

Research paper

Use of heavy liquid density separation to remove pyrite from sediment samples for radiocarbon dating

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ABSTRACT

Sample preparation protocols for concentrating organic material from sediments for radiocarbon dating often include a large number of steps and the use of hazardous chemicals. Thus, these protocols are often problematic for pollen-poor sediments as material can get lost or may become degraded. Pyrite in samples for radiocarbon dating hinders an effective graphitisation process and thus needs to be removed during sample preparation. Standard protocols require the use of nitric acid, a strong oxidant that corrodes organic material. Therefore, the use of nitric acid needs to be avoided when preparing pollen-poor but pyrite-rich sediments.

We present a method that minimises the sample preparation steps by replacing acid treatment with heavy liquid separation. Using non-toxic LST at a density of 2.0 g cm^{-3} is shown to be effective for separating the organic fraction from sulphides, silicates and carbonates. We applied this method to pyrite-rich and pollen-poor sediments from Western Australia that had previously been problematic to date. The successful AMS radiocarbon dating of all samples pre-treated with LST demonstrates the effectiveness of this method.

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1. Introduction

Deposits containing sulphides such as pyrite (FeS_2) are common in coastal settings worldwide where pyrite forms under reducing conditions during the bacterial breakdown of organic material in the sediment (Dent and Pons, 1995). Such sediments commonly form in tidal swamps, such as mangroves, and coastal lagoons. The radiocarbon dating of these deposits using bulk material can be problematic for two reasons:

- 1) When sulphidic sediments are subjected to aerobic conditions, strong acid is released which can lead to the corrosion of organic material (Dent and Pons, 1995). Sediments affected by this acid sulphate soil formation are often poor in organic material which means that large sample volumes need to be processed to obtain enough material for radiocarbon dating (Dent and Pons, 1995; Jaffé et al., 2013).
- 2) Pyrite releases sulphur oxides when combusted which can hinder the reduction of the carbon dioxide to graphite. During radiocarbon dating, organic samples are often converted to

graphite for measurement in an accelerator mass spectrometer (AMS) by reacting carbon dioxide, produced by combustion of the sample, with hydrogen over an iron catalyst (Vogel et al., 1984). The carbon dioxide is collected cryogenically. Unfortunately, sulphur dioxide has a similar freezing point to carbon dioxide (-72°C versus -78°C) and is transferred to the reaction vessel where it is reduced to elemental sulphur, coating the iron catalyst and preventing the reduction of carbon dioxide to graphite (Pohlman et al., 2000; Thomsen and Gulliksen, 1992).

Ideally, short-lived plant remains, such as leaves or twigs, should be used to date sediment cores (Tornqvist, 1992; Walker, 2005). In many contexts this type of material is rare, and so charcoal, potentially affected by the 'old-wood' effect, shells, often affected by a radiocarbon reservoir effect, or roots, which can introduce large vertical errors, may be dated (e.g. Crowley and Gagan, 1995; Kermode et al., 2013; Woodroffe et al., 1985). In the absence of macroscopic organic remains, pollen concentrates have been shown to provide reliable radiocarbon dates in some cases (Brown et al., 1989; Newnham et al., 2007; Prior and Chester, 2001; Vandergoes and Prior, 2006; Zhou et al., 1997), although problems do remain with pollen concentrates being both erroneously old (Kilian et al., 2002; Li et al., 2014) and erroneously young (e.g. Neulieb et al., 2013; Vasil'chuk et al., 2005), even where individual

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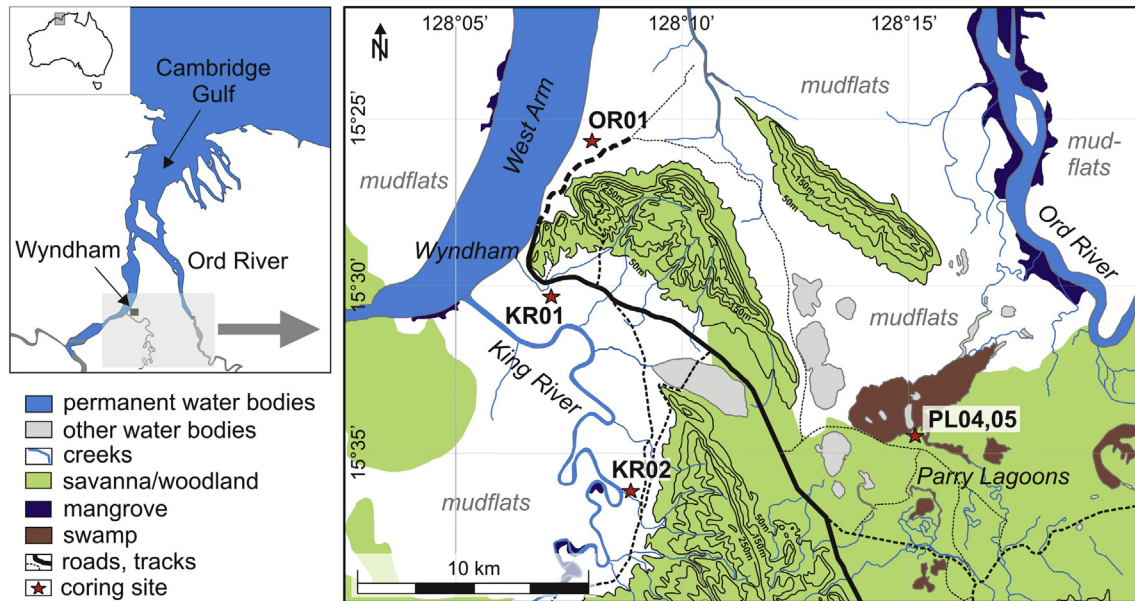


Fig. 1. Overview of the coring sites. The left-hand map shows the study area within the regional context. The right-hand map shows the coring locations and major landscape and vegetation units in the Wyndham region (adapted from Topographic Map of Mt Erskine and Beard, 1979).

pollen grains are selected (Mensing and Southon, 1999; Neulieb et al., 2013). It is possible that these concentrates are affected by ancient pollen from old sediments, reworking of sediment and possibly contamination during storage. In addition, unless hand picked these pollen concentrates contain a mixtures of pollen with varying amounts of microcharcoal and other small organic particles and are therefore subject to the ‘old-wood’ effect. Despite these problems, where macroscopic organic remains are absent this fraction provides the only opportunity to produce a chronology, and multiple radiocarbon dates on samples from the same core can increase confidence in this sample type. As a result, considerable research has been undertaken to improve pre-treatment protocols.

The methods proposed for concentrating pollen from sediment usually include sample treatment with a number of chemical such as NaOH, HCl, KOH, HNO₃ and hot HF or rely on manual separation (Brown et al., 1989; Eriksson et al., 1996; Howarth et al., 2013; Mensing and Southon, 1999; Newnham et al., 2007; Piotrowska et al., 2004; Prior and Chester, 2001; Regnell, 1992; Regnell and Everitt, 1996; Tennant et al., 2013; Vandergoes and Prior, 2006; Xu et al., 2013). In these protocols, treatment with dilute nitric acid is recommended for the removal of sulphides such as pyrite. Nitric acid, however, reacts with organic material and can degrade the sample substantially. This implies that it should best be avoided, in particular when the sample is poor in organic material.

The use of heavy liquid in environmental and geological research dates back to at least the early 20th century (Strutt, 1906). In the late 1980s and 1990s, new and non-toxic heavy liquids such as sodium polytungstate and lithium heteropolytungstate have become available (Munsterman and Kerstholt, 1996; Patrick and Patrick, 1997). Most commonly heavy liquids are used for

- the density separation of heavy minerals in sedimentological and petrographical research (Alexanderson and Murray, 2012; Krumbein and Sloss, 1951; Sakoda et al., 2010; Tomkins et al., 2003),
- the preparation of sediment and soil samples for palynological, palaeontological and biological analysis (Bolch, 1997; Faegri and Iversen, 1989; Mallinson et al., 2010; Morley et al., 2004; Munsterman and Kerstholt, 1996; Riding and Kyffin-Hughes, 2004; van Leeuwen et al., 2000; Wang et al., 2001),

- the separation of tephra for geochemical analysis and tephrochronology (Blockley et al., 2005) and
- the separation of carbonates and other particles from soil and sediment samples for radiocarbon dating (Douka et al., 2010; Howarth et al., 2013; Newnham et al., 2007; Piotrowska et al., 2004; Prior and Chester, 2001; Regnell and Everitt, 1996; Vandergoes and Prior, 2006; Zhou et al., 1997).

Particle separation by heavy liquid uses the specific gravity (and thus buoyancy) of the particle that needs to be isolated from its surrounding matrix. Following sample preparation (crushing, sieving etc.), the sample is added to the heavy liquid (Krumbein and Sloss, 1951). The gravity (or density) at which the heavy liquid is set depends on the particle that needs to be isolated. For example, to separate heavy minerals out of rock or sediment samples, the heavy liquid needs to have the specific density of 2.9 g cm⁻³. Lighter particles such as quartz and feldspar (specific gravity around 2.6–2.7 g cm⁻³) will float whereas heavier particles such as zircon (4.6 g cm⁻³), magnetite (5.2 g cm⁻³) and tourmaline

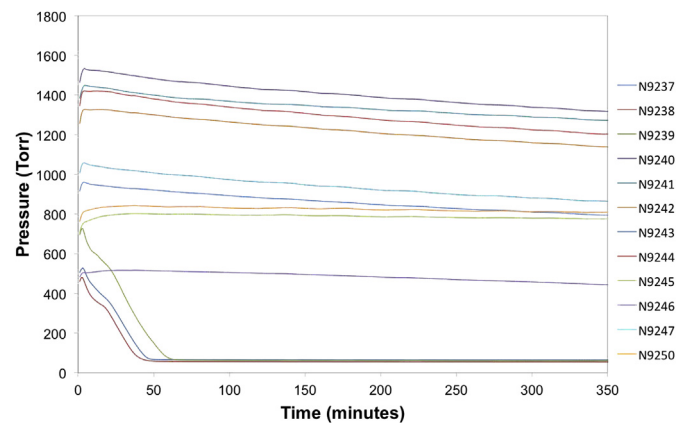


Fig. 2. Pressure within reaction vessels during the first attempt at graphitising the pollen concentrates. During this reaction CO₂ and H₂ are heated to 570 °C over an iron catalyst to form graphite. The three lowermost samples have graphitised, with the pressure dropping to <100 Torr. The remaining samples have not graphitised.

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