QUATERNARY RESEARCH **47,** 337–343 (1997)
ARTICLE NO. QR971894

Potential for ^{14}C Dating of Biogenic Carbonate in Hackberry (*Celtis*) Endocarps

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Received July 10, 1996

Hackberry endocarp (*Celtis* sp.) contains significant amounts (up to 70 wt%) of biogenic carbonate that is nearly pure aragonite ($CaCO_3$). Because of their high mineral content, hackberry endocarps are found abundantly in Tertiary and Quaternary sediments and are very common in many North American archaeological sites. We analyzed the ^{14}C content of different components of modern hackberries including the biogenic carbonate in hackberry endocarps collected at known times over the past century. The ^{14}C content of the endocarp carbonate accurately records the ^{14}C content of the atmosphere. ^{14}C dates of fossil endocarp carbonates compared favorably with dates obtained by other means at archaeological and geological sites ranging in age from the late Pleistocene through the early Holocene. We therefore suggest that hackberry endocarp is a suitable substrate for ^{14}C dating provided that its morphological and mineralogical integrity is preserved.

INTRODUCTION

Age-determination of archaeological and many late Quaternary deposits is dependent upon the availability of suitable carbon-containing substrates for radiocarbon dating. A variety of carbon-bearing compounds has been used for dating over the years, each having various attributes and limitations (Sellstede *et al.*, 1966; Haynes, 1968, 1980; Amundson *et al.*, 1994; Wang *et al.*, 1994, 1996). Many of the most common carbon-bearing materials (e.g., bone collagen, soil organic matter, pedogenic carbonate) suffer from two common problems: (1) susceptibility to carbon exchange and adsorption, and/or (2) formation over a long and/or indefinite time span. Here we discuss the morphological, mineralogical, and chemical composition of modern hackberry endocarps, and suggest that they can provide a less ambiguous method of ^{14}C dating than some of the other commonly used substrates.

Hackberries are the annual fruits produced by *Celtis* sp. trees; they are a common, well-preserved plant remnant in many archaeological and geological deposits (Segal, 1966; Thomasson, 1985; Yanovsky *et al.*, 1932; Haffner *et al.*, 1991; Houle and Bouchard, 1990). The hard layer surrounding the endosperm, known as the ''endocarp'' (Fig. 1) has a peculiar chemical composition, which was first reported in 1932 by Yanovsky *et al.* An interesting feature of the endocarp is its high mineral content, which contributes to preservation in geological and archaeological deposits. The primary mineralogical component of hackberry endocarp is carbonate, but smaller amounts of opal and organic matter are present (Yanovsky *et al.*, 1932; Jahren *et al.*, 1994). Both modern (Table 1) and fossil endocarps have been reported to be composed of up to 70 wt% calcium carbonate, initially in the form of aragonite (Jahren, 1996). The high carbonate content raises the possibility that hackberry endocarp is a stable substrate for isotopic analyses and paleoclimate reconstruction.

We report here on ^{14}C systematics of modern hackberry endocarp carbonate and examine its potential as a ^{14}C dating substrate. This research complements our ongoing work on the stable carbon and oxygen isotope systematics of modern hackberries (Jahren *et al.*, 1994, 1995). Through multiple isotopic analyses, we hope to establish hackberry endocarps as self-contained terrestrial paleoclimate and chronological indicators.

METHODS

To determine if hackberry endocarp carbonate reflects the ^{14}C content of contemporaneous atmospheric CO_2, we analyzed 36 samples from the University of California Herbaria collection of *Celtis.* These samples are of known age (1889 to 1993) and from various localities in Minnesota, New Mexico, California, Arizona, Virginia, Oregon, Wyoming, Texas, Missouri, Georgia, Indiana, Oklahoma, Louisiana, Kansas, North Dakota, and South Carolina. We also analyzed different components of modern *Celtis* collected in 1993: endocarp carbonate, endocarp organic matter, external fruit, and small stems. In addition to isotopic analysis, morphological and mineralogical studies were performed on modern and selected fossil endocarps. Fossil hackberry endocarps from

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0033-5894/97 $25.00

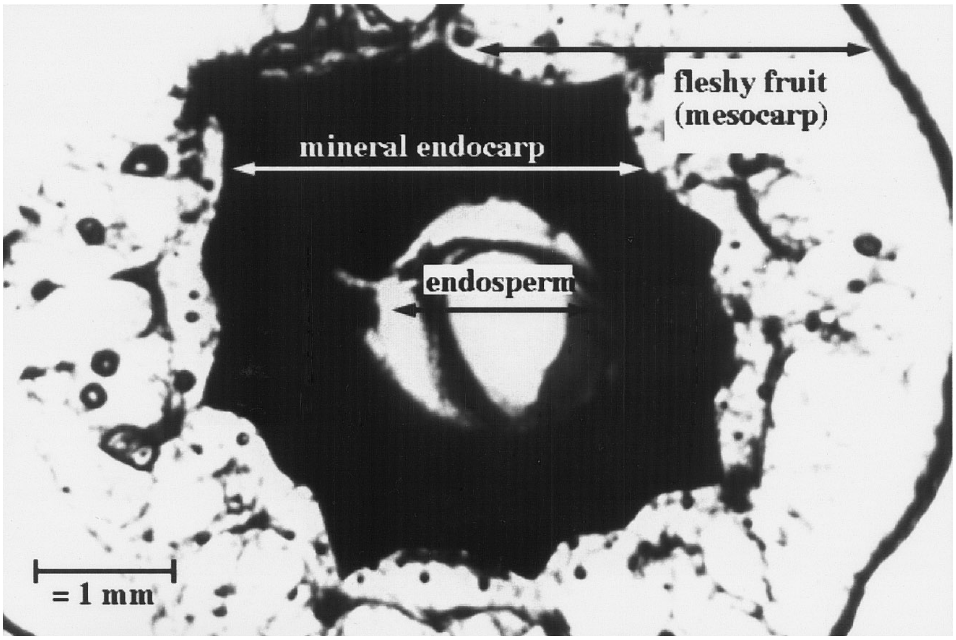

FIG. 1. Photograph of modern hackberry in cross section.

several archaeological and geological sites were also studied and results were compared to ^{14}C dates obtained by analysis of other materials, in order to determine the reliability of hackberry endocarp carbonate for radiocarbon dating.

Modern hackberry endocarps (and enclosed endosperm) were manually separated from external fruit (the mesocarp), soaked in bleach for 2 hr to help remove the remaining external organic matter on the pit surface, rinsed, and dried. The endocarp was then separated manually from the endosperm and ground into powder. Fossil endocarps (which lacked endosperm and mesocarp) were also treated with bleach for 2 hr, rinsed, dried, and ground into powder. The powder was baked under vacuum at 425°C for 1 hr to carbonize any organic carbon and then reacted with 100% phosphoric acid to release CO_2. Endocarp organic matter was isolated by soaking the endocarp in 6 *M* HCl to remove $CaCO_3$, rinsed, and dried. Other modern plant components such as stem and fruit were treated with 10% HCl overnight and then rinsed and dried. CO_2 was produced by combustion of the sample with CuO and silver foil at 875°C (Minagawa *et al.*, 1984). The CO_2 was then purified cryogenically and reduced to graphite with H_2 over Co (Vogel *et al.*, 1989). The $^{14}C/^{13}C$ ratio of the graphite was measured on an accelerator mass spectrometer (AMS) at the Center for Accelerator Mass Spectrometry of Lawrence Livermore National Laboratory. The ^{14}C data are reported as $\Delta^{14}C = [A_{SN}/A_{ABS} - 1] \times 1000‰$, where A_{SN} is the specific activity of a sample (which is proportional to the $^{14}C/C$ ratio in the sample) normalized to $\delta^{13}C = -25‰$, and A_{ABS} is the absolute international standard activity which is equal to 0.95 times the specific activity of the international standard (NBS oxalic acid) in 1950 normalized to $\delta^{13}C = -19‰$ (Stuiver and Polach, 1977; Donahue *et al.*, 1990). In addition, thin sections of both modern and fossil endocarps were examined under a petrographic microscope.

RESULTS AND DISCUSSION

Composition of Hackberry Endocarp

Modern hackberry (*Celtis* sp.), a small tree that annually (or sometimes less frequently) produces berries known as ''hackberries,'' is widely distributed from eastern North America to semiarid regions of the western United States (Lanner, 1983; Little, 1976; Stephens, 1973). A single hackberry consists of a dark-purple, fleshy fruit (mesocarp) which covers the mineral endocarp. The endosperm is encased by the endocarp (Fig. 1). Examination of the endocarp in thin section reveals a ''honey-comb'' morphology in the mineral fraction (Fig. 2a). X-ray diffraction patterns reveal that the endocarps contain aragonite and, commonly, opal. Sequential dissolution experiments (involving hydrogen peroxide, followed by hydrochloric acid and hydrofluoric acid) revealed that endocarps consist of an organic framework surrounded by biogenic carbonate (aragonite) and opal (Jahren, 1996). The carbonate content ranges from 41 to 70 wt% in the endocarp. The high concentration of biogenic carbonate makes the endocarp a unique plant component for ^{14}C dating.

^{14}C Content of Modern Hackberries

The ^{14}C analyses of samples collected in 1993 (Table 1) show that endocarp organic carbon, endocarp carbonate,

TABLE 1
^{14}C Content of Modern Hackberries (*Celtis* sp.)

Sample No.	Sample date	$\Delta^{14}C$	$\Delta^{14}C$ (age-corrected)[a]	$CaCO_3$ (wt%)	Sample type	Notes	CAMS No.
RA-C0241	1993	113.6 ± 6.4	113.9		Small stem	Minnesota	12734
RA-C0244	1993	112.5 ± 7.7	112.8		Mesocarp	Minnesota	12735
RA-C0247	1993	104.1 ± 9.1	104.4		Small stem	Minnesota	12736
RA-C0249	1993	107.6 ± 7.1	107.9		Endocarp O.M.[b]	Minnesota	11868
RA-C0250	1993	109.6 ± 11.6	109.9		Endocarp O.M.	Minnesota	11873
RA-C0259	1993	115.0 ± 7.1	115.3	64.9	Endocarp CC.[c]	Minnesota	11864
RA-C0260	1993	111.9 ± 7.1	112.2	70.2	Endocarp CC.	Minnesota	11865
RA-C0258	1992	120.3 ± 7.2	120.7	66.0	Endocarp CC.	Minnesota	11863
RA-C0259	7-1993	115 ± 7.1	115.3	64.9	Endocarp CC.	Minnesota	11864
RA-C0260	8-26-1993	111.9 ± 7.1	112.2	70.2	Endocarp CC.	Minnesota	11865
RA-C0261	1983	247.4 ± 8.9	249.2	65.5	Endocarp CC.	Minnesota	11866
RA-C0264	7-2-1889	−4.3 ± 6.8	8.5	57.8	Endocarp CC.	UC5862, Arizona	12743
RA-C0265	8-30-1897	−10.8 ± 6.5	1.0	50.5	Endocarp CC	UC141978, New Mexico	12744
RA-C0266	8-1910	−29.5 ± 6.3	−19.5	64.9	Endocarp CC.	UC148612, California	12745
RA-C0267	8-2-1912	−25.3 ± 6.4	−15.5	40.5	Endocarp CC.	UC202461, Virginia	12746
RA-C0268	9-14-1925	−24.2 ± 6.3	−15.9	69.0	Endocarp CC.	UC848411, Missouri	12747
RA-C0269	7-25-1926	−18.2 ± 6.3	−10	69.7	Endocarp CC.	UC M083152, Oregon	12748
RA-C0270	2-22-1930	−28.7 ± 6.3	−21	62.1	Endocarp CC.	UC500701, Wyoming	12749
RA-C0271	7-5-1931	−28.1 ± 5.4	−20.5		Endocarp CC.	UC471178, Texas	12751
RA-C0272	8-11-1937	−15.9 ± 6.7	−9		Endocarp CC.	UC738007, Missouri	12752
RA-C0273	8-28-1940	−32.6 ± 7.1	−26.1		Endocarp CC.	UC724799, Georgia	12753
RA-C0274	8-10-1943	−32.4 ± 6.6	−26.3		Endocarp CC.	UC990769, Texas	12754
RA-C0275	8-8-1948	−26.8 ± 7.5	−21.3		Endocarp CC.	UC914435, California	12755
RA-C0276	9-8-1951	−52.8 ± 6.3	−47.7		Endocarp CC.	M056513, Indiana	12756
RA-C0277	6-23-1955	−9.4 ± 9.1	−4.6	61.1	Endocarp CC.	UCM086341, Oklahoma	12761
RA-C0278	6-8-1956	1.9 ± 6.9	6.6		Endocarp CC.	UCM246531, Louisiana	12757
RA-C0279	7-11-1957	120.4 ± 8.2	125.6	63.7	Endocarp CC.	UC1364682, California	12762
RA-C0281	6-1-1959	202.5 ± 8.7	207.7	65.1	Endocarp CC.	UCM227592, Texas	12764
RA-C0280	10-1-1960	162.9 ± 8.5	167.8	66.6	Endocarp CC.	UCM211831, Kansas	12763
RA-C0282	7-26-1961	199.9 ± 8.8	204.8	67.5	Endocarp CC.	UCM211831, North Dakota	12765
RA-C0283	6-14-1963	445 ± 8.1	450.6	65.6	Endocarp CC.	UC1351236, Texas	12741
RA-C0284	7-17-1963	512.9 ± 10.7	518.8	58.4	Endocarp CC.	UC1339525, Texas	12742
RA-C0285	7-14-1964	720 ± 11.8	726.5	57.1	Endocarp CC.	UC1339672, Louisiana	12758
RA-C0286	9-28-1973	458.5 ± 10	462.4	69.1	Endocarp CC.	UC1437775, South Carolina	12759
RA-C0287	7-23-1981	251.1 ± 10	253.2	62.2	Endocarp CC.	UC1489582, Texas	12760
RA-C0288	8-30-1986	188.2 ± 7.7	189.5	65.5	Endocarp CC.	UC1588721, Missouri	12750

[a] ^{14}C data corrected for decay between year of collection and 1950.
[b] Organic C in hackberry endocarp.
[c] Biogenic carbonate in hackberry endocarp.

mesocarp, and small stems, which are all the products of one growing season, have essentially the same ^{14}C content. This indicates that these plant components all derive their carbon through the fixed atmospheric CO_2, and do not incorporate remobilized carbon that was photosynthetically fixed during past years.

The ^{14}C content of carbonate in endocarps (Fig. 3) collected over the past 120 yr parallels the reported ^{14}C variations of the atmosphere during that time span (Suess, 1955; Stuiver, 1965; Manning *et al.*, 1990; Levin *et al.*, 1985), providing more definitive proof that the ^{14}C content of endocarp carbonate accurately reflects the ^{14}C content of atmospheric CO_2 for a given growing season. The following unique features of the atmospheric $^{14}CO_2$ trends are reflected by the endocarps: (1) the decreasing $\Delta^{14}C$ values from late 19th century until 1955 as a result of the input of ''dead'' CO_2 by combustion of fossil fuel (the ''Suess Effect'') (Suess, 1955; Damon *et al.*, 1978); (2) the very positive $\Delta^{14}C$ values after 1955 due to the production of ''bomb'' $^{14}CO_2$ by nuclear weapons testing; (3) the general decline, after the test-ban agreement in 1963, of $\Delta^{14}C$ values which continues toward the prebomb levels. These results suggest that, as long as the endocarps are not altered by diagenesis, the biogenic carbonate in hackberry endocarp should be desirable for radiocarbon dating because it reflects atmospheric $\Delta^{14}C$ values of a single growing season.

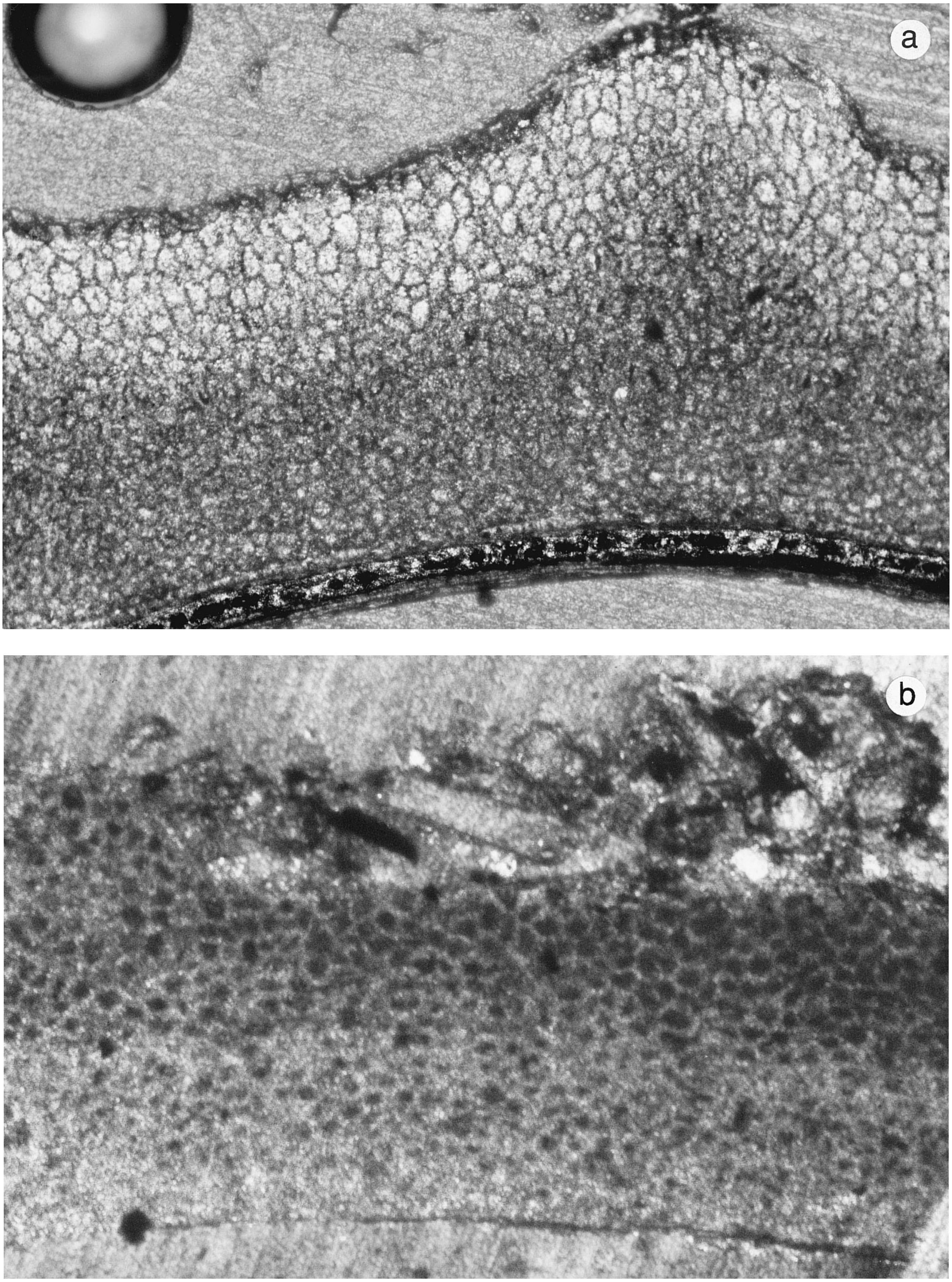

FIG. 2. Petrographic thin section photos of (a) modern endocarp and (b) fossil endocarp (short axis = 0.9 mm).

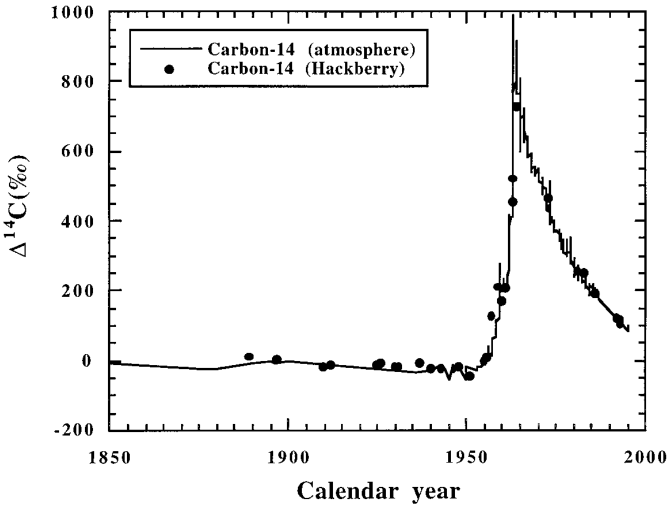

FIG. 3. Comparison of age-corrected $\Delta^{14}C$ values (corrected for decay between year of collection and 1950) of hackberry endocarp carbonate with ^{14}C levels of the atmosphere (Suess, 1955; Stuiver, 1965; Levin *et al.*, 1985; Manning *et al.*, 1990; Y. Wang *et al.*, unpublished data).

^{14}C Content of Fossil Hackberry Endocarps

In order to test the reliability of endocarp carbonate for ^{14}C dating, we selected fossil endocarps from dated archaeological and geological sites in central North America for ^{14}C analysis (Table 2). Samples RA-C0289, RA-C0416, RA-C0418, RA-CO420, RA-C0436, and RA-C0437 yielded X-ray powder diffraction patterns typical of aragonite; no calcite was detected. The persistence of aragonite mineralogy is likely a crucial indication of minimal chemical alteration. It has been observed that aragonite readily converts to calcite upon even minimal alteration, which might result in an opening of the chemical system and allow exchange of isotopes, especially when a fluid phase was involved (Folk and Assereto, 1976). X-ray powder diffraction method offers a quick and easy way of identifying calcite and aragonite. Because calcite and aragonite have very strong and distinct reflections, the sensitivity of the X-ray diffraction method in detecting the presence of calcite in aragonite matrix is below the 1% level (Zussman, 1967). The presence of 1% modern calcite in an 11,000 yr B.P. sample would cause a dating error of ~240 yr. Thin section photos of the fossil endocarp (Fig. 2b) also reveal the same morphological structure observed in modern endocarps, further suggesting insignificant alteration by diagenesis. The other samples dated had insufficient material for X-ray analysis or petrographic examination.

The Burnham site is located in western Woods County of northwestern Oklahoma, and has yielded important late Pleistocene human artifacts (Wyckoff *et al.*, 1991, 1994). Initially, this site was thought to contain pre-Clovis artifacts (Wyckoff *et al.*, 1991), but recent excavations determined that these were probably Folsom-age artifacts redeposited during early Holocene gullying (Wyckoff *et al.*, 1994). We analyzed six hackberry endocarps from this site (Table 2). Samples RA-C0353 and RA-C0289 are from a rodent burrow. RA-C0353 yielded a ^{14}C date of 40,130 ± 1280 yr B.P. which is similar to a previous date (37,790 ± 680 yr B.P.) for charcoal fragments from the same burrow. RA-C0289 yielded a ^{14}C age of 30 ± 60 yr B.P. which clearly indicates recent emplacement. Possibly the ^{14}C content of the endocarp carbonate had been altered and contaminated with modern carbon after burial. However, the endocarp was in excellent condition and looked fresher than other specimens in the collection. Inasmuch as hackberry trees grow in the arroyo near the site today, it is likely that this is a 20th-century endocarp that was reworked into the deposits by rodents. Other fossil endocarp samples are from a layer of alluvium that contains human artifacts. Radiocarbon dating of gastropod shells and charcoal fragments from the alluvial deposits yielded a variety of dates between 40,900 and 10,210 yr

TABLE 2

^{14}C Ages of Biogenic Carbonate in Fossil Hackberry Endocarps from Three Locations in North America

Sample No.	^{14}C age (yr B.P.)	Museum No.	Site	X-ray patterns	CAMS No.
RA-C0291	37,590 ± 820	34WO73/94-3	Burnham, OK	nd[a]	13755
RA-C0353	40,130 ± 1280	34WO73/94-5	Burnham, OK	nd	15979
RA-C0354	35,680 ± 710	34WO73/94-6	Burnham, OK	nd	15980
RA-C0352	22,670 ± 330	WO73/94-10	Burnham, OK	nd	15978
RA-C0419	40,190 ± 870	34WO73/94-7	Burnham, OK	nd	17602
RA-C0289	30 ± 60	34WO73/94-1	Burnham, OK	Aragonite	13773
RA-C0416	7880 ± 50		Homestead, UT	Aragonite	17599
RA-C0418	8110 ± 60		Gillespie Hill, UT	Aragonite	17601
RA-C0420	8240 ± 60		Devils Gate 1A	Aragonite	17603
RA-C0436	8900 ± 60	I77-20-4	Hudson-Meng, NE	Aragonite	18893
RA-C0437	8970 ± 60	I77-20-2	Hudson-Meng, NE	Aragonite	18894

[a] nd, not determined.

B.P., with an average age of 33,000 yr B.P. (B. Carter, personal commun. 1996). Our ^{14}C measurements of endocarp carbonate yielded ^{14}C ages ranging from 22,670 ± 330 to 40,190 ± 870 yr B.P., which are comparable to the estimated ages based on ^{14}C dates on the other carbon-bearing materials within the same sedimentary sequence. This suggests that hackberry endocarp carbonate is very resistant to diagenetic alteration under the geochemical conditions at the site.

Sample RA-C0416 is from Homestead Cave, a small cavern in the Lakeside Mountains of northwestern Utah (near Great Salt Lake). The shelter has an extended late Pleistocene/Holocene sedimentary record, including numerous animal bones and hackberry endocarps. The stratum containing RA-C0416 has one conventional ^{14}C date of 8840 ± 240 yr B.P. for the organic fraction of a mixed sample of hackberry remains from the entire stratum, which is about 17 cm thick (D. Rhode, personal commun. 1996). The measured ^{14}C age of endocarp carbonate from this sample is 7880 ± 50 yr B.P., which is slightly younger than the date obtained for the mixed sample. The hackberry ^{14}C date is likely comparable in that the prior date was for a composite sample from a 17-cm-thick stratum that probably represents hundreds to thousands of years of depositional history. AMS ^{14}C dating of individual endocarp carbonate from different stratigraphic levels could provide better chronological control than a mixed sample from an entire stratum. In addition, the organic fraction of hackberries is likely to be far more susceptible to contamination than the inorganic portion by a variety of organic compounds in the sediments.

Samples RA-C0418 and RA-C0420 are also from Utah. Sample RA-C0418 is from a packrat midden in Gillespie Hill near Homestead Cave. The ^{14}C measurement on the endocarp carbonate gave a ^{14}C age of 8110 ± 60 yr B.P. A conventional radiocarbon date for a mixed sample of the organic fraction of *Celtis* endocarps from the same packrat midden yielded a date of 6670 ± 60 yr B.P. (D. Rhode, personal commun. 1996), which is younger than our ^{14}C date for the endocarp carbonate. Sample RA-C0420 is from another packrat midden (Devils Gate 1A) located in the Stansburry Mountains south of Great Salt Lake. The ^{14}C age of associated limber pine needles is 12,370 ± 60 yr B.P. (Rhode and Madsen, 1995). The ^{14}C measurement of a hackberry endocarp carbonate yielded a ^{14}C age of 8240 ± 60 yr B.P., which is ~4000 yr younger than the age of the needles. One possible explanation for the discrepancies is that these different materials indeed have different ages, which is expectable in packrat middens. It is not possible to resolve the reasons for these age discrepancies. However, we believe that the ages we measured reflect unaltered samples because X-ray analysis (Table 2) of the samples suggests that they are still aragonite and are little altered by diagenesis.

The Hudson-Meng site in Sioux County, Nebraska, is the largest Paleoindian-period bison bone bed in North America. Important artifacts, including Alberta points and other stone tools, have been found associated with the bone bed. Age estimates for the site have been obtained from ^{14}C dating of charcoal from numerous locations within a paleosol encasing the bone bed. The charcoal ^{14}C ages range from 10,540 ± 70 to 9380 ± 70 yr B.P. (L. C. Todd, personal commun., 1995). Hackberry endocarps were obtained from the same paleosol about 5 cm above a charcoal sample with a ^{14}C age of 9600 ± 60 yr B.P. Two endocarps (RA-C0436 and RA-C0437) yielded similar ^{14}C ages of 8900 ± 60 and 8970 ± 60 yr B.P., which agree well with the constraining charcoal ^{14}C age of 9600 yr B.P. at lower stratigraphic level within the paleosol.

CONCLUSIONS

Radiocarbon analysis of different components of modern hackberries (i.e., endocarp carbonate, endocarp organic carbon, mesocarp, and small stems) suggests that hackberry carbonate derives its carbon from contemporary atmospheric CO_2 through photosynthesis, and not from other carbon sources within the tree. Analysis of archived hackberry endocarps from the past century indicates that the ^{14}C content of hackberry endocarp carbonate accurately reflects the ^{14}C variations of the atmosphere. Carbonate of fossil hackberry endocarps yielded ^{14}C dates that are consistent with ^{14}C dates obtained from other materials within the same site or the same stratigraphic unit (given uncertainties in the ages of these other materials).

Radiocarbon dating of endocarp carbonate should be a useful tool not only for archaeologists but for Quaternary geologists and soil scientists. It has the advantage of being very straightforward compared with ^{14}C dating of many other carbonates (e.g., soil carbonate) (Wang *et al.*, 1993, 1994, 1996; Amundson *et al.*, 1994), because (1) it forms over a single growing season and (2) the initial ^{14}C content of endocarp carbonate is the same as that of the contemporaneous atmospheric CO_2. In addition, a series of tests can be conducted to check the mineralogical and morphological integrity of a fossil endocarp before ^{14}C dating, whereas most organic materials lack similar criteria. Furthermore, the oxygen isotopic composition of hackberry endocarps has been reported to be a potential indicator of rainfall composition (Jahren *et al.*, 1994, 1995). Application of ^{14}C dating to the same substrate can provide important age control for many terrestrial paleoclimate records on millennial to decadal scales. However, as with other ^{14}C dating methods, caution must be exercised when collecting fossil hackberry endocarps in the field and with data interpretation because hackberry endocarps, like many other fossils, can be eroded, transported, and redeposited in sediments.

ACKNOWLEDGMENTS

We thank Barbara Ertter (University of California, Berkeley Herbaria), Don Wyckoff (Oklahoma Archaeological Survey), Larry Todd (Colorado

State University), and David Rhode (Desert Research Institute) for kindly offering hackberry specimens analyzed in this study and for their helpful comments. We also thank Michaele Kashgarian and John Southon (Lawrence Livermore National Laboratory) for assistance in ^{14}C measurements and helpful discussions. We thank C. E. Jahren for his assistance in collecting 1993 modern hackberry materials. This research was funded by NSF Grant 9219519.

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