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Research article

Properties of seawater neutralized bauxite residues and changes in chemical, physical and microbial properties induced by additions of gypsum and organic matter

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ABSTRACT

Seawater neutralization is a technique that can be used to treat bauxite residue prior to its storage but, as yet, no attempts have been made to revegetate it. Seawater neutralized bauxite residue was found to have a $\text{pH}_{1:5}$ of 9.3 and to be highly saline ($\text{EC}_{1:5}$ 16.5 dS m^{-1}). After leaching $\text{pH}_{1:5}$ rose to 9.7 and the residue was still highly sodic ($\text{ESP} = 64\text{--}69\%$). Addition of 1% gypsum, prior to leaching, arrested this increase in pH while with 5% gypsum addition the $\text{pH}_{1:5}$ was lowered to 8.9. Addition of 5% gypsum also reduced ESP to 38% and increased watercross germination in the residue from 58% in control treatments to 88%. The major ions in leachates were Na^+ and Cl^- and gypsum application increased the quantities of Na^+ , Ca^{2+} and SO_4^{2-} leached. Addition of 6% biosolids or 6% poultry manure added exchangeable cations to the mud and lowered ESP by 5–11%. The EC was 2.8–3.7 (mean 3.1) times higher and pH 0.2–0.7 (mean 0.43) units lower in saturation paste compared with 1:5 soil-water extracts. Addition of amendments had only small effects on physical properties. While organic C content was increased more by biosolids than poultry manure addition the reverse was the case for soluble organic C, microbial biomass C and basal respiration. It was concluded that although seawater neutralization initially lowers the pH of bauxite residues it is unlikely to increase the ease with which they can be revegetated.

1. Introduction

For every tonne of alumina produced in an alumina refinery during processing of bauxite, 1–2 tonnes of bauxite processing waste is also produced. This is deposited in land-based lagoons and impoundments adjacent to the refinery. Bauxite residues have a high pH (11–13) and high salinity and sodicity (Jones and Haynes, 2011; Grafe et al., 2011; Xue et al., 2016a) and the drainage from storage areas can pose an environmental risk (Oleszewska et al., 2016; Higgins et al., 2017). For this reason, there is an increasing trend towards partial neutralization of residues prior to their deposition in storage areas (Grafe et al., 2011). Seawater neutralization is increasingly being used by refineries close to the marine environment and it results in the pH being reduced from 11 to 13 down to about 9.0 and the exchangeable Na percentage is also reduced (by addition of Ca, Mg and K in the seawater) (Hanahan et al., 2004; Palmer et al., 2009).

Although seawater neutralization has been promoted as a method of converting bauxite residues into a relatively benign material (Hanahan et al., 2004) to date, there are no reports of its revegetation or what

treatments/management practices are required. Since revegetation of residue disposal areas is considered a key component for the closure plan of a refinery, revegetation strategies need to be developed. Because the residue has already been partially neutralized it can be argued that there will be no need for *in situ* neutralization with gypsum prior to revegetation (A. Boullemant, personal communication, 2016) as is commonly carried out for un-neutralized mud (Jones and Haynes, 2011). Nevertheless, leaching of the material is likely to be necessary in order to remove soluble salts (residual NaOH and seawater) prior to revegetation (Menziez et al., 2004, 2009). Limitations to revegetation are expected to be similar, although possibly less marked, to those for un-neutralized mud including salinity, sodicity, alkalinity, lack of microbial activity and a compact, fine-textured structure (Jones et al., 2012). Gypsum might need to be applied in order to further neutralize the residue and lower the pH to between 7 and 8. Addition of organic amendments to the surface 10–15 cm of mud may also be desirable in order to increase microbial activity and nutrient supplying capacity and improve physical conditions (Jones et al., 2012, 2015). Before attempting to grow plants in seawater neutralized residues, their

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properties should be defined and the effects of leaching and addition of amendments quantified.

In this study, the properties of seawater neutralized bauxite residues were examined and the effects of amending it with gypsum (1% w/v), poultry manure (6% w/v) and biosolids (6% w/v), followed by leaching, on soil chemical, physical and microbial properties were investigated. In a second experiment, the effects of 0, 1% and 5% gypsum application on chemical properties after leaching were studied.

2. Materials and methods

2.1. Materials used

Residue was collected from an alumina refinery in northern Queensland after seawater neutralization and immediately prior to its deposition into the residue disposal area. The residue contained 3% residue sand (> 150 µm dia.) and the remainder was composed of silt and clay-sized particles (i.e. residue mud; < 150 µm dia.). The residue was air-dried and crushed (< 2 mm) prior to use. Biosolids were collected from the Oxley Creek Wastewater Treatment Plant (Brisbane). At this plant, the sewage sludge is treated by the Cambi process (155 °C at 4.5 bar) and digested anaerobically prior to dewatering. Poultry manure was collected from a commercial egg producer. Organic materials were air-dried and ground/sieved (< 2 mm) prior to use. Gypsum was purchased from a local garden centre.

2.2. Experiment 1

There were 6 treatments consisting of addition of (i) control, (ii) poultry manure (6% w/v), (iii) biosolids (6% w/v) with or without the addition of gypsum (1% w/v). Amendments were thoroughly mixed with the residue (1 L) and placed in 2 L plastic containers with mesh bottoms to allow drainage into polypropylene collecting containers. The mixtures were rewetted to 70% of water holding capacity. The pots were arranged in a randomized block design with 3 replicates and incubated at room temperature (24–30 °C) for 4 weeks to give time for the gypsum and other amendments to react with the residue. Containers were opened and mixed each week to ensure adequate aeration. At the end of this period, samples were leached slowly over a 96-h period with 6 pore volumes of distilled water. Preliminary studies by Li et al. (2016) showed that after being leached with 6 pore volumes, greater than 95% of leachable ions had been removed from residue mud. Leachates were stored at 5 °C until analysed. Following leaching the samples were incubated for a further four weeks in order to allow time for the samples to re-equilibrate. After incubation, samples were split into two subsamples. One was stored at 4 °C for microbial and physical analysis and the other was air-dried and stored for chemical analysis.

2.3. Experiment 2

Because the addition of 1% gypsum did not produce a pronounced improvement in chemical properties of the residue mud a second experiment was initiated which consisted of 3 treatments: control, 1% gypsum and 5% gypsum added to the residue. The experimental procedure was identical to that of experiment 1. After incubation, leaching and final incubation, samples were air-dried and stored for chemical analysis.

2.4. Chemical analysis

To determine their mineralogy, bauxite residue were subjected to X-ray diffraction analysis. Amorphous content was calculated by difference using a corundum internal standard of known amorphous content. The total content of P, K, Ca, Mg, Si, Fe, Al and S in residue was determined after microwave digestion using HF, HCl and HNO₃ and that in organic materials after nitric-perchloric acid digestion. Elemental

content of extracts was determined by ICP-AES. Total C was measured by a dry combustion method using a Carlo Erba C, H, N analyser (Rayment and Higginson, 1992). In order to calculate the organic C content, inorganic C (HCO₃⁻/CO₃²⁻) was removed from samples by treatment with HCl to lower pH to 4.0 over a 4-day period (with intermittent vigorous mixing). Residual acid was then neutralized by raising pH to 5.5 using 0.5 M NaOH. Samples were dried at 70 °C before analysis.

The pH and EC were analysed in a 1:5 w/v water extract (i.e. pH_{1:5} and EC_{1:5}) using a pH/conductivity meter (Rayment and Higginson, 1992). Exchangeable cations (Ca²⁺, K⁺, Mg²⁺, Na⁺) were extracted with 1 M ammonium acetate (pH 7.0) (Rayment and Higginson, 1992) and analysed by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). Effective cation exchange capacity was calculated as the sum of exchangeable cations (Ca, Mg, K and Na). Exchangeable sodium percentage (ESP) was calculated as the percentage of exchangeable bases present as Na. Extractable Al was extracted with 1 M KCl (1:10 ratio for 1 h) and Al analysed by ICP-AES. Saturated paste extracts were prepared (Rhoades, 1982) and extracted under vacuum. The pH and EC in saturation extracts (i.e. pH_{SE} and EC_{SE}) were measured using a glass electrode and Ca, Mg, K, Na and Al were analysed by ICP-AES. The Sodium Adsorption Ratio (SAR) was calculated as the relative concentration of Na compared to Ca and Mg in the saturation paste extracts (Rengasamy and Churchman, 1999). Colwell Colwell-extractable P was extracted with 0.5 M NaHCO₃ (pH 8.5) (1:100 w/v ratio for 16 h) (Colwell, 1963) and measured colorimetrically by the molybdenum blue method. Leachates were analysed for Al, Ca, K, Mg, Na, P (ICP-AES) and pH and EC using a pH/conductivity meter.

2.5. Microbial analysis

Microbial biomass C was estimated based on the difference between organic C extracted with 0.5 M K₂SO₄ from chloroform-fumigated and unfumigated samples using a K_c factor of 0.45 (Wu et al., 1990). Soluble C in the K₂SO₄ extracts was analysed using a Shimadzu 5000A soluble C/N analyser. Values for the unfumigated samples were used as a measure of soluble C. Basal respiration was determined by placing 30 g oven-dry equivalent of moist residue in a 50 mL beaker and incubating the sample in the dark for 10 days at 25 °C in a 1 L air tight jar along with 10 mL of 1 M NaOH. The CO₂ evolved was determined by titration (Anderson, 1982). The metabolic quotient was calculated as basal respiration (mg CO₂-C per day) expressed per gram of microbial biomass C.

2.6. Physical analysis

Bulk density was determined on naturally compacted samples (Haynes and Goh, 1978), particle density by the pycnometer method (Blake and Hartge, 1986) and total porosity by difference. Soil water content in samples was determined at -10 and -1500 kPa using a pressure plate apparatus. Pore size distribution was calculated as macropores (> 29 µm diameter, air-filled pores at -10 kPa), mesopores (0.2–29 µm diameter, drained between -10 and -1500 kPa) and micropores (< 0.2 µm diameter, water-filled pores at -1500 kPa). Field capacity was determined as the volumetric water content at -10 kPa and available water as that held between -10 and -1500 kPa.

2.7. Germination assay

A germination assay was carried out (in quadruplet) on residue treatments. 2 mL of aqueous extract (1/10 w/v) from treatments was added to dishes (Belyaeva and Haynes, 2009). Ten seeds of watercress (*Lepidium sativum*) were placed on filter paper and dishes placed in the dark at 28 °C. The germination percentages with respect to control (distilled water) and root lengths were determined after 5 days. The

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