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Paleoclimatic Reconstruction Using the Correlation in δ^{18}O of Hackberry Carbonate and Environmental Water, North America

A. Hope Jahren

Department of Earth and Planetary Sciences, The Johns Hopkins University, Baltimore, Maryland 21218

E-mail: jahren@jhu.edu

Ronald Amundson

Division of Ecosystem Sciences, Department of Environmental Science, Policy and Management, University of California, Berkeley, California 94720

Carol Kendall

Water Resources Division, United States Geological Survey, Menlo Park, California 94025

and

Peter Wigand

Great Basin and Mojave Paleoenvironmental Consulting, Reno, Nevada 89506

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Celtis **sp. (commonly known as "hackberry") fruits were collected from 101 North American sites located in 13 states and one Canadian province between the years of 1979–1994. The biomineralized carbonate endocarp of the hackberry, which is a common botanical fossil found throughout the Quaternary sediments of the Great Plains, was analyzed for its δ^{18}O value and plotted against the δ^{18}O value of site environmental water to demonstrate the potential of the hackberry as a paleoclimate indicator. This correlation was reinforced by intensive studies on extracted tissue-water δ^{18}O value and hackberry endocarp carbonate δ^{18}O value from three trees in Sterling, Colorado. The observed correlation in the large data set between hackberry endocarp carbonate δ^{18}O value and environmental water is [endocarp $\delta^{18}\mathrm{O} = 38.56 + 0.69 \times$ environmental water δ^{18}O] ($R = 0.88$; $R^2 = 0.78$; p value < 0.0001). The relation of the hackberry carbonate to temperature in the Great Plains was the following: (average daily-maximum growing season temperature [°C]) $= 6.33 + 0.67$ (δ^{18}O of endocarp carbonate) ($R = 0.73$; $R^2 = 0.54$; p value $= 0.0133$). The δ^{18}O value of early Holocene fossil hackberry carbonate in the Pintwater Cave, southern Nevada, suggested precipitation δ^{18}O values more positive than today ($\sim -4‰$ early Holocene vs ~ -9 to $-10‰$ today). This shift, combined with paleobotanical data, suggests an influx of summer monsoonal moisture to this region in the early Holocene. Alternatively, the more positive δ^{18}O values could be viewed as suggestive of warmer temperatures, although the direct use of Great Plains hackberry/temperature relationships to the Great Basin is of debatable value.** © 2001 University of Washington.

Key Words: **oxygen isotope; paleoclimate; hackberry carbonate.**

INTRODUCTION

Celtis occidentalis L. (known commonly and referred to hereafter as "hackberry") is a deciduous tree in the elm family (Ulmaceae). This irregularly shaped tree sometimes occurs as a shrub and has a wide but fragmented distribution in North America (Little 1971: map 121; see Little, 1976, maps 33N, 33NW, and 33SW for the distribution of *C. reticulata* and Little, 1971, maps 122W and 122E for the distribution of *C. laevigata*). The hackberry produces a fleshy, globose drupe containing a single seed. Fruits ripen in late fall and are dispersed by birds, rodents, other small mammals, and gravity. The drupe contains a fleshy mesocarp and a biomineralized aragonite-rich endocarp, all of which encase the endosperm. The mineralogy, morphology, and isotope chemistry of the hackberry during development have been described elsewhere (Cowan *et al.*, 1997; Jahren *et al.*, 1998). The durability of the biomineralized aragonite endocarp contributes to its preservation in the fossil record: fossil hackberry endocarps are found throughout the Quaternary sediments of the Great Plains and are common botanical remnants in sediments of the Miocene and Oligocene epochs. Because the hackberry fruit is edible and was a favorite condiment of some American Indian groups, fossil endocarps are commonly found in Great Plains Holocene archeological sites (Thomasson, 1991).

We first pursued the idea that hackberry endocarps might be a valid paleotemperature indicator by examining several *Celtis occidentalis* individuals and comparing the isotopic composition of

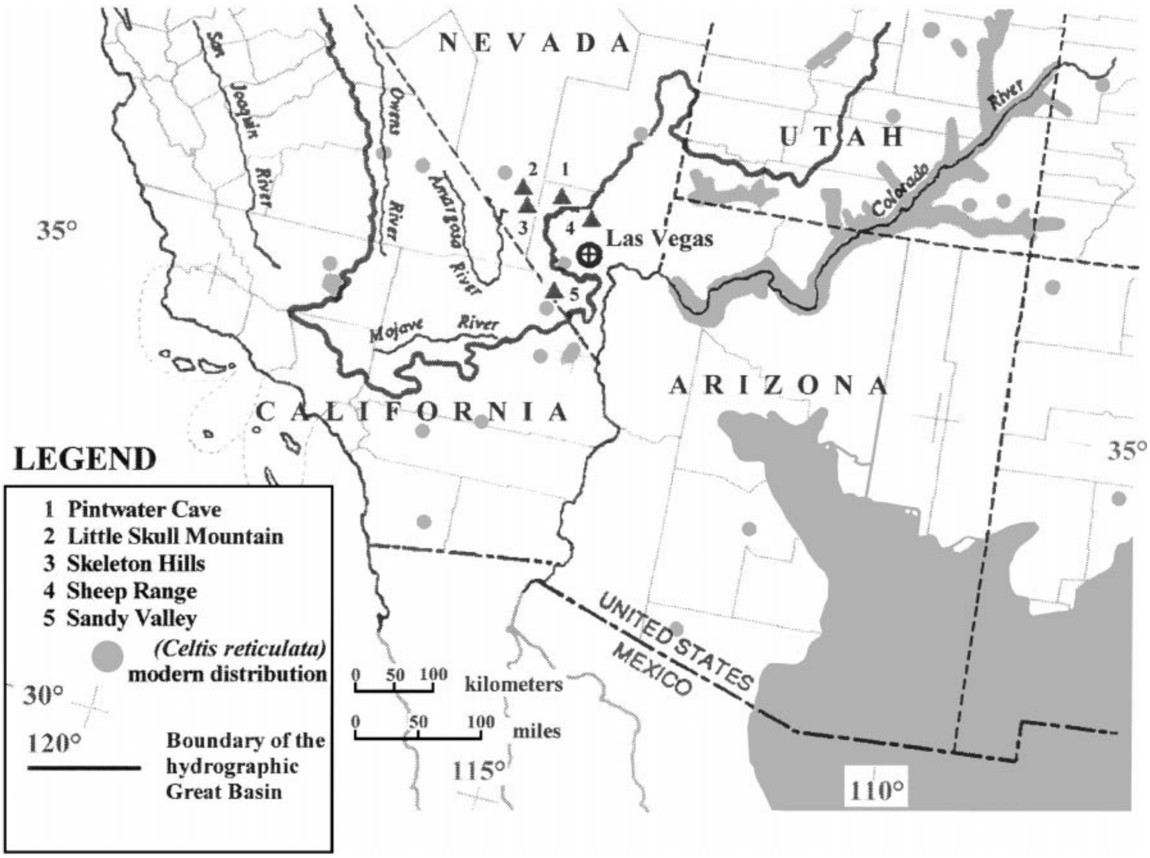

FIG. 1. Current distribution of *Celtis reticulata* in the southernwestern United States and localities from which fossil endocarps have been recovered in the northern Mojave Desert. These endocarps all date between 9000 to 10,000 ^{14}C yr B.P.

water extracted from tree tissues with that of local precipitation and long-term reservoirs of environmental water, such as streams and lakes. It is the demonstrated relationship between environmental water and site temperature that forms one basis of terrestrial paleoclimate reconstruction (Craig, 1961; Dansgaard, 1964; Yurtsever, 1975; Gat, 1981). Since the ultimate source of oxygen for hackberry endocarp carbonate is plant cell water (Cowan *et al.*, 1997), the isotopic composition of biomineralized endocarp aragonite might be strongly affected by water type and source. In addition, many photosynthetic tissues transpire, which dramatically enriches the isotopic composition of plant fluids in δD and δ^{18}O value (reviewed in Ehleringer and Dawson 1992). The δ^{18}O value of tree ring holocellulose has been shown to be well correlated with environmental temperature in modern fir trees (Ramesh *et al.*, 1986). Here we quantify the effect that source water has upon endocarp oxygen isotope composition through comparison of isotopic composition of source water and carbonate values at the Sterling, Colorado, field site.

We test the relationship of environmental water to hackberry carbonate δ^{18}O values *via* a database of carbonate δ^{18}O values from 101 North American sites. Using 30-yr-averaged temperature data from these sites we then construct a predictive relationship between hackberry endocarp carbonate δ^{18}O values and average daily maximum growing season temperature.

We applied the relationship revealed by this set of modern hackberry endocarps to the δ^{18}O values of early Holocene fossil hackberry endocarp carbonate from Pintwater Cave in the Pintwater Range of Southern Nevada (Fig. 1). This period is ideal for testing our results because other significant evidence is available from the northern Mojave Desert and the Intermountain West regarding the climate of this period. Conclusions derived from this application of our method have particular importance with regard to global temperature estimates and inferred climatic regimes for this period referred to as the “Holocene thermal maximum.”

METHODS

Field Collection

Hackberry occurs in a wide range of habitats, including riparian woodlands, various shrub communities, rocky ravines, and as scattered individuals in grasslands. This range of habitats demonstrates the hackberry’s “wide ecological tolerances” (DeBolt, 1992), as is evidenced in the tree’s tendency to occupy all types of mesosites, including draws, talus slopes, river banks, and terraces (DeBolt, 1992), and to grow at elevations ranging from 200 to 2000 m asl (Elias, 1980).

To determine the magnitude and consistency of the fractionation factor between hackberry carbonate and environmental water, three hackberry (*Celtis occidentalis*) trees near Sterling, Colorado, were identified for intensive study of stem tissue and hackberry fruits. Sampling was performed during the 1994 growing season, between the months of May and October. None of the trees used in the study had been irrigated since planting. During the hackberry growing season, the average daily temperature in Sterling, Colorado, is 18°C and the average daily relative humidity is 39% (Spangler and Jenne, 1990). Three trees >7 yr of age (i.e., not of juvenile age) were chosen for sampling at each site and flagged for identification. During a sampling session, stem tissue and hackberry fruits were removed from two locations on each of the three trees. Stems and fruits were then packed into 40-mL borosilicate vials until headspace was minimal and sealed with Teflon-coated caps to inhibit evaporation. Water from local wells, streams, lakes, and rivers was collected in 40-mL borosilicate vials and sealed with Teflon-coated caps; snow was sampled in the same manner. Precipitation was collected in two rain gauges consisting of a 1-L Teflon bottle with funnel attachment. Inside the bottle, approximately one inch of silica oil was inserted to prevent evaporation. Precipitation samples were removed by withdrawing water using a large-gauge syringe and injecting it into 40-mL borosilicate vials and sealing with Teflon-coated caps. During transport to the laboratory and before analysis, plant tissue samples were kept at $\sim -15°$C and water samples were kept at $\sim 5°$C.

For the large database, hackberry (mostly *Celtis occidentalis*, with a minor component of *Celtis reticulata*) fruits were collected from 101 North American sites located in 13 states and one Canadian province (Table 1). Collection took place between the years of 1979–1994 and was performed by the USDA Great Plains Tree Improvement Committee and also by Dr. Mark Gabel of Black Hills State University, South Dakota. Specimens were air dried in between collection and analysis.

The January 1996 excavations in Pintwater Cave, Nevada, resulted in the recovery of 227 fossil hackberry endocarps. These were retrieved from four of eight depositional horizons (Table 2). Radiocarbon dates from four of the five excavation units (primarily from sheep fecal pellets) indicate that most of the hackberry endocarps recovered date from 10,000 to 9,000 ^{14}C yr B.P. (Table 5; Buck *et al.*, 1997).

Pintwater Cave lies on the west slope of the Pintwater Range ~25 km north northeast of Indian Springs, Nevada (Fig. 1; Buck *et al.*, 1997: Fig. 4). The cave's orchestra-shell-shaped opening faces west at an elevation of 1268 m. Its only access is a very steep 200-m climb up the limestone cliffs from the playa below. The cave tapers from a 12.2-m-wide entrance to a steeply rising rock–fall talus that joins the ceiling at the back wall over 57.9 m away. At its highest point, the roof of the cave is about 5 to 6 m above the floor. Radiocarbon ages of over 32,000 ^{14}C yr B.P. at 1 to 2 m below the surface suggest that much of the late Quaternary may be represented in the deeply stratified rock fall and aeolian fill of the cave floor. The modern plant community growing in the silt-rich soils around the cave entrance is comprised of a xeric shrubs dominated by shadscale (*Atriplex confertifolia*), snakeweed (*Gutierrezia sarothrae*), and creosote bush (*Larrea tridentata*). This species configuration reflects the alkaline soils derived from the playa that lie in the Indian Springs Valley below and west of the cave entrance. Playa deposits are lifted by the wind to blanket the west slopes of the Pintwater Range. Where soils are well-leached or silts less abundant, pigmy-cedar (*Peucephyllum schottii*) is the dominant vegetation. Native stipa grasses have been almost entirely displaced by invading cheatgrass of Eurasian origin. Hackberry does not occur anywhere in the Pintwater Range today. Current interest in the cave has been primarily generated by abundant middle Holocene archaeological remains that lie in its surface deposits.

We submitted 147 endocarps for δ^{18}O analysis. Based on examination in thin-section and upon their X-ray diffraction pattern, two separate fossil hackberry endocarps (designated "A" and "B" in Table 3) were selected from each depositional layer of Pintwater Cave and were analyzed for δ^{18}O value of carbonate (Table 3). The criteria used for selection were based on visual similarity in thin-section and diffraction pattern with modern hackberry endocarps. All fossil endocarps chosen for study had percentage carbonate (by mass) composition within 5% of values seen in modern endocarps. Using these selection criteria, more than 85% of fossil hackberry endocarps examined met the evaluation standards, suggesting that endocarps from the Pintwater Cave site are predominantly well preserved with respect to carbonate diagenesis.

Isotopic Analyses

Hackberry endocarps from all sites were prepared for carbonate analysis by cutting away the fleshy layers, scraping to remove as much organic material as possible, and further processing using a preparation technique designed to remove residual organic material chemically without altering the isotopic signature of the carbonate (Jahren, 1996). This involved completely pulverizing the isolated mineral endocarp to maximize surface area and then exposing the material to standard household bleach (sodium hypochlorite, ~5%) for 1 h at room temperature before rinsing and lyophilization. Carbonate standards subjected to this preparation technique were found to retain characteristic δ^{13}C and δ^{18}O values within 0.1 and 0.2‰, respectively. Hackberry carbonate was then reacted with 100% phosphoric acid (McCrea, 1950) at 90°C in an automated common acid bath carbonate device. Each fossil endocarp from Pintwater Cave was prepared for analysis using the method described above and was analyzed for δ^{18}O value in triplicate. Variability in δ^{18}O value between replicates was much less than the precision associated with measurement on the mass spectrometer (± 0.10‰ for δ^{18}O values).

Water was extracted from plant stems and fruits *via* vacuum distillation at 100°C for 6 h, as described in Jahren (1996).

TABLE 1
δ^{18}O and δ^{13}C Carbonate Values and Site Locations of Hackberry Endocarps

	Collection information			Endocarp carbonate	
Sample code[a]	County	State (or Province)	Year	$\delta^{18}O_{(VSMOW)}$ [‰]	$\delta^{13}C_{(VPDB)}$ [‰]
USDA 2499	Fulton	AR	1985	35.59	−10.49
USDA 2131	Cleburne	AR	1985	34.55	−9.33
USDA 2496	Yell	AR	1985	33.92	−11.58
MG 2655	Yuma	CO	1992	32.07	−9.58
SC celtis	Logan	CO	1993	32.47	—
USDA 3976	Monona	IA	1983	31.08	−12.42
USDA 3954	Sioux	IA	1983	31.05	−15.19
USDA 3986	Mitchell	IA	1984	30.19	−14.14
USDA 3881	Barber	KS	1982	33.50	−10.00
USDA 4054	Lvnworth.—1	KS	1984	33.22	−12.61
USDA 4055	Lvnworth.—2	KS	1984	33.26	−11.80
USDA 4005	Nemaha	KS	1983	33.43	−12.17
USDA 4042	Jackson	KS	1984	33.52	−11.65
USDA 4063	Harvey	KS	1984	36.07	−12.54
USDA 3870	Clark—site 1	KS	1982	35.94	−10.67
USDA 2126	Stafford	KS	1985	36.26	−12.95
USDA 3981	Wabaunsee	KS	1983	36.20	−11.64
USDA 4015	Summer	KS	1984	35.91	−13.49
MG 2625	Clark—site 2	KS	1992	34.41	−12.40
USDA 3879	Jewell	KS	1985	34.29	−9.10
USDA 4079	Cherokee	KS	1985	35.51	−10.08
USDA 3942	Pratt	KS	1983	35.11	−9.41
USDA 3856	Delta, Canada	MB	1982	30.28	−12.35
USDA 2116	Big Stone—2	MN	1985	31.24	−13.14
USDA 3847	Lac Qui Parle	MN	1982	29.47	−10.19
Austin 805	Mower	MN	1994	31.19	—
USDA 3939	Kandiyohi—1	MN	1983	30.59	−12.00
USDA 3878	Polk	MN	1982	29.90	−11.74
USDA 3836	Big Stone—1	MN	1982	33.15	−11.90
USDA 2138	Dodge	MN	1985	31.54	−13.15
USDA 3940	Kandiyohi—2	MN	1983	30.82	−12.98
USDA 4090	Henry	MO	1984	33.46	−10.83
USDA 4081	Carter	MO	1984	33.42	−13.23
USDA 4101	Franklin	MO	1984	33.67	−11.11
USDA 3854	Ramsey	ND	1982	29.72	−10.55
USDA 3864	Morton	ND	1982	29.20	−14.30
USDA 3834	Barnes—1	ND	1982	29.03	−14.64
USDA 2134	Barnes—2	ND	1985	29.85	−9.98
USDA 2114	Logan	ND	1985	29.91	−10.20
USDA 3867	McHenry—2	ND	1982	29.38	−9.62
USDA 3829	Cass	ND	1982	29.50	−14.83
USDA 3853	McHenry—1	ND	1982	29.39	−11.80
USDA 2497	Stutsman	ND	1985	30.73	−10.50
MG 2619	Custer—2	NE	1992	30.95	−11.63
USDA 3882	Custer—1	NE	1982	30.92	−13.51
USDA 3941	Boyd	NE	1983	30.16	−13.88
USDA 3865	Boone	NE	1982	31.16	−13.89
MG 2616	Cherry—2	NE	1992	30.96	−11.23
USDA 3911	Thomas—1	NE	1983	30.76	−13.27
USDA 2121	Knox	NE	1985	34.95	−11.97
USDA 3863	Dawson	NE	1982	30.62	−10.58
MG 2618	Thomas—3	NE	1992	30.67	−17.14
USDA 3926	Franklin	NE	1983	34.29	−13.27
USDA 3937	Pawnee	NE	1983	29.62	−13.09
USDA 4001	Nemaha	NE	1983	33.09	−11.16

TABLE 1—*Continued*

	Collection information			Endocarp carbonate	
Sample code[a]	County	State (or Province)	Year	$\delta^{18}O_{(VSMOW)}$ [‰]	$\delta^{13}C_{(VPDB)}$ [‰]
USDA 4012	Box Butte	NE	1984	31.98	−13.42
USDA 3877	Hall	NE	1982	32.57	−13.16
USDA 3844	Thayer	NE	1982	32.72	−11.90
USDA 4045	Cherry—1	NE	1984	31.88	−13.46
USDA 4032	Dodge	NE	1984	31.51	−12.39
MG 2617	Thomas—2	NE	1992	30.17	−12.63
C. reticulata only in NM					
MG 2644	San Miguel—1	NM	1992	36.65	−11.98
MG 2651	Union—2	NM	1992	35.16	−12.41
MG 2641	Curry—2	NM	1992	36.48	−12.43
MG 2642	Quay	NM	1992	36.31	−10.27
MG 2650	Union—1	NM	1992	36.71	−12.88
MG 2646	San Miguel—3	NM	1992	37.69	−10.89
MG 2645	San Miguel—2	NM	1992	39.24	−9.68
MG 2653	Union—4	NM	1992	39.18	−12.63
MG 2640	Curry—1	NM	1992	35.51	−10.35
MG 2652	Union—3	NM	1992	34.08	−10.41
C. occidentalis area					
USDA 3968	Rogers	OK	1983	34.27	−10.79
USDA 4112	Osage	OK	1985	34.48	−11.32
USDA 4044	Pawnee—2	OK	1984	34.72	−9.75
USDA 3967	Pawnee—1	OK	1983	34.83	−11.33
USDA 4067	Haskell	OK	1984	35.12	−13.61
USDA 4053	Pottawatomie	OK	1984	34.84	−11.82
Both species occur in these counties					
USDA 3963	Kingfisher—1	OK	1983	35.55	−11.55
USDA 4061	Kingfisher—2	OK	1984	35.88	−14.31
USDA 4062	Kingfisher—3	OK	1984	36.56	−12.57
MG 2628	Woodward	OK	1992	35.51	−9.96
MG 2631	Ellis	OK	1992	36.92	−14.00
C. reticulata area					
USDA 4022	Beaver	OK	1984	37.00	−11.49
USDA 4033	Comanche	OK	1984	38.24	—
USDA 2136	Walworth	SD	1985	32.48	−11.86
USDA 2492	Deuel	SD	1985	32.50	−11.66
USDA 4043	Bennett	SD	1984	32.13	−13.56
USDA 4035	Charles Mix	SD	1984	30.33	−14.62
USDA 3923	Potter—2	SD	1983	30.34	−11.86
USDA 3783	Potter—1	SD	1979	29.91	−12.63
USDA 4039	Sully	SD	1984	31.17	−12.45
USDA 4034	Gregory	SD	1984	30.35	−11.51
USDA 4071	Jackson	SD	1984	30.27	−12.10
USDA 3924	Stanely	SD	1983	29.96	−14.90
USDA 3848	Hand	SD	1982	32.06	−10.31
Both *C. reticulata* and *C. occidentalis* can occur					
MG 2634	Lopscomb	TX	1992	33.92	−13.75
MG 2635	Hemphill	TX	1992	35.58	−10.58
MG 2636	Gray	TX	1992	34.51	−12.49
MG 2637	Randall—1	TX	1992	34.07	−12.75
MG 2639	Randall—2	TX	1992	34.47	−14.29
Only *C. reticulata* occurs in WY					
Cheyenne 93	Laramie	WY	1993	35.35	—

[a] MG-coded samples were collected by Dr. Mark Gabel; USDA-coded samples were collected by the USDA Great Plains Tree Improvement Committee; other samples were collected by the authors.

TABLE 2
^{14}C Age of *Celtis Reticulata* Endocarps in the Sediments of Pintwater Cave

Horizon #	^{14}C age^{a} (^{14}C yr B.P.)	^{14}C age provenience	Unit #1	Unit #2	Unit #3	Unit #4	Unit #5
1	7349 ± 64	Unit # 3, H #1					
	8312 ± 130	Unit #3, H #1					
2	8608 ± 88	Unit #2, Top H #2		1	10		2
	9513 ± 129	Unit #1, Top H #3					
3	9042 ± 72	Unit #3, Top H #3			9	184	5
	9200 ± 100	Unit #4, H #3					
	9300 ± 100	Unit #4, H #3					
	10,063 ± 90	Unit #3, H #3					
	10,109 ± 92	Unit #1, Bottom H #3					
4				1			
5	15,379 ± 111	Unit #4, H-5				15	
6							
7	28,410 ± 250	Unit #2, H-7					

[a] Radiocarbon ages and their provenience from Buck *et al.*, 1997; provenience of *Celtis* endocarps from unpublished data of Wigand. Provenience is recorded by Test Pit # (Unit #) and stratigraphic depth Horizon (numbered from the surface). Radiocarbon ages are uncalibrated radiocarbon ages.

Collected and extracted water samples were prepared for measurement of oxygen isotope composition *via* equilibration with CO_2 of known composition and subsequent measurement of the postequilibration CO_2 (Epstein and Mayeda, 1953). Water samples were prepared for measurement of the hydrogen isotopic composition by reacting $3\,\mu$L of water with purified Zn in an evacuated tube at 500°C to yield hydrogen gas (Coleman *et al.*, 1982).

Measurement of stable isotope ratios was performed on a VG Prism stable isotope mass spectrometer at the Lawrence Berkeley National Laboratory. All isotope values are reported in the delta notation,

$$\delta = \frac{R_{sample} - R_{standard}}{R_{standard}} \times 1000\,[‰],$$

where the standard is VSMOW for $R = {}^{18}\mathrm{O}/{}^{16}\mathrm{O}$ and $R = \mathrm{D/H}$; and VPDB for $R = {}^{13}\mathrm{C}/{}^{12}\mathrm{C}$. The precision associated with measurement on the mass spectrometer is ±0.10‰ for δ^{18}O values, ±2.0‰ for δD values, and ±0.05‰ for δ^{13}C values.

The isotopic variability in the carbonate of a typical modern hackberry endocarp is demonstrated by the following data for one fruit collected in Mower County, Minnesota, that was divided into nine parts: total range of values of $\delta^{18}\mathrm{O} = 1.07‰$ ($M = 31.1‰$, $SD = 0.39‰$, $n = 9$); total range of values of $\delta^{13}\mathrm{C} = 0.93‰$ ($M = -14.8‰$, $SD = 0.34‰$, $n = 9$) (Jahren, 1996). For all hackberries used in this study, the entire endocarp of one fruit was homogenized, prepared, and analyzed.

Within-site isotopic variability has been characterized in two ways: variability due to the process of endocarp biomineralization and variability in mature endocarps taken from different trees within the same site (Jahren *et al.*, 1998). Total range of values in endocarp carbonate during 120 days of development was assessed as 3.0‰ for δ^{18}O and 8.0‰ for δ^{13}C, based on eight measurements made at each of three different sites. Total range of values seen in mature endocarp carbonate within a site was assessed as 1.2‰ for δ^{18}O and 3.0‰ for δ^{13}C, based on six measurements made at each of two different sites. All endocarps used in this study were mature and were analyzed in entirety; therefore, we estimate the total within-site variability that could be assigned to each data point to be 1.2‰ for δ^{18}O and 3.0‰ for δ^{13}C. Maturity in endocarps can be recognized by the developed reticulate pattern apparent in endocarps, which is also preserved in fossil specimens of mature endocarps.

Water oxygen isotope data used for the large data set correlation were obtained from a set of depth-integrated stream water samples collected in two USGS monitoring networks (NASQAN and Benchmark) in the United States (Smith and Alexander, 1987). Each site was sampled on average 12 times over 2–3 years in the mid-1980s, and the data were averaged. These stream data show the same general isotopic patterns as precipitation at the same location (Kendall and Coplen, 2001). We chose to use stream data for this correlation in preference to precipitation data because stream data are a better estimate of the composition of water residing in soil (and thus available for plant uptake); stream water, like soil water, is slightly affected by relative humidity. The average δ^{18}O value of the closest USGS site on the same major river, stream, or drainage basin to each hackberry site was used for the correlation. In most cases, hackberry and stream sites were located within 100 km of each other, and given that both hackberry carbonate and stream water δ^{18}O values are not highly variable over 100-km geographic scales, a broad regional study is justified.

RESULTS AND DISCUSSION

Stable isotope analysis of well water, surface water, precipitation, and snow samples obtained at and near the Colorado

TABLE 3
Pintwater Cave Growing Season Paleotemperature Prediction Based on Fossil Hackberry Endocarps

Pintwater cave site ID	Pintwater cave provenience	Hackberry carbonate $\delta^{18}O_{VSMOW}$ [‰]	Growing season paleotemperature prediction °C (±0.8°C)
2072-2 A	Unit #3, H #3	41.90	34.4
2072-2 B	Unit #3, H #3	40.11	33.2
2100-2 A	Unit #4, H #3	36.41	30.7
2100-2 B	Unit #4, H #3	29.21	25.9
2105-2 A	Unit #4, H #3	35.99	30.4
2105-2 B	Unit #4, H #3	35.89	30.4
2146-1 A	Unit #4, H #3	39.84	33.0
2147-2 A	Unit #4, H #3	32.38	28.0
2147-2 B	Unit #4, H #3	32.48	28.1
2149-2 A	Unit #4, H #3	36.98	31.1
2149-2 B	Unit #4, H #3	37.87	31.7
2152-2 A	Unit #4, H #3	41.24	34.0
2152-2 B	Unit #4, H #3	33.13	28.5
2153-2 A	Unit #4, H #3	38.50	32.1
2153-2 B	Unit #4, H #3	31.88	27.7
2157-2 A	Unit #4, H #3	35.44	30.1
2157-2 B	Unit #4, H #3	38.85	32.4
2161-2 A	Unit #4, H #3	32.59	28.2
2161-2 B	Unit #4, H #3	36.96	31.1
2169-2 A	Unit #4, H #3	33.88	29.0
2169-2 B	Unit #4, H #3	34.97	29.8
2175-2 A	Unit #4, H #3	37.25	31.3
2175-2 B	Unit #4, H #3	36.09	30.5
2177-2 A	Unit #4, H #3	36.03	30.5
2177-2 B	Unit #4, H #3	35.38	30.0
2189-2 A	Unit #4, H #3	33.50	28.8
2189-2 B	Unit #4, H #3	35.12	29.9
2194-2 A	Unit #4, H #3	37.78	31.6
2194-2 B	Unit #4, H #3	34.51	29.4
2198-2 A	Unit #4, H #3	36.08	30.5
2198-2 B	Unit #4, H #3	38.21	31.9
2203-2 A	Unit #4, H #3	38.12	31.9
2203-2 B	Unit #4, H #3	35.61	30.2
2246-2 A	Unit #4, H #5	33.87	29.0
2246-2 B	Unit #4, H #5	36.26	30.6

TABLE 4
Stable Isotope Composition of Environmental Water at Colorado Field Site

Site	Date of collection	$\delta^{18}O_{VSMOW}$ [‰]	δD_{VSMOW} [‰]
Sterling, CO, Site Well	5/31/94	−12.54	−97.0
	8/29/94	−12.82	−96.5
	9/10/94	−12.81	−92.9
South Platte River, near Sterling, CO	8/29/94	−10.15	−86.3
	9/10/94	−11.13	−91.2
	3/30/95	−11.66	−90.1
	7/19/95	−13.96	−102.0
Precipitation from Sterling, CO, site	6/29/94	−7.55	+0.4
	7/29/94	−3.03	+0.7
	8/10/94	−7.18	−51.4
	8/29/94	−2.54	−17.8
	9/10/94	−8.16	−57.1
Snow from Sterling, CO	3/30/95	−21.15	—
Snow from Merino, CO	3/30/95	−20.33	−148.8

Note. CO = Colorado, USA.

field site are presented in Table 4. Values presented represent averages of four measurements at each site; in each case the total variability in individual subsamples was much less than the precision associated with measurement on the mass spectrometer (±0.10‰ for $\delta^{18}O$ values, ±2.0‰ for δD values). The results in Table 4 show that the $\delta^{18}O$ value of well water changes by less than 0.4‰ during the course of the growing season, in agreement with previous work at other sites demonstrating that the δD value of groundwater was unchanging over a 1-yr period (Flanagan and Ehleringer, 1991). Reservoirs within several miles of the field sites also exhibit unchanging isotopic composition, albeit they may be up to 2.6‰ different than well water at the site (Table 4). Significantly, the isotopic composition of precipitation is highly variable, even on a short-term basis. In terms of $\delta^{18}O$ value, growing season precipitation is relatively isotopically heavy, compared with winter snow, which is as much as 18‰ isotopically lighter. However, groundwater has been shown to be the source of water for large plants, with no fractionation of isotopes by plant roots (Dawson and Ehleringer, 1991), suggesting that the Sterling site well water reflects most closely the composition of water within the nontranspiring tissues of site hackberry trees.

We compared the $\delta^{18}O$ value of water extracted from hackberry stems and fruits with the $\delta^{18}O$ value of endocarp carbonate within the same fruits (Table 5). Each value represents the average of all trees sampled at the Sterling site; variability between samples was less than the precision associated with measurement on the mass spectrometer (±0.10‰ for $\delta^{18}O$ values, ±2.0‰ for δD values). The difference between $\delta^{18}O$ value of the fruit water and $\delta^{18}O$ value of the endocarp carbonate is 38.42‰ for Sterling, Colorado. Hackberries were sampled in September, well after the fruits had entered Stage 3 of drupe formation, the final stage of fruit maturity (Williamson and Coston, 1989; Jahren *et al.*, 1998). Fruits collected at their most mature state

TABLE 5
Comparison of Hackberry Endocarp Carbonate Stable Isotope Values and Other Components of the Hackberry Tree

Site	Year collected	Green stem water $\delta^{18}O_{VSMOW}$ [‰] (δD_{VSMOW} [‰])	Fruit water $\delta^{18}O_{VSMOW}$ [‰] (δD_{VSMOW} [‰])	Hackberry carbonate $\delta^{18}O_{VSMOW}$ [‰] ($\delta^{13}C_{VPDB}$ [‰])
Sterling, CO	1994	−8.91 (−80.7)	−5.40 (−52.3)	+33.12 (−12.7)

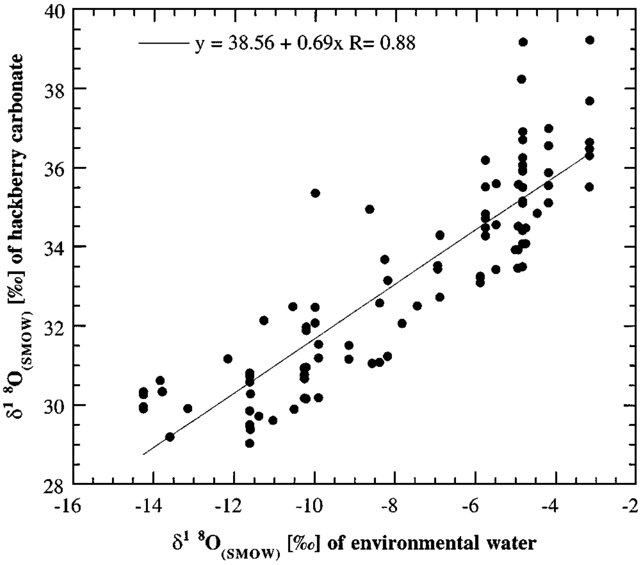

FIG. 2. Correlation between the δ^{18}O value of hackberry endocarp carbonate at a site and the δ^{18}O value of environmental water at the same site, or at the nearest existing site within the same drainage area.

(Stage 3 or beyond) show a consistent fractionation between δ^{18}O of the fruit water and δ^{18}O of the endocarp carbonate (Jahren *et al.*, 1998).

The correlation seen between the δ^{18}O value of hackberry endocarp carbonate at a site and the δ^{18}O value of environmental waters at the same site, or at the nearest existing site within the same drainage area is described by [endocarp $\delta^{18}O = 38.56 + 0.69 \times$ environmental water δ^{18}O] ($R = 0.88$; $R^2 = 0.78$; $p < 0.0001$; Fig. 2, data in Table 1). Figure 2 shows a strong positive relationship between hackberry endocarp δ^{18}O values and the δ^{18}O values of site environmental waters. In general, the endocarp δ^{18}O value increases as the environmental water δ^{18}O value increases, over a 10‰ range in endocarp carbonate δ^{18}O values and a 11‰ range in environmental water δ^{18}O values.

In addressing the variability around the linear relationship, it must be noted that Figure 2 is subject to the limitations of the environmental water δ^{18}O value data set. These water data represent a best estimate of the environmental water in the vicinity of the sampled hackberry tree and may have actually been a water sample from several counties away. However, after careful analysis of site drainage areas, we consider these water data the best available for this comparison. The apparent fractionation between hackberry endocarp carbonate δ^{18}O value and environmental water δ^{18}O value ($\alpha_{cc\leftrightarrow water}$ ranges from 1.0380 to 1.0430 for the data shown in Fig. 2) is much greater than that reported for the precipitation of inorganic carbonate from water ($\alpha_{cc\leftrightarrow water} = 1.0286$ at 25°C; O'Neil and Clayton, 1964) and increases with decreasing precipitation δ^{18}O values. Included in the large data set are hackberries collected at two sites during the 1994 and 1995 growing season (Austin, Minnesota, and the Sterling, Colorado, site described above) which exhibited a 38 to 44‰ difference between extracted plant stem water δ^{18}O value and hackberry endocarp carbonate δ^{18}O value (Jahren, 1996). Therefore, these data represent field validation of the values of α derived using river water data as opposed to *in vivo* plant water. The difference between hackberry and inorganic fractionation factors is likely due to biological effects, such as evaporation of water in the fruit during carbonate development, but gives rise to a reasonably uniform α across the sites studied that is specific to hackberry endocarp carbonate. The fact that the slope of Fig. 2 is not equal to one is also most likely attributed to biological effects, as environmental conditions such as relative humidity partially control the status of transpiration in the hackberry fruit. Under this scenario, α should decrease with increasing humidity, which may be apparent.

The validity of the relationship between hackberry endocarp carbonate δ^{18}O value and environmental water δ^{18}O value is further reinforced when one notes the strong geographical tendency of the data (Table 6). Averaging isotopic values by state merely represents one possible way of dividing the data set into subclimates (e.g., see Kendall and Coplen, 2001, for an analysis based on averaging by hydrologic unit) but the expected regional differences associated with higher growing season average temperatures in the south versus north can be easily recognized in such an analysis.

Stable carbon isotope values were also measured in hackberry endocarp carbonate and the resulting values and means are presented in Tables 5 and 6, respectively. δ^{13}C values in hackberry endocarp carbonate do not correlate with site environmental water δ^{18}O value, endocarp carbonate δ^{18}O value, or site growing season temperature. Table 6 shows that the standard deviation in the δ^{13}C value of endocarp carbonate is larger

TABLE 6
Mean Hackberry Endocarp Carbonate δ^{18}O and δ^{13}C Values by State or Province

	Endocarp carbonate					
	$\delta^{18}O_{VSMOW}$			$\delta^{13}C_{VPDB}$		
State or province	Mean [‰]	*SD* [‰]	*n*	Mean [‰]	*SD* [‰]	*n*
Northern states or province						
Iowa	30.76	(0.51)	3	−13.92	(1.39)	3
Manitoba	30.28	—	1	−12.35	—	1
Minnesota	30.99	(1.12)	8	−12.16	(1.06)	7
Nebraska	31.61	(1.44)	18	−12.87	(1.46)	18
North Dakota	29.64	(0.50)	9	−11.83	(2.16)	9
South Dakota	31.05	(1.05)	11	−12.50	(1.37)	11
Southern states						
Arkansas	34.69	(0.85)	3	−10.47	(1.13)	3
Colorado	32.27	(0.28)	2	−9.58	—	1
Kansas	34.76	(1.22)	14	−11.46	(1.38)	14
Missouri	33.52	(0.13)	3	−11.72	(1.31)	3
New Mexico	36.70	(1.65)	10	−11.39	(1.19)	10
Oklahoma	35.69	(1.18)	13	−11.87	(1.48)	12
Texas	34.51	(0.65)	5	−12.77	(1.43)	5
Wyoming	35.35	—	1	—	—	0

than the standard deviation in δ^{18}O value of endocarp carbonate in the same sets of samples, and as previously mentioned, the within-site variability of endocarp carbonate δ^{13}C value is large (=3.0‰). Across the whole data set there is a range in endocarp carbonate δ^{13}C values of 9‰, and yet stable carbon isotope value in the endocarp carbonate does not appear to be at all determined by environmental waters or any obvious climate factor. The δ^{13}C values in hackberry endocarp carbonates in northern states are, on average 1‰ lower than δ^{13}C values in hackberry endocarp carbonates in southern states (Table 6), perhaps reflecting increased water stress in the more arid southern states (Condon *et al.*, 1993).

Carbon in hackberry carbonate is ultimately derived from the atmosphere as the plant precipitates aragonite from a mixture of respired and directly assimilated CO_2 into organic carbon compounds. With respect to direct assimilation, increased water stress results in reduced stomatal conductance, which in turn increases CO_2 assimilation efficiency and simultaneously reduces effective descrimination against $\delta^{13}CO_2$. In general, the δ^{13}C value of plant tissue increases as water stress increases. The carbon isotopic composition of plant-dissolved CO_2 is observed to vary greatly over small time spans due to the effect of changing stomatal conductance in response to various environmental stresses (Farquhar *et al.*, 1982).

Conversely, it may also reflect a difference in the species of hackberry represented in the data set. Based on the spatial distributions of *Celtis reticulata* and *C. occidentalis,* δ^{13}C values in hackberry endocarp carbonates from New Mexico, Texas, and Wyoming are clearly from *C. reticula.* δ^{13}C values from Oklahoma may be from either species depending on where the samples were obtained. *C. reticulata* is distributed west of a line running along a roughly southeast to northwest line through Oklahoma City. *Celtis occidentalis* occurs north of a line running through Oklahoma City. Examination of the values on a county-by-county basis clearly demonstrates the difference with highest values from counties where *C. reticulata* occurs and lowest values in areas where *C. occidentalis* is the only species occurring. The area where both occur show transitional values, but suggests primarily *C. reticulata.*

Another test of hackberry endocarp carbonate as a paleoclimate indicator is illustrated by Fig. 3, in which the δ^{18}O value of endocarp carbonate is plotted against the 30-yr average (1961–1990) of growing season daily maximum temperature (Spangler and Jenne, 1990) for the town or city nearest to the collection site. This relationship exhibits reasonable correlation ($R = 0.73$; $R^2 = 0.54$; $p = 0.0133$). Growing season (June–August) mean temperatures were used instead of mean annual temperature because the hackberry endocarp is completely constructed during the growing season and is therefore expected to most closely reflect environmental climate during endocarp formation.

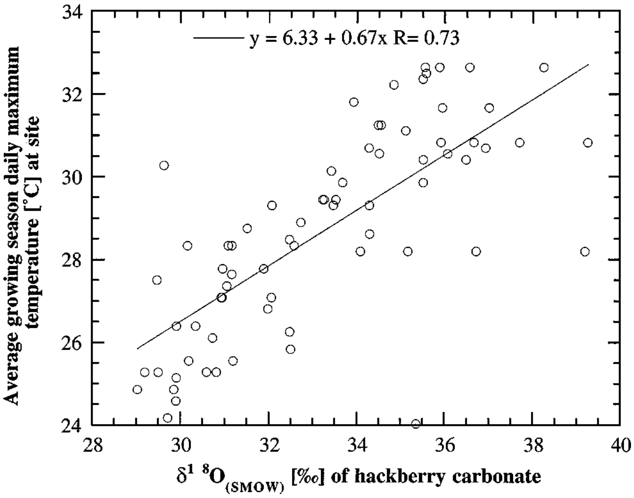

FIG. 3. Correlation between the δ^{18}O value of hackberry endocarp carbonate at a site and the 30-yr average growing season daily maximum temperature (°C) at a site.

Hackberry carbonate δ^{18}O value was not found to correlate with site mean annual temperature, relative humidity, mean annual precipitation, or growing season mean precipitation (all 30-yr average); all of the above comparisons exhibited $R < 0.10$ ($R^2 < 0.01$). The apparent correlation between the δ^{18}O value of endocarp carbonate and environmental temperature is lower than the correlation between the δ^{18}O value of endocarp carbonate and the δ^{18}O value of environmental water. There are several possible reasons. First, average growing season temperature may not reflect the actual temperature that occurred during the year of hackberry growth and collection. Second, the correlation between the growing season temperature of these sites and the δ^{18}O value of environmental differs from standardly observed relationships: on average, there is a correlation of δ^{18}O value in precipitation with mean average temparature (MAT) = ∼0.6‰/°C at the mid latitudes (Fricke and O'Neil, 1999). The correlation seen in this study between δ^{18}O value of environmental water with mean maximum growing season temperature = 1.0 ‰/°C ($R^2 = 0.65$). In addition, the environmental temperature and the δ^{18}O value of precipitation are not perfectly correlated for central North America (Amundson *et al.*, 1996; Yu *et al.*, 1997): in the Great Plains, the observed strong correlation between environmental temperature and the δ^{18}O value of precipitation may be a partial artifact of the fact that the relative total contribution of Gulf-derived precipitation varies in concert with mean annual temperature (Nativ and Riggio, 1990). Recent work has shown excellent correlation ($R^2 = 0.85$ for eastern sites) between the isotopic composition of U.S. river waters and that of 30-yr MAT (1961–1990; Kendall and Coplen, 2001). The correlation observed in this study allowed us to form the following predictive relationship: (average daily maximum growing season temperature [°C]) = 6.33 + 0.67 × δ^{18}O of endocarp carbonate). Confidence interval analysis of Fig. 3 suggests that average daily maximum growing season temperature can be predicted within ±0.82°C at a 95% confidence level from hackberry endocarp carbonate δ^{18}O values.

APPLICATION: FOSSIL HACKBERRY ENDOCARPS IN SOUTHERN NEVADA

Paleotemperture Analysis

Triplicate analysis of δ^{18}O value in Pintwater Cave fossil endocarps yielded the following: variability in δ^{18}O value between endocarps from the same depositional layer was determined for 17 separate layers and was large in many cases (8.1‰ = maximum difference). The average variability in δ^{18}O value between endocarps from the same depositional layer was 2.8‰, much larger than the average variability across one modern site (=1.2‰), suggesting that each depositional layer contains hackberry endocarps formed during varied yearly climate fluctuations. For this reason, the average values from all depositional layers refects the long-term average of endocarp carbonate δ^{18}O value (=36.01‰) for the period 9500 to 9000 ^{14}C yr B.P. By applying this value to the paleotemperature relationship shown in Fig. 3, we predict a mean growing season (June + July + August) temperature of 30.46 ± 0.8°C for the latter early Holocene.

This value carries considerable uncertainty associated with the fact that we have imported the carbonate temperature correlation from the Great Plains to the Great Basin. According to Kendall and Coplen (2001), there are significantly different slope and intercept of the δ^{18}O value of precipitation versus MAT relationship between the eastern and western United States due to differences in moisture sources and transport pathways. Taken at face value, the Great Plains correlation suggests a paleotemperature that correlates with additional climate information for the northern Mojave during the period 9500 to 9000 ^{14}C yr B.P. Although *Celtis reticulata* can occur over a broad range of habitats south and east of the Mojave Desert it is a facultative phreatophyte in much of the West including the Great Basin and the valleys of the Colorado, Columbia, and Snake Rivers (Spaulding, 1994; Fig. 1), restricted to springs and the banks of perennial streams. Climate parameters generated by Thompson *et al.* (2000b) for *Celtis reticulata* indicate that mean annual temperature in comparison to those for *C. occidentalis* are as much as 6°C warmer. *C. reticulata* occupies areas with significantly warmer summers, but slightly cooler winters. Rainfall for both species is centered during the summer months (the growing season, but *C. reticulata* occupies regions that have as much as 50% less rainfall than those where *C. occidentalis* occurs (Thompson *et al.*, 2000b).

In comparison, the 30-yr average (1961–1990) of growing season mean high and low temperatures (National Weather Service Web site) available at the nearest location (Indian Springs, Nevada) = 27.226°C; A second climate record from Indian Springs for the period from 1948 to 1964 indicates a growing season mean of the mean highs and lows = 27.29°C, implying >3°C warmer growing season temperatures during the latter early Holocene, relative to today. This suggests that the modern climate is actually about 3°C cooler than that of the period often referred to as the thermal maximum.

While the δ^{18}O values of the Pintwater hackberry carbonate may be suggestive of temperature change, we outline below what is likely a less ambiguous interpretation: changes in early Holocene circulation and precipitation patterns.

Paleocirculation Analysis

Application of the mean endocarp carbonate δ^{18}O value (=36.01‰) of hackberries from Pintwater Cave to the relationship shown in Fig. 2 yields a prediction of environmental water δ^{18}O value = −3.70‰. Friedman *et al.* (1992) reported δD_{SMOW} values in modern environmental water from southern Nevada to be dominated by winter precipitation and strongly controlled by elevation, ranging from ∼−55.2 at 0 m asl to −87.7‰ at 2500 m asl (−73.4‰ at 1268 m asl, the elevation of Pintwater Cave). Using the approximation $(\delta D + 9.7)/6.5 = \delta^{18}O$ (Amundson *et al.*, 1996), this suggests a modern environmental water δ^{18}O value = −9.80 for Pintwater Cave, substantially isotopically lighter than the environmental water inferred for the early Holocene by the composition of hackberry endocarps. Present southwestern monsoonal moisture is derived from the Gulf of California and elsewhere. The δ^{18}O value of this rain is variable but commonly falls between −2 to −6‰ (Arizona values within IAEA, 1981). In contrast, the present precipitation in southern Nevada is almost entirely derived from Pacific frontal systems, storms which pass over multiple moutain ranges and have experienced extensive rainout by the time they reach Nevada. The δ^{18}O value of this precipitation in Nevada is related to elevation but, as mentioned earlier, is probably close to −9‰.

We believe that the most probable explanation for the relatively ^{18}O-enriched Pintwater carbonates is that monsoonal rains during the early Holocene were far stronger than they are today. This hypothesis is supported by paleovegetational data: based on the incidence of grass and succulent components within packrat middens throughout the Mojave early Holocene record, Spaulding and Graumlich (1986) suggested that increased summer insolation during the early Holocene likely increased summer monsoonal flow, especially in intermontane troughs such as southern Nevada. In addition, accelerated erosion rates observed in alluvial fans of the Mojave Desert also suggest that monsoonal rains were becoming characteristic of the climate in the region during this time (Harvey *et al.*, 1999).

These results concur with at least three other lines of evidence indicating increased rainfall during the summer growing season in the latter early Holocene, relative to today. The first line of evidence involves organic mats (an accumulation of organic residues) dating primarily from 10,200 to 8600^{14}C yr B.P. (Quade *et al.*, 1998), which imply the generation and preservation of organic material under a regime of relatively higher rainfall. The second set of observations is of intermittent lakes that appeared in the Lake Mojave Basin between during the early Holocene. Brown *et al.* (1990) reported six lakes with intervening drying that occupied the Lake Mojave Basin between 11,500 and ∼8500^{14}C yr B.P. and interpreted this as evidence of moisture incursion. The third line of evidence involves the presence of the hackberry tree itself, which is at its greatest regional abundance between 9500 and 9000^{14}C yr B.P. (Spaulding, 1990; summarized in Spaulding, 1994: Table 11; Wigand and Rhode, in press). Fossil hackberry endocarps have been recovered from woodrat middens located on the xeric southeast slope of Little Skull Mountain (Wigand *et al.*, 1995: 9480 ± 370^{14}C yr B.P.), from middens in the Sheep Range north of Las Vegas (Spaulding, 1990: 9560 ± 180^{14}C yr B.P.), from the Sandy Valley (Spaulding, 1994: 9400 ± 90^{14}C yr B.P.), in addition to hundreds collected at Pintwater Cave; all of these areas are currently too dry to support hackberry trees. All three of these independent lines of evidence point to a wetter latter early Holocene, relative to today.

CONCLUSIONS

This study, with sampling area ranging from 35°–50°N latitude and from 90°–105°W longitude, tests a terrestrial paleoclimate indicator over a large land area. Similar studies have been conducted for other carbonate-bearing entities. Quade *et al.* (1995) sampled soil carbonate at many sites between 34°–40°S latitude and from 135°–150°E longitude. Abell (1985) examined the isotopic composition of modern freshwater gastropods collected from over 80 sites scattered over the African continent. Given the large land area and variety of climate environments represented by the hackberry collection used in this study, the correlation seen between δ^{18}O values in endocarp carbonate and δ^{18}O values of environmental water bodes well for the use of the fossil hackberry endocarp as a paleoclimate indicator. We have shown that, with some complexities, the δ^{18}O value of endocarp carbonate reflects the δ^{18}O value of the precipitation. Therefore, hackberry endocarps sampled across gradients of space and time provide a fairly unambiguous link to precipitation values—environmental data of great interest in deriving patterns of circulation and rainfall sources. In a limited geographical and temporal range, the δ^{18}O value of rainfall (and carbonate) is commonly correlated to MAT. However, across broader ranges of space and time, the δ^{18}O value of precipitation (and hence carbonate) may not be directly, or unambiguously, related to MAT without considerable supplementary information.

Fossil hackberry endocarps are a substrate of great utility in paleoclimate research. In addition to the stable isotope information they contain, they have been shown to precisely mimic the ^{14}C content of atmospheric CO_2 (Wang *et al.*, 1996) and, because they form over a very limited time span during the growing season, are ideal substrates for radiocarbon dating provided they have escaped alteration. The morphological and mineralogical integrity of a fossil endocarp can be determined through a combination of X-ray diffraction, percent-carbonate determination, and thin-section petrography which take advantage of the unique biomineralized morphology and chemistry of the hackberry endocarp (Jahren *et al.*, 1998).

The quantitative isotopic information provided by the δ^{18}O value of fossil hackberry endocarp carbonate provides an added robustness to our reconstruction of paleoclimates and allows for a new avenue of terrestrial paleoclimate reconstruction at a wealth of archaeological and geological sites in the Great Plains and the Great Basin of North America.

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