A radiocarbon chronology for Sanamere Lagoon, Cape York Peninsula, using multiple organic fractions

Maria Rivera-Araya[a,*], Cassandra Rowe[a], Vladimir Levchenko[b], Sean Ulm[c], Michael I. Bird[a]

a ARC Centre of Excellence of Australian Biodiversity and Heritage, College of Science and Engineering, James Cook University, PO Box 6811, Cairns, QLD, 4870, Australia

b Centre for Accelerator Science, Australian Nuclear Science and Technology Organisation (ANSTO), Locked Bag 2001, Kirrawee, DC, NSW, 2232, Australia

c ARC Centre of Excellence for Australian Biodiversity and Heritage, College of Arts, Society and Education, James Cook University, PO Box 6811, Cairns, QLD, 4870, Australia

Abstract

The selection and pre-treatment of reliable organic fractions for radiocarbon age determination is fundamental to the development of accurate chronologies. Sampling from tropical lakes is particularly challenging given the adverse preservation conditions and diagenesis in these environments. Our research is the first to examine and quantify the differences between radiocarbon ages from different carbon fractions and pretreatment protocols from tropical lake sediments. Six different organic fractions (bulk organics, pollen concentrate, cellulose, stable polycyclic aromatic carbon (SPAC), macrocharcoal >250 µm and microcharcoal >63 µm) were compared at six different depths along a 1.72 m long core extracted from Sanamere Lagoon, Cape York Peninsula, northern Australia. Acid-base-acid (ABA), modified ABA (30% hydrogen peroxide + ABA), 2chlorOx (a novel cellulose pre-treatment method) and hydrogen pyrolysis (hypy) were used to pre-treat the organic fractions. The oldest date is ~31,300 calibrated years before present (cal yr BP) and the youngest is ~2800 cal yr BP, spanning ~28,500 years. The smallest offset between the minimum and the maximum age for different fractions and across pretreatment methods at a given depth was found to be 832 years (between SPAC and pollen) and the largest ~16,750 years (between pollen concentrate and SPAC). The SPAC fractions pre-treated with hypy yielded older ages compared to all other fractions in most cases, while bulk organics yielded consistently younger ages. The magnitude and consistency of the offsets and the physical and chemical properties of the tested organic fractions suggest that SPAC is the most reliable fraction to date in tropical lake sediments and that hypy successfully removes exogenous carbon contamination.

1. Introduction

Developing an accurate chronology is fundamental to placing any reconstruction of past environmental change within a robust temporal framework (Zimmerman and Wahl, 2020). Selecting a reliable carbon fraction and a pre-treatment technique that removes carbon contaminants are key to obtaining reliable radiocarbon dates (Pettitt et al., 2003; Bronk Ramsey 2008). Although there is currently no agreement about which is the most reliable fraction to date to avoid these issues, short-lived plant macrofossils and charcoal, if present, tend to be among the most favoured materials for dating (Cohen 2003; Martin et al., 2019). This selection becomes particularly challenging in the tropics where high annual temperatures negatively and differentially influence the preservation of organic fractions. Furthermore, the seasonal nature of the tropical hydrological cycle in northern Australia results in high rates of weathering and

variable pH and redox conditions, in turn altering carbon cycling and preservation in lake sediment columns (Bird et al., 2002; Higham et al., 2009). Collectively, these conditions limit the availability of those organic components considered more reliable and the dating of other fractions can result in aberrant radiocarbon results that are linked to, for example, poorly preserved or degraded charcoal, identifiable when combustion yields are lower than expected (50–60% carbon by weight; Higham et al., 2009).

Despite the importance of choosing a carbon fraction that represents the age of contemporaneous sediment deposition, few studies have focused on sediments in the tropics. Those studies available from the tropics, and based on multiple organic fractions, have focused on swamps (May et al., 2018), peats (Wüst et al., 2008) and organic springs (Field et al., 2018). No studies have been undertaken using lake sediments and no studies in any tropical environment compare more than four fractions (May et al., 2018). Results from the available studies (as mentioned above) are contradictory regarding the reliability of macro-charcoal and pollen concentrates (yielding anomalously older or younger ages compared to other fractions). Studies in temperate areas have also reported significant discrepancies between radiocarbon dates for different, supposedly contemporaneous, carbon fractions in lake sediments from the same depth. For example, in boreal and arctic lake sediments wood and charcoal were found to be older than other organic materials such as conifer and deciduous periderms, usually by several hundred years (Oswald et al., 2005).

When no macro-organic remains are visible in lake sediment matrices, dating bulk sediments is the one of the most commonly used options. Given the multiple sources from which carbon could have derived (Bronk Ramsey 2008), the contribution of contaminants increases considerably for any bulk organic matter fraction. Several studies have found this fraction to be consistently younger than other fractions given the possibility of contamination with younger carbon (Barnekow et al., 2009; Wang et al. 1996; Pessenda et al. 2001).

Discrepancies have likewise been found between different types of macrofossils, with some more prone to an apparent ‘reservoir effect’ (Turney et al., 2000). Similarly, pollen, charcoal and bulk sediment are recognized to be potentially highly mobile, porous and heterogeneously sourced, all of which complicates their reliability for radiocarbon dating. Additionally, several studies have demonstrated that there are significant uncertainties associated with simply dating bulk sediment, regardless of the geographical location from where the sample was collected (Bj¨orck et al., 1998; Wüst et al., 2008; Xu and Zheng 2003). Extensive research has shown how the effect of contamination depends on the age of the sample (Pettitt et al., 2003; Higham et al., 2011; Wood 2015). Thus, dating beyond ~10,000 years becomes considerably more problematic as even minimal percentages of contamination can cause hundreds to thousands of years of offset (Aitken 1990; Wood 2015). In an extreme example, offsets of up to 16,000 years between radiocarbon ages from bark and pollen from a tropical peat have been obtained in a previous study, with the pollen extracts yielding older dates (Wüst et al., 2008).

Pre-treatment procedures are also fundamental to the generation of accurate radiocarbon dates. While acid-base-acid (ABA treatment) is routinely employed to remove contaminants, it is not always effective (Chappell et al. 1996; Gillespie et al., 1992). Notably, the determination of when decontamination is complete can be technically challenging. The base treatment during ABA can also further remove charcoal by solubilizing it to ‘humic acid’, resulting in considerable sample loss. As an alternative to ABA, acid-base-wet oxidation (ABOX) has been found to be more appropriate and an effective method to

remove contamination, especially for old samples (Bird et al., 1999, 2014). However, the harshness of the technique can similarly remove excessive carbon and this may limit the application of the technique in the tropics where macro-remains are scarce (Bird et al., 1999). Recent studies have shown that ABA and hydropyrolysis (hypy) treated pairs can produce comparable results in some contexts (Bird et al., 2014; Alex et al., 2017; David et al., 2019). Hypy (to isolate stable polycyclic aromatic carbon: SPAC) has shown promising results in mound spring deposits, where the technique appeared to remove the effects of post-depositional modification to the ages obtained from different organic fractions from the sediment (Field et al., 2018). Given this promising result, hypy pretreatment might be expected to be similarly effective in lake sediments.

Given the fact that most studies rely on the ages from one organic fraction over the entire sequence, it is imperative to test whether alternative protocols using different carbon fractions and pre-treatment protocols would yield more accurate results across a range of depositional ages. This paper examines six carbon fractions in samples from a sediment core from Sanamere Lagoon on Cape York Peninsula in tropical northern Australia to determine which fraction and technique pretreatment is most appropriate to obtain reliable radiocarbon ages.

2. Methodology

2.1. Site description, fieldwork and core processing

Sanamere Lagoon (11.123030◦S 142.359470◦E; 15 m above sea level) is located close to the northern tip of Cape York Peninsula and 1 km north of the W-E flowing perennial Jardine River (Fig. 1). The lagoon is 1.5 km in diameter. The climate of the region is monsoonal with 88% of an annual 1753 mm rainfall falling between December and April. Mean annual temperature is high at 27°C (BOM 2018). Vegetation surrounding the lagoon is dominated by low-shrub heathlands (incorporating, for example, species of *Asteromyrtus*, *Jacksonia*, *Hibbertia*, *Thryptomene*, *Allocasuarina* and *Grevillea*). Eucalyptus woodlands, and to a lesser extent grass ground cover, fringe the outer catchment (Neldner et al., 1995). Given the lack of studies comparing the results from radiocarbon dating from more than four carbon fractions in tropical lake sediments, Sanamere Lagoon is an ideal study case to better understand the use of different carbon fractions and pretreatments in tropical environments.

Sanamere Lagoon is considered a sub-coastal wet heath swamp, a palustrine system (a wetland with more than 30% emergent vegetation) (Environment & Science, 2018) and is also valued as a “wilderness wetland area” (Abrahams et al., 1995). We also acknowledge the Traditional Owners of the lands and waters of the Sanamere Lagoon region, with many thanks to Charles Woosop and the Apudthama Land Trust.

Fieldwork was undertaken in July 2017. Sanamere Lagoon was cored using a floating platform with hydraulic coring-rig, positioned at the approximate centre of the water body. The coring location was chosen based on previous reconnaissance work in May and April 2016. At the time of sampling, water depth was 1.2 m. A single sediment core of 1.72 m was collected (terminating at bedrock). This column represents the entire sediment sequence in the lagoon. Evidence from satellite observations suggests that the lagoon does not dry completely during the dry season (Mueller et al., 2016).

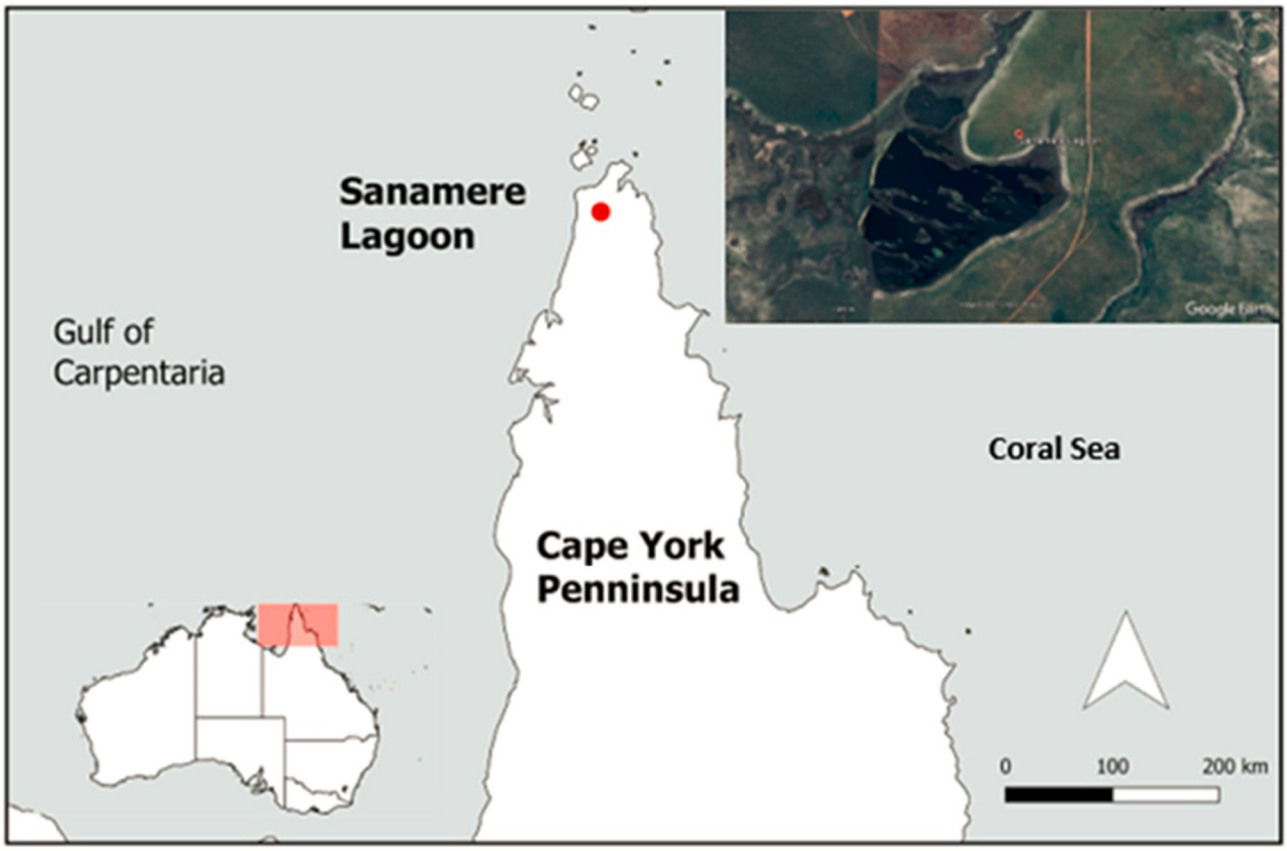

Fig. 1. Sanamere lagoon on Cape York Peninsula, Australia.

The core was cut on-site into four sections that were frozen upright, also on-site. They were later described according to standard descriptors (Schnurrenberger et al. 2003) and scanned using the ITRAX micro X-ray Fluorescence (μXRF) core scanner at the Australian Nuclear Science and Technology Organisation (ANSTO). The scans included optical and X-ray photographs of all sections.

The sedimentary sequence for Sanamere Lagoon is shown in Figs. 2 and 3. Five stratigraphic units were identified according to variation in physical and chemical parameters. Munsell soil colour codes (e.g., 10 YR 2/1) are included in brackets for reference. The first 43 cm of the core consists of a black (10 YR 2/1), organic (5–40% C), silty layer. From 43 cm, a marked decrease in organic content (0.5–1%) occurs down-core whilst sediment grain size decreased. Between 65 and 71 cm a series of sequential orange (7.5 YR 6/8) bands are evident. Between 71 and 140 cm a brown, silty clay layer dominates the sequence. Starting at 140 cm a dark brown layer appears (7.5 YR 3/3), and gravel content also increases, indicating the presence of laterite (bedrock), while the carbon content remains low.

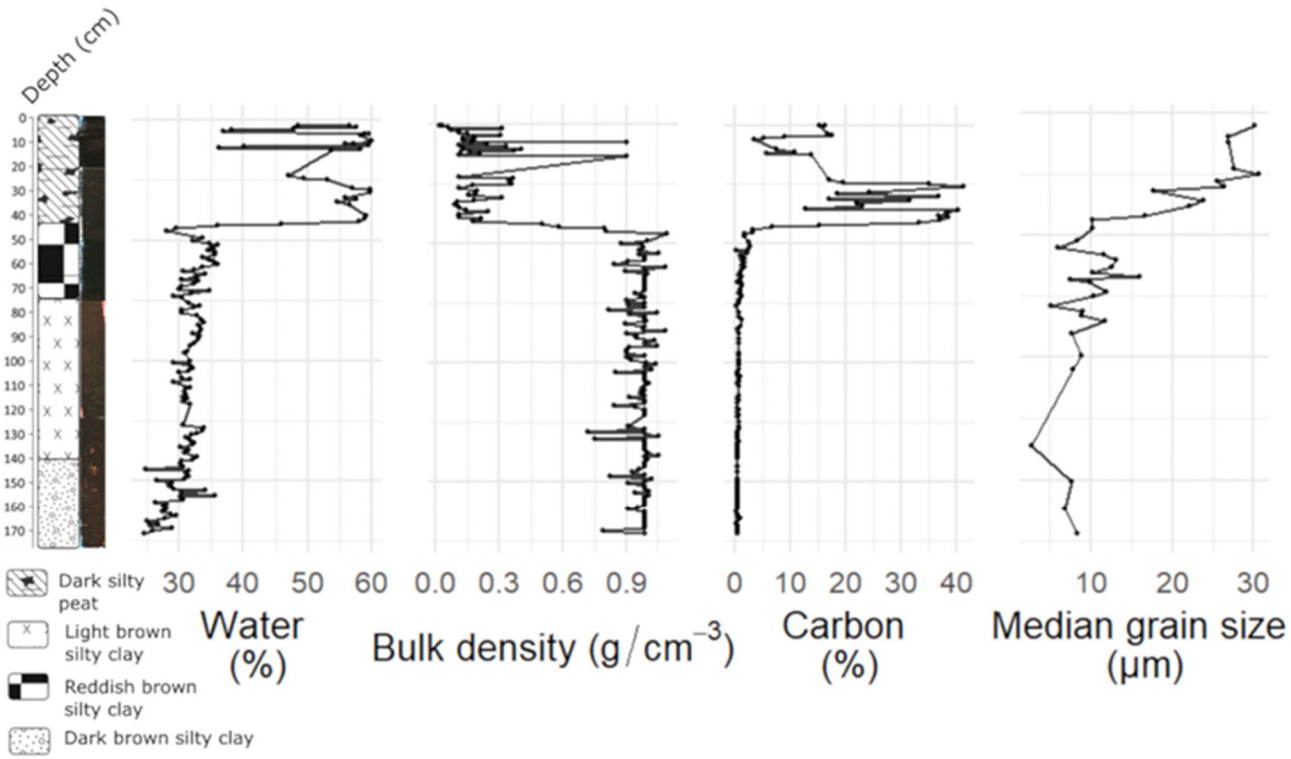

Fig. 2. Stratigraphy of the Sanamere sequence.

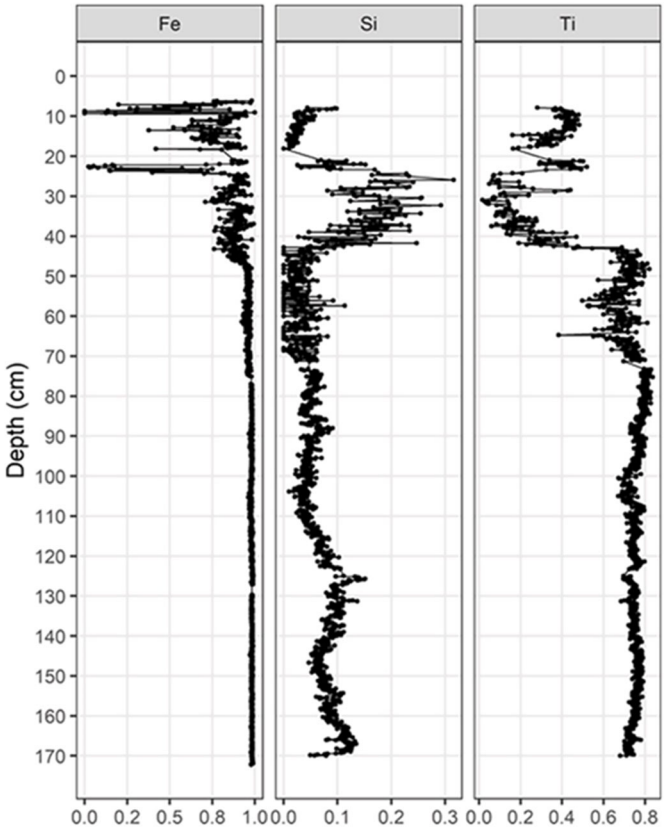

Fig. 3. Fe, Si and Ti counts on the Sanamere sequence.

2.2. Sampling and pretreatment for radiocarbon dating

Samples for ^{14}C AMS dating were obtained for six different carbon fractions (SPAC, microcharcoal (>63 µm), macrocharcoal (>250 µm), pollen concentrate, 'cellulose' and bulk organics) for six depths spread along the core (Table 1, Figs. 4–5). These depths were chosen after consideration of the stratigraphic changes observed along the sequence (texture, colour, elemental abundance). Although attempts to extract all fractions from the same depth were made, this was not always achieved as samples from some depths yielded insufficient amounts of carbon after pretreatment to be processed for radiocarbon dating (Supplementary material). In order to address this issue, samples from additional depths immediately above or below the original sample were analysed to complete the age-depth model. The studied carbon fractions were selected according to their availability in the sequence. For example, macrofossils were not included as they were absent from the sediment sequence.

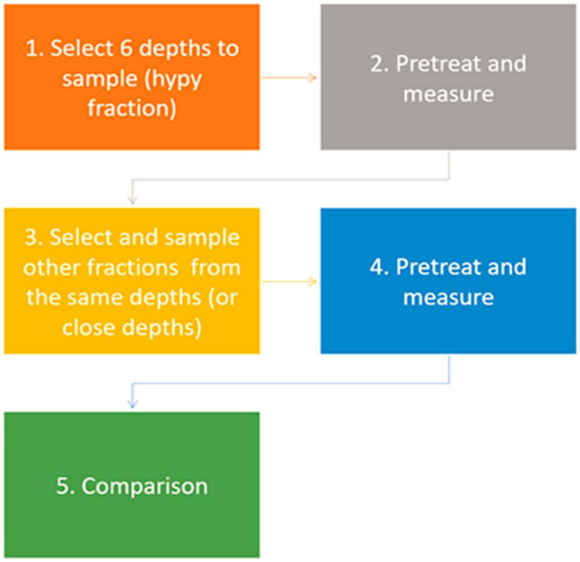

Fig. 4. Summary of methods. Radiocarbon results from the hypy fraction were initially used as a reference to the age of the sediment sequence. This step allowed samples to be represented homogenously along the core.

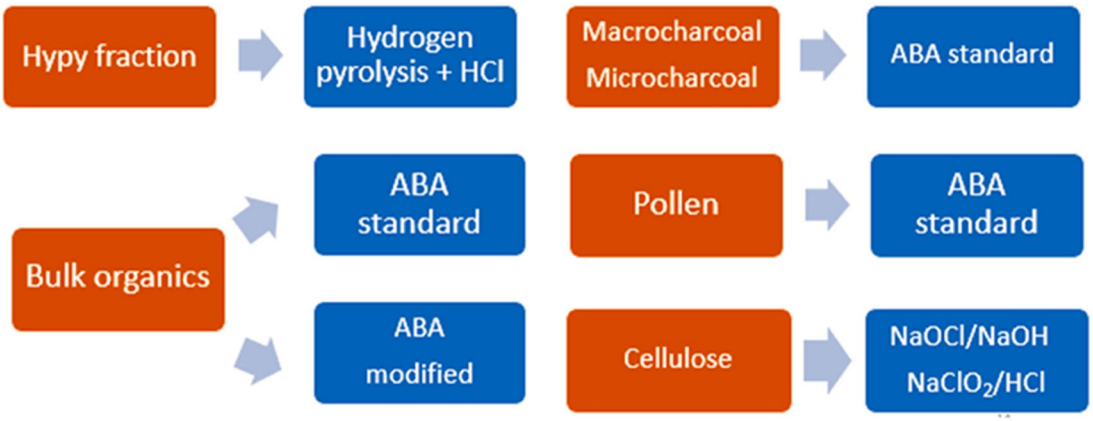

Fig. 5. Carbon fractions and corresponding pretreatment techniques (after isolation of fractions).

Table 1. Conventional and calibrated dates from all samples tested in the study.

Laboratory Code	Depth (cm)	Conventional radiocarbon dates	Calibrated age range (95%)	Carbon fraction	Pretreatment	Carbon mass (mg)
OZY416	3	2690 ± 70	2710.5–2991.5	SPAC	Hypy	0.031
OZY423	3	10,200 ± 140	11329.5–12477.5	Charcoal >63 um	ABA	0.014
OZX672	6	3840 ± 45	4096–4410	SPAC	Hypy	0.040
OZY131	6	4450 ± 30	4885.5–5284.5	Pollen	ABA	0.970
OZX765U2	6	4180 ± 140	4295.5–5270	Charcoal >63 um	ABA	0.015
OZX765U1	6	4120 ± 100	4405–4866	Charcoal >250 um	ABA	0.017
OZX765	6	4165 ± 35	4579–4830	Bulk organics	ABA	0.240
OZY417	12.5	4180 ± 70	4523.5–4856	SPAC	Hypy	0.083
OZY418	23	7040 ± 80	7694–8007	SPAC	Hypy	0.081
OZY333	32	6435 ± 30	7279.5–7424.5	SPAC	Hypy	0.310
OZY419	42	5990 ± 80	6638–7154.5	Cellulose	Gillespie	0.039
OZX673	43	8660 ± 40	9766–10,172	SPAC	Hypy	0.180
OZX766	43	7750 ± 35	8429.5–8593.5	Bulk organics	ABA	1.880
OZX674	67	15,240 ± 60	18,283–18,713	SPAC	Hypy	0.330
OZX767	67	9900 ± 50	11,205–11604.5	Bulk organics	ABA	0.290
OZY420	76	17,360 ± 160	20,562–21,391	SPAC	Hypy	0.142
OZY758	82	10,590 ± 100	12103.5–12750.5	Charcoal >63 um	ABA	0.044
OZY421	90	19,450 ± 80	23,166–23756.5	SPAC	Hypy	0.230
OZY422	105	17,960 ± 160	21,373–22262.5	SPAC	Hypy	0.125
Wk50327	114	23,549 ± 126	27,400–27,893	Bulk organics	H2O2 + ABA	0.576
OZX675	137	24,340 ± 190	27964.5–29,017	SPAC	Hypy	0.340
OZX768	137	15,560 ± 90	18,695–19,020	Bulk organics	ABA	0.137
OZX676	146	26,290 ± 260	30091.5–31029.5	SPAC	Hypy	0.055
OZY132	146	11,920 ± 40	13606.5–14009.5	Pollen	ABA	0.240
OZX769	146	16,270 ± 70	19485.5–19,860	Bulk organics	ABA	0.135
Wk50328	150	25,931 ± 167	29,940–30,735	Bulk organics	H2O2 + ABA	0.840
OZX677	162	27,100 ± 140	31,014–31,451	SPAC	Hypy	0.410

2.3. Pre-treatment

Pre-treatment of radiocarbon samples for pollen, cellulose, macrocharcoal, microcharcoal and bulk organics (standard method) followed the ANSTO protocols detailed below.

2.3.1. Pollen

Extraction of pollen was adapted from Bennett and Willis (2002). Three sediment samples were washed with 10% HCl and then passed through a 150 µm sieve. The smaller fraction was retained and then washed with 10% NaOH several times until the supernatant was clear. Subsequently, 20 mL of 40% HF was added and the sample left overnight. Following HF treatment, samples were first washed two times with 2 M HCl and finally with Milli-Q water, until neutral. Lithium heteropolytungstate (LST) with a specific gravity of 1.8 g/cm3 was used for density separation, with the floating fraction retained for examination under the microscope. The resultant pollen concentrate was dried overnight at 60°C.

2.3.2. Bulk organics (standard method)

Five sediment samples were processed at ANSTO following the ABA pre-treatment detailed in Hatté et al. (2001). First, visible contaminants (roots, rocks) were removed and then a wash with 2 M HCl was performed (to remove carbonates), followed by sequential washes of 0.5%, 1%, 2% and 4% NaOH, until the supernatant liquid was clear (to remove fulvic and humic acids). A wash with 2 M HCl to remove any atmospheric carbon dioxide (CO_2) absorbed during alkali treatment was performed, followed by three washes with Milli-Q water, and the samples were finally oven-dried at 60°C overnight.

2.3.3. Bulk organics (modified method)

Pretreatment of samples with hydrogen peroxide has proven to be effective in removing contaminant organic matter in radiocarbon dating samples (Chiu et al., 2005). In order to further test the ability of this approach for removing exogenous organic matter, two additional sediment samples were first pretreated with 30% hydrogen peroxide overnight at the Advanced Analytical Centre at James Cook University Cairns, freeze dried and then sent for standard ABA pre-treatment at the University of Waikato Radiocarbon Dating Laboratory. This procedure involved the removal of visible contaminants, following with the samples washed with hot HCl, then rinsed and treated with multiple hot NaOH washes. The NaOH insoluble fraction was treated with hot HCl, filtered, rinsed, and dried.

2.3.4. Macro and microcharcoal

A sample at 6 cm was pre-treated with 30% hydrogen peroxide for 2 h and then passed through 250 µm and 63 µm sieves. These two fractions were then examined under the microscope, and pieces of charcoal were recovered using tweezers (in the case of the >250 µm fraction) and an Eppendorf InjectMan® 4 micromanipulator (for the 63–250 µm fraction). Samples were then washed with 0.5% NaOH and 2 M HCl. Samples at 3 and 82 cm were pre-treated with 10% hydrogen peroxide and the pre-

treatment followed as above. Samples at 3 and 82 cm were more fragile than the other samples. The use of 10% hydrogen peroxide prevented further mass loss in the pretreatment process.

2.3.5. Cellulose

Extraction of cellulose was using the method 2chlorOx, adapted from Gillespie (2019). Three sediment samples were pre-treated with 1 M NaOCl/NaOH for 2 h and washed with 1 M HCl. The cellulose extraction procedure did not yield enough mass to be measured by AMS in the rest of the depths. The samples were then reacted with 1 M $NaClO_2$/HCl for another 2 h and then washed with 1 M HCl. This procedure was then repeated, and the samples were then washed with water three times. Finally, samples were oven-dried at 60°C overnight.

2.3.6. Hypy fraction

Fourteen sediment samples were pretreated using hydrogen pyrolysis (hypy) to isolate the pyrogenic carbon fraction (PyC; Ascough et al., 2009; Meredith et al., 2012). Initially, 30% hydrogen peroxide was added to the samples. The samples were left overnight then washed with 2 M HCl and freeze dried. Aliquots of each sample were then loaded with a catalyst and 20% MeOH/H2O solution, sonicated for 15 min and dried over a hotplate at 60°C. These samples were placed in the HyPy reactor, pressurized with hydrogen (H_2) to 150 bar with a gas flow of 4 L/min over 40 min. Finally, samples were washed for 2 h with 6 M HCl at 60°C.

2.4. Graphitisation and measurement

Samples were combusted at 900°C to convert them to CO_2, followed by graphitisation using the H_2/Fe method (Hua et al., 2001). Targets that yielded <10 µg of carbon were not analysed.

Carbon-14 measurement of all samples was undertaken by Accelerator Mass Spectrometry (AMS) on the VEGA and ANTARES accelerators at ANSTO (Fink et al., 2004; Wilcken et al., 2015) except for samples Wk50327 and Wk50328, which were processed at the University of Waikato Radiocarbon Dating Laboratory.

All analyses are also potentially subject to the introduction of contamination during pretreatment and further processing to graphite. Bird et al. (2014) have analysed the probable contamination in ABA and hypy procedures on pyrogenic carbon and found it negligible in comparison with combustion-graphitisation stage, especially for small size samples. Other studies (e.g. Orr et al., 2021; Haig et al., 2020; Turney et al., 2021) support this conclusion. The ANSTO combustion-graphitisation blank at ANSTO laboratories in routinely determined for every studied batch of samples as described in Bird et al. (2014), and the final results of this study were corrected based on these blank measurements. The sedimentary record of Sanamere Lagoon does not extend back to a time beyond the limit of radiocarbon. The absence of definitively radiocarbon-dead material from the Sanamere core, that is, from a comparable matrix to the samples analysed, precluded the direct determination of a processing blank for each pretreatment technique directly on the Sanamere Lagoon material.

2.5. Calibration

All samples were calibrated to calendar years (cal BP) using the Oxcal Program and the IntCal13 calibration curve (Reimer et al., 2013) with 0 calibrated years before present representing 1950 AD. IntCal13 was used rather than SHCal13. IntCal13 was used due to the influence of Northern Hemisphere air masses on the Tropical North of Australia, when the Inter Tropical Convergence Zone moves southwards during the Australian-Indonesian summer monsoons (Hogg et al., 2013). The rbacon R package (Blaauw and Christen 2019) was used to develop the age models for the core.

3. Results

A total of 27 radiocarbon dates were obtained for 17 different depths along the core. The final mass of graphitized carbon ranged from 31 μg (OZY416; Hypy) to 1.88 mg (OZX-766; ABA) (Table 1). Hypy targets were generally the smallest in carbon mass (31–330 μg), and ABA treated bulk organics the largest (135–1880 μg).

Overall, the calibrated ages ranged from 2710–2991 cal BP at 3 cm (hypy fraction) to 31,014–31,451 cal BP at 162 cm (hypy fraction), spanning 28,741 years in total. As discussed above, ages were obtained for more than one fraction from six depths. Table 1 shows the results from the radiocarbon dating measurements and the calibrated results. From the bottom of the core to 82 cm, the availability of fractions to compare was reduced to bulk organics, the hypy fraction and pollen concentrates, as these were the only fractions to yield sufficient carbon to be analysed by AMS. Therefore, charcoal fractions >63 μm and cellulose could not be analysed below 82 cm. From the three depths originally processed with the cellulose pre-treatment method only one sample (OZY419) retained enough carbon to be measured, given the aggressiveness of the pretreatment.

The pollen result at 146 cm showed the largest age reversal overall, 16,752 cal years older than the hypy date at the same depth. Two pairs of hypy ages also showed age reversals, with the smallest (a difference of 505 cal years) between the dates at 23 and 32 cm and the largest between the dates 90 and 105 cm (a difference of ~1600 cal years). The only cellulose sample yielded a younger date in comparison to what was expected for that depth based on the other results (Table 1, Fig. 6).

The difference between the ages from different fractions generally increased with depth (Table 2). The only section of the core where all dating results overlap (except for the pollen concentrate) is at 6 cm, while the other five depths showed large differences between the results from different fractions, with the largest offset at 146 cm (a difference of ~16,700 years), between hypy and pollen. The bulk organic fractions pretreated with standard ABA procedures also differed significantly from the hypy fraction (being up to ~9600 years younger than the former). Although not from the same depths (but within 10 cm), the modified ABA pretreatment (samples Wk50327 and Wk50328) yielded results that aligned closely with the hypy results at the two depths where they could be compared.

In four out of six cases (the deepest samples), hypy yielded the oldest date from those available at that depth, and in two cases (the shallowest samples), hypy yielded the youngest results. Bulk organics (standard ABA) yielded the youngest result for four depths (43, 67, 137 and 146 cm). The pollen results were inconsistent, yielding the oldest and youngest dates at 6 cm and 146 cm, respectively (Table 2).

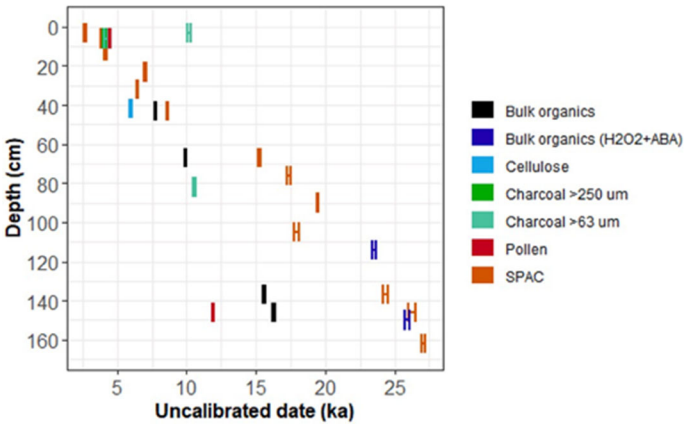

Fig. 6. Uncalibrated ages by depth and carbon fraction along the Sanamere sediment core.

Table 2. Offset, minimum and maximum calibrated ages by depth.

Depth (cm)	Minimum age (cal BP)	Carbon fraction (min)	Maximum age (cal BP)	Carbon fraction (max)	Offset	Number of dates
3	2710	Hypy	12,477	Charcoal >63 um	9052	2
6	4096	Hypy	5284	Pollen	832	5
43	8429	Bulk organics	10,172	Hypy	1457	2
67	11,205	Bulk organics	18,713	Hypy	7093	2
137	18,695	Bulk organics	29,017	Hypy	9633	2
146	13,606	Pollen	31,029	Hypy	16,752	3

4. Discussion

4.1. Assessment of reliability

All ages returned (except at 6 cm) following hypy pre-treatment are considerably older than dates from comparable levels for other carbon fractions. This finding is consistent with the results obtained from the radiocarbon dates from organic spring deposits in northwest Australia, where hypy ages were found to be older than other fractions, but were also considered more reliable based on their more consistent age-depth relationship within each core (Field et al., 2018). Hypy reduces labile organic matter to volatile products (Ascough et al., 2009) and removes >92% and up to 100% of all labile carbon (Bird et al., 2014). Hypy represents the carbon fixed by pyrolysis at the time of a burning event, therefore the 'indigenous' or core component of the original charcoal (Ascough et al., 2009, 2010).

The hypy dates at 23 cm (~7700–8000 cal yr BP) and 32 cm (7300–7400 cal yr BP) show an age reversal of 506–870 years (Fig. 5). These dates are from the section of the core that has high organic content, high pyrogenic carbon mass accumulation rates and large fluctuations in the normalized titanium counts (range 0.02–0.45) (Fig. 3). Both changes in erosion (as evidenced by clastic input) and high organic input at this time suggest that some reworking could have occurred during this period, which coincides in timing with the flooding of the adjacent continental shelf in response to an increase in sea level during the early Holocene in north Australia (Sloss et al., 2018; Chivas et al., 2001; Yokoyama et al., 2001; Reeves et al., 2013), and therefore to a period of wetter climate, with potentially more intense seasonal rainfall events.

More overland transport of soil material from the catchment, may well have transported 'old' charcoal into the lake at this time. A second age reversal is present between 90 cm and 105 cm (1100 years) in the hypy results, with the only apparent sedimentological indication of change being a slight decrease in the titanium counts down-core between these two depths. It is possible that the date at 105 cm exhibits an incomplete removal of exogenous carbon, the main issue identified when applying hypy pre-treatment to charcoal formed at 400°C or below (Bird et al., 2014).

The results from fractions other than hypy at the same or comparable depths (except at 6 cm) were uniformly younger than the hypy results., and generally considerably younger that could be explained by introduction of contaminant carbon during pretreatment or graphitisation. As well as the possibility of the physical mobility of material through the relatively short sediment column, it is likely that the differences between the ages of fractions at 43, 67, 137 and 146 cm are the result of unremoved contamination by carbon of a different, generally younger age in the bulk organics (standard treatment) and pollen fractions. The mobilization of materials and therefore, the contamination of samples with exogenous carbon is made more likely in the Sanamere sequence because the entire 31,000 years of accumulation is represented in only 172 cm of sediment. The top 43 cm contains high proportions of water, which also facilitates the mixing of materials by physical translocation and/or solubilisation. The high water content of this section of the core (>40%) allowed for components to mix downwards and upwards.

The results suggest that, for most samples in this study, the standard ABA pretreatment was ineffective in removing younger contamination from the bulk organic fraction. An inbuilt 'reservoir' age associated with a period of storage in the lake catchment may have biased the hypy results towards older ages by an unknown amount. However, it is unlikely that this could cause such large offsets between the hypy dates and dates from the other fractions. If there was a reservoir offset relating to a period of storage of up to ~9500 years in the catchment, this would manifest itself in the hypy dates throughout the sequence, whereas what is observed is a generally smaller offset up the core. Indeed, the uppermost hypy date is younger than that of the other fractions, implying the circulation of carbon of an apparent age representative of all carbon in the sediment through the relatively thin sediment sequence, such that dates high in the sequence are biased towards older ages and dates deeper in the sequence are biased towards younger ages.

Further evidence that there is no substantial reservoir effect on the hypy dates is provided by the observation that the two samples subject to the modified bulk organic pretreatment, where peroxide was used before standard ABA, yielded results close to those obtained from the hypy fraction. Except for the anomalous charcoal result at 3 cm, and the slightly older pollen date at 6 cm, all dates from all fractions and pretreatments between 23 cm and the top of the core overlapped (when they derived from the same depth) and did not show any age reversals. From 23 cm to the bottom, large offsets between samples from the same depth and age reversals were observed.

The increasing magnitude of the difference between hypy and the other fractions ages with depth/age is consistent with the younger samples at each depth having between 2 and 5% unremoved equivalent modern contamination (Wood 2015), or more contamination with an older aggregate apparent age. The results strongly indicate the presence of unremoved exogenous carbon contamination in some of the fractions. For example, the largest offset between two fractions was found at 146 cm (~16,700 years) between pollen and hypy, which suggests that pollen from higher depths mobilized down the core

and/or the concentrate contained exogenous younger materials. The two pollen concentrate samples available show inconsistent results, as has been found in previous studies being younger (Field et al., 2018) or older (Neulieb 2013; Fletcher et al., 2017), with the sample at 6 cm having the oldest date compared with the other fractions at the same depth, while the sample at 146 cm is the youngest compared to the bulk organics and hypy results. When inspected under the microscope, these samples appeared to comprise ~50% pollen, with the rest of the residue being other plant material and charcoal. This addition of heterogeneously sourced materials could have added exogenous carbon contamination resulting in the aberrant results. Other studies also have identified anomalous, generally younger ages derived from pollen concentrates (May et al., 2018; Clymo and Mackay 1987) due to the high physical mobility of small particles but also the high porosity of the pollen walls, which can absorb and accumulate exogenous carbon (Kilian et al., 2002). The existence of clearly anomalously young pollen dates strongly suggests that there is potential for contamination by young carbon in all the organic fractions.

Large offsets (up to ~ 9600 years) were also found between bulk organics and hypy, with the former showing younger ages. This finding is consistent with previous studies which found that bulk organics pretreated with standard ABA can yield anomalously young dates (Wang et al. 1996; Wüst et al., 2008; Pessenda et al. 2001). Although measured at 42 cm (not 43 cm as the other two fractions), the cellulose extraction yielded an age younger compared to what was expected from the bulk organics and hypy age-depth relationship.

The hypy dates yielded the oldest dates and the most consistent internal chronology with only two minor age reversals (at 23 and 90 cm), with the caveat that some previous studies suggest that hypy dates could be biased to older ages due to potential for an inbuilt 'reservoir' age (Field et al., 2018).

While the results from micro- and macro-charcoal were expected to be tested against each other along the entire core to better understand and differences in their sources, availability was limited below 82 cm, indicating comminution into particles <63 µm, measurable only as the hypy fraction. As the age ranges obtained for both overlapped at 6 cm, the possibility that they represent different sources is low. The charcoal samples (>63 µm fraction) at 3 cm and 82 cm showed older and younger dates (respectively), compared to the age/depth relationships expected from the relationship determined by samples treated by hypy or modified ABA. It is possible that the incomplete removal of organic contaminants by the standard ABA method caused this offset. Given its large surface area and porosity, charcoal is known to absorb exogenous carbon, which can undergo irreversible reactions with the charcoal surface. Charcoal is also suitable for microbial colonization, and microbial carbon cycling could also lead to the incorporation of exogenous carbon (Bird, 2007).

4.2. Developing a robust chronology

While two age-depth models were built with the results from bulk organics (ABA) and hypy dates (Fig. 7), the model including only the hypy dates was the most consistent with the stratigraphic and sedimentologic changes identified in the Sanamere sequence. This evidence is indirect, and not as robust as independent evidence derived, for example, from varved sequences that are available temperate environments, but this again is a common issue with tropical lacustrine records.

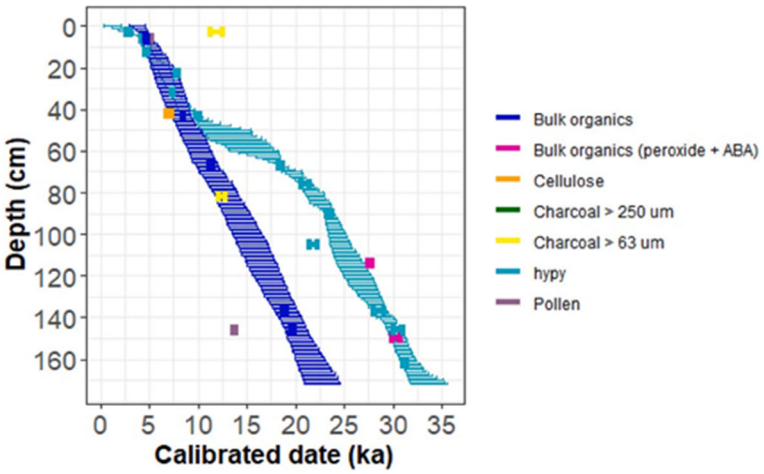

Fig. 7. Age-depth models (95% confidence error bars) constructed using Bayesian age modelling with rbacon package in R. Chronology based on Hypy is represented in turquoise and chronology based on bulk organics in blue, along with the calibrated dates from additional carbon fractions, none used in the construction of either chronology.

The selected model included 13 hypy samples (Table 1), with all but two dates (23 cm and 105 cm) fitting within the 95% confidence interval. The modelled ages ranged between 400 (3 cm) and 33,000 cal yr BP (162 cm). The peroxide + ABA pretreated bulk organics samples also agree reasonably well with the hypy-only model, including one of the dates fitting within the error range, and the other yielding a slightly older date (by ~300 years). This further supports the reliability of the hypy results and bolsters the conclusion that old contamination has not affected the hypy results to any significant degree.

The timing of changes in sedimentation rate inferred using the hypy model is consistent with the observed changes in sedimentology observed at 43 cm and 71 cm, which are the upper and lower bounds of two of the stratigraphic units observed in the Sanamere sequence (Fig. 7). Between 71 and 172 cm, there are no major sedimentological changes, other than the inclusion of more coarse clastic fragments at 140 cm down-core. The charcoal samples (both particle sizes) were consistent with hypy dates at 6 cm, but not down-core. This inconsistency suggests that dates from unique pieces of charcoal pre-treated only by ABA could lead to biased results. Again, the hypy samples appear more reliable, as total pyrogenic carbon content measured using the hypy technique is less likely to be biased by individual charcoal fragments, each of which may have some (unknown) residence time prior to deposition in the lake sediment and/or degree of unremoved contamination. Evidence from proxies measured at the lagoon (Rivera-Araya, unpublished data) suggest the occurrence of environmental changes between 9700 cal BP and 18,000 cal BP based on the hypy chronology. For example, between 12,800 cal BP and 9700 cal BP, the trends in the biological and geochemical indicators suggest the existence of episodic wet events derived from an increase in rainfall associated with a stronger monsoon. From 10,800 cal BP, increases in organic matter content and diatom diversity suggest increased terrigenous organic input, resulting from an increase in local biomass. Both of these timings are consistent with what is generally known about the broad timing of major environmental changes following the LGM into the Holocene in northern Australia.

Although the age-depth model built with the standard ABA pretreated bulk organics samples had no age-reversals, and a range between 4100 and 22,300 cal BP, the modelled ages were not consistent with the stratigraphic changes in the Sanamere sequence (in contrast with the hypy model). Additionally, none of the pollen or cellulose samples fit within the age-model derived from the organics or bore any relationship to the observed stratigraphic units in the sequence, regardless of depth. These inconsistencies between pollen, cellulose and bulk organics dates suggest that results from any of these fractions are likely biased by unremoved contamination or physical translocation of younger material to a variable degree.

Additional evidence to support the choice of the hypy model is its consistency with the timing of environmental events. For instance, the layer 65–71 cm matches the time period between 18,000–20,000 cal BP, identified as a period of environmental change in regional studies. Strong evidence from palaeoenvironmental studies has been found to support the dominance of cooler and drier conditions during this period in tropical Australasia (Reeves et al., 2013; Turney et al., 2006; Burrows et al., 2016). In contrast, this layer was modelled as 11,000–11,600 cal BP based on the age model derived from the bulk organics (ABA) curve. Moreover, the hypy chronology is consistent with the most likely timing for the formation of the lake ~33,000 cal BP. In this instance, a collapse in the underlying laterite karst formed a depression, as a result of lowered water tables that would accompany the rapid drop in sea level and drier conditions moving from MIS3 into MIS2 (Xu et al. 2019).

Finally, the results from this study highlight the importance of comparing the results from different fractions (when using radiocarbon dating) or contrasting the dates from multiple dating methods when building to support the construction of palaeoenvironmental proxy records and comparing results with those derived from other dating methods. Caution should be exercised when interpreting chronologies derived from bulk organic carbon fractions. Although the chronology obtained using bulk organics with the standard ABA-pretreatment method appeared to be consistent and free of age reversals, hypy provides the most robust chronology for the Sanamere Lagoon sequence and indicates the bulk organic chronology to be incorrect.

5. Conclusions

The selection and pre-treatment of a reliable organic fraction from which to acquire radiocarbon dates is fundamental to the development of accurate and precise chronologies. Sampling from tropical lakes is particularly challenging given the adverse preservation conditions and potential diagenesis in these hot environments. This paper examines and quantifies the differences between the radiocarbon date results and reliability from different carbon fractions and pretreatments from the same depths from Sanamere Lagoon, a tropical lake sediment core (1.72 m long) located in north Australia. Six different organic fractions (bulk organics, pollen concentrate, cellulose, stable polycyclic aromatic carbon (SPAC), charcoal >250 μm and charcoal >63 μm), for a total of 27 radiocarbon dates, were compared in six different depths along the core. Acid-base-acid (ABA), modified ABA (30% hydrogen peroxide + ABA), 2chlorOx (a novel cellulose pre-treatment method) and hydrogen pyrolysis (hypy) were used to pre-treat the correspondent organic fractions.

The chronology spanned ~29,000 years.The SPAC fractions pretreated with hypy yielded older ages compared to all other fraction in most cases, while bulk organics consistently yielded younger ages. The

magnitude and consistency of the offsets and the physical and chemical properties of the tested organic fractions suggest that SPAC is the most reliable fraction to date in tropical lake sediments and that hypy successfully removes contamination sourced from exogenous carbon.

The reasons behind the range of dates obtained using different techniques from the same stratigraphic level are multiple and, to some degree, site specific. The comparatively low sedimentation rate and low degree of compaction of the Sanamere Lagoon sediments means that carbon of very different age in dissolved and particulate form is in close proximity in the sediment sequence. Hence translocation of particulate or dissolved material up or down the sequence will have a comparatively large effect on the apparent age of the sample. In addition, some materials dated have potentially variable inbuilt radiocarbon ages, and each of the pretreatments applied is subject variable degrees of bias due to incomplete removal of younger/older contaminants.

The final Sanamere Lagoon chronology was built exclusively using samples pretreated with hypy, an approach that removes contamination with a high degree of certainty (Orr et al., 2021) and where the main source of uncertainty is the degree to which a potentially variable inbuilt reservoir age could bias the results to older ages, but probably by less than a few hundred years. The chronology based on hypy was also the most consistent with the stratigraphic changes observed in the core.

The record spans the last ca. 33,000 cal BP, based on a robust chronology established by comparing age-models derived from multiple carbon fractions (pyrogenic carbon derived from hydrogen pyrolysis – hypy fraction -, macrocharcoal, microcharcoal, pollen concentrates, bulk sediment and cellulose). The age-depth model derived from the hypy fraction was the most reliable and consistent with the stratigraphic changes identified along the core, removing allochthonous carbon and post-depositional contamination.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary material. Extracted radiocarbon samples with insufficient carbon after combustion.

Lab Code	ID	Carbon fraction	Pretreatment	Final weight
Y131U1	32	Charcoal <63 um	ABA	1.□g
Y133	171	Charcoal >250 um	ABA	3.67□g
Y134	171	Charcoal >63 um	ABA	1.52□g
Y135	171	Charcoal <63 um	ABA	0.46□g
Y136	94	Charcoal >250 um	ABA	3.35□g
Y137	94	Charcoal >63 um	ABA	1.05□g
Y138	94	Charcoal <63 um	ABA	6.03□g
Y139	94	Pollen	ABA	1.71□g
Y331	170	Charcoal <63 um	ABA	NA
Y332	282	Charcoal <63 um	ABA	NA
Y330	71	Charcoal <63 um	ABA	NA
NA	56	PyC	Hypy	NA
NA	56	Charcoal <63 um	ABA	NA

NA	32	Charcoal <63 um	ABA	NA
NA	171	Pollen	ABA	NA
NA	94	Pollen	ABA	NA