PRELIMINARY ^{14}C DATES ON BULK SOIL ORGANIC MATTER FROM THE BLACK CREEK MEGAFAUNA FOSSIL SITE, ROCKY RIVER, KANGAROO ISLAND, SOUTH AUSTRALIA

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ABSTRACT. Radiocarbon age determinations and stratigraphy suggest that the deposits in Black Creek Swamp on Kangaroo Island record 3 phases of deposition and associated soil development which spanned at least the last 20,000 yr. Four new ^{14}C age determinations on bulk soil organic matter and their stratigraphic context are presented in this paper. Three of these age determinations (FP6: 15,687 ± 110 BP [WK11487]; FP7: 16,326 ± 385 BP [WK11488]; and FP8: 17,618 ± 447 BP [WK11489]), are from the organic-rich fossil layer located 45–75 cm below the current floodplain surface. The fourth, a much younger date, FP5: 5589 ± 259 BP (WK11486), was obtained from the base of the overlying modern soil. The dates for the fossil layer increase systematically with depth and correlate well with 5 previous ^{14}C dates (Hope et al., unpublished), ranging between 15,040 ± 120 BP and 19,000 ± 310 BP. This suggests that the data set represents a possible minimum age of the bulk organic matter, and considering the high organic matter contents of approximately 8%, has implications for the age of the megafauna buried in this layer. The overlying modern soil, with its much younger date, contains lower levels of organic matter (3–7%) and gastropods not seen in the fossil layer. This suggests a substantial change in environmental conditions probably due to an alteration in the floodplain drainage conditions. This chronological and sedimentalogical discontinuity indicates that 2 distinct depositional regimes existed and were separated by up to 10,000 ^{14}C yr. A calcareous, sandy silt deposit underlying the fossil layer is a calcarenite deposit with low total organic content and is considered the base of the section; it suggests a third separate depositional episode. As such, the Black Creek Swamp in the southwest corner of Kangaroo Island formed intermittently over at least the last 20,000 yr during 3 distinct depositional phases, one of which was the formation of the fossil-laden, organic-rich floodplain surface, which has a possible minimum age of approximately 15,000 to 19,000 BP.

INTRODUCTION

The Black Creek Swamp, located in the Rocky River catchment in the southwest region of Kangaroo Island South Australia (Figure 1), has had intermittent geological (Tindale et al. 1935; Hope et al., unpublished; Tyler et al. 1979), paleontological (Tindale et al. 1935; Hope et al., unpublished), archaeological (Tyler et al. 1979), and paleoenvironmental (Gröcke 1996, 1998) research over the last 90 yr. The Black Creek Swamp is nestled between the calcareous dune ridges of the west and south coasts and the laterite plateau that comprises the northern two-thirds of Kangaroo Island (Figure 1). It is a unique deposit because it contains a significant, dense, and well-preserved megafauna fossil deposit (including *Diprotodon*, *Zygamaturus*, and *Sthenurus*). Chaplin (1972) has showed that highly alkaline conditions, similar to those in the Black Creek Swamp, contribute to the high level of bone preservation.

A description of the stratigraphy of Black Creek Swamp by Tindale et al. (1935) included 4 sedimentary horizons (from top to bottom: A, B, C, and D): a brown earth (A, 0–10 cm) and a reddish-colored swamp earth (B, 10–30 cm), followed by the fossil-bearing black swamp earth (C, 30–60 cm). Underlying these deposits and representing the base of the swamp was a greyish calcareous sand (layer D), consisting of worn marine shells, foraminifera, freshwater shell fragments (*America pyramidatus, Sphaerium sp.*), and salt-lake-frequenting *Coxiella sp*. The varied suite of shells in this basal layer indicated a sediment accumulation well below water, most likely under estuarine conditions (Tindale et al. 1935). Importantly, 2 key erosional boundaries were

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Kangaroo Island
South Australia

Rocky River Catchment including the Black Creek Swamp fossil site

Australia

Kingscote

Laterite Plateau

Rocky River

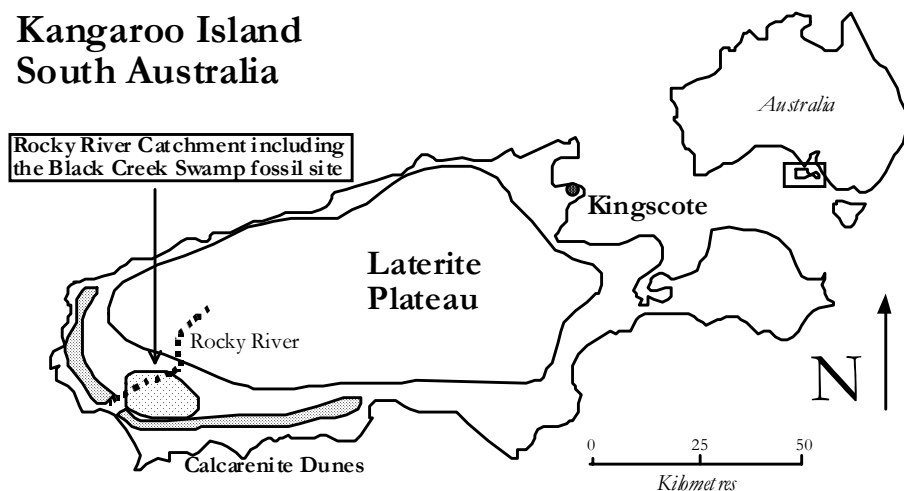**Calcarenite Dunes**

N

| 0 | 25 | 50 |

Kilometres

Figure 1 Location map of the Black Creek Swamp in the Flinders Chase National Park, Kangaroo Island, South Australia.

identified in the stratigraphic section. A marked change in coloration was seen between layers B and C. Also noted was the existence of boulders and cobbles of hardened calcrete crust at the top of layer D, consolidated during a period of sub-aerial denudation and kunkarization. Tindale et al. (1935) tentatively suggested a Pleistocene age for the deposit based on a geomorphological comparison of the Rocky River site with the Pleistocene coastal limestone hills located on the island.

Research by Hope et al. (unpublished) recognized layers A, B, and C; however, they divided the calcareous layer D into 2 separate horizons consisting of a grey silt sand overlying a light grey calcareous marl which graded into decomposed calcarenite. Five dates, which span 15,040 ± 120 BP and 19,000 ± 310 BP, were obtained for layer C (the fossil horizon), both from the excavation pit itself and a sediment core taken nearby for pollen analysis. An age of 1960 ± 120 BP (ANU 2170) for the overlying layer B, obtained by Hope et al. (unpublished), further supported an early Holocene depositional hiatus in the stratigraphic section, as suggested by Tindale et al. (1935). Another radiocarbon date of 4260 ± 90 BP was obtained for the organic material extracted from the calcarenite layer (layer D), which is stratigraphically the oldest of the units. This anomalous ^{14}C age was primarily attributed to carbon contamination from humates in groundwater.

Recent age determinations of the site using other methods, such as Optical Stimulated Luminescence (OSL), are proving to be problematic due to the high uranium content of the deposit. This amounts to 10 parts per million (ppm) for groundwater sampled from the site (Gill 1996), as well as 37.9 ppm (FP6) and 21.7 ppm (FP8) obtained from recent X-ray fluorescence analysis for bulk soil samples taken from the fossil pit (this study). An attempt to obtain ^{14}C dates from collagen extracted from the fossil material has also proved to be difficult due to the minimal amounts of dateable material preserved in the excavated bones. Hence, the ^{14}C dating of bulk soil organic matter (SOM) is a first step in determining the approximate age of the site. More detailed age determinations currently being undertaken are aimed at placing the geomorphological, paleoenviromental, and paleontological interpretations to be put into a chronological context and will allow for a comparison to other late Pleistocene megafauna sites and paleoenviromental databases.

METHODS

A stratigraphic profile was constructed from a section located in the southwestern corner of the 4×4-m 2003 fossil excavation pit (FP). A 120-cm vertical trench was excavated in order to obtain fresh samples. These were described in cm scale using standard geological techniques to estimate grain size, texture, mineralogy, and color, following methods described by Retallack (1988), while color codes follow Munsell (1994). Inspection of numerous samples in each horizon was undertaken in order to determine the representative features. Twelve samples (FP1 to FP12) were taken for geochemical analysis at 10-cm intervals.

Four of these samples (FP5, FP6, FP7, and FP8) were hand-sieved to remove root material, bone and shell fragments, with the portion ≤1 mm in diameter being retained and milled to a fine powder. These samples were forwarded to the Radiocarbon Laboratory at the University of Waikato in Hamilton, New Zealand for dating. Physical pretreatment at the Waikato laboratory involved removal of any visible contaminants (roots and stones). This was followed by a chemical pretreatment involving an initial acid wash using 10% concentrated HCl (to remove carbonates), followed by a DI rinse. Samples were then washed in hot 0.5% NaOH solution (to remove humic acids), then again washed in 10% concentrated HCl, rinsed and dried. From this, the base soluble fraction was selected for ^{14}C age determination (Alan Hogg, personal communication, 2003).

^{14}C age determinations in this paper (Table 1) follow Godwin (1962), Stuiver and Polach (1977), and Gillespie (1986). They are based on the Libby half-life of ^{14}C (5568 yr), the assumption of constancy of ^{14}C atmospheric concentrations during the past, and the use of oxalic acid (direct or indirect) as the modern ^{14}C standard. Isotopic fractionation normalization of all sample activities to the base of $\delta^{13}C = -25‰$ (relative to the $^{13}C/^{12}C$ ratio of PDB) is assumed. Finally, BP ages are presented with the year 1950 automatically designated as the base year (i.e. present is AD 1950). Dates are given ±1 standard deviation (±1 σ) due to counting statistics multiplied by an experimentally determined laboratory error multiplier of 1.217 (Alan Hogg, personal communication, 2003).

Table 1 $\delta^{14}C‰$, $\delta^{13}C‰$, $\Delta^{14}C‰$, and % modern carbon (pMC) for the 4 ^{14}C dates WK11486 (FP5), WK11487 (FP6), WK11488 (FP7), and WK11489 (FP8) provided by the University of Waikato, Radiocarbon Dating Laboratory, Hamilton, New Zealand. Note: WK numbers are catalog numbers provided by the University of Waikato Radiocarbon Dating Laboratory.

Sample	Depth	$\delta^{14}C‰$	$\delta^{13}C‰$	$\Delta^{14}C‰$	pMC	Result
FP 5	40 cm	−504.6 ± −12.9	−28.3 ± −0.2	−501.3 ± −15.8	49.9 ± −1.6	5589 ± −259 BP
FP 6	50 cm	−858.6 ± −0.2	−26.8 ± −0.2	−858.1 ± −1.9	14.2 ± −0.2	15,687 ± −110 BP
FP 7	60 cm	−870.8 ± −5.0	−32.0 ± −0.2	−869.0 ± −6.1	13.1 ± −0.6	16,326 ± −385 BP
FP 8	70 cm	−889.7 ± −4.9	−30.7 ± −0.2	−888.4 ± −6.0	11.2 ± −0.6	17,618 ± −447 BP

RESULTS

The 4 main units initially identified by Tindale et al. (1935) and expanded on by Hope et al. (unpublished) are further described (Figure 2). The modern soil horizon grades from a 10YR brown to a 10YR 4/2 dark greyish brown through approximately the top 40 cm of the profile. It is moderately calcareous with a PH_w of 8.57 and contains a substantial modern root system, charcoal fragments, and sub-rounded quartz grains of sand and silt. A visual non-quantitative inspection of mega-scopic charcoal content indicates that it increases down-profile to 40 cm. Total organic carbon (TOC) content varies between 7% at the top, decreasing down to 3% at the base of the horizon. These values were derived from the non-calcareous residual portion of the sample retained after 1M hydrochloric

acid treatment at 85 °C for 45 min. Of particular note is the presence of the gastropod species *Hydrococcus brazieri* in the top 40 cm of the profile (layers A, B) of the profile (John Cann, personal communication, 2003). This small species has a wide temperature and salinity tolerance and prefers marginal salt lakes, back dune swamps, estuaries, and tidal flats (Ludbrook and Gowlett-Holmes 1989).
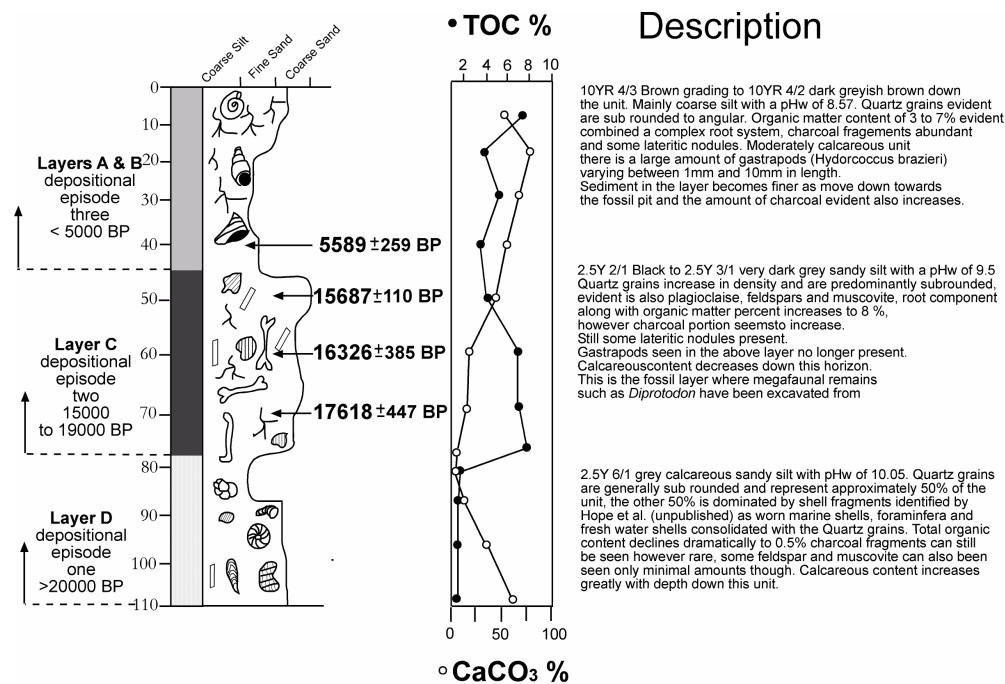

Figure 2 Stratigraphical reconstruction of the Black Creek Swamp fossil excavation pit. Included are PH_w values, 4 ^{14}C dates, and TOC data provided with $\delta^{13}C$ values from the Rafter Isotope Lab, Lower Hutt, New Zealand.

A distinct yet gradational boundary between this unit and the underlying 2.5Y 2/1 black to 2.5Y 3/1 very dark grey, silty organic-rich fossil layer is evident between 30 and 40 cm. Below this boundary, there is a total lack of megascopic gastropods, suggesting a different depositional regime. The fossil layer, which extends down to 70~75 cm, has a PH_w of 9.5 and a TOC of between 6 and 8% for the HCl-treated residual portion of the samples. The percentage of quartz grains in this unit is greater than in the overlying modern soil and there are minor occurrences of feldspars and muscovite. The quantity of modern root material decreases significantly down-profile, while macroscopic charcoal content increases and the calcareous content decreases. A distinct boundary is evident between the dark fossil horizon (layer C) and the underlying 2.5Y 6/1 grey calcareous silt and sand (layer D) identified by Tindale et al. (1935). It is recognized by the existence of boulders and cobbles of hardened calcrete crust, clearly visible in the fossil pit stratigraphy. Quartz grains are sub-rounded and represent approximately 40% of the horizon decreasing in abundance with depth and corresponding with an increase in the percentage of shell fragments. The latter include worn and rounded marine shell fragments, freshwater shells, and foraminifera of likely calcarenite aeolian origin. Both the calcareous component and the PH_w (10.05) of the unit corresponding to increasing shell fragment content. Macroscopic charcoal is almost non-existent in this unit and TOC on the residual sample decreases to 0.5%. There is also a small percentage (1–2%) of feldspars and muscovite. The distinct variations between this layer and the overlying organic-rich soil (layer C) infer the existence of 2 separate depositional modes: the first being aeolian deposition of calcarenite grains and the second being a floodplain, organic-rich soil.

Table 1 presents the $\delta^{14}C$‰, $\delta^{13}C$‰, $\Delta^{14}C$‰, and pMC for the 4 dates obtained from the Waikato Laboratory in New Zealand. Three of the dates (FP6, FP7, and FP8) were taken at 10-cm intervals from the 2.5Y 2/1 black peat-like horizon at depths from the top datum of 50, 60, and 70 cm, respectively (Figure 2). The fourth date was obtained at the base of the overlying 10YR 4/2 dark greyish brown silt at a depth of 40 cm (sample FP5).

DISCUSSION

^{14}C dating of organic matter soils, sediment, and their individual components is not a routine procedure. This is due largely to difficulties in determining the origin of the carbon in these materials (Gillespie 1986). Although treated to remove sources of modern carbon (plant rootlets, humic acids, and carbonates), some level of contamination of the ^{14}C sample will occur. This becomes a problem for samples older than 20,000 yr, due to the small amounts of original ^{14}C carbon that remain (Roberts et al. 1994; Fauve 1986; Gillespie 1986). However, for samples younger than 20,000 yr in age, it is still necessary to have an independent check of the dates obtained.

Radiocarbon (^{14}C) dating has been used in conjunction with other dating methods, such as uranium-thorium (U/Th), thermoluminescence (TL), electron spin resonance (ESR), and more recently, optical stimulated luminescence (OSL). ^{14}C dates have been compared to these methods at many archaeological sites in Australia with varying degrees of success (Turney et al. 2001; Gillespie 2002; Bowler et al. 2003; Stone and Cupper 2003). Cave sediments in Western Australia's Devil's Lair produced strong correlation between ^{14}C dates of charcoal, and OSL and ESR ages for sediments (Turney et al. 2001). This was consistent for ages up to approximately 30 ka BP; however, older dates obtained produced differences of up to 10,000 yr between the various techniques. At Lake Mungo in western New South Wales, site of Australia's oldest human remains, both comparative and non-comparative results are informative (Bowler et al. 2003). Good correlation existed between ^{14}C dates of charcoal and TL dates of sediment spanning the Lake Mungo geomagnetic excursion (Barbetti and McElhinny 1972; Huxtable and Aitken 1977; Bell 1994). However, ^{14}C estimates for the Mungo I cremation site are approximately 15 to 20 ka younger than those provided by OSL dating (Bowler et al. 2003). Another archaeological site at Kow Swamp was dated, giving ^{14}C ages of 15–9 ka BP obtained from charcoal, shells, and bone apatite, and OSL dates of 22 and 19 Ka from the surrounding sediment (Stone and Cupper 2003). At both Lake Mungo and Kow Swamp, differences in ages were attributed primarily to contamination by younger carbon that had moved downward through the profile.

Investigations into the soil organic matter (SOM) grain size fractions have been undertaken to better appreciate the contribution of carbon fractions to ^{14}C age determinations (Gillespie et al. 1992; Trumbore 1993; Bol et al. 1996; Leavitt et al. 1996; Paul et al. 1997). Acid hydrolysis, humus fractionation, density separation, and size separation are all examples of methods that have been employed to isolate and date certain components of SOM. In general, the residual or recalcitrant components retained after separation provided older ages than those obtained for bulk SOM. Lipid fractions (Bol et al. 1996), fine silt and coarse clay fractions (Anderson and Paul 1984), denser particle fractions (Trumbore et al. 1989), and fine-grained charcoal (Gillespie et al. 1992) are examples of SOM components that contain carbon older than the bulk SOM from which they were extracted. The method employed to extract this older carbon fraction from SOM varies with environment of deposition. Hence, it is important to identify the carbon components that exist in the SOM before an extraction processes is chosen. ^{13}C nuclear magnetic resonance (NMR) spectra of bulk and treated SOM is one such method that can achieve this.

^{13}C NMR spectroscopy is a technique that can be used to characterize SOM (Baldock and Skjemstad 2000; Mathers et al. 2000; Quideau et al. 2000; Krull and Skjemstad 2002). This technique produces spectra that roughly divide SOM samples into chemical shift regions in which the chemistry of the C atoms within each region is similar. Examples of such shift regions are alkyl, o-alkyl, aromatic, methoxyl, and carbonyl C atoms. Hence, the application of ^{13}C NMR to the dated bulk SOM from the Black Creek Swamp will help to identify the SOM components present. This will allow for a better appreciation of in situ and contaminant carbon constituents in the sediment. As such, the extraction and dating of these in situ fractions will provide an age comparison to the most likely minimum ages provided by the bulk SOM dating process. This type of work is now in progress for the Black Creek Swamp SOM samples. Preliminary results indicate a substantial aromatic and lignin component, as well as an absence of any significant carbohydrate component (Jan Skjemstad and Evelyn Krull, personal communications, 2003). This suggests that the carbon of the organic-rich layer has not been contaminated extensively by more modern SOM, and that the ^{14}C age determinations are a reasonably accurate age estimation of the SOM accumulation in the fossil layer soil zone.

CONCLUSION

The ^{14}C dates presented in this paper give an approximate age of the bulk SOM contained within the organic-rich layer of the Black Creek Swamp excavation site at Rocky River, Kangaroo Island. The dates for FP6: 15,687 ± 110 BP (WK11487); FP7: 16,326 ± 385 BP (WK11488); and FP8: 17,618 ± 447 BP (WK11489) correlate with the time frame of 15,000 to 19,000 BP obtained by Hope et al. (unpublished). These dates also combined with a fourth, FP5: 5589 ± 259 BP (WK11486, base of Layer B), have been useful in placing the site's stratigraphy into a better chronological context. It seems clear that the Black Creek Swamp experienced 3 separate depositional periods that spanned at least the last 20,000 yr. The 3 dates (FP6, FP7, and FP8) obtained for the overlying organic-rich horizon (layer C) suggest a possible minimum age of between 15,000 and 18,000 BP for this unit. Thus, it can be assumed that the underlying calcareous, sandy silt layer D was most likely deposited prior to 20,000 BP. The ^{14}C age for the base of layer B constrains the horizons above it to a possible mid-Holocene age of 5000 BP. It also suggests that the erosional boundary between layers A and B and the fossil layer below spans a minimum of approximately 10,000 ^{14}C yr.

It should be made clear, however, that these ^{14}C dates are of bulk SOM only, and do not imply a similar age for the fossils excavated from this site. The ^{14}C ages given are purely from the TOC, which is most likely residual carbon retained since the time of deposition. It is also likely that the ages also reflect some component of younger carbon that has been translocated by surface or groundwater processes. Hence, these preliminary dates are likely to represent a possible minimum age of the bulk SOM. In situ and contaminant carbon components contained in the SOM will be quantified using ^{13}C nuclear magnetic resonance (NMR). In turn, the extraction of these older carbon constituents identified for ^{14}C age determinations will allow the on-going palaeoenvironmental reconstruction to be put into the clearest chronological context yet.

ACKNOWLEDGEMENTS

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