



## Loss and gain of carbon during char degradation



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### ARTICLE INFO

#### Article history:

Received 28 March 2016

Received in revised form

23 November 2016

Accepted 18 December 2016

Available online 26 December 2016

#### Keywords:

Biochar

Radiocarbon

Carbon-isotope

Degradation

Carbon sequestration

### ABSTRACT

We report results of a study examining controls on the degradation of chars produced at 300, 400 and 500 °C from radiocarbon-free wood, deployed for three years in a humid tropical rainforest soil in north Queensland, Australia. The chars were subjected to four treatments (i) no litter (ii) covered by leaf litter, (iii) covered by limestone chips to alter local pH, and (iv) covered by limestone chips mixed with leaf litter. Radiocarbon, stable isotope and proximate analyses indicate significant ingress of exogenous (environmental) carbon and mineral material, strongly correlated with loss of indigenous (char) carbon from the samples. While indigenous carbon losses over three years were generally <8% for the char produced at 500 °C char under any treatment, chars formed at lower temperatures lost 5–22% of indigenous carbon accompanied by ingress of up to 7.5% modern exogenous carbon. The data provide clear evidence of a direct link between the ingress of exogenous carbon, likely at least partly due to microbial colonization, and the extent of char decomposition. Failure to account for the ingress of exogenous carbon will lead to a significant under-estimate of the rate of char degradation.

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## 1. Introduction

Pyrogenic carbon (PyC; soot, char, black carbon) is the product of incomplete combustion of organic material during biomass burning, controlled pyrolysis (biochar) and fossil fuel consumption. Natural and anthropogenic PyC is pervasively distributed throughout the terrestrial and marine environments, atmosphere and the cryosphere (Bird et al., 2015; Santín et al., 2016).

PyC is significant in the global carbon cycle as a potentially recalcitrant form of carbon that is stable in the environment over long timescales (centuries to millennia; Preston and Schmidt, 2006; Zimmerman, 2010; Singh et al., 2012). The environmental stability of PyC has attracted attention as a means of sequestering atmospheric carbon dioxide into the soil in the form of biochar, thereby reducing the global warming impacts of anthropogenic CO<sub>2</sub> emissions (e.g. Jeffery et al., 2015). While it is clear that a

component of PyC exhibits a high degree of resistance to degradation in the environment, it is also clear that there is a PyC 'degradation continuum' (Bird et al., 2015), with the rate of degradation of PyC determined by complex interactions between, at least, the feedstock type and temperature of pyrolysis on the one hand, and the environmental conditions to which the PyC is exposed after production in terms of parameters such as ambient temperature (Zimmermann et al., 2012), moisture availability and soil type (texture, pH, organic carbon and microbial activity).

There is a growing body of evidence that chemical modifications of the surface of the charcoal occur rapidly upon environmental exposure (<1yr) and on longer timescales PyC can undergo significant environmental degradation, (eg. Cheng and Lehmann, 2009; Ascough et al., 2011a, 2011b), ultimately leading to virtually complete loss of macroscopic PyC (Bird et al., 1999a,b). Degradation is usually framed in terms of loss of carbon by mineralization and/or dissolution. However, it also appears to be the case that at least in the short term, exogenous carbon and mineral material can become strongly associated with PyC surfaces and pores (Jaafar

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et al., 2015a; b; Lehmann et al., 2011). PyC has known ability to adsorb organic compounds from the environment to varying degrees (e.g. Mohan et al., 2014) and indeed biotic degradation of PyC implies at least an initial addition of exogenous carbon during microbial colonization.

It seems likely that PyC degradation is the result of continual carbon exchange between PyC surfaces and the local environment, leading over time to the mineralization/dissolution of a variable amount of PyC-derived carbon. Elucidating the mechanisms by which degradation occurs and the parameters governing the rates at which degradation occurs remains challenging. Previous studies have attempted to investigate these issues through laboratory incubations (Singh et al., 2012; Kuzyakov et al., 2014), field emplacement and monitoring (Major et al., 2010; Zimmermann et al., 2012) and observation of natural systems using a space for time substitution approach (Cheng et al., 2008; Bird et al., 1999a,b; Cheng and Lehmann, 2009).

Isotope labelling with either stable carbon isotopes or radiocarbon has also been used to separately quantify soil and PyC-derived components in both laboratory incubations and field studies (Singh et al., 2012; Kuzyakov et al., 2014; Sagrilo et al., 2015). This study utilizes both stable and radiogenic carbon isotope labelling and a mass balance approach to separately quantify indigenous (char) carbon loss from degradation, and exogenous (environmental) carbon gain from active colonization by soil (micro)biota or passive sorption, through a three-year field deployment of laboratory-produced, radiocarbon-free char on the soil surface of a humid tropical rainforest. Treatments with and without litter, to provide a range in labile carbon supply and limestone chips, to provide a range of local pH conditions, were applied to determine the degree to which priming by labile carbon and/or local pH exert an influence on degradation behaviour. Preliminary radiocarbon results from these samples after one year of environmental exposure have been previously presented (Bird et al., 2014), focusing on the ability of pre-treatment techniques to decontaminate the char for radiocarbon dating.

## 2. Materials and methods

### 2.1. Samples

A c.8 million year old log (*Nothofagus* spp.; 10 kg) was obtained from the upper 10m of the 70m thick Yallourn brown coal seam in Victoria, Australia. The log was dried at 105 °C, chipped and sieved, with the >2 mm fraction retained for pyrolysis. Previous <sup>13</sup>C NMR analysis of the wood demonstrated that the material was dominated by lignocellulosic components similar to fresh wood (Bird et al., 2014). Aliquots of the wood (~100 g) were pyrolysed under a 3.5 L min<sup>-1</sup> flow of nitrogen at 305, 414 or 512 °C for 1 h, using the system described in detail by Bird et al. (2011). The resulting char was lightly crushed and sieved to yield approximately 100 g each of the 0.5–2 mm fraction for each char. The pyrolysis conditions were chosen to represent a typical range of common temperatures at which char is produced (McBeath et al., 2013) and thereby a range of physico-chemical characteristics in the resultant char that can be expected to influence subsequent colonization by microorganisms and degradation processes (Ascough et al., 2010a,b).

### 2.2. Field deployment

The site used for these trials was located at the James Cook University Daintree Rainforest Observatory, Cape Tribulation, Queensland (16.103 S; 145.447 E; 70m asl). This site was chosen because numerous soil and atmospheric parameters are routinely monitored, and the site is in a hot (mean monthly temperature

ranging from 22–28 °C; mean annual temperature 25 °C), humid (3,500 mm annual rainfall, with a pronounced wet season from December to March) rainforest environment where interactions between char and environment can be expected to be comparatively rapid. This humid tropical forest site has not burned in historical times, and it is likely that natural char abundance in the soil is very low.

Vegetation at the site is an evergreen mesophyll to notophyll vine 'tall forest' (Torello-Raventos et al., 2013) that produces abundant broadleaf litter throughout the year. This accumulates to a depth of 5–10 cm in the 'dry' season, decaying away over a period of a few months after the onset of the wet season. The soil is an Acidic, Dystrophic, Brown Dermosol (Isbell, 2002) or a Haplic Cambisol (Hyperdystic, Alumatic, Skeletic) based on the World Reference Base soil classification (Deckers et al., 1998), developed on colluvium derived from metamorphic and granitic mountains to the west of the site. The upper A-horizon is dark greyish brown silty loam to silty clay loam with many (20–50%) cobbles and stones throughout the profile. The <2 mm fraction of the soil (0–10 cm) is 28% clay, 54% silt and 19% sand, mildly acid (pH 5.5–6.5) and relatively organic-rich (3.7% C, 0.3% N).

Approximately 5 g aliquots of each char type were weighed into triplicate 125 µm aperture nylon mesh bags. The litter bags were pegged to the soil surface and subjected to one of the following four treatments:

- (i) NL – all litter removed from the surface and aliquots laid directly on the soil surface;
- (ii) L – as for NL but aliquots then covered with a ~5 cm thick layer of local leaf litter replenished each six months;
- (iii) NL-LM – as for NL but aliquots then covered with a ~5 cm thick layer of limestone chips (sieved at 2–10 mm);
- (iv) L-LM – as for NL but aliquots covered with a layer of limestone chips (sieved at 2–10 mm) mixed with an equal volume of periodically replenished local leaf litter each six months. A volume of litter equivalent to that applied to L was re-mixed with the limestone chips each six months.

The samples were emplaced in June 2009 and recovered in July 2010 (1 year) and August 2012 (3 years). The area where the samples were emplaced was covered with polyester shade cloth and enclosed in a wire mesh cage to exclude disturbance by foraging wildlife. The purpose of the limestone chips was to increase local pH, as alkaline conditions have been shown to be a significant determinant of PyC degradation behaviour (Braadbaart et al., 2009; Huisman et al., 2012). Determination of the exact pH experienced by the samples is not possible, but analysis of surface soil and leachate water at the end of the experiment suggests the treatments without limestone addition (L and NL) experienced a local pH between 5.6 and 6.5, while the limestone treatments (L-LM and NL-LM) experienced local pH between 6.6 and 8.0. Upon recovery, the samples were returned to the laboratory, removed from the mesh bags, gently washed free of loosely adhering soil particles, dried at 80 °C and weighed.

### 2.3. Laboratory analysis

#### 2.3.1. Characterization

Total organic carbon abundance and isotope composition of samples were determined using a Costech elemental analyzer fitted with a zero-blank auto-sampler coupled via a ConFloIV to a ThermoFinnigan DeltaV<sup>PLUS</sup> using continuous-flow isotope ratio mass spectrometry (EA-CF-IRMS) at James Cook University's Cairns Analytical Unit. Stable isotope results are reported as per mil (‰) deviations from the VPDB reference standard scale for δ<sup>13</sup>C values.

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