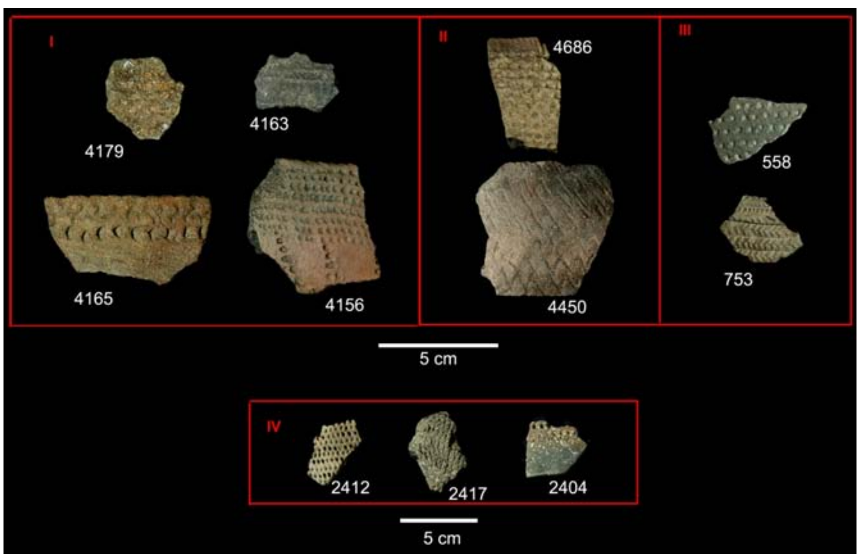

Figure 2 Pottery samples from the Tin Tartaït (I), In Itinen (II), Tissoukaï (III), and Idjabarene (IV) rock-art shelters.

METHODS

Potsherds were observed both macro- and microscopically. Thin sections were examined using polarized light microscopy (PLM), and the plant fraction of pre-Pastoral sherds was observed using a scanning electron microscope (SEM). Only sherds with plant temper or having numerous/sizeable organic inclusions were selected for ^{14}C dating. Samples were pretreated with diluted HCl acid (1.2N) and NaOH (0.1N) to remove contaminants such as carbonates and organic acids from the soil originating from decomposed organic matter. Food deposits, organic coatings, and carbon arising from fuel, where present in any significant degree, are contemporaneous with pottery manufacture or subsequent use and were not removed. The accuracy of pottery ^{14}C dates is a direct function of the "temper"/clay organic matter ratio: the higher it is, the weaker the influence of the organic matter bound to the clay and the more reliable the ^{14}C dating. In practice, our technique consists of removing a thin section of potsherd, eliminating the surface layers, and heating it at a low temperature (450 °C) in the presence of oxygen (Delqué-Količ 1995). Under these conditions, we facilitate the combustion of the non-occluded carbon, such as plants remains or early degraded plants, to the detriment of the occluded carbon associated in clay. The CO_2 produced is then graphitized before being dated by accelerator mass spectrometry (AMS) at the LMC14 (Cottereau et al. 2007). An aliquot of CO_2 was used to perform $\delta^{13}C$ analyses. Carbon isotope ratios were measured at the LOCEAN Lab (Univ. Paris VI, France) using a VG Optima stable isotope mass spectrometer.

RESULTS

Microscopic Analysis

The results are summarized in Table 1 and presented in Figure 3. There is a diversity of organic elements of the metaphases. Most of the plant remains are dilacerated and fragmented in form, often millimetric in size, totally preserved and perfectly determinable. Recognizable macro-remains represent tubular and flat foliar elements showing axes with sclerenchymae and conducting elements in central position (stems/twigs) and leaves with parallel nervures (2–6 × 30 mm) visible as porosities with ribbed contours on the section, characteristic of leaves of monocots. A detailed examination suggests the *Poaceae* family; *Cyperaceae* is another possibility. The presence of bilobate phytoliths along with bands of epidermal cells, often showing stomata and the bases of hairs in internal view, indicates leaves of grasses (Figure 3a) as attested by some determinable surface imprints and casts on thin sections (M A Magid, personal communication). These phytoliths may correspond to at least 4 taxonomic groups: *Panicoïdeae* subfamily (mainly C_4 plants); *Arundinoïdeae* subfamily (mainly C_3 plants); *Aristidoïdeae* subfamily (mainly C_4 plants); and *Stipa* genus (C_3 plants). When considering morphological characteristics, *Stipa*, which presents a clear asymmetry (Fredlund and Tieszen 1994), can be rejected. The use of temper is attested to in the form of a dense to very dense pattern (Figure 3c) with high porosity, abundant large flat and tubular fragments, discrete lineal distribution, and strong horizontal orientation. A thorough examination suggests that a dung-like temper is likely present: a feature characterized by abundant, evenly dispersed, fibrous to tubular fragments or their imprints (ribs and grooves). These fragments are 1 mm wide and several mm long, and present a strong horizontal orientation on sections in parallel to potsherd faces, and a rather random orientation visible on abraded surfaces (Livingstone-Smith 2001; van Doosselaere and Hayes 2005–2006; van Doosselaere 2011). Other sherds contain unidentifiable elements with vague outlines and heterogeneous size. These elements are spread throughout the mass, sometimes in the form of clumps, and in the best cases are recognizable by their erratic distribution (Figure 3d, left), their small size, and occasionally by tubular sub-mm elements also indicating phytoliths of the bilobate morphotype, either *in situ* in the residual epidermis or in their casts left in the matrix (Figure 3d, right).

Geochemical Analyses

Results are presented in Table 1. Potsherd organic carbon content ranges from 0.33% to 1.20% for the pre-Pastoral samples, and from 0.34% to 2.90% for the Pastoral samples. The $\delta^{13}C$ values vary between –22.2‰ and –17.7‰ and between –19.6‰ and –15.7‰ for the pre-Pastoral and Pastoral samples, respectively. These values indicate a C_3-C_4 mixture ranging from a C_3-dominated (#4179) to a C_4-dominated (#2412) signal. ^{14}C ages measured on Tin Tartaït pottery samples range between 8160 and 6740 BP, and indicate an age comprised between the end of the 10th and the middle of the

Table 1 Macro- and microscopic observations, as well as geochemical analysis, of pre-Pastoral and Pastoral pottery from Tassili n'Ajjer (central Sahara).

Site	Lab ID (SacA)	Sample ID	Decoration + (tool)	Typological attribution	Temper	Family	Taxa	% C	$\delta^{13}C$ ‰	^{14}C age BP	cal age BP (1σ)
Tin Tartaït[a]	18176	4156	Spaced-serrated zigzag impressions (evenly serrated comb)	pre-Pastoral	Flat and tubular fragments, mm to sub-mm. + casts and porosities. Tubular axis with vascular tissues (sclerenchyma and conducting elements in central position) + bilobate phytoliths on the epidermis.	*Poaceae Cyperaceae?*	*Panicoïdeae Arundinoïdeae Aristidoïdeae*	1.00	–20.9	8160 ± 45	9190–9015
	18173	4165	Plain rocker + simple impressions (plain edge + punch/wedge)	pre-Pastoral	Flat dilacerated and tubular fragments, mm to sub-mm, describing a dense feature. Stomata, bases of hairs and bilobate phytoliths *in situ* on the epidermis.	*Poaceae*	*Panicoïdeae Arundinoïdeae Aristidoïdeae*	1.20	–17.7	7700 ± 45	8537–8429
	18782	4179	Dotted wavy lines (bifid comb?)	pre-Pastoral	Flat dilacerated and tubular fragments, mm + erratic porosities of flat foliar fragts. Tubular axis cast (Ø>1–2mm) showing both negative imprint of hairs and bilobate phytoliths on the clayey matrix.	*Poaceae*	*Panicoïdeae Arundinoïdeae Aristidoïdeae*	0.33	–22.2	7645 ± 40	8507–8391
	18175	4163	Plain rocker + cord impressions or rouletting (?) (plain edge + cord or cord-wrapped comb?)	pre-Pastoral/Pastoral?	Dilacerated fragts, foliar(?), and fine tubular fragments + erratic porosities + clumps indet. Bilobate phytoliths *in situ* on lumps of tissues.	*Poaceae*	*Panicoïdeae Arundinoïdeae Aristidoïdeae*	0.55	–20.8	6740 ± 40	7651–7572
In Itinen[b]	18172	4686	Alternately pivoting stamp (bifid comb)	Pastoral	Thin dilacerated and tubular fragments + casts.	—	—	2.76	–17.3	6095 ± 45	7146–6891
	18169	4450	Plain rocker (plain edge)	Pastoral	Dilacerated fragments, rather long + casts.	—	—	0.75	–18.2	5380 ± 40	6277–6034
Tissoukaï[c]	18167	558	Return technique (bifid comb)	Pastoral	Abundant, thin tubular and fibrous fragments and casts.	—	—	2.90	–19.2	5735 ± 40	6625–6474
	18174	753	Plain rocker (narrow plain edge)	Pastoral	Thin dilacerated and tubular fragments.	—	—	0.41	–18.2	5620 ± 45	6444–6320
Idjabarene[d]	18168	2404	Alternately pivoting stamp (bifid comb)	Pastoral	?	—	—	0.34	–19.6	5405 ± 40	6277–6191
	18170	2412	Plain rocker (plain edge)	Pastoral	Thin dilacerated and tubular fragments.	—	—	1.10	–15.7	5375 ± 40	6276–6030
	18171	2417	Alternately pivoting stamp (bifid comb)	Pastoral	Thin dilacerated and tubular fragments.	—	—	0.90	–19.2	5270 ± 35	6276–5948

[a]Inner trench, layer II (Alimen et al. 1968); [b]Excavation, layers I-II (Delibrias 1966; Alimen et al. 1968); [c](Unpublished; Delibrias 1964; Thommeret and Rapaire 1964); [d](Unpublished; Delibrias 1971).

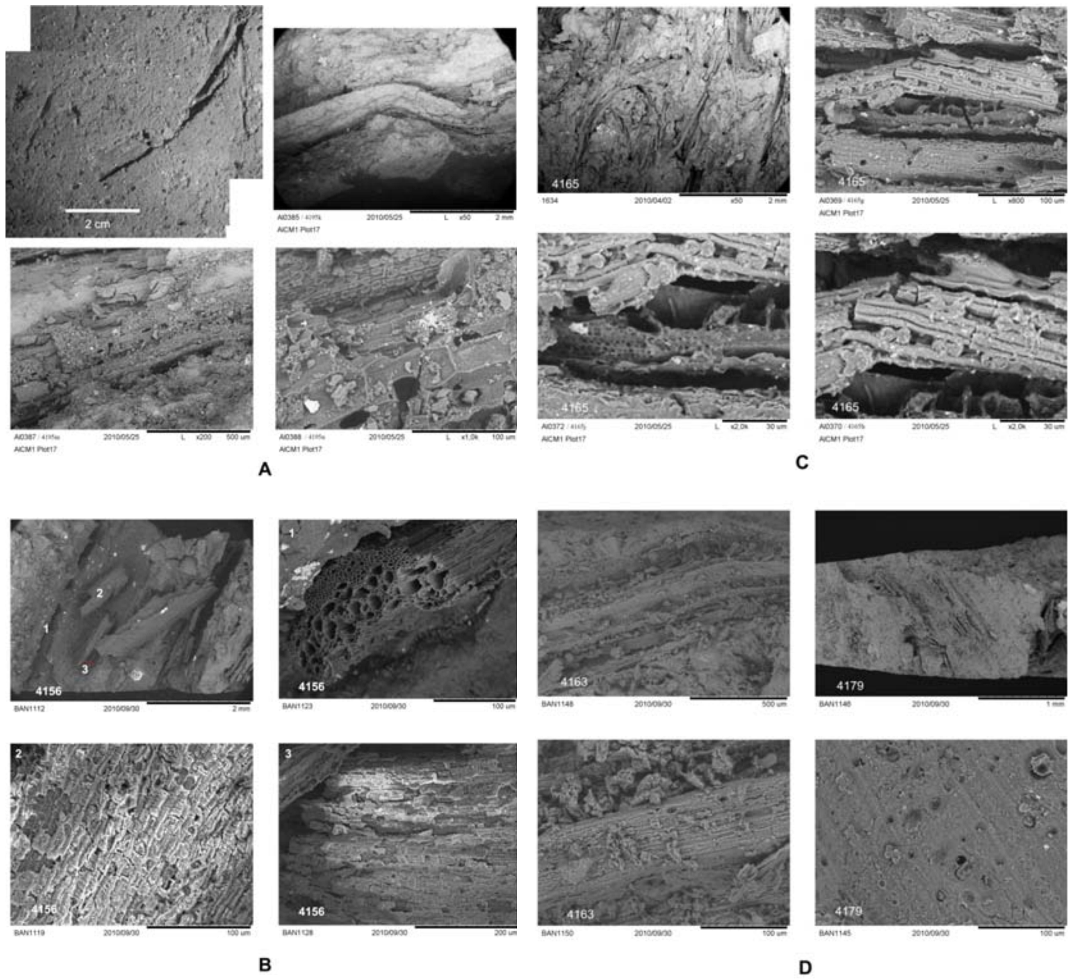

Figure 3 Pottery plant remains: (a) Foliar surface imprints and epidermis showing bands of cells and *in situ* bilobate phytoliths; (b) Axes with sclerenchymae and conducting elements in central position (stems/twigs), showing bilobate phytoliths; (c) A very dense feature of temper; (d) Casts with residual organic matter and lumps of dilacerated vegetal fragments.

9th millennium cal BP for the pre-Pastoral period. A sherd with a more uncertain typological attribution (#4163; Haour et al. 2010) returns the youngest age, around the middle of the 8th millennium cal BP. ^{14}C ages measured on Pastoral sherds from In Itinen, Tissoukaï, and Idjabarene range between 6095 and 5270 BP, and indicate an age comprised between the end of the 8th and the end of the 6th millennium BP, in line with their cultural chronology.

DISCUSSION

Identical or very similar ages were obtained within the same site on sample pairs with very different carbon contents. This is the case for samples #4179 (0.33% C_{org}, 7645 ± 40 BP) and #4165 (1.20% C_{org}, 7700 ± 45 BP) in Tin Tartaït, and samples #2404 (0.34% C_{org}, 5405 ± 40 BP) and #2412 (1.10% C_{org}, 5375 ± 40 BP) in Idjabarene. At In Itinen, the age difference measured between samples #4450 (5380 ± 40 BP) and #4686 (6095 ± 45 BP) is significant, but could correspond to 2 distinct and well-documented occupation phases (Alimen et al. 1968). Overall, this consistency demonstrates the weak influence of the carbon bound to the clay, similar to what has been previously observed in Sub-Saharan Africa (Sereno et al. 2008), and confirms that pottery organic temper can be reliably dated in these contexts. The ^{14}C activities of our pottery assemblage can thus be considered as being contemporaneous with the age of their manufacture.

This new chronometric evidence places the Tin Tartaït series at the transition between the early and mid-Holocene, i.e. the passage from pre-Pastoral to early Pastoral. There is a good chronological correspondence with pre-Pastoral and Pastoral records in Tadrart Acacus (Libya) (Barich 1987; Cremaschi 2001; Cremaschi and di Lernia 1998, 1999; di Lernia and Manzi 1998; di Lernia 1999; Garcea 2001; Biagetti et al. 2004; Biagetti and di Lernia 2007; Biagetti et al. 2009) and with the available data from southernmost Tassili-Tadrart (Algeria) (Hamoudi 2002; Tauveron et al. 2009) (Figure 4). Until recently, the stratigraphic continuity between pre-Pastoral and Pastoral contexts has been observed across very few sites. This period of transition between pre-Pastoral and Pastoral lifestyles corresponds to the interval of stratigraphic interruption recorded in southernmost Tassili between 7900 and 7200 BP (Aumassip and Delibrias 1982–1983; Aumassip and Heim 1989) and between 8000 and 7400 BP in the Tadrart Acacus (Cremaschi 2001; Cremaschi and di Lernia 1998, 1999). Therefore, the Tin Tartaït pottery has provided us with the opportunity to date the shift in pottery decoration from a rocker comb pattern, predominant among broadly contemporaneous contexts dating to the early Holocene, to the rocker bifid comb pattern, prevalent since the beginning of pastoralism (Caneva 1987). Our results confirm that this shift, starting from the 8th–7th millennia BP, is contemporary with the early-mid Holocene transition observed in many areas of central Sahara across the N-NE Niger/SE Algeria/SW Libya transect.

The relatively high residual carbon content in the pottery reveals the importance of the organic fraction in the samples after firing, but what does this fraction correspond to? We found no correlation between the carbon content and the $\delta^{13}C$ values. In the vast majority of our samples, the $\delta^{13}C$ values indicate a C_3-C_4 mixture in varying proportions, ranging from a C_3-dominated to C_4-dominated assemblage. $\delta^{13}C$ values reaching –20‰ are generally interpreted as indicating a mixture of C_3 and C_4. Due to water stress, C_3 plants in arid zones have less negative $\delta^{13}C$ values than in humid zones and the C_3 contribution could have been more important than indicated by the $\delta^{13}C$ values (Wittmer et al. 2008). While this is evidence of the important contribution of the C_3 component to the mixture, the microscopic data so far available indicate a grass element among fabrics (phytoliths of the bilobate morphotype along with the evidence of leaf and stem/twig fragments), either as part of a vegetal additive or perhaps a dung-like temper. Among the plant fragments, the bilobate phytoliths assigned to 3 subfamilies of *Poaceae* (*Panicoïdeae, Arundinoïdeae,* and *Aristidoïdeae*) may correspond to both C_3 and C_4 grasses, with the majority being C_4 grasses (Watson and Dallwitz 1992). Based on the isotopic and phytolith data, these fabrics could have been composed by a mixture of C_4 grasses with C_3 dicotyledonous plants fragments as well as a mixture of C_4 and C_3 grasses. Currently, the C_4 photosynthetic pathway is largely predominant in central Sahara amongst grasses, but C_3 grasses do exist as well (Quézel and Santa 1962–1963; Ozenda 2004). While the abundance of C_3 plants in early-mid Holocene pottery in the Sahelo-Saharan zone can be explained by wetter climatic conditions (Bernus et al. 1999:156), the trend towards less negative $\delta^{13}C$ values recorded between pre-Pastoral and Pastoral cultures could indicate increasing aridity, which, in turn, could be associated with the adoption of a seminomadic way of life.

Grasses form the largest group of macro-remains and are an important anthropogenic clue among site palynomorphs (Wasylikowa 1992; Aumassip and Tauveron 1993; Cremaschi and di Lernia 1999; Mercuri 1999, 2001, 2008). Although preliminary, our data suggest that grasses were being used by human communities from the early Holocene. This finding clearly correlates with the synthesis of

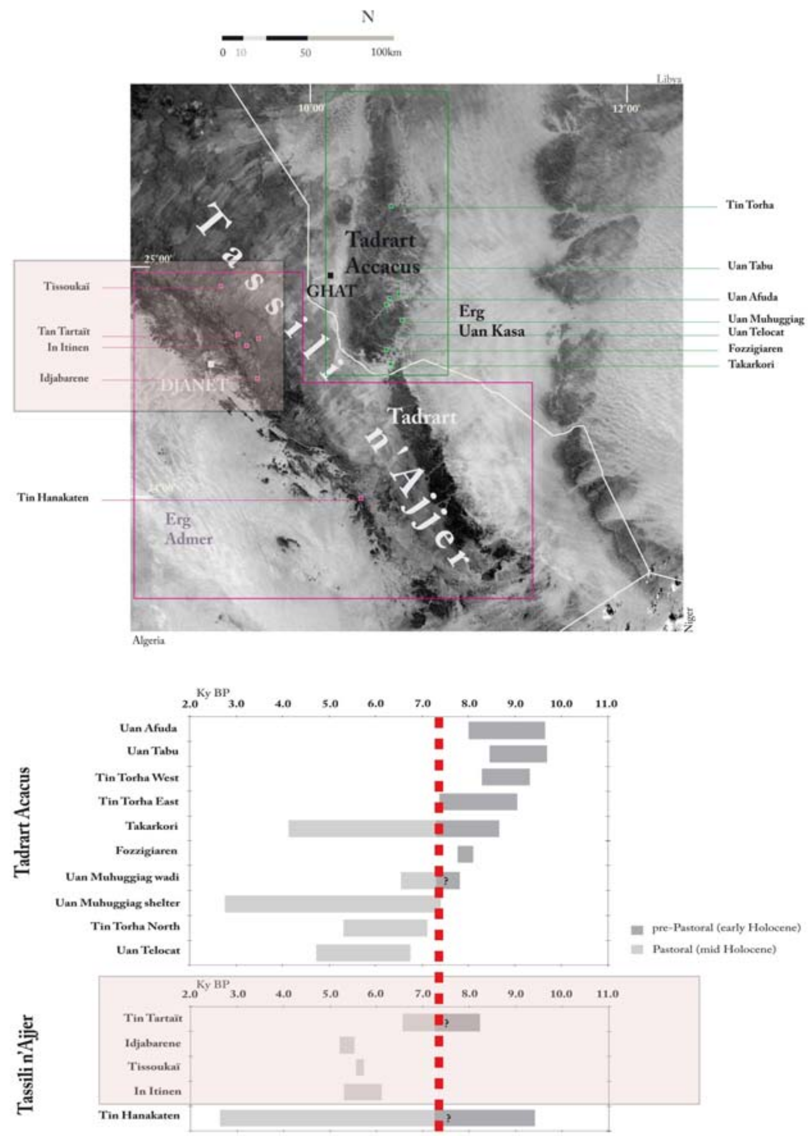

Figure 4 Time intervals (uncalibrated ^{14}C age BP) of human occupation in the Tadrart Acacus-Tassili n'Ajjer area during the early to mid-Holocene: (a) Map of the main sites; (b) Time intervals corresponding to the Tadrart Acacus and Tassili n'Ajjer (dashed line: limit early-mid Holocene; gray shaded: this study).

Mercuri (1999, 2008), regarding the neighboring Tadrart Acacus, which demonstrated that *Poaceae* show up frequently as food, fodder, and pasture in pre-Pastoral and early Pastoral contexts in particular. At Uan Afuda, for example, a dung layer dated to 8000 ± 100 BP attests to the ingestion of wild grass derived from waste products (di Lernia 1999). Our results are consistent with the alternative data currently available on pre-Pastoral and early Pastoral pottery in the Acacus and in the southernmost Tassili: (i) the use of straw (*Gramineae*) and dung as a tempering media (Magid 1999; Livingstone-Smith 2001;), (ii) comparable textural and geochemical characteristics (Senasson 1995).

Both Libyan and southern Tassili paleobotanical and paleopalynological records (Hachi 1983; Schulz 1987, 1994; Mercuri et al. 1998; Trevisan Grandi et al. 1998; Castelletti et al. 1999; Mercuri 2008) are interpreted as typical wadi vegetation, with *Acacia-Panicum* or *Tamarix-Stipagrostis* as main units, the saharomontane wadi type, and a wet to Saharo-Sahelian savannah prevailing during the early Holocene. Water promotes the development of vegetal communities associated with riparian zones, characterized by lines of trees with a ground layer of tufted grasses, shrubs, and herbs (*Typha*, *Phragmite*, and *Cyperaceae* near water holes; Schulz 1987). Landforms such as wadis, interdune corridors, rock pools, ponds, etc. provide desert dwellers with direct access to water, fuel, and different sources of supply of pottery raw materials. So can these pottery plant remains be traced to a particular vegetational/geographical zone? Do they indicate a settlement pattern potentially overlapping with the lowlands or mountain areas? Can cultural inferences be drawn from them or do they reflect an opportunistic behavior? These questions are left open pending future studies.

CONCLUSION

Direct ^{14}C dating of potsherds allowed us to establish a chronology for a series of pre-Pastoral and Pastoral Saharan pottery. The coherency of ^{14}C ages obtained from different sites on pairs of sherds with very different carbon contents validated the method used. Occupation of Tin Tartaït covers a period comprised between the end of the 10th and the mid-8th millennium cal BP and includes the transition between the pre-Pastoral and Pastoral periods. At this site, our approach allowed us to directly date a sherd with imprecise typological attribution (#4163) to the Pastoral period (first half of mid-Holocene), confirming the potential of this approach to refine or test existing ceramic typologies. Macro- and microscopic investigations highlight the presence of *Poaceae* among pottery fabrics, while carbon isotope analysis of organics in pottery suggests that a mixture of C_3 and C_4 plants (or dung) was used as temper. Finally, the shift towards less negative $\delta^{13}C$ values observed between the pre-Pastoral and Pastoral period is in good agreement with the general increase in aridity, which, in turn, could be associated to the adoption of a semi-nomadic way of life.

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