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Micro-flotation removal of coal contaminants from archaeological radiocarbon samples from Chaco Canyon, New Mexico, USA

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ABSTRACT

Micro-flotation, a specific gravity separation technique, was successfully used to remove coal contaminants from radiocarbon samples obtained from profiles, unit excavations, and solid sediment cores in Chaco Canyon, New Mexico, USA. Coal from the Cretaceous Menefee Formation occurs throughout Chaco Canyon in aeolian, alluvial, colluvial, and anthropogenic sediments. The Menefee Formation contains carbonized broadleaf angiosperm and gymnosperm plants and, as such, paleobotanical analysis was not effective in the identification and removal of coal contaminants. The effectiveness of micro-flotation as a pretreatment procedure was evaluated by: i) comparing AMS radiocarbon ages on processed and unprocessed samples from the same archaeological contexts; ii) comparing a processed sample of carbonized hardwood charcoal with a sample of uncarbonized hardwood from the same archaeological context; and iii) comparing radiocarbon ages on a split sample of processed bulk carbon. The comparisons confirmed the effectiveness of micro-flotation in processing samples for radiocarbon dating in Chaco Canyon and would be applicable for similar locations elsewhere in the world.

1. Introduction

Coal occurs in sedimentary and occasionally in low-grade metamorphic rocks. Coals are composed of macerals (inertinite, liptinite, vitrinite), which are predominantly plant matter, tissues, spores, pollen, resins, and humus that were inundated, buried and compressed (Thomas, 2012). In time, macerals are carbonized with increasing pressure, temperature, and acidity. As these processes increase, the plant remains are transformed into different grades of coal—progressively peat, lignite (also known as brown coal), sub-bituminous, bituminous, anthracite, and graphite (Stach et al., 1975).

Coal deposits occur around the world and date from the Proterozoic (~2 Ga) to the Pliocene (~2 Ma) (Tyler et al., 1957). Consequently, the original atmospheric radioisotope ^{14}C is absent in coal since its half-life is ~5730 years. While trace amounts of ^{14}C occur in coal, they are attributed to cluster decay from radiogenic isotopes, which create ^{14}C from ^{12}C (Beck, 2011). Additionally, bacteria (*Diplococcus* sp.) and fungus (*Trametes versicolor*) metabolize and degrade coal (Potter, 1908), which can artificially enrich the ^{14}C content of coal (Campbell et al., 1988).

Chemically, coal is composed of C, H, N, S, O, and about 140 different hyrdrocarbons such as anthrocene, benzene, ethylbenzene, n-hexane, 2-hexene, methyl ethylbenzene, naphthalene, propylbenzene, and toluene (Tankersley and Munson, 1992). The quantities of these chemicals vary from one grade of coal to the next and even from one lump of coal to the next (Tankersley and Munson, 1992). Isotopically, the composition of coal includes ^{12}C, ^{13}C, and trace amounts of post-depositional ^{14}C.

Radiocarbon ages are calculated by determining the ratio of ^{12}C to ^{14}C in an archaeological or geological sample. Consequently, a natural or anthropogenic admixture of coal would likely increase the quantity of ^{12}C and result in an age determination that was older than expected (Tankersley et al., 1987). The geologic distribution of coal is pan-global and poses a significant contamination threat to accurate radiocarbon

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Table 1
Specific gravity of wood charcoal and different grades of coal.

Sample	Specific gravity[a]
Wood charcoal	0.40
Peat	1.24
Lignite (Jet)	1.29
Sub-bituminous coal	1.30
Bituminous coal	1.32
Anthracite	1.47
Graphite	2.25

[a] Averages after Wood et al. (1983).

dating in many regions. Coal can occur in samples collected for radiocarbon dating as the result of weathering, erosion, deposition processes, groundwater transport, and human introduction through its procurement as a fuel source or a manufacturing raw material (Tankersley, 1984; Tankersley et al., 1987).

Archaeologically, the oldest use of coal is uncertain. Jet was used in carvings in the Shenyang region of China ~4000 BCE and as a fuel source by ~1000 BCE (Golas and Needham, 1999). In eastern North America, bituminous coal was used to manufacture beads as early as the Late Archaic cultural period (e.g., the DuPont site, 33Ha11, ~3000 BCE; Dalby, 2007). By ~1000 CE, coal, coal ash, and "clinkers" occur in hearth features of Late Woodland and subsequent Fort Ancient and Mississippian sites suggesting that it was used as a fuel source (Tankersley et al., 1987).

Coal can be indistinguishable from more recent carbonized plant remains in archaeological and geological radiocarbon samples, even under high magnification scanning electron microscopy, because it is composed of carbonized plant matter. While prolonged acid-base-acid sample pretreatment can eliminate contamination from coal humates, it is an ineffective procedure for the removal of particulate and larger coal particles (Tankersley et al., 1987). Paleobotanical analysis can be used to successfully identify distinctive residues of fossil algae and spores if radiocarbon sample contaminants are from Proterozoic and Paleozoic coals, respectively (Tankersley et al., 1987; Tankersley and Munson, 1992). However, contamination of radiocarbon samples from Cretaceous and Tertiary coals pose a greater challenge because of the presence of angiosperm and conifer plant fossils that may overlap with archaeological plant assemblages. In these situations, density or specific gravity—the ratio of the mass of a material to the mass of a reference material for the same given volume—can be used to separate coal from particles of more recent carbonized plant remains of a differing specific gravity (Table 1).

Flotation is the most common specific gravity separation technique used by archaeologists and geologists to recover botanical remains. Samples are placed in water, which is gently circulated, separating carbonized plant remains from sediments with a higher specific gravity onto a filter by using a fine mesh screen. Radiocarbon sample sizes (≥50 g for conventional β-decay and ≥5 mg for accelerator mass spectrometry [AMS]) are far less than those obtained for archaeobotanical and paleobotanical analyses so flotation separation systems need to be greatly scaled down to what we call micro-flotation to recover all possible usable samples.

2. Methods

2.1. Chaco Canyon

We collected a suite of radiocarbon samples from Chaco Canyon, located in the central San Juan Basin of northwest New Mexico, to determine whether micro-flotation can be successfully used to separate archaeological and geological carbonized plant remains from coal contaminants (Fig. 1). Chaco Canyon is formed in the Cretaceous Cliffhouse

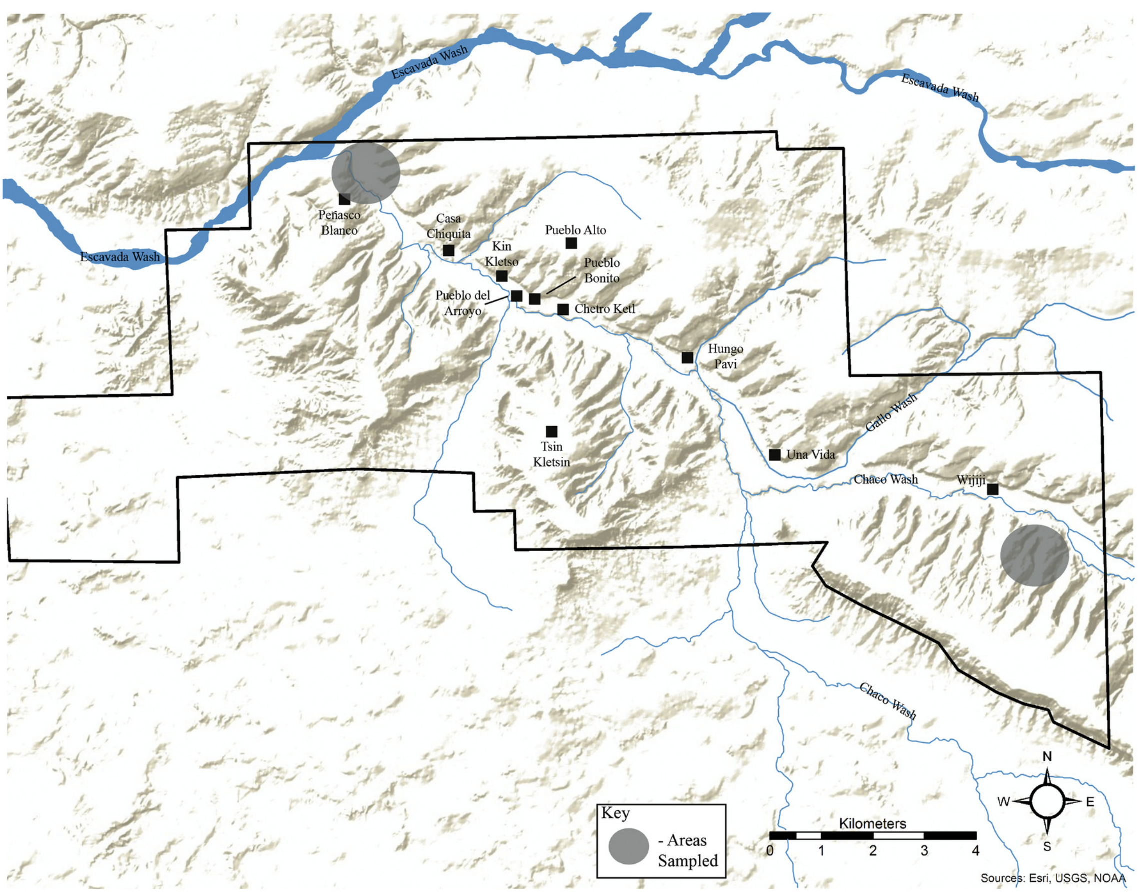

Fig. 1. Geographic location of the study area.

Sandstone and Menefee formations, which are dated to ~80 Ma (Spencer et al., 2005). The Menefee Formation is composed of siltstone, mudstone, carbonaceous shale, jet, and sub-bituminous coal. The bituminous coal is composed of carbonized trunks, logs, wood debris, and leaves from broad leaf angiosperms and gymnosperm trees (Miller, 1984). Coal derived from the Menefee Formation is ubiquitous in the Quaternary age aeolian, alluvial, colluvial sediments in Chaco Canyon (Fig. 2).

Archaeologically, Chaco Canyon is best known for the massive, complex, multistory masonry buildings known as Great Houses built by Ancestral Puebloans during the Pueblo II cultural period (~850 CE to 1150) in a high elevation dry land setting (Lekson, 2006; Vivian, 1990; Vivian and Hilpert, 2012). In the Jeddito Valley of Arizona, some 200 km west of Chaco Canyon, Ancestral Puebloans used coal as a raw material for carving and as a source of fuel during the Pueblo III (~1150 CE to 1350), Pueblo IV (~1350 CE to 1600), and Pueblo V (~1600 CE to present) cultural periods (Hack, 1942; Ward 1976). Since the beginning of Pueblo III time, coal was mined from mesa outcroppings, requiring significant economic and social coordination (Brew, 1937; Colton, 1936; Hack, 1942).

Coal ash and clinkers have been recovered from firepits in pueblos and kivas in the Jeddito Valley and was used to fire pottery during the Pueblo IV cultural period (Hack, 1942). The pre-Hispanic use of Menefee Formation coal by Ancestral Puebloans is not surprising given that it co-occurs with high quality kaolinite shale, which was used in manufacturing pottery. Coal firing of ceramics became particularly prevalent at Hopi villages beginning in the 14th century and, likely due to its hardness, durability, and distinctive yellow hue, has been found at archaeological sites throughout the American Southwest (Adams, 2002; Hays-Gilpin, 1996). Ancestral Puebloans are considered among the world's first cultures to use coal for firing pottery (Brew, 1937; Colton, 1936; Hack, 1942; Turner and Lofgren, 1966).

In Chaco Canyon, frog ornaments carved out of jet from the Menefee Formation were found at Pueblo Bonito and other Great Houses (Reed, 2004). They are similar to the frog ornaments, which occur at the freshwater springs discharging at the Menefee Formation coal-shale contacts. To the Pueblo people, frogs are symbols of both water and fertility. The Zuni also use them as symbols of *Tak'yakwe*, the frog clan (Plog et al., 2012). Carving of jet results in contamination of organic matter that might be used for radiocarbon dating.

2.2. Field methods

Over a three-year period (2013, 2014 and 2015), AMS radiocarbon samples (i.e., carbonized plant remains) were collected from profile excavations, test unit excavations and solid sediment cores placed at the opposite ends of Chaco Canyon to increase the chronometric resolution of the Quaternary stratigraphy, and to better understand Holocene hydrology and its relationship to human occupation (Figs. 3 and 4). A portable JMC hand-operated soil sampler was used to extract 15 cores in the "Dune Dam" area near the juncture with Escavada Wash and 12 cores in the Roberts Great House area well upstream along the Chaco Wash in the Canyon. Three cm-diameter and 1 m-long stainless steel core-tubes were pounded into the ground using a 5.0 kg slide-hammer to a depth of 3.0 m or to refusal. Samples were collected directly into clear, PETG co-polyester liners with red (top) and black (bottom) color-coded vinyl caps.

Four cut bank profile and six test unit excavation locations in the Dune Dam area and two profiles and one test unit excavation in the Roberts Great House area were selected to collect stratigraphic and chronometric data to anchor and supplement the solid sediment cores and previous archaeological investigations. Profile and excavation units were hand dug and all sediments were screened through 0.25 cm screens. Sedimentary units were numbered based on their stratigraphic sequence and soils were characterized using Munsell soil color, sedimentary structures, and particle size.

2.3. Laboratory analysis

Twenty-eight samples of carbonized plant remains, seven from the Roberts Great House area and 21 from the Dune Dam area, were hand-selected for AMS radiocarbon dating (Table 2). Aliquots of exemplary carbon were selected and used for species

Fig. 2. Menefee Formation coal in the Chaco Wash alluvium.

Fig. 3. Sample collection from the Chaco Wash alluvium.

identification with an environmental scanning electron microscope (Fig. 5). Twenty-four of these samples were subjected to micro-flotation analysis. Four samples were not processed and served as controls. Three of these control samples were carbonized plant remains and a fourth was uncarbonized hardwood. As an additional control, a particulate bulk carbon sample subjected to micro-flotation was split into two samples and each sample dated to determine the homogeneity of radiogenic carbon (^{14}C).

Micro-flotation was accomplished using pure water, two 100 ml glass beakers, a small (23.1 mm) magnetic stirrer, a 225 ml glass funnel with a plain 60° angle, accurate fitting 11 μm filter paper or a 10 μm nylon mesh, a ring stand (lab stand and ring clamp), a 150 mm watch glass, tweezers, and a scalpel (Fig. 6). Samples were first weighed in a nonslip 200 ml hexagonal polystyrene-weighing dish with a flat bottom for stability. Then a polygonal magnetic stirrer bar was gently placed at the bottom of a 10 ml glass beaker and placed on the magnetic stirrer. Approximately 75 ml of pure water was poured into the beaker. The hexagonal weighing dish was used to carefully dispense or pour the radiocarbon sample into the pure water.

Wood charcoal has a specific gravity of ~0.4 (with reference to water) and a density of ~0.4 g/cm^3 so it floats in water that has a density of 1.0 g/cm^3 at 4 °C and wood charcoal is positively buoyant. Coal has a specific gravity of 1.2 to 2.3 and so it is negatively buoyant in pure water and will sink if there is no surface tension. The magnetic stirrer was set to a slow speed (45–60 rpm) to break surface tension. Within 5 min, buoyant carbonized plant remains were separated from coal particles and carbonized plant remains. The buoyant carbonized plant remains were carefully decanted into an 11 μm filter paper or a 10 μm nylon mesh lined glass funnel held by a lab stand and ring clamp. The filtered water was collected in a second 100 ml glass beaker. After all of the water drained through the filter paper, it was removed and placed on a 150 mm watch glass and allowed to air dry. All carbonized particles were physically separated and removed from uncarbonized materials such as rootlets using a tweezers and a scalpel.

The resulting charcoal samples were subjected to a standard acid-base-acid pretreatment, washed with hot (70 °C) 1 N HCl for 30 min to dissolve carbonates followed by a hot (70 °C) 1 N NaOH wash for 30 min repeated until the liquid was clear to remove organic acids, and a final 30 min 1 N HCl rinse to neutralize the NaOH. The resulting samples were washed in pure water until the pH was neutral. The samples were sent to W. M. Keck Carbon Cycle AMS Laboratory at

Fig. 4. Menefee Formation coal in anthropogenic sediments of a hydraulic feature.

Table 2
Radiocarbon samples from Chaco Canyon, New Mexico.

Field sample number	Location	Depth (cm)	Composition	Sample weight (g)	Coal removed (g)	^{14}C sample weight (g)	UCI[a] AMS lab number
DD-117	Dune Dam	28–37	*Juniperus* sp. charcoal	0.21	0.01	0.20	Not dated
DD-88	Dune Dam	34–50	*Sambucus* sp. charcoal	5.44	3.51	1.93	167241 167242
DD-87	Dune Dam	39–56	*Pinus* sp. charcoal	0.04	0.02	0.02	Not dated
DD-96	Dune Dam	42–55	Hardwood charcoal	0.06	0.01	0.05	167245
DD-90	Dune Dam	53–57	Hardwood charcoal	2.14	2.12	0.02	167243
DD-93	Dune Dam	55–71	*Sambucus* sp. charcoal	18.10	15.14	2.96	167244
DD-97	Dune Dam	60	*Sambucus* sp. charcoal	0.14	0.01	0.13	167246
DD-98	Dune Dam	60–82	*Juniperus* sp. charcoal	10.86	10.80	0.06	167247
DD-95	Dune Dam	71–112	Spermatophyte charcoal	9.39	9.11	0.28	Not dated
DD-89	Dune Dam	74	*Sambucus* sp. charcoal	1.96	1.62	0.34	Not dated
DD-176	Dune Dam	80	Spermatophyte charcoal	2.25	2.23	0.02	Not dated
DD-114	Dune Dam	84	Hardwood charcoal	29.45	29.25	0.20	Not dated
DD-92	Dune Dam	90	Hardwood charcoal	3.21	3.21	0.00	Not dated
DD-116	Dune Dam	98	Conifer charcoal	0.02	0.00	0.02	Not dated
DD-175	Dune Dam	106	*Pinus* sp. charcoal	3.32	3.23	0.09	Not dated
DD-110	Dune Dam	125	Conifer charcoal	4.39	4.39	0.00	Not dated
DD-173	Dune Dam	138	Hardwood charcoal	0.25	0.19	0.06	167250
DD-109	Dune Dam	142	*Populus* sp. charcoal	2.70	1.92	0.78	167249
DD-107	Dune Dam	150	Spermatophyte charcoal	59.97	59.97	0.00	Not dated
DD-174	Dune Dam	153	Conifer charcoal	13.64	13.64	0.00	Not dated
DD-103	Dune Dam	240	Hardwood charcoal	8.81	8.80	0.01	167248
RGH-1	Roberts Great House	30–40	Unidentified carbon	3.80	Not processed	3.80	135117
RGH-2	Roberts Great House	63–73	Unidentified carbon	2.20	Not processed	2.20	135118
RGH-3	Roberts Great House	73–83	Unidentified carbon	0.95	Not processed	0.95	135119
RGH-4	Roberts Great House	93–103	Hardwood charcoal	5.40	3.50	1.90	135120
RGH-5	Roberts Great House	118–198	Hardwood charcoal	0.30	0.2	0.10	135121
RGH-6	Roberts Great House	130–157	Uncarbonized hardwood	5.00	Not processed	5.00	150903
RGH-7	Roberts Great House	157–250	Hardwood charcoal	0.30	0.20	0.10	150904

[a] University of California, Irvine.

the University of California at Irvine for the follow-up AMS radiocarbon dating.

3. Results

3.1. Micro-flotation analysis

The resulting charcoal fragments were composed of carbonized wood, which included 24 samples identified as *Juniperus* sp. (n = 2), *Pinus* sp. (n = 2), *Populus* sp. (n = 1), *Sambucus* sp. (n = 4), Spermatophyte (n = 3), unidentified conifer (n = 3), and unidentified hardwood (n = 9) ranging in mass from 59.97 to 0.02 g (Table 2). Following micro-flotation, the resulting radiocarbon samples ranged in mass from 2.96 to 0.00 g (Table 2). All of the samples exhibited some degree of coal contamination with the exception of a single specimen of conifer charcoal (DD-116). The quantity of coal contaminants ranged from 100% to 4% and averaged 70%. Three of the processed samples were composed entirely of coal (Tables 2 and 3).

3.2. AMS radiocarbon dating

Of the 28 samples of carbonized plant remains, 17 were selected for AMS radiocarbon dating on the basis of the integrity of their composition and archaeological and geological setting (Table 2). All of the samples subjected to micro-flotation analysis produced radiocarbon ages consistent with their stratigraphic position and archaeological context (Archaic to Historic Navajo) (Table 3).

Bulk carbon sample DD-88, which was split and dated produced identical radiocarbon ages (UCIAMS-167241 and UCIAMS-167242, 170 ± 20 yr BP). The sample of uncarbonized hardwood (RGH-6), which came from the same archaeological stratum (Pueblo II) as the carbonized hardwood charcoal sample (RGH-7) produced radiocarbon ages that overlapped at 1σ (Table 3).

The three samples of carbonized plant remains, which were not subjected to micro-flotation analysis produced ages beyond the limits of radiocarbon dating (>46,000 yr BP) (Table 3). These samples were collected from a transitional Basketmaker III-Early Pueblo I stratum, which contained Lino Gray earthenware pottery

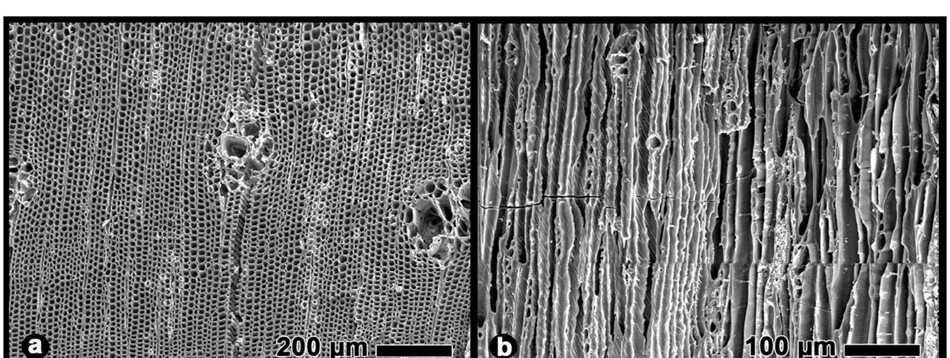

Fig. 5. Environmental scanning electron micrographs of hardwood charcoal.

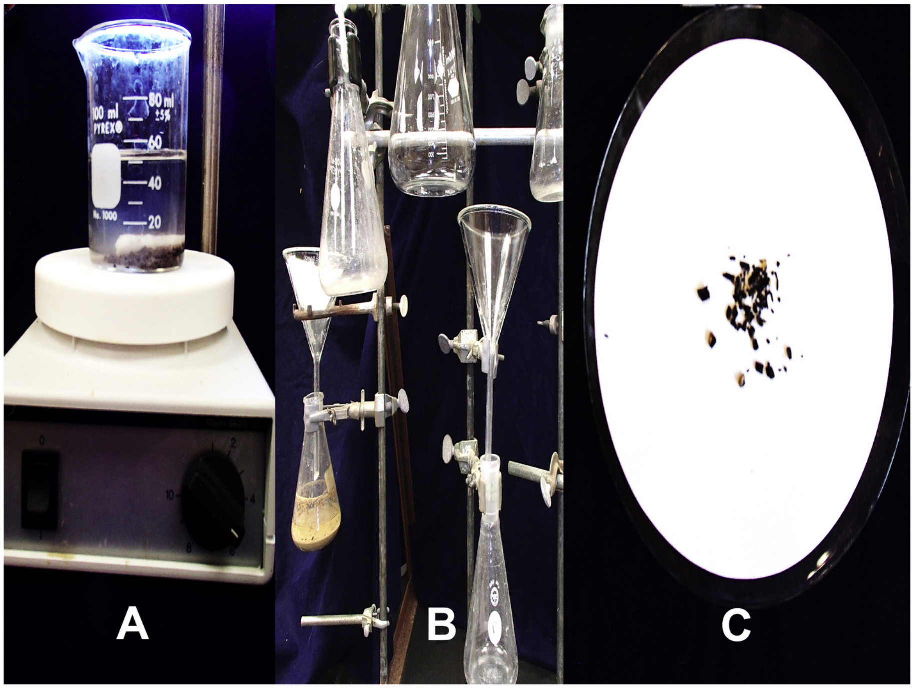

Fig. 6. Micro-flotation laboratory components: (A) glass beaker, stirrer bar, and magnetic stirrer; (B) glass funnels with filter paper on a lab stand; and, (C) watch glass, filter paper, and a processed radiocarbon sample.

(~500 CE to 800–1450 to 1150 cal yr BP; Hays-Gilpin and van Hartesveldt, 1998:135; Lekson, 2006; Vivian, 1990; Vivian and Hilpert, 2012).

4. Discussion

The coal-bearing Cretaceous Menefee Formation outcrops along the entire length of Chaco Canyon. Coal is widespread in Quaternary aeolian sediments, alluvium, and colluvium, as well as Ancestral Puebloan anthropogenic deposits. The Menefee Formation contains the carbonized remains of broadleaf angiosperm plants and conifers, and therefore conventional paleobotanical analysis and standard chemical pretreatments were ineffective in identifying and completely removing contaminants.

The issue of coal contamination is not unique to radiocarbon samples obtained from Chaco Canyon and Ancestral Puebloan archaeological sites in the American Southwest. Indeed, coal is a common contaminant of radiocarbon samples worldwide (Taylor and Bar-Yosef, 2014; Vogel, 1969). If coal is present at an archaeological site or occurs in a spatially correlated geological stratum, then it should be suspected as a radiocarbon sample contaminant (Cresswell, 1992; Tankersley et al., 1987).

The redeposition of detrital coal is a common source of contamination of radiocarbon ages obtained from organic rich Quaternary sediments (Godwin and Willis, 1959; MacDonald et al., 1991). Radiocarbon ages obtained from bulk sediment samples and bulk core samples of particularly low organic content are particularly vulnerable to coal contamination (Pilcher, 2003). While all fractions of bulk samples are susceptible to coal contamination, the 0.25 to 0.50 mm fractions are particularly problematic (Nelson et al. 1988). In these situations, it is not uncommon that 50% or more of the sample is coal from a dated interval (Holmes and Creager, 1974).

Previous attempts to remove coal contaminates from other organic matter have been only partially successful as a consequence of the wide grain-size distribution of the coal (Snyder et al., 1994; Tankersley et al., 1987; Tankersley and Munson, 1992; Waters and Stafford, 2014). Micro-flotation analysis provides a simple and cost-effective means of removing coal contaminants regardless of whether the samples were obtained from excavated contexts or from solid sediment cores. This technique is particularly useful when working with bulk samples from archaeological sites and/or geomorphological contexts.

5. Conclusions

Coal is a significant contaminant for conventional β-decay and AMS radiocarbon dating, which can bias archaeological interpretations and geomorphic and paleoenvironmental reconstructions. Micro-flotation, a specific gravity separation technique, was successfully used to remove coal contaminants from radiocarbon samples obtained from excavated and bulk solid sediment cores in Chaco Canyon, New Mexico in the USA.

While this study focused on Ancestral Puebloan archaeological sites located in the American Southwest, micro-flotation can be used to pretreat radiocarbon dating samples obtained from archaeological and geomorphic sites anywhere in the world that coal is present. Micro-flotation can also be used to remove dispersed thermally mature organic matter, which may be present as dispersed matter in sedimentary rocks, such as inertinite, pre-Quaternary megaspores and spores, pollen and other microfossils. These contaminants are possibly an even bigger problem because they are less easy to see, and have been responsible for many so-called "bad ages" by archaeologists and geologists who did not consider the issue when submitting their organic matter for analysis.

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Table 3
AMS radiocarbon ages of Chaco Canyon samples.

Field specimen number	Lab number UCIAMS[a]	Composition	^{14}C age BP (1 σ)	Calibrated age BP (2 σ)[b]	Probability distribution	Cultural stage
DD-88	167241	*Sambucus* sp. charcoal	170 ± 20	0–31	0.197	Historic Navajo
				138–157	0.112	
				165–222	0.505	
				258–285	0.186	
DD-88	167242	*Sambucus* sp. charcoal	170 ± 20	0–31	0.197	Historic Navajo
				138–157	0.112	
				165–222	0.505	
				258–285	0.186	
DD-96	167245	Hardwood charcoal	180 ± 20	0–23	0.190	Historic Navajo
				142–219	0.619	
				265–286	0.192	
DD-97	167246	*Sambucus* sp. charcoal	185 ± 20	0–21	0.190	Historic Navajo
				143–217	0.609	
				266–287	0.201	
DD-93	167244	*Sambucus* sp. charcoal	200 ± 20	146–189	0.484	Historic Navajo
				193–213	0.086	
				268–296	0.267	
DD-173	167250	Hardwood charcoal	970 ± 25	796–875	0.595	Pueblo II
				892–933	0.405	
DD-90	167243	Hardwood charcoal	985 ± 20	800–813	0.058	Pueblo II
				826–865	0.263	
				901–939	0.665	
				946–953	0.013	
RGH-7	150904	Hardwood charcoal	1095 ± 20	958–1014	0.603	Pueblo II
				1017–1057	0.397	
RGH-6	150903	Hardwood	1120 ± 20	970–1062	1.000	Pueblo II
RGH-5	135121	Hardwood charcoal	1235 ± 15	1081–1160	0.278	Pueblo I
				1172–1187	0.190	
				1203–1258	0.532	
DD-109	167249	*Populus* sp. charcoal	1245 ± 20	1086–1112	0.057	Pueblo I
				1122–1159	0.078	
				1172–1266	0.866	
RGH-4	135120	Hardwood charcoal	1260 ± 15	1179–1263	1.000	Pueblo I
DD-98	167247	*Juniperus* sp. charcoal	1690 ± 20	1545–1624	0.926	Basketmaker II
				1671–1690	0.074	
DD-103	167248	Hardwood charcoal	3150 ± 25	3272–3285	0.038	Archaic
				3339–3445	0.962	
RGH-1	135117	Unidentified carbon	>47,200	Unknown	Unknown	Unknown
RGH-2	135118	Unidentified carbon	>47,800	Unknown	Unknown	Unknown
RGH-3	135119	unidentified carbon	>46,100	Unknown	Unknown	Unknown

[a] University of California, Irvine.
[b] Calib 7.10.

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