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Research paper

Effect of feedstock and pyrolysis temperature on properties of biochar governing end use efficacy

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ABSTRACT

The aim of this study was to systemically evaluate how feedstock characteristics and temperature influence biochar evolution during pyrolysis and to establish their relationships with biochar potential for soil amendments. We produced four biochar thermosequences from oak, pine, sugarcane and peanut shell at twelve temperatures (350–900 °C), and characterised them by yield, proximate analysis, elemental analysis, pH, electrical conductivity (EC) and carbon (C) functional groups using FTIR and solid-state ¹³C CPMAS NMR spectroscopy, along with principal component and cluster analyses. The results showed that not all biochar properties changed consistently with increasing pyrolysis temperature during slow pyrolysis. The significant increase in biochar pH with increasing pyrolysis temperature, was only observed between 350 and 500 °C ($p < 0.05$). Three principal components (PC) explained 88.9% of the variances in biochars. PC1 (ash, Mn, K, Fe, N, P and EC) and PC3 (Ca, Na and Mg) scores were essentially determined by feedstock type with notable influence of low pyrolysis temperatures. PC2 (O, H, C, volatile matter, fixed matter and pH) score was greatly influenced by pyrolysis temperature. Sugarcane derived biochars had high PC1 score but low PC3 score, while peanut shell derived biochars were contributed by both PC1 and PC3. The wood derived biochars had low scores of both PC1 and PC3, but the low temperature end of wood derived biochar thermosequences had significant higher PC2 scores than the high temperature end ($p < 0.05$). Understanding how biochar feedstock characteristics and pyrolysis temperature regulate biochar evolution will benefit tailored biochar manufacture.

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1. Introduction

Biochar is a by-product of other value added products from biomass via thermochemical processes (e.g. bioenergy and chemicals) in the modern biorefinery. It has attracted increasing attention in recent years due to its great potential in providing solutions to environmental and agricultural issues. Organic carbon (C) in biomass is converted into the form of char with long half-life under certain thermal decomposition conditions [1]. This means that C can be kept in the solid state so as to decrease the amount of C released into atmosphere and then mitigate global warming [2,3]. Biochar can directly introduce nutrients to soil [4,5], adjust soil pH [6], enhance soil porosity [7], increase soil cation exchange capacity (CEC) [8], stimulate plant growth [9,10] and reduce greenhouse gas

emissions from soil [11–13]. However, the real-world performance of biochar as a soil amendment and in mitigating soil greenhouse gas emissions is inconsistent among different studies. For example, Omondi et al. [14] quantified biochar effects on selected soil physical properties using a meta-analysis of literature data published by October 2015. With 95% confidence intervals, the response ratios of aggregate stability to amendment of biochar produced at 250–500 °C, porosity to amendment of biochar derived from wood, and saturated hydraulic conductivity to amendment of biochar derived from crop residues all overlapped one, indicating presence of both increase and decrease observations in the tested variables. Cayuela et al. [15] undertook a meta-analysis using publications from 2007 to 2013 to assist in elucidating key mechanisms in which biochar may act in mitigating N₂O emission. They found that the confidence interval of the mean effect size of N₂O emission to manure derived biochar amendment ranged from –46% to +39%.

The properties and structure of a biochar determine its

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performance in soil amendments [16]. The feedstock type and pyrolysis conditions applied in the biochar manufacture, especially the pyrolysis temperature, have significant impacts on biochar characteristics [17–21]. Numerous studies reported that high pyrolysis temperature led to increased biochar surface area [22,23], excellent stability in the environment [3] and higher pH [4] and mineral nutrient contents [24], but lower N content and lower nutrient availability for plant uptake [25,26]. However, due to the large increments in the pyrolysis temperature and limited temperature ranges employed in these studies (generally 100–200 °C and 300–700 °C, respectively), the changes in biochar properties with increasing pyrolysis temperature have probably been overgeneralized.

There have been several studies investigating the mechanisms of biochar-induced soil amendments. Atkinson et al. [27] summarised potential mechanisms for achieving agricultural benefits from application of biochar. For example, the high porosity and large surface area of biochars provided a refuge for beneficial soil microorganisms (away from grazing animals) and enhanced macro-nutrient availability (through the provision of binding sites for them). Anderson et al. [28] suggested that the biochar-induced decrease in nitrifying community and increases in mycobacterial nitrate reduction to ammonium (NH_4^+), N_2 fixation reductions and NH_4^+ adsorption by biochars have led to reductions in soil N_2O emissions. Biochar could provide phosphate adsorption sites in the form of CaCO_3 [29] or Mg crystals [30] to increase phosphorus (P) retention in soils, thereby increasing P availability and stimulating plant growth. Despite these achievements, how to produce a biochar to guarantee a desirable result after application is still largely unclear, due to the lack of clear relationships between biochar properties and their functions in soils and implications in agricultural and environmental areas.

Based on the current knowledge, this study aimed to (a) investigate the evolution of biochars derived from four plant materials during slow pyrolysis at different peak pyrolysis temperatures, (b) identify feedstock characteristics and pyrolysis temperatures explaining the changes in biochar thermosequences and classify biochars with similar characteristics, and (c) establish relationships between biochar type classified by feedstock type and pyrolysis temperature and its agronomic and environmental implications. This knowledge will not only inform biochar producers with respect to the selection of the feedstocks and pyrolysis conditions appropriate for biochar manufacture with a specific purpose, but will also increase the efficiency of biochar selection for future studies on the mechanisms of soil amendment through biochar application.

2. Material and methods

2.1. Biochar feedstock

Four types of biomass waste materials were selected to produce biochar thermosequences in this study: oak (*Allocasuarina torulosa*), pine (*Pinus radiata*), sugarcane (*Saccharum officinarum*) and peanut (*Arachis hypogaea*) shell. The native forest residues (e.g. oak and pine chips) were collected from Brisbane, Australia. The crop residues, i.e. sugarcane and peanut shell, were obtained from Bundaberg, Queensland, Australia and the Peanut Company of Australia, Kingaroy, Queensland, Australia, respectively.

2.2. Biochar manufacture

Slow and fast pyrolysis were two processes widely used in biomass pyrolysis [31]. The focus of this study was on biochar, so slow pyrolysis was selected to thermal decompose biomass in this

study due to its higher biochar yield, compared to fast pyrolysis [32]. The biochars derived from oak (*Allocasuarina torulosa*), pine (*Pinus radiata*), sugarcane (*Saccharum officinarum*) and peanut (*Arachis hypogaea*) shell, were manufactured at laboratory scale, using slow pyrolysis technology in a horizontal tube furnace (HTF 80/12–3/1, Laboratory Equipment Pty. Ltd., Australia) under oxygen-free conditions. Pyrolysis temperatures were controlled using three Eurotherm 3216 PID controllers (Eurotherm, Australia) to maintain a hot zone 300 mm long having temperature uniformity within 5 °C. The heating rate used in the biochar manufacture was 10 °C min^{-1} with a residence time of 1 h at the targeted peak temperatures ranging from 350 to 900 °C with increments of 50 °C. The feedstock was cut into small pieces (2–3 cm) and oven-dried at 65 °C for one week before pyrolysis. A portion of 60–100 g feedstock, depending on feedstock type, was loaded for each run. The furnace was purged continuously with high purity N_2 gas at a flow rate of 2 l min^{-1} for 30 min before pyrolysis and at 0.5 l min^{-1} during pyrolysis. Once the pyrolysis was completed, the furnace was shut off and the produced biochar was allowed to cool down in the furnace overnight in a flowing nitrogen atmosphere (0.5 l min^{-1}). After cooling, the biochar was stored in a plastic container under a nitrogen atmosphere before analysis.

A preliminary experiment was carried out to determine the reliability of the pyrolysis system used in this study. Two batches of peanut shell derived biochar thermosequence were produced in the preliminary experiment. The manufacture conditions and procedures employed in the preliminary experiment were as the same as described above. The characterisation methods of the properties of two repeated biochar thermosequences were described as below. The results were presented in Table A.1. The uncertainty of pyrolysis and analysis combination was better than $\pm 10\%$ of the measured value for biochar yield, volatile matter, fixed matter, ash, C, N and P, suggesting that the pyrolysis system was reliable to produce repeatable biochars. Thus, due to the limited feedstock stock, there was only one set of biochar manufactured at each pyrolysis temperature for each feedstock material.

2.3. Biochar characterisation

Before analysis, the biochar samples were homogenised and ground to <1 mm with a mortar and pestle.

2.3.1. Yield

Biochar yields are given as feedstock recovery and expressed as a percentage of weight of dry feedstock.

2.3.2. Proximate analysis

Moisture, volatile matter, fixed matter and ash content of the biochar samples were determined using the modified thermal analysis method [33]. Moisture was determined by calculating the weight loss after heating samples at 105 °C for 24 h, while volatile matter content was then determined as the weight loss after heating the samples at 450 °C for 1 h. The ash content was determined by the weight of the residues after then heating the samples at 750 °C for 6 h. The fixed matter content was calculated by the difference in moisture, ash and volatile matter contents. The proximate analysis was performed in duplicate for each sample.

2.3.3. Elemental analysis

Before elemental analysis, the biochar samples were further finely ground for 20 s using a puck and ring grinding mill. Total C and N contents of the biochar samples were determined by dry combustion using a TruMac CN analyser (Leco Corporation, USA) with routine analytical uncertainty better than $\pm 4\%$ of the measured value. The analyses of total H and O contents were

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