Materials and Methods

Site 67 Sampling

The column samples from the wall of Unit 1 from Site 67 that are labeled 80 to 90 cm b.s. correspond to the portion of the midden with the most dense concentration of shell, bone, stone artifacts, fire-altered stone, and bits of burnt earth. This midden deposit is at least 40 cm thick and has no visible stratigraphy. The column sample was excavated in artificial 10 cm levels within this midden. The 7890 ± 70 date on shell, sampled at about the same depth as the column sample from the wall, is 3000 ^{14}C yrs. younger than the phytolith date from the 80 to 90 b.s. column sample. The difference probably reflects the deposition of shell in highly compressed midden characterized by horizontal heterogeneity.

Phytolith Analysis and Dating

Phytoliths were isolated from modern plants and archaeological sediments using standard methods of analysis (ref. *4* in the print manuscript). As described therein, discrete separations of phytoliths from fine silt, coarse silt, and sand fractions of sediment were achieved by gravity sedimentation and wet sieving over a 270 mesh geological screen.

Conventional and accelerator mass spectrometry (AMS) analysis of the carbon-14 trapped within phytoliths was originally described and applied to sediment phytoliths from archaeological and paleoecological contexts in North America (*S1-S3*). We dated phytolith samples by AMS using refinements of these methods developed by Darden Hood of the Beta-Analytic Inc. radiocarbon laboratory, Miami, Florida and DRP, which allow age determinations on smaller numbers of phytoliths, including reasonably discrete separations of *Cucurbita*. Phytoliths for ^{14}C study were isolated from 5 to 10 grams of sediment using the following modifications to standard methods:1) sterile test tubes were used for all steps, 2) oxidation in a solution of concentrated HNO3 and $KClO_3$ was preceded by a 10 minute warm bath in 10% KOH to enhance removal of humic acids, and 3) after phytoliths were removed from sediment by chemical flotation, they were washed and re-heated in concentrated HNO3 and $KClO_3$ to ensure removal of any organic material that might be clinging to surficial crevices. Before submission to the radiocarbon facility, a sub-sample was weighed and then mounted on a microscope slide, and all phytoliths were counted and identified. The number of phytoliths dated was estimated by extrapolation to the sample weight combusted and graphitized at the radiocarbon laboratory.

Once at the radiocarbon facility, phytolith samples were combusted to carbon dioxide in a closed chemistry line under active oxygen flow. Tin was added to the samples prior to combustion so that upon application of heat, the temperature of combustion would elevate to +1400 C via the exothermic reaction between tin and oxygen. This temperature completely melted the phytoliths and thoroughly combusted the target organic carbon within them. Comparisons of SEM photos taken before and after combustion demonstrating an almost pure separation of phytoliths before and only melted silica afterward, confirmed this result (Figs. S1A and B).

In cases where available carbon was below about 250 micrograms after combustion, the small volume of evolved sample CO2 was measured and then diluted with a known quantity of radiocarbon dead CO2 (called the Micro-sample AMS or MS-AMS). This step ensured that enough graphite, about 0.5 to 1 mg, was available for a standard AMS analysis with appropriate fractionation correction. The fraction of sample gas within the diluted gas was calculated as a "dilution factor" to use in the final data calculation. The sigma on the date was derived by combining the counting errors and uncertainties in the dilution step.

Validation of the dating was performed by analyzing a known age reference standard in conjunction with each unknown sample analysis. The reference sample used was TIRI wood "B" (Belfast Pine) with a consensus age of 4503 ± 6 B.P. The standard was analyzed using approximately the same quantity of carbon as the unknown sample and was diluted with the same gas used to dilute the unknown sample. All wood dilution results were in very close agreement with the known age of the reference sample.

Fig. S1. (**A**) The nearly pure separation of phytoliths that was submitted for carbon-14 study from the fine and coarse silt fraction of Vegas Site M5A4-67, Unit 1, 40-50 cm beneath surface. (**B**) The same preparation of phytoliths as above after combustion at Beta Analytic Inc. radiocarbon facility showing how the phytoliths are completely melted, liberating the carbon within them for carbon-14 study. The carbon that was isolated from this treatment returned an age of 8070 ± 160 B.P.

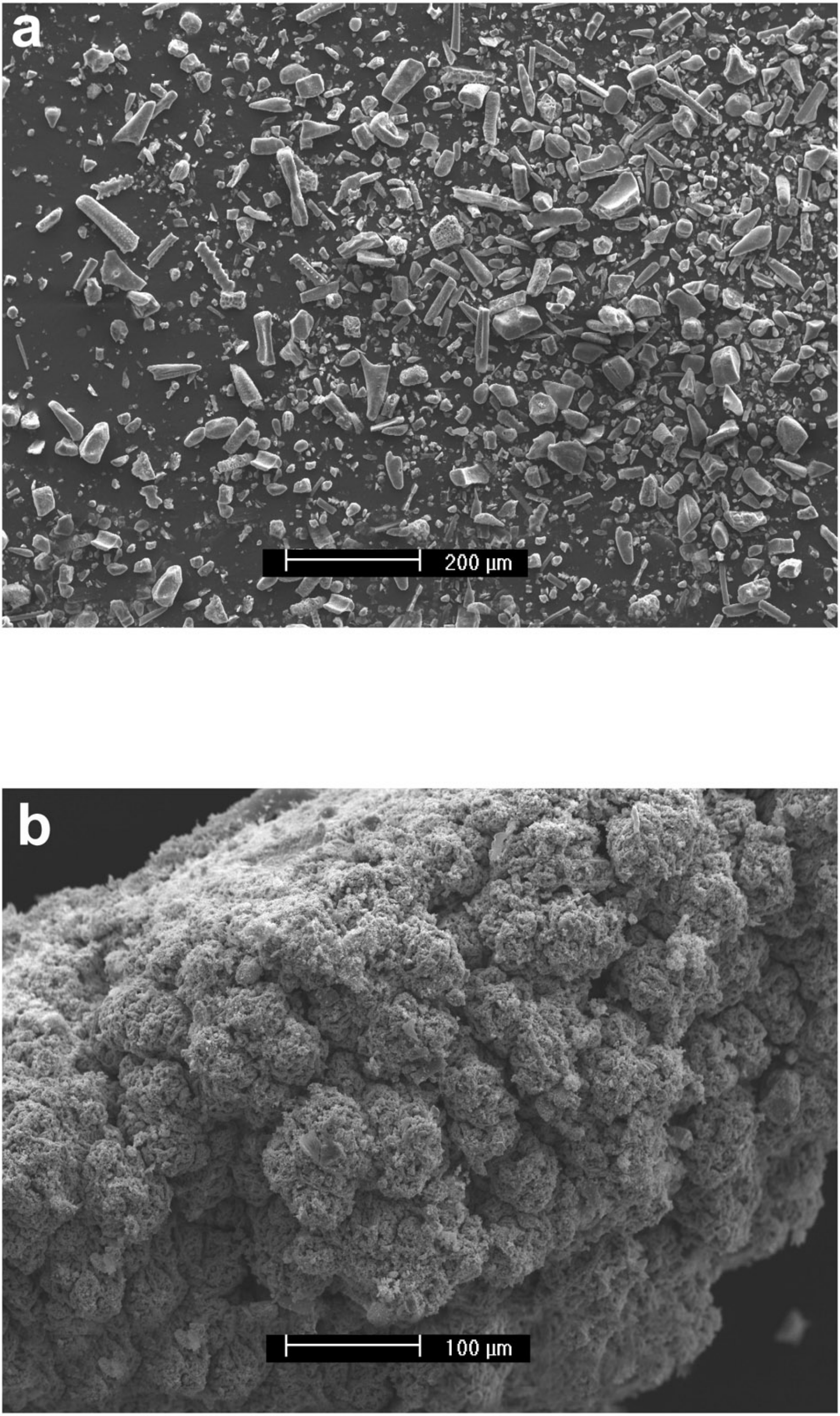
a
200 µm
b
100 µm

Table S1. Phytolith Age and Size in Vegas Sites OGSE-80 and M5A4-67

^{14}C yr. B.P. Phytoliths	Dendrocalibrated, cal-BP 2 S.D. age range	Lab number	Phytolith Length µm	Range	Phytolith Thickness µm	Range	*n*	Sample Provenience cm beneath surface/datum
Site OGSE-80								
7170 ± 60*	8105 to 8095 and 8055 to 7860	UCR-3282; CAMS-14216	94*	64-116	78*	64-95	12, 6	GH8-9, 105-110 fs+cs
10130 ± 40	12120 to 11560		86*		68*	42-93	66, 32	E8-9,110-120, s
9320 ± 250**	11210 to 9900		-		-			E8-9, 110-120, cs
9080 ± 60*	10365 to 10325 and 10275 to 10170		-		-			E8-9, 110-120, fs+cs
7990 ± 220**	9480 to 8370		-		-			E8-9,110-120, fs
9740 ± 60*	11225 to 11095 and 10915 to 10895	UCR-3284; CAMS-14218	72*	48-108	55*	36-76	51, 45	F8-9, 110-120, fs+cs
			65	56-92	55	41-80	9, 3	C113, 210
			72	56-106	56	36-72	14, 10	CH112, 230
			69	40-108	59	36-84	16, 9	C112, 268

Samples from C113, CH112, and C112 are associated with dates on charcoal and shell of 10,840 ± 410, 10,300 ± 240, and 10,500 ± 130 ^{14}C yr. B.P. (13,790 to 10,870 calendar years ago).

^{14}C yr. B.P. Phytoliths	Dendrocalibrated, cal-BP 2 S.D. age range	Lab number	Phytolith Length µm	Range	Phytolith Thickness µm	Range	*n*	Sample Provenience cm beneath surface/datum
Site M5A4-67, Unit 1 Column Sample								
3810 ± 40	4350 to 4330, 4300 to 4090	Beta-168374	86	64-104	70	56-84	15, 13	3-10, fs+cs
5900 ± 40	6780 to 6650	Beta-169266	-	-	-	-	-	3-10 + 20-30 s
	-	-	88	64-104	77	52-96	16, 16	10-20
5910 ± 40	6790 to 6650	Beta-168269	-	-	-	-	-	20-30, fs+cs
7250 ± 190**	8400 to 7760	Beta-167241	88	68-108	73	55-92	18,15	20-30, cs
-			90	58-116	75	44-96	18, 14	30-40
8070 ± 160**	9450 to 8530	Beta-161403	Few *Cucurbita* phytoliths are present					40-50, fs+cs
8240 ± 170**	9540 to 8650	Beta-170223	-	-	-	-	-	50-60 + 60-70, cs
	-	-	88	52-104	75	46-104	24,19	50-60
	-	-	83	56-108	72	48-100	16, 14	60-70
10,820 ± 250**	13420 to 11950	Beta-167239	79	45-112	68	40-84	32,17	70-80, fs+cs
	-	-	77	48-104	63	44-80	24,17	80-90

-		-	73	54-88	63	44-74	13, 6	90-100
M5A4-67, Other Samples								
-		-	86	64-112	71	44-100	48, 35	Unit E3, 60-90
8980 ± 40	10220 to 10140 and 10000 to 9960	Beta-167242	83	52-116	70	48-92	33, 27	Unit E4,110-120, fs+cs+s

*Phytolith date and size data reported previously in ref. 9 of the print manuscript. **MS-AMS ^{14}C phytolith method. fs=fine silt, cs=coarse silt, s=sand. Under sample number (*n*), the first is for phytolith length and the second is for thickness. All Site 80 samples are in cm beneath datum (b.d.). Unit 1 column samples from Site 67 are in cm beneath surface (b.s.); the other two are in cm. b.d. The date of 3810 ± 40 B.P. from 3-10 b.s. of Site 67 is consistent with the presence of a few ceramic sherds dating to the late Valdivia period in superficial contexts at this site.

References

S1. Wilding, L.P., *Science* **156**, 66 (1967).

S2. Kelly, E.F., R.G. Amundson, B.D. Marino, M.J. Deniro, *Quat. Res.* **35**, 222 (1991).

S3. Mullholland, S.C., C. Prior, in *Current Research in Phytolith Analysis:Applications in Archaeology and Paleoecology*, D.M. Pearsall and D.R. Piperno, Eds. (The University Museum of Archaeology and Anthropology, Philadelphia, 1993), pp. 21-23.