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DATING HUMAN OCCUPATION ON DIATOM-PHYTOLITH-RICH SEDIMENT: CASE STUDIES OF MUSTANG SPRING AND LUBBOCK LAKE, TEXAS, USA

Christine Hatté[1] • Gregory Hodgins[2] • Vance T Holliday[3,4] • A J Timothy Jull[2,4]

ABSTRACT. The Great Plains of North America have a rich archaeological record that spans the period from Late Glacial to Historic times, a period that also witnessed significant changes in climate and ecology. Chronometric dating of archaeological sites in many areas of the Great Plains, however, is often problematic, largely because charcoal and wood—the preferred materials for radiocarbon dating—are scarce in this grassland environment with few trees. Two reference archaeological sites are studied here: Mustang Spring and Lubbock Lake, Texas, USA. We carry out a geochronological approach based on a cross-study of carbon-derived data: combustion yield, $\delta^{13}C$, ^{14}C age differences between high temperature and low temperature released carbon, and the ^{14}C age itself. A study that incorporates multiple approaches is required to solve issues induced by the sedimentological context, which is rich in both freshwater diatoms and phytoliths from quite different origins. Analysis of carbon-derived data allows us to draw a succession model of dry and wet episodes and to associate it with a chronological framework. In this way, we can assert that, for the Mustang Spring site, several human occupations existed from ~11 kyr BP to ~8.7 kyr BP along the 110-cm-long series with an interruption of ~150 yr that is associated with a palustrine environment between the Plainview and Firstview occupations.

INTRODUCTION

The Great Plains of North America has a rich archaeological record that spans the period from Late Glacial to Historic times, a period that also witnessed significant changes in climate and ecology. Chronometric dating of archaeological sites in many areas of the Great Plains, however, is often problematic, largely because charcoal and wood—the preferred materials for radiocarbon dating—are scarce in this grassland environment with few trees. Bone can be abundant in the archaeological and paleontological sites of interest but often is not suitable for ^{14}C dating because the organic carbon is poorly preserved. Recently, luminescence dating has been applied successfully to eolian sediments on sites of the Southern High Plains (Feathers et al. 2006). Optically simulated luminescence (OSL) dating precision has improved, but ^{14}C dating is still more precise than OSL by a factor 2 to 4. By default, ^{14}C dating of bulk organic matter from sediment remains the most common method used, but results are often problematic (Holliday 1995, 2000b; Holliday et al. 1983, 1985). A common difficulty in ^{14}C dating sediment is the presence of carbon from different sources that would give very different and misleading ^{14}C signatures (e.g. McGeehin et al. 2001; Walker et al. 2007). Sources of carbon include residual geological material (e.g. clay and organic matter); younger plant material (e.g. phytoliths and organic macromolecules); and, specifically for settings with extreme or seasonal floods, algal input (e.g. diatoms and algal organic matter).

In this study, we assess the ages of diatomaceous samples from the Southern High Plains by multiple ^{14}C-dating approaches. The Lubbock Lake site (Holliday et al. 1985 and references therein) and the Mustang Spring site (Meltzer 1991) are two of the principal archaeological localities in the region and contain previously dated diatomaceous deposits, making them suitable for this study.

GEOLOGICAL SETTING

The Southern High Plains is a semi-arid plateau of mixed prairie grasslands. The low relief surface is underlain by an extensive eolian deposit that is heavily modified by pedogenesis, known as the

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[1] Laboratoire des Sciences du Climat et de l'Environnement, UMR CEA-CNRS-UVSQ 1572, Domaine du CNRS, bâtiment 12, F-91198 Gif-sur-Yvette Cedex, France. Corresponding author. Email: hatte@lsce.ipsl.fr.
[2] NSF-Arizona AMS Laboratory, Physics Building, University of Arizona, Tucson, Arizona 85721, USA.
[3] Department of Anthropology, University of Arizona, Tucson, Arizona 85721, USA.
[4] Department of Geosciences, University of Arizona, Tucson, Arizona 85721, USA.

Blackwater Draw Formation. This deposit accumulated episodically through the Pleistocene and forms the "bedrock" for the archaeological and other late Quaternary deposits of the region (Holliday 1989). Sediments are predominantly fine sand with increasing amounts of clay to the northeast. The sands are mostly quartz with minor amounts of mica and feldspar. The clay minerals are predominantly illite and smectite. Topographic relief on this otherwise flat landscape is provided by small basins containing seasonal lakes ("playas"), dry valleys ("draws"), and dunes. Dominant geomorphic and sedimentologic processes in the region include wind erosion, dust production, and eolian sedimentation (Holliday 1987; Holliday and Gustavson 1991), all of which involve sediment reworking and thus input of previously eroded particles that contain geological organic material (adsorbed on clay). These sandy and clayey sediments contain the archaeological sites that formed the basis of this study.

ARCHAEOLOGICAL FRAMEWORK

Lubbock Lake in Yellowhouse Draw (a tributary of the Brazos River) contains a thick, well-stratified record of cultural, faunal, and floral change spanning the past 11,500 ^{14}C yr. Five major stratigraphic units (1–5) were defined: Strata 1 and 2 contain the Paleoindian record, with the Clovis occupation (~11,100 ^{14}C yr BP) in alluvial gravel, sand, and clay of Stratum 1; Folsom features (~10,500 ^{14}C yr BP) in bedded diatomite (Stratum 2A); and Plainview (~10,000 ^{14}C yr BP) and Firstview (~8600 ^{14}C yr BP) occupations in Stratum 2B. Strata 3–5 contain the Archaic, Ceramic, Protohistoric, Apache, Comanche, and Euro-American archaeological records (Johnson 1987). Because the sample we analyzed (LL65-2A) was extracted from the base of Stratum 2, very close to the top of Stratum 1, it is expected to exhibit an age of ~11,000 yr BP.

Mustang Spring in Mustang Draw (a tributary of the Colorado River) contains a stratigraphic record similar to Lubbock Lake (Holliday 1995; Meltzer 1991). Archaeologically, the site is best known for Archaic-age water wells (Meltzer 1991; Meltzer and Collins 1987). Our sampling focused on Stratum 2, a bedded diatomite that is stratigraphically and lithologically identical to Stratum 2A at Lubbock Lake. We sampled and analyzed Stratum 2 from top to bottom, which, in aggregate across the site, spans the period between approximately 10,130 to 8080 ^{14}C yr BP (Haynes 1992; Holliday 2000b). In our core, the base of Stratum 2 is dated to ~10,130 ^{14}C yr BP; the top may be older, similar to the basal dates at Lubbock Lake (~11,000 yr BP) (Haynes 1975, 1995).

METHODS

On each sample, we apply several treatments in order to establish treatment efficiency and reliability. Protocols are summarized in Figure 1. Sediment is demineralized with HCl 1M at room temperature and is then divided into 2 parts (at a ratio of about 1/4–3/4). The first part of the leached sample then undergoes step-combustion under O_2; the low-temperature fraction evolves up to 400 °C (hereinafter called "bulk LT"), whereas the high-temperature fraction evolves from 400 to 850 °C (hereinafter called "bulk HT").

The second part of the leached sample undergoes clay removal to concentrate diatoms and phytoliths according to the protocol of Hatté et al. (2008). Disaggregation of the sediment is conducted by placing the sediment in 5% sodium hexametaphosphate solution in an ultrasonic bath for 3 min and then by vigorous boiling the sample for 15 min. After dilution with ultrapure water, the sample is allowed to settle for at least 3 hr. The supernatant is discarded and the sample is sieved at 38 µm. The fine fraction is retained. Differential settling with 1% boiling sodium hexametaphosphate solution is then applied several times (typically 15 times for these terrestrial samples) to reach the best compromise between clay removal and small diatom retention. All phytoliths, freshwater diatoms, and most of

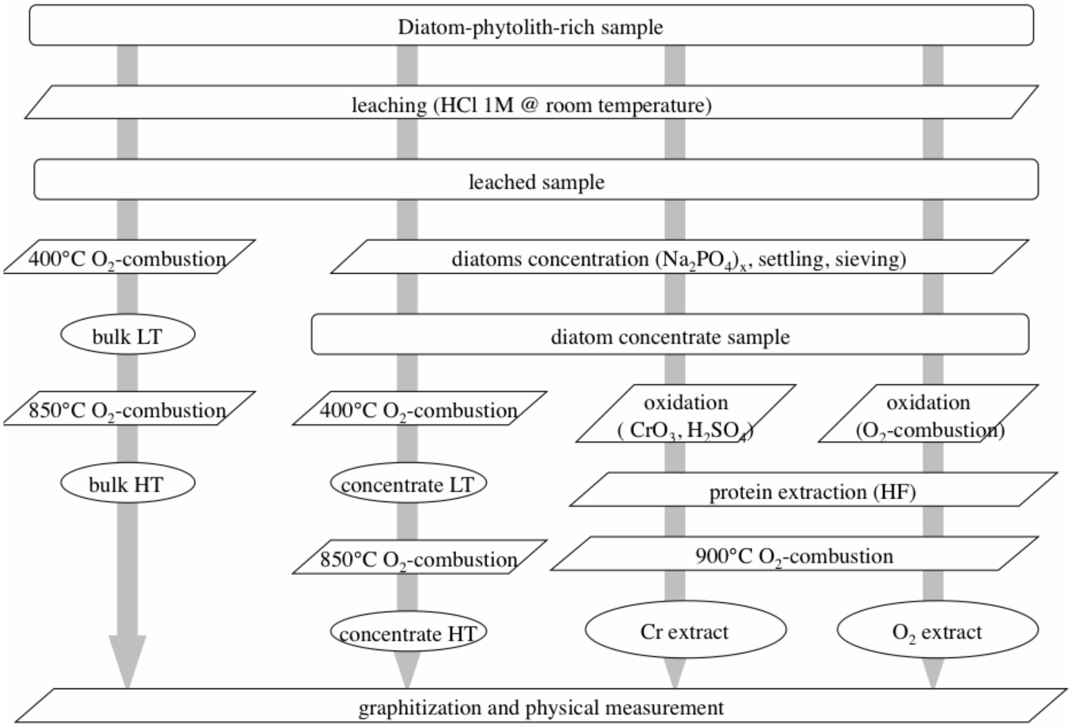

Figure 1 Schematic representation of the applied protocols

the clay particles have similar shapes and/or densities; therefore, heavy-liquid separation with sodium polytungstate (SPT) does not give better results. Phytoliths and diatoms cannot be definitively separated from one another. Sample concentration of diatoms and phytoliths is controlled step-by-step by observation under a light microscope (after freeze-drying) of the supernatant and the pellet. The Lubbock Lake sample was also checked by SEM photo (Figure 2). This diatom and phytolith-enriched sediment, hereinafter called the "concentrate," is divided into 3 roughly equal parts (about 1/3 each). The first part of the concentrate undergoes step-combustion with the same 400 °C and 850 °C threshold temperatures to evolve "concentrate LT" followed by "concentrate HT."

Both remaining parts of the "concentrate" sample are further treated, in order to release phytolith and diatom proteins. Both undergo a purification step to remove all free organic matter present in the sediment, including free organic matter linked to diatoms (mucilage and frustuline, i.e. inner and outer organic matter), free organic matter that coats phytoliths, and free organic matter linked to the still-present clay layers. The oxidative treatment is either a wet oxidation with 1M CrO_3/6N H_2SO_4 (Hatté et al. 2008) or a gaseous oxidation by combustion at 600 °C under an O_2 stream (this study). The treated materials are hereinafter called "Cr extract" and "O_2 extract," respectively. Both fractions subsequently undergo dissolution in 50% vol. HF to release proteins and polyamines. This step is performed under a N_2 stream.

Evolved CO_2 is quantified and transferred into a vessel. Splits for $\delta^{13}C$ are collected for larger samples. Stable isotope measurement is performed on a VG Optima with a typical error of 0.1‰. Evolved CO_2 for ^{14}C measurement is then converted into graphite over iron (Fe/C = 2 with a minimum of 0.2 mg Fe) at 575 °C in the presence of ZnO at 425 °C. All ^{14}C analyses were carried out at

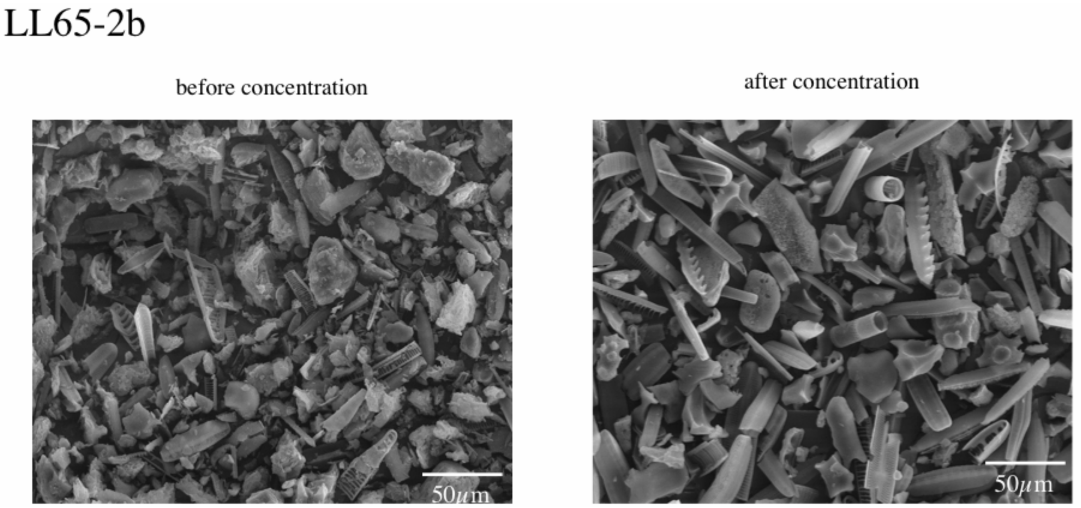

Figure 2 SEM photo performed on Lubbock Lake LL65-2A sample before and after concentration with the same magnification (×400). Note the disappearance of clay after concentration treatment.

the NSF-Arizona AMS Facility (Jull et al. 2003, 2004). Dates are reported in conventional ^{14}C yr. We choose not to calibrate them to facilitate easy comparison with chronological tie-points available in literature, which are all provided in units of yr BP.

RESULTS

Combustion yield, δ^{13}C, and ^{14}C results obtained on Lubbock Lake and Mustang Spring samples are presented in Tables 1 and 2, and are illustrated within a 1-σ uncertainty range in Figures 3 and 4, respectively. As added illustration, ^{14}C and δ^{13}C differences between HT and LT fractions of the Mustang Spring series are shown in Figure 4c.

Table 1 Raw data (white area) and processed data (shaded area) obtained on Lubbock Lake sample are gathered here for the different applied protocols: sediment mass, carbon mass evolved by combustion, δ^{13}C in ‰, ^{14}C age in yr BP, then combustion yield and difference between ^{14}C age obtained at high temperature and ^{14}C age obtained at low temperature.

		Raw data					Processed data	
					^{14}C age (yr BP)			
		Sediment mass (mg)	Carbon mass (μg)	δ^{13}C (‰)	Mean	±1 σ	Combustion yield (%wt)	$^{14}C_{HT}$–$^{14}C_{LT}$ (yr BP–yr BP)
Bulk organic matter	Low temperature	256	996	–24.6	9507[a]	48	0.41	417
	High temperature		639	–23.6	9924[a]	50	0.28	
Concentrate	Low temperature	333	63	–24.9	8580[a]	180	0.03	2200
	High temperature		89	–24.6	10,780[a]	300	0.03	
Extract	O_2 oxidation	60	54	na	14,091[b]	2088	1.81	
	CrO_3/H_2SO_4 oxidation	81	35	na	10,791[b]	422	0.87	

[a] Casual lab blank subtracted, measured δ^{13}C corrected.

[b] ^{14}C age correction according to a previous diatom protein study (Hatté et al. 2008): F blank = 0.1268 ± 0.0066 and δ^{13}C = –22.1‰.

Table 2 Results gathered according to the applied protocol (see text and Figure 1). Both raw data (white area) and processed data (shaded area) obtained on Mustang Spring samples are shown: sediment mass, carbon mass evolved by combustion, $\delta^{13}C$ in ‰, ^{14}C age in yr BP, then combustion yield and difference between ^{14}C age obtained at high temperature and ^{14}C age obtained at low temperature.

Bulk organic matter		Raw data								Processed data		
		Low temperature				High temperature				Combustion yield (%wt)		
Level (cm)	Sed. mass (mg)	Carbon mass (µg)	$\delta^{13}C$ (‰)	Mean ^{14}C age (yr BP)[a]	±1 σ	Carbon mass (µg)	$\delta^{13}C$ (‰)	Mean ^{14}C age (yr BP)[a]	±1 σ	LT	HT	$^{14}C_{HT}$–$^{14}C_{LT}$ (yr BP–yr BP)
215–225	250	401	–25.1	8015	50	415	–24.7	8690	50	0.16	0.17	675
245–255	250					406	–22.9	7968	59		0.16	
255–265	279	803	–22.1	9028	48	863	–21.2	9090	52	0.29	0.31	62
265–275	255					1184	–21.2	9421	48		0.47	
275–285	269	1189	–21.3	9304	49	681	–21.3	9437	49	0.44	0.25	133
285–295	224					876	–21.3	9417	48		0.39	
295–305	414	1105	–22.1	9576	48	1814	–21.3	9639	59	0.27	0.44	63
305–315	223	605	–24.2	9212	66	296	–23.9	9890	100	0.27	0.13	678
315–325	285	105	–23.0	9630	160	54	–22.9	10820	240	0.04	0.02	1190

Concentrate		Raw data								Processed data		
		Low temperature				High temperature				Combustion yield (%wt)		
Level (cm)	Sed. mass (mg)	Carbon mass (µg)	$\delta^{13}C$ (‰)	Mean ^{14}C age (yr BP)[a]	±1 σ	Carbon mass (µg)	$\delta^{13}C$ (‰)	Mean ^{14}C age (yr BP)[a]	±1 σ	LT	HT	$^{14}C_{HT}$–$^{14}C_{LT}$ (yr BP–yr BP)
215–225	270	28	–26.7	*na*		156	–25.9	8690	170	0.01	0.06	
245–255	32	231	–24.3	6260	120	328	–24.5	9100	130	0.07	0.10	2840
255–265	313	111	–22.6	7298	140	104	–23.1	9830	190	0.04	0.03	2532
265–275	346	93	–22.0	8760	160	192	–22.3	9720	110	0.03	0.06	960
275–285	318	94	–21.8	8500	140	225	–22.1	9770	110	0.03	0.07	1270
285–295	316	104	–21.1	8890	430	216	–22.2	9570	160	0.03	0.07	680
295–305	322	89	–23.5	8760	230	165	–23.5	9950	130	0.03	0.05	1190
305–315	32	94	–24.3	8840	190	159	–24.8	11,270	640	0.03	0.05	2430
315–325	319					163	–25.1	10,730	160		0.05	

Extract	Raw data										Processed data	
	O_2 oxidation					CrO_3/H_2SO_4 oxidation					Combustion yield (%wt)	
Level (cm)	Sed. mass (mg)	Carbon mass (µg)	$\delta^{13}C$ (‰)	Mean ^{14}C age (yr BP)[b]	±1 σ	Sed. mass (mg)	Carbon mass (µg)	$\delta^{13}C$ (‰)	Mean ^{14}C age (yr BP)[b]	±1 σ	O_3 oxidation	CrO_3/H_2SO_4 oxidation
215–225	22	32	na	11,199	1075	46					0.15	
245–255	22	32	*na*	3180	322	53	72	*na*	10,551	238	0.15	0.14
255–265	40	51	*na*	2494	340	22	58	*na*	9807	213	0.13	0.26
265–275	43	71	*na*	4700	650	55	52	*na*	4873	617	0.17	0.09
275–285						59	87	*na*	15,513	1765		0.15
285–295	26					40	68	*na*	3498	523		0.17
295–305	32	41	*na*	10,881	1001	55					0.13	
305–315	20					54						
315–325	23					54						

[a] Casual lab blank subtracted, measured $\delta^{13}C$ corrected.

[b] ^{14}C age correction according to Hatté et al. (2008): F blank = 0.1268 ± 0.0066 and $\delta^{13}C = -22.1‰$.

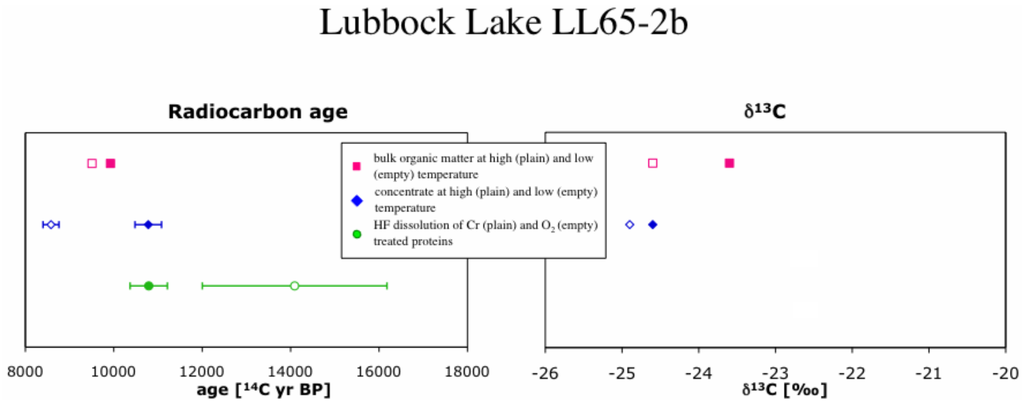

Figure 3 Lubbock Lake. Left: ^{14}C results according to the applied protocol: pink squares are for bulk organic matter at low (empty square) and high (plain square) temperatures, blue diamonds are for concentrate at low (empty diamond) and high (plain diamond) temperatures, green circles are for HF dissolution of Cr (plain circle) and O_2 (empty circle) extracts. See text and Figure 1 for details on protocol. Right: δ^{13}C results according to the applied protocol. Same symbols as left panel.

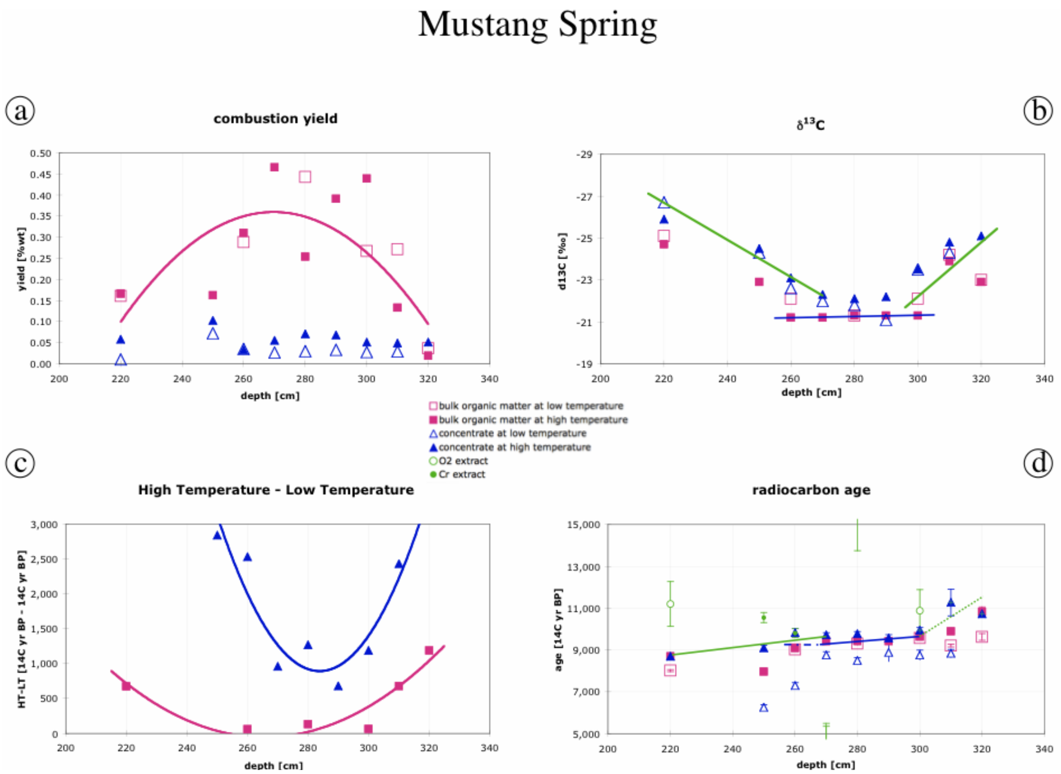

Figure 4 Mustang Spring. (a) Combustion yield according to the depth and the applied protocol: pink squares are for bulk organic matter at low (empty square) and high (plain square) temperature, blue diamonds are for concentrate at low (empty diamond) and high (plain diamond) temperatures. The pink line highlights trends within bulk yields. (b) δ^{13}C results according to the depth and the applied protocol. Same symbols as "a" panel. The blue line is for constant δ^{13}C during the wet period; green lines highlight dry periods with δ^{13}C as results of the mixture between palustrine and open grassland organic matter. Note that y axis is reversed. (c) Difference between ^{14}C dating at high and low temperature, noted $^{14}C_{HT}$-$^{14}C_{LT}$ in the text, for bulk (blue diamonds) and concentrate (pink squares) according to the depth. The blue and pink lines highlight trends. (d) ^{14}C results according to the depth and the applied protocol: same symbols as "a" panel plus green circles are for HF dissolution of Cr (plain circle) and O_2 (empty circle) extracts. See text and Figure 1 for details on protocol. This panel focuses on the 5000–15,000 ^{14}C yr time slice and thus does not show all concentrate dating. See Table 2 to find all results.

As shown in Tables 1 and 2 and Figure 4a, the yield obtained on bulk sediment and "concentrate" are very different: yields are about 5 times higher for bulk than for concentrate. As a consequence of the applied protocol, the concentrate is only a small fraction of initial bulk sediment. Organic matter linked to both diatoms and phytoliths only contributes a negligible amount to the CO_2 evolved from bulk sediment. A large majority of bulk OC is associated with clay. With "bulk" and "concentrate" samples we thus face 2 independent chronologies. The first one is mostly based on free organic matter and organic matter that is associated with clay, and reflects primary production of aerian plants and/or freshwater living organisms to which illuvial carbon might be added. The second chronology characterizes solely the organic matter directly embedded into and adsorbed on both diatoms and phytoliths.

Bulk organic matter (from both the LT and HT fractions) of the Mustang Spring series demonstrates relatively high yield in the middle of the series than at the beginning or the end of the series (Figure 4a). This suggests deposition into different environments with changes in either sedimentation rate or primary production between the middle of the series on one hand and the end and beginning of the sequence on the other hand. "Concentrate" does not present the same features, which is in agreement with expected results from a pure mixture of phytoliths and diatoms. This attests the reliability and efficiency of the concentration treatment even in these unfavorable conditions.

All Mustang Spring samples (bulk, "concentrate," LT and HT fractions) yield similar $\delta^{13}C$ data; the lightest values (to –26‰) are at the top and the bottom of the section and the heaviest values (to –21‰) are found between 300 and 260 cm (Figure 4b). These data suggest that environmental conditions at the beginning and end of the series were similar to one another and different from conditions in the middle the series. The Lubbock Lake sample exhibits a $\delta^{13}C$ value similar to that recorded during deposition of the beginning and the end of the Mustang Spring sample.

Neither of the ^{14}C results obtained on "O_2 treated proteins" nor "Cr treated proteins" presents a coherent pattern (Tables 1 and 2, Figure 3, and partly in Figure 4d). These results vary from ~2400 ^{14}C yr BP to ~15,500 ^{14}C yr BP for Mustang Spring and Lubbock Lake. As an example, "O_2 extract" of Lubbock Lake and "Cr extract" of Mustang Spring 275–285 samples show unexpectedly old ages of a ~14,100 ^{14}C yr BP and ~15,500 ^{14}C yr BP, respectively, which do not fit with the chronological framework of the sequence. The results also show a large number of inversions. "Cr extract" of Lubbock Lake is the only sample that shows the expected age of ~11,000 ^{14}C yr BP. We cannot rule out that this result could be obtained by chance. Rejuvenation and aging can be attributed either to lab contamination or, more likely, to an inadequate blank correction. No ^{14}C-free sample from Great Plains was available and we thus applied a blank correction that was previously defined for marine samples (Hatté et al. 2008). Furthermore, we assume that differential settling contained an identical number of steps and was applied on an identical mass of sample; however, this was not the case and this likely induced a variable level of potential chemistry-induced contamination. Considering the minute amount of carbon present, even using a mass dependent blank correction, a slight shift in the blank value may result in high shift in the resulting age. Obtained ages is in this condition are not reliable. We will not further discuss ^{14}C on proteins. Nevertheless, further investigation would have to decipher whether any oxidant treatment, most likely "Cr" extract, could produce a reliable chronology in a terrestrial environment as it did in a marine one. In such a case, this treatment on bulk sediment would be a rapid and reliable way to remove all of labile organic matter as well as adsorbed organic molecules on silicate biological and mineral supports, in order to ensure preservation of only the targeted carbon.

In contrast, ^{14}C measurements on bulk and "concentrate" samples are coherent and roughly in agreement with archaeological settlement. Mustang Spring ^{14}C dating covers the 6300–11,300 ^{14}C yr BP range and returns older dates for HT than for LT. The difference between HT and LT fractions (hereinafter called $^{14}C_{HT}$–$^{14}C_{LT}$) is much higher for concentrate than for bulk, but both sediment components present a similar pattern with depth: larger differences (to 2800 ^{14}C yr for bulk and to 1200 ^{14}C yr for concentrate) are observed at the top and the bottom of the section and smaller values (to 700 ^{14}C yr for bulk and to 0 ^{14}C yr for concentrate) are observed between 300 and 260 cm. As is observed in δ^{13}C patterns, this represents different environments between the top and the bottom of the section on one hand and the middle of the section on the other hand.

The Lubbock Lake sample displays a large difference between HT and LT fractions in the concentrate component (2300 ^{14}C yr), while the $^{14}C_{HT}$–$^{14}C_{LT}$ for bulk is much smaller (400 ^{14}C yr). Based on the Mustang Spring series pattern, the Lubbock Lake sample is similar to the top and bottom of the Mustang Spring sample series. The "concentrate HT" fraction yields the expected age of 11,000 ^{14}C yr expected age, whereas "concentrate LT" and both bulk fractions exhibit ages that are quite younger than expected.

DISCUSSION

Since the Lubbock Lake sample appears to reflect a specific point of the natural succession recorded by the Mustang Spring series, we will first focus the discussion on Mustang Spring and then consider the specific case of Lubbock Lake.

The geological setting of Mustang Spring indicates a succession of wet and dry episodes. During wet periods, small lakes developed, allowing for large production of diatoms that settled into diatomite layers. Those episodes were associated with high primary production, essentially diatom blooms as shown by the color of the white to light gray sediment. Resulting aquatic OC is associated with high consumption of nutrients and thus heavy OC (~ –20‰). Levels with a depth between 260 and 300 cm can thus be associated with these wet episodes. They are typical diatomite levels. Their linked bulk organic matter results from a mixture between organic carbon contemporaneous to the deposits and subsequent carbon that is likely added by illuviation. Thanks to the dilution effect of the fresh carbon by the in-place carbon, ages of diatomite levels are not very likely to show rejuvenation. This is corroborated by similar ages obtained for both LT and HT fractions. Hence, we can assert that bulk OM are highly likely to provide a reliable chronology of the diatomite deposits. Nevertheless, to be more rigorous and to account for possible added fresh carbon, we would rather recommend "bulk HT."

Dry periods appear to be synchronous with grassland expansion leading to grassland soil development into diatomite layers. This induces injection of graminae organic matter and phytoliths into diatomite layers. Dry intervals are thus associated with an isotopic shift towards lighter δ^{13}C values as result of a mixture between aquatic and aerial organic matter. Aerial OC isotopic signatures fluctuate between isotopic pools of both potential photosynthetic pathways, namely, δ^{13}C of approximately –25‰ for C_3 plants and a δ^{13}C of about –12‰ for C_4 plants. Furthermore, aside from this regular isotopic definition of C_3 and C_4 plants, Krull et al. (2003) reported a 9‰ shift towards lighter values for phytoliths compared to whole-plant δ^{13}C. It is very likely that we did not face an arboreal environment but open grassland. The remaining OC that can be saved during diagenesis and fossilization processes for the most part must have been derived from phytolith rather than lignin. The isotopic signature of an aerial pool thus falls into [–34‰; –21‰] range. Levels at the top and the bottom of the Mustang Spring section can thus be attributed to these dry episodes.

Open grassland is associated with low primary production not large enough to dilute potential illuviated fresh carbon as it does for diatomite so that using bulk as dating support may allow for rejuvenation. This is clearly expressed by the high difference between the HT and the LT fractions, which can reach ~3000 yr. Indeed, the HT fraction is regularly associated with the refractory carbon whereas LT is associated with labile carbon, or relatively "fresh" carbon. Because the soil developed into diatomite layers, bulk HT would include algal carbon deposited earlier in large amounts. The only solution is to work on "concentrate" where clay and associated organic carbon are taken off. Looking at the most recent part of the Mustang Spring series shows that "concentrate LT" and "concentrate HT" fractions both have similar $\delta^{13}C$ but have quite different chronologies. Indeed "concentrate LT" fraction would give a lower accumulation rate of about 10 mm/100 ^{14}C yr whereas the "concentrate HT" chronology would be for 35 mm/100 ^{14}C yr. Mustang Spring's geomorphological context is characterized by storms, wind erosion, dust production, and eolian sedimentation (Stout et al. 1996). This suggests a high sedimentation rate and thus a more likely chronology based on "concentrate HT" fraction. This agrees with the Lubbock Lake sample for which "concentrate HT" effectively provides the expected age. The "concentrate LT" fraction likely includes "fresher" carbon from subsequent phases of open grassland soil development. This carbon presents a similar $\delta^{13}C$ than the considered phase but slightly or largely rejuvenates the chronology of diatomite or soil, respectively. This carbon that evolves at low temperature might likely come from free molecules that move downward and are absorbed by walls of diatoms and phytoliths.

In summary, pure diatomite deposits can be dated with bulk HT, whereas dry deposits should be chronologically characterized with "concentrate HT." The open grassland phases are associated with light $\delta^{13}C$ and high $^{14}C_{HT}$–$^{14}C_{LT}$.

All of these carbon-derived data allow us to draw a deposition model for the Mustang Spring succession (Figure 5). During a wet episode, diatomite accumulated with a $\delta^{13}C$ close to the aquatic pool $\delta^{13}C$ (about –20‰) and with $^{14}C_{HT}$–$^{14}C_{HT}$ close to zero. A dry period followed inducing soil development into the diatomite layers. Aquatic carbon of the upper layers mixed with aerial carbon. This resulted in a $\delta^{13}C$ shift towards lighter values and an increase of $^{14}C_{HT}$–$^{14}C_{HT}$. Revolution goes ahead with a new wet period and diatomite settlement, giving a return to $\delta^{13}C$ close to –20‰ and a decrease of $^{14}C_{HT}$–$^{14}C_{HT}$ and so on. The part of the Mustang Spring succession we studied here is highlighted in Figure 5 by the red rectangle.

An open grassland covered the Mustang Spring area from ~11,500 yr BP by extrapolation of the obtained dating and lasted until 9950 yr BP (level 295–305 cm), then a wet period began as shown by the heavy value of $\delta^{13}C$. A mixture between aquatic and aerian organic matter occurs at ~10 cm as shown by the ~2‰ difference between $\delta^{13}C$ of "concentrate HT" and "bulk HT" fractions. Based on "bulk HT" $\delta^{13}C$, diatomite layers accumulated by ~40 cm until ~9800 yr BP at a 2.7 mm yr^{-1} rate. Then, another dry episode began with grassland soil development that goes back to about 10 cm deep into diatomite layers and lasted until ~8700 yr BP, the upper end of the studied series.

This succession of sedimentological features agrees with the archaeological framework and further suggests human occupation during dry episodes. The chronology series studied fits with a duration of the human occupation from ~11 to 8.7 kyr BP, from the Late Clovis to the Middle Archaic periods. Mustang Spring was furthermore exposed to a short interruption in human occupation during the wet period, i.e. 9950 and 9800 yr BP, between the Plainview and Firstview occupation.

Based on this model, our Lubbock Lake sample corresponds to a dry environment as shown by light $\delta^{13}C$ and a large shift between HT and LT ^{14}C fractions of both bulk and concentrate. We can thus date sample LL65-2A to 10.8 ± 0.3 kyr BP, agreeing with the original ^{14}C dating of ~11,000 yr BP and associated with Folsom archaeology. The drying trend indicated by our data also agrees with other paleoenvironmental interpretations for this time period (Holliday 2000a).

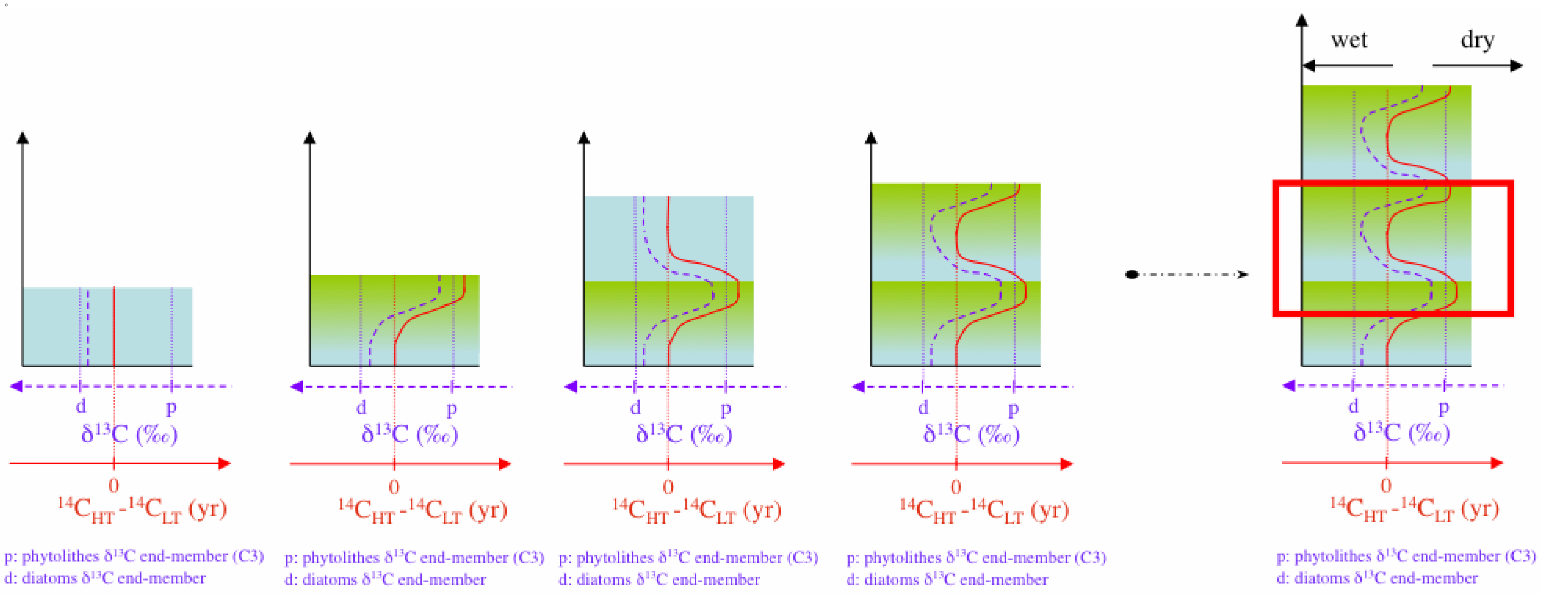

Figure 5 Schematic view of the succession phases of Mustang Spring accumulation, with representations of δ13C and $^{14}C_{HT}$-$^{14}C_{LT}$. Since the δ13C scale is not numerically defined, the δ13C representation is for both bulk and concentrate and both HT and LT fractions. Likewise, $^{14}C_{HT}$-$^{14}C_{LT}$ is for both bulk and concentrate. From left to right: 1- a wet episode with diatomite accumulation (blue) characterized by $^{14}C_{HT}$-$^{14}C_{LT}$ close to zero and a relatively heavy δ13C close to the diatom end-member; 2 - a dry episode with soil development (green) into diatomite layers characterized by increasing $^{14}C_{HT}$-$^{14}C_{LT}$ and decreasing δ13C towards the phytolith end-member; 3 - a new wet episode with diatomite accumulation on the previous soil characterized by a return of $^{14}C_{HT}$-$^{14}C_{LT}$ to 0 and an increase of δ13C, then another dry period, and so on to show the Mustang Spring series. The red rectangle in the last panel shows the Mustang Spring fraction that we study here.

CONCLUSION

With the Mustang Spring and Lubbock Lake studies and the derived chronology, we have shown that considering a geological framework is a key point to understanding ^{14}C signals even in an archaeological context. As an alternative to the common "bulk" ^{14}C, we prove that in the case of sediment richness in both phytoliths and diatoms, bulk HT and concentrate LT provide reliable support for ^{14}C dating to characterize the palustrine and grassland environments, respectively.

In the case of Mustang Spring, we propose a chronological model that asserts several human occupations from ~11 to ~8.7 kyr BP along the 110-cm-long series with an interruption of ~150 yr associated with the palustrine environment between the Plainview and Firstview occupations.

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