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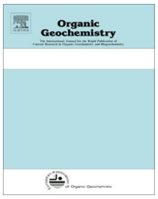

An irrigation experiment to compare soil, water and speleothem tetraether membrane lipid distributions

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ABSTRACT

Measurement of glycerol dialkyl glycerol tetraethers (GDGTs) preserved in speleothems offers a potential proxy for past temperature but, in general, their origin is unknown. To understand the source of speleothem GDGTs, we undertook an irrigation experiment to activate drip sites within a hydrogeochemically well characterised cave. The cave drip water was analysed for GDGTs, inorganic elements (major ions and trace elements), stable isotopes and dissolved organic matter concentration and character. Published speleothem GDGT records from the site have been observed to be dominated by isoprenoid GDGTs and interpreted as deriving from in situ microbial communities within the cave or vadose zone. The drip water in our irrigation experiment had a GDGT distribution distinct from that of soil and speleothem samples, providing direct evidence that the distinctive GDGT signature in speleothems is derived from a subsurface source. Analysis of GDGTs in this context allowed further elucidation of their source and transport in cave systems, enhancing our understanding of how they might be used as a temperature proxy.

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1. Introduction

There has been increasing interest in dissolved organic matter (DOM) in cave drip water and the potential use of organic markers in speleothems as paleoclimate proxies (Blyth et al., 2008; Fairchild and Baker, 2012). While initial studies focussed on fluorescent OM (Baker et al., 1997), subsequent investigations have focussed on the potential of lipid biomarkers (Xie et al., 2003; Blyth et al., 2007, 2011; Rushdi et al., 2011), $\delta^{13}C$ measurements of OM (Blyth et al., 2013a,b; Li et al. 2014), lignin phenols (Blyth and Watson 2009; Blyth et al., 2010) and trace elements associated with organic colloids (Hartland et al., 2012). Lipids such as *n*-alkanes have been shown to record vegetation and land use change (Blyth et al. 2007, 2011) and glycerol dialkyl glycerol tetraethers (GDGTs) have shown potential as a palaeotemperature proxy (Yang et al., 2011; Blyth and Schouten, 2013; Blyth et al., 2014).

GDGTs are widely used as a palaeoenvironmental proxy, predominantly for temperature reconstruction from lake and ocean sediments and soils (Schouten et al., 2013). They are divided into two classes. One, containing isoprenoid alkyl moieties (iGDGTs), is produced by Crenarchaeota, Thaumarchaeota and Euryarchaeota; the second comprises methyl branched alkyl chains with 0–2 cyclopentane moieties (brGDGTs) and is believed to be produced by bacteria (Schouten et al., 2013). These branched compounds were originally considered to be associated with soil derived terrigenous input, although recent studies have now found that they can also be produced in situ in aquatic environments (for a review see Schouten et al., 2013). The brGDGTs are more abundant than iGDGTs in soil (e.g. Weijers et al., 2006; Naeher et al., 2014). Ajioka et al. (2014) investigated GDGT depth profiles in soil

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in the Lake Biwa basin and found that the maximum brGDGT concentration occurred in the surface organic ('O') horizon, decreasing with depth. In contrast, iGDGTs increased with depth. Huguet et al. (2014) demonstrated that, in a peat soil, brGDGTs had a turnover of at least 1 yr and that the source organisms rapidly (between 3 months and 1 yr) adjusted their membrane lipid composition to their environment. Weijers et al. (2010) used a natural labelling experiment using a change between C_3 and C_4 crop types to determine a turnover time of ca. 18 yr for brGDGTs in arable soil.

In soil, branching ratios have been shown to relate to temperature and pH (Weijers et al., 2007; Peterse et al., 2012). Blyth and Schouten (2013) and Blyth et al. (2014) have also demonstrated that a temperature signal is contained within GDGTs preserved in speleothems. In that case, the GDGTs were dominated by iGDGTs, despite the drip water supplying the speleothems initially having a soil source and, in most cases, both the iGDGTs and brGDGTs had distinct compositions compared with overlying soil (Blyth et al., 2014). Blyth et al. (2014) hypothesised that the GDGTs in speleothems derive predominantly from a source further down the transport pathway from the soil, although comparison of speleothems and soils could not distinguish whether the source was in situ in the cave itself or derived from the vadose zone of the limestone.

To understand the processes determining the GDGTs in speleothems and to examine sources and transport of the compounds during a controlled recharge event, we used an artificial irrigation experiment. The site had been used for an artificial irrigation experiment in January 2013 that determined the karst hydrogeochemical processes occurring from surface to cave (Rutlidge et al., 2014). This and subsequent irrigation experiments in January and June 2014 were undertaken to improve understanding of various cave processes and potential speleothem proxies. These included heat transport processes (Rau et al., 2015), evaporative cooling of drip water (Cuthbert et al., 2014b), water isotope evaporation and vadose zone flow paths (Markowska et al., 2016) and OM characterisation using liquid chromatography – organic carbon detection (LC-OCD; Rutlidge et al., 2015). Here, we report the GDGT results obtained from the January 2014 irrigation experiment.

2. Material and methods

2.1. Study site

Cathedral Cave at Wellington Caves Reserve, NSW, Australia (32 37′S; 148 56′E) was the location for our irrigation experiments (Fig. 1). It formed within massive Devonian limestone (Johnson, 1975) and the irrigation site is close to the cave entrance. Overlying the limestone is a thin layer of red-brown soil, comprising clays, iron oxides, fine quartz sand and calcite nodules (Frank, 1971), with an aeolian contribution (Hesse and McTanish, 2003). The site is within a temperate semi-arid region, with a mean annual precipitation of 619 mm (1956–2005) and pan evaporation of 1825 mm (1956–2005) recorded nearby at Wellington (Bureau of Meteorology, 2015). There is a significant seasonal temperature variation, with the monthly mean maximum ranging from 15 °C

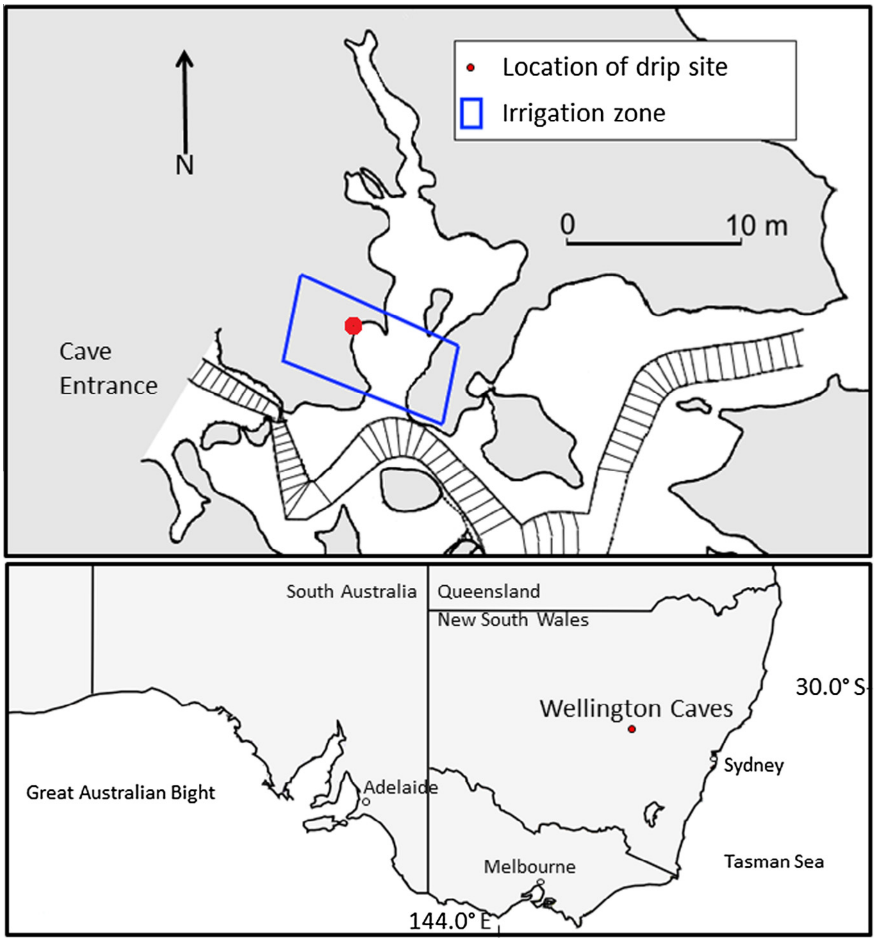

Fig. 1. Plan view of study site at Cathedral Cave, Wellington Caves (top), with a boxed area indicating where surface irrigation was performed, and location of the drip site (adapted from Sydney University Speleology Society survey map 2006–2007). Location of the study site in New South Wales in southeastern Australia (bottom).

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