



Aged acidic biochar increases nitrogen retention and decreases ammonia volatilization in alkaline bauxite residue sand



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ABSTRACT

Bauxite residue sand (BRS) is the primary growth medium for rehabilitating Alcoa's residue storage areas in south-west Western Australia. Successful revegetation of highly alkaline BRS can be hindered by its low nitrogen (N) use efficiency. Biochar, a carbon (C)-rich material, has been suggested to have the potential to improve water and nutrient retention in soil. However, little is still known about the effect of biochar amendment on N use efficiency in the alkaline BRS environment. This incubation study aimed to evaluate the impact of biochars with different characteristics on N retention and dynamics in BRS. The BRS (pH 9.5 after being pre-treated with 1% gypsum and leached with water) was amended with the acidic biochar (pH 3.86; AC, from wild fire) and alkaline biochars (pH 9.58–10.8; greenwaste, GW; Jarrah, JL; mallee, ML) at a rate of 10% (w/w). The N loss via NH₃ volatilization was much lower from the AC treatment (24% of di-ammonia phosphate (DAP)-N added) than the alkaline biochars treatments (76–80% of the DAP-N added). The AC treatment can retain about 73% of N added to BRS, compared with <25% in alkaline biochar treatments. This can be attributed to the acidic nature and the greater NH₄⁺-N sorption capacity arising from the presence of a high density of the oxygen-containing functional groups on the surface of acidic biochar as revealed by the FTIR spectroscopy. These results imply acidic biochar can be used as an effective amendment for increasing N use efficiency by plants growing in alkaline BRS.

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1. Introduction

The environmental concern regarding long management of bauxite residue is increasing. Globally, it is estimated about 2.7 billion tons of bauxite residue are placed in storage facilities, increasing by ca. 120 mega tons each year (Gräfe and Klauber, 2011). Australia is the world's largest producer of bauxite, with an annual output of approximately 69 million metric dry tons (Chen et al., 2013). Alcoa of Australia refineries in Western Australia produce approximately 15 MT of residue each year from its three refineries (Kwinana, Pinjarra and Wagerup) (Chen et al., 2010). These residues are usually divided into two distinct fractions: coarse (bauxite-processing residue sand; BRS) and fine (red mud) residues (Courtney and Timpson 2005; Chen et al., 2010). The

residue sand is used to construct impoundments to store the residue mud, and as such is the primary growth medium for establishing a vegetation cover. Alcoa's current rehabilitation program aims to establish a sustainable ecosystem at each residue storage area (RSA) as part of progressive closure of these facilities. The establishment of a sustainable vegetation cover can enhance physical stability in terms of erosion and dust control, reduce deep drainage within the RSA, and encourage *in-situ* remediation (Wong, 2003; Courtney and Timpson 2005; Courtney and Mullen 2009; Banning et al., 2011).

The combined effects of alkalinity, salinity, sodicity, negligible organic matter, poor water retention and low nutrient availability can negatively impact the establishment of a vegetation cover in residue sand (Goloran et al., 2014a). The N content of bauxite residues is very low, ranging from 0.002% to 0.03%, depending on its sources (e.g. Courtney et al., 2008; Chen et al., 2010; Jones and Haynes 2011). As in other alkaline environments, NH₃ volatilization is the key pathway for N loss in bauxite residue disposal areas

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(He et al., 1999; Chen et al. 2010; Dendooven et al., 2010; Chen et al., 2013). To improve the characteristics of BRS as a growth medium, Alcoa's rehabilitation strategy currently incorporates 100 t/ha of phosphogypsum per 1 m depth, and about 2.7 ton ha⁻¹ of DAP (di-ammonia phosphate)-based fertilizer to help overcome these limitations (Chen et al., 2010). Previous studies have shown a substantial proportion (>85%) of N applied as NH₄⁺-N was lost through NH₃ volatilization within one week after application (Chen et al., 2010). Therefore, increasing NH₄⁺-N use efficiency is critical for ensuring long term survival of the vegetation cover.

Biochar is a stable carbon (C)-rich material derived from combusting biomass in an oxygen limited environment, and can be used to enhance soil C sequestration due to its stability and long residence time (>100 years) in soil (Lehmann et al., 2006). It has been suggested that biochar application can increase nutrient retention capacity and N use efficiency (Eldridge et al., 2010; Knowles et al., 2011), improve water-holding capacity and drought tolerance (e.g. Laird et al., 2010; Kammann et al., 2011), enhance biological fertility (Lehmann et al., 2011; Thies and Rillig 2012), and reduce greenhouse gas emissions (Yanai et al., 2007; Singh et al., 2010; Van Zwieten et al., 2010; Taghizadeh-Toosi et al., 2012). However, these effects vary depending on biochar application rate, feedstock type, pyrolysis condition and soil type (Van Zwieten et al., 2015).

Currently there is little published information on the benefits of biochar in improving water and nutrient use efficiency in the rehabilitation of mine tailings such as alkaline BRS. Chen et al. (2013) showed the benefits of alkaline biochar on reducing NH₃ volatilization in BRS was influenced by pH. At low pH (pH 5), NH₃ volatilisation increased with increasing pH despite the presence of biochar, suggesting that pH rather than adsorption was the primary cause of volatilisation. At near-neutral (pH 7) and slightly alkaline (pH 8) pH, adsorption of NH₄⁺-N by biochar reduced the loss via NH₃ volatilization. At highly alkaline pH (pH 9), the majority of NH₄⁺-N/NH₃-N pools was lost via NH₃ volatilization due to the strong acid-base reaction (Chen et al., 2013).

Fourier transform infrared spectroscopy (FTIR) has been applied to elucidate characterization of organic substances and identify the surface functionality (Cao et al., 2009; Teixidó et al., 2013). Analysis of the FTIR spectrum is useful with respect to the structural sorption behavior of NH₄⁺ on chars. This is because the vibration bands which coordinates with NH₄⁺ are sensitive to hydroxyle and carboxyle bonding, which can result in splitting of the vibrational modes (Chourabi and Fripiat, 1981). Cai et al. (2016) studied sorption behavior and mechanism of ammonium sulphate in three alkaline biochars derived from common agricultural wastes under different pyrolysis temperatures from 200 to 500 °C. They found that biochar produced at 200 °C had better retention ability in holding NH₄⁺ (about 90% after 21 days) which can be deduced from carboxyl and keton functional groups. However, effects of aged acidic biochar and its functional groups on NH₄⁺-N adsorption and NH₃ volatilization are largely unknown.

The aim of this study was to compare the adsorption capacity and acid-base reaction of acidic and alkaline biochars produced from different feedstock materials and under different pyrolysis

conditions in terms of N availability and transformation in BRS. It was hypothesized that aged acidic biochar would significantly increase N adsorption and decrease NH₃ volatilization in BRS due to its stronger adsorption capacity and acidic nature.

2. Materials and methods

2.1. Biochar and bauxite residue sand materials

Four biochar materials exhibiting differing physiochemical characteristics were used (Table 1): aged acidic biochar (AC), green waste biochar (GW), jarrah biochar (JR) and mallee biochar (ML). The AC was produced from an intense wildfire that occurred in 1969 at Peachester State Forest, situated in the Sunshine Coast hinterland, Queensland, Australia (26°50'S, 152°53'E). This biochar was collected from the stem of *Eucalyptus Pilularis* about 44 years after the wildfire. The GW was produced from green waste at 450 °C, while JL (*Eucalyptus marginata*) and ML (*Eucalyptus polybractea*) were both produced at 700 °C from jarrah and mallee mulches, respectively. Concentrations of sorbed NH₄⁺-N and NO₃⁻-N in biochar samples were measured by cold and hot water extraction. In brief, the biochar NH₄⁺-N and NO₃⁻-N were extracted by 2 M KCl at 1:10 (biochar: solution ratio), and shaken at 70 RPM on an end-to-end shaker for 1 h, then the extracts were filtered through Whatman 42 paper after being spun at 4000 rpm for 5 min, and the biochar samples were recovered for further extraction using hot 2 M KCl for 16 h (Rayment and Lyons, 2011). The hot water extraction involved incubating the biochar samples at 95 °C in 2 M KCl at 1:10 (biochar: solution ratio) for 16 h. Concentrations of NH₄⁺-N and NO₃⁻-N in both cold and hot water extracts were determined by SmartChem®200 Discrete Chemistry Analyser (WESTCO Scientific Instruments Inc.). Cold extractable NH₄⁺-N and NO₃⁻-N pools represent weekly-adsorbed by the biochar surface, while hot water extractable NH₄⁺-N and NO₃⁻-N the pools strongly sorbed in the inner layers of biochar samples.

Briefly, the biochar samples were placed into the oven which temperature was set at 95 °C incubating for 16 h. Both cold and hot 2 M KCl extracts were analysed for NH₄⁺-N and NO₃⁻-N. Concentrations of NH₄⁺-N and NO₃⁻-N were determined by SmartChem®200 Discrete Chemistry Analyser (WESTCO Scientific Instruments Inc.).

The BRS material was collected from Alcoa's Kwinana Refinery in Western Australia. At the time of collection, the BRS had a high pH (pH 11.8), high EC (34 dS m⁻¹) and elevated extractable (exchangeable + pore water) Na (12.8 cmol/kg), but had low organic C and nutrient contents (0.07 g kg⁻¹ C, 1 mg kg⁻¹ NH₄⁺-N, 2 mg kg⁻¹ Colwell P) (Table 1; Chen et al., 2010). Greater than 93% of the BRS comprised the 20–2000 μm particle size fraction (Chen et al., 2010). The BRS sample was air-dried and passed through a 2-mm sieve and amended with gypsum at a rate of 1% (w/w%) and leached with the equivalent of the average annual rainfall experienced at the Kwinana RSA (i.e. 758 mm) (Goloran et al., 2013). This treatment reduced pH of the BRS to ca. 9.5, after which the samples were air-dried and stored at room temperature for 12 weeks prior to commencing the experimental work.

Table 1
Selected properties of biochars and bauxite residue sand (BRS) used this study.

Sample type	Pyrolysis temperature (°C)	Moisture%	BET surface area (m ² g ⁻¹)	pH	EC (dS m ⁻¹)	pH _{PZC}	C%	N%	δ ¹³ C‰	δ ¹⁵ N ‰
AC	350–550	4.4	108	3.10	0.11	3.8	51.7	0.16	–27.2	47.1
GW	450	8.2	271	9.58	0.72	4.0	50.5	0.21	–27.6	–64.3
JR	720	2.3	205	10.8	0.32	5.7	60.8	0.16	–28.4	–0.8
ML	720	21.4	233	10.2	1.37	6.5	54.9	0.21	–27.5	–3.8
BRS	N/A	4.5	N/D	11.3	34.00	N/D	0.07	0.01	–12.7%	N/D

AC, aged acidic biochar produced from the stem of *Eucalyptus Pilularis* during wildfire at Peachester state forest, Queensland in 1969; GW, greenwaster biochar; JR, Jarrah (*Eucalyptus marginata*) biochar; ML, mallee (*Eucalyptus polybractea*) biochar. PZC, point of zero charge. N/A, not applicable; N/D, not determined.

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