Effects of Leaching on the Chemical and Physical Properties of Bauxite Residue Mud and Sand

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Abstract—The major mineralogical constituents for residue mud and sand were amorphous material 34 and 23%, goethite 22 and 10% and quartz 7.1 and 48% respectively. Mixtures consisting of 0:100, 25:75, 50:50, 75:25 and 100:0% v/v residue mud: residue sand were incubated for four weeks and a portion was then leached bi-weekly for another 14 weeks. Residue sand had a much lower EC, exchangeable Na, ESP and short-term and residual acid neutralizing capacity than mud. Leaching caused a decrease in the EC of residues by at least one order of magnitude and ammonium acetate-extractable Na levels were reduced by more than half. The pH of the residue decreased by about one unit after leaching. This was attributed to leaching of soluble alkalinity (HCO_3^-/CO_3^{2-}) with the mobile Na^+ ion. Both short-term and residual acid neutralizing capacity were lowered following leaching. The macroporosity of sand was much greater than that of mud whilst available water holding capacity was greater for mud. Adding increasing proportions of mud to sand decreased macroporosity and increased available water but also increased EC, soluble and exchangeable Na, ESP and acid neutralizing capacity. If mud were to be added to sand prior to revegetation more consideration would need to be directed towards management of chemical constraints to plant growth.

Index Terms—Bauxite processing mud, bauxite processing residue, red mud, red sand, leaching.

I. INTRODUCTION

Alumina is produced in alumina refineries by the Bayer Process in which Al-containing minerals in bauxite are digested in hot NaOH [1], [2]. The insoluble solids (bauxite processing residues) are washed, sometimes partially neutralized and then deposited in impoundments surrounding the refinery. In Western Australia, bauxite from the Darling Range is mined and refined. This deposit has a relatively low Al content (27-30 % Al_2O_3) but contains little reactive Si (i.e. layer silicate clay minerals) and most of the Si is present as quartz which is relatively insoluble during Bayer digestion [3].The processing residue produced is separated into sand (> 150 um; which makes up about 30% of the residue) and mud (<150 um). The residue sand is used to construct the outer embankments of residue storage areas and the residue mud is deposited within them. A stack is built progressively whereby the perimeter embankments are periodically built higher with sand and the volume within is filled with mud. The sand embankments are progressively revegetated with a

mixture of shrubs and herbs native to a Western Australian sand dune ecosystem.

Establishment and of growth of vegetation on residue is typically limited by the presence of residual NaOH in residues which confers on them a high pH (10-13) and saline/sodic properties. Because of this, leaching of the residue profile to remove excess Na and accompanying alkalinity (HCO_3^-/CO_3^{2-}) is essential prior to attempting revegetation [4]. The residue sand also has a low water holding capacity and high leaching potential. Addition of residue mud to the sand could potentially increase the amount of available water held [5].

The purpose of this study was to investigate the effect of adding residue mud to sand (or sand to mud) on chemical and physical properties of the substrate and also to study the effect of leaching of residues on their chemical properties. The combinations used were: 100% mud, 75% mud/25% sand, 50% mud/50% sand, 25% mud/75% sand and 100% sand.

II. METHODOLOGY

A. Samples and Experiment Design

Bauxite residues were obtained from an alumina refinery in Western Australia. The sand made up approximately 30% of the total residue mass. Sieve analysis showed that particle size distribution of residue sand was : >1mm, 7.8%; 0.5 – 1mm, 24.6%; 0.25 – 0.5mm, 36.0%; 0.125 – 0.5mm, 21.5%; <0.125mm, 10.1% and that of mud was < 2 μm 24%, 2-20 μm 54%, and 20-200 μm 22%.

For each residue source, five treatments were prepared consisting 0:100, 25:75, 50:50, 75:25 or 100:0% v/v residue mud: residue sand. Samples (1kg dry weight) were thoroughly mixed, rewetted to 70% water holding capacity and incubated for four weeks in a randomized block design with three replicates. A subsample was air-dried for subsequent chemical analysis while part of the moist sample was used for measurement of physical parameters and another (200 cm^3) was transferred to polypropylene leaching tubes (12 cm long and 5 cm diameter). A plug of glass wool was placed at both the top and bottom of the incubation column to prevent loss of material by downward movement of fine particles during leaching. Samples were incubated for two weeks and then leached slowly (over a 24 hour period) with one pore volume (calculated for residue mud) of distilled water. Leachate was collected in polypropylene collecting containers and stored at 5 ℃ until analysed. Following leaching, a tension of 17 kPa was applied to each column for 10 minutes to remove excess water. This process

was repeated every 2 weeks for 7 leachings. At the completion of leaching, residue was removed from tubes and air-dried for subsequent chemical analysis

B. Chemical Analysis

In order to determine their mineralogy, residues were subjected to X-ray diffraction analysis. Amorphous content was calculated by difference using a corundum internal standard of known amorphous content. Exchangeable bases (Ca^{2+}, K^+, Mg^{2+}, Na^+) were extracted with 1M ammonium acetate (pH 7.0) [6] and analysed by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). EC and pH were analysed in a 1:5 w/v water extract using a pH/conductivity meter [6]. Saturated paste extracts were prepared and extracted under vacuum. pH and EC in extracts were measured using a glass electrode and Ca, Mg, K, Na and Al by 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. Bicarbonate-extractable P was extracted with 0.5M $NaHCO_3$ (pH 8.5) (1:100 w/v ratio for 16h) [6] and measured colorimetrically. Diethylenetriminepentaacetic acid (DTPA)-extractable metals were extracted according to Lindsay and Norvell [7] (0.005 M DTPA, 0.01 M $CaCl_2$ and 0.1 M TEA: 1:2 ratio for 2 h) and Zn, Cu, Mn and Fe were analysed by ICP-MS. Acid neutralizing capacity of samples before and after leaching was measured by titrating a mud/water slurry to pH 7.0, using 0.5 M HCl, over a 24 and 120-hour period. Short term (24 h) and residual (120 minus 24 h) acid neutralizing capacity were calculated (mol H^+ kg^{-1} solid) [8].

The total content of P, K, Ca, Mg, Si and heavy metals in residue mud and sand was determined by ICP-AES after microwave digestion using HF, HCl and HNO_3. Total C and N were measured by dry combustion using a Carlo Erba C, H, N analyser [6]. Leachates were analysed for Al, Ca, K, Mg, Na, P (ICP-AES) and pH and EC using a pH/conductivity meter.

C. Physical Analysis

Bulk density was determined on naturally compacted samples, particle density by the pycnometer method [9] 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). Available water was calculated as as that held between -10 and -1500 kPa.

D. Germination Assay

A germination assay was carried out (in quadruplet) on residues before and after leaching using filter paper in petri dishes. Five mL of aqueous extract (1/10 w/v) from residues was added to dishes [10]. Ten seeds of watercress (*Lepidium sativum*) were placed on filter paper and dishes placed in the dark at 25 °C. The germination percentages with respect to control (distilled water) and root lengths were determined after 5 days. The germination index (GI) was calculated as *GI = %G x Le/Lc,* where *%G* is the percentage of germinated seeds in each extract with respect to control, *Le* is the mean total root length of the germinated seeds in each extract, and *Lc* is the mean root length of the control [9]. The control GI value is considered as 100%.

E. Statistical Analysis

The statistical significance of experimental treatments was determined by subjected the data to Analysis of Variance using the Minitab Software Package. Differences were calculated at the 5% level using Tukey's test.

III. RESULTS AND DISCUSSION

A. Properties of the Residues

Because the sand fraction is essentially undigested quartzite rock it contained a much greater Si and quartz content than mud (Table I). Goethite was the dominant crystalline mineral in mud while amorphous minerals accounted for 23-34% of the mineral content of residues. This range is similar to that found by other workers [10] and demonstrates that a substantial portion of the mineral fraction has yet to be characterized. The sodalite content of the mud was very low (4%) and in the sand it was not detected. A low sodalite content is a potential advantage since long-term dissolution of this compound will supply alkalinity and Na to the deposit [11], [12].

The EC values in saturation paste extracts prior to leaching were very high ranging from 5.2-13.2 dS m^{-1} (Table 2). Such values are likely to be detrimental to plant growth since an EC of > 2 dS m^{-1} is considered saline and values > 12 dS m^{-1} are generally considered too saline for most plants [13]. Thus, leaching of excess salts out of the profile will be an important management practice prior to revegetation. Values for SAR (512-604) (Table II) and ESP both before (77-95%) and after (22-69%) leaching (Table III) were also high since SAR values above 13 and ESP values above 10-15% are normally considered likely to reduce plant growth [14]. Thus, the large quantities of both soluble (Table II) and exchangeable Na present on cation exchange sites will need to be displaced and then leached from the surface horizons prior to revegetation. Normally this is achieved by adding gypsum ($CaSO_4.2H_2O$) to the residue. The added Ca^{2+} displaces exchangeable Na^+ which leaches down the profile with the added SO_4^{2-}. Because the ECEC is much higher for mud than sand (Table 3), the quantity of exchangeable Na^+ retained following leaching was also greater. Thus, the quantity of gypsum added will need to be greater for mud and combinations containing a high proportion of mud.

Residue mud had a notably high extractable-P content (i.e. 116.9 mg kg^{-1}; data not shown) which is greater than many fertile agricultural soils [15]. This is because the bauxite from the Darling Range is high in P (it contains 195 mg P kg^{-1}) and in in the refining process used, P is added as dihydrogen phosphate to control calcia [16]. Thus, although the mud is high in Fe oxides (which characteristically adsorb and sequester P) the extractable P content is still very high and higher than that for residue sand. For agricultural plants, critical bicarbonate-extractable P levels are usually in the

range of 15-30 mg kg^{-1} [13] so that, at least in the short-term, P fertilizer applications are unlikely to be required for plant growth in mud but may well be required in sand.

TABLE I: PH, EC, ELEMENTAL AND MINERALOGICAL COMPOSITION OF THE MATERIALS USED

Residue	pH	EC	Total Analysis (g kg^{-1})							
			Si	Fe	Al	Ca	K	Mg	Na	P
Mud	11.0	5.65	104	191	67.1	12.5	7.3	0.6	37.2	0.7
Sand	11.3	1.35	247	158	50.4	0.8	0.9	0.04	3.3	0.1

Residue	Mineralogical composition (%)												
	Quartz	Hematite	Goethite	Maghemite	Calcite	Anatase	Rutile	Boehmite	Gibbsite	Kaolin	Illite	Sodalite	Amorphous
Mud	7.1	8.5	22.2	-	6.5	0.6	0.6	3.3	1.3	-	11.7	4.1	34.2
Sand	47.6	11.2	9.9	0.9	0.2	0.1	0.5	0.4	4.3	0.5	0.8	-	23.4

TABLE II: EFFECT OF ADDITION OF DECREASING PROPORTIONS OF RESIDUE MUD TO RESIDUE SAND ON PH, EC, EXTRACTABLE CATIONS AND SAR IN SATURATED PASTE EXTRACTS

Mud addition (% v/v)	pH	EC (dS m^{-1})	Saturated paste extractable cations (mmolc L^{-1})				SAR
			Ca	Mg	K	Na	
100	10.2a	13.2c	0.33b	0.07a	0.32a	225b	518a
75	10.3a	12.8c	0.31b	0.03a	0.28a	225b	586b
50	10.4ab	12.2b	0.26ab	0.03a	0.27a	219b	604b
25	10.5ab	8.3ab	0.22a	0.02a	0.22a	175ab	556b
0	10.6b	5.2a	0.15a	0.02a	0.16a	117a	512a

Means followed by same letters within one column are not significantly different at $P \leq 0.05$.

TABLE III: EFFECT OF ADDITION OF DECREASING PROPORTIONS OF RESIDUE MUD TO RESIDUE SAND ON PH, EC, ESP, ECEC AND GERMINATION INDEX

Mud addition (% v/v)	pH		EC (dS m^{-1})		ESP (%)		ECEC (cmol$_c$ kg^{-1})		Germination index (%)	
	Initial	Final	initial	Final	Initial	Final	Initial	Final	Initial	Final
100	11.0b	10.2a*	5.6e	1.0d*	95d	69d*	37.7e	15.3d*	10a	86a*
75	11.0b	10.2a*	4.8d	0.94d*	93cd	62d*	29.0d	11.5c*	12a	83a*
50	11.0b	10.1a*	3.6c	0.61c*	90c	49c*	22.2c	8.1bc*	30b	94b*
25	11.0b	10.0a*	2.5b	0.39b*	85b	33b*	17.7b	5.5b*	72c	94b*
0	11.3b	9.9a*	1.4a	0.13a*	77a	22a*	11.7a	2.8a*	90d	120c

Means followed by same letters within one column are not significantly different at $P \leq 0.05$.
* denotes a significant effect ($P \leq 0.05$) of measurement time (initial versus final) for that particular property.

TABLE IV: EFFECT OF ADDITION OF INCREASING PROPORTIONS OF RESIDUE MUD TO RESIDUE SAND ON DTPA-EXTRACTABLE MICRONUTRIENTS AND SHORT-TERM AND RESIDUAL ACID NEUTRALIZING CAPACITY (ANC) BEFORE (INITIAL) AND AFTER (FINAL) LEACHING

Mud addition (% v/v)	DTPA – extractable metal (mg kg^{-1})				ANC (short-term)		ANC (residual)	
	Fe	Mn	Zn	Cu	Initial	Final	Initial	Final
100	21.5d	0.29d	0.31a	1.01e	0.28d	0.12d*	0.26e	0.15d*
75	20.7d	0.24c	0.27a	0.76d	0.19c	0.08c*	0.18d	0.10c*
50	17.4c	0.18b	0.25a	0.53c	0.11b	0.05b*	0.11c	0.07b*
25	11.4b	0.10a	0.21a	0.22b	0.06ab	0.02a*	0.08b	0.04ab*
0	9.21a	0.05a	0.22a	0.06a	0.04a	0.01a*	0.06a	0.02a*

Means followed by same letters within one column are not significantly different at $P \leq 0.05$.
* denotes a significant effect ($P \leq 0.05$) of measurement time (initial versus final) for ANC.

Concentrations of extractable Mn and Zn were low in both mud and sand and Cu was also low in the sand (Table 4). That is, critical levels of DTPA-extractable Fe, Mn, Zn and Cu are about 4.5, 1.0, 0.8 and 0.2 mg kg^{-1} [7]. In addition, critical levels of exchangeable K and Mg for adequate plant growth are about 0.2-0.5 and 0.14-0.30 cmol$_c$ kg^{-1} respectively [17,18] so that after leaching levels of K were extremely low while Mg status was low for the sand (Table 5). The lack of organic matter in the residue means there is minimal N supply (total N content 0.20-0.30 g kg^{-1}) so fertilizer N will also be required for adequate plant growth. Thus, a basal fertilizer application of both macro- and micronutrients will be required prior to revegetation.

B. Effect of Leaching

Leaching caused a decrease in residue pH of about one unit (Table III). This is attributable to leaching of soluble alkalinity (HCO$_3^-$/CO$_3^{2-}$) as counterions for the mobile Na

cation [4]. That is, at the high pH of residue mud (11.0-11.3), HCO_3^-/CO_3^{2-} are the main forms of soluble alkalinity present [12], [19].

TABLE V: EFFECT OF ADDITION OF INCREASING PROPORTIONS OF RESIDUE MUD TO RESIDUE SAND ON AMMONIUM ACETATE-EXCHANGEABLE CATIONS BEFORE (INITIAL) AND AFTER (FINAL) LEACHING

Mud addition (% v/v)	Exchangeable cations ($cmol_c$ kg^{-1})							
	Ca		Mg		K		Na	
	Initial	Final	Initial	Final	Initial	Final	Initial	Final
100	0.9a	0.70a	0.79c	0.56d*	0.05a	0.02a*	36d	14d*
75	1.4b	1.0a	0.55bc	0.44c*	0.04a	0.01a*	27c	10c*
50	1.8bc	1.3a	0.39b	0.36bc	0.03a	0.01a	20bc	6.4b*
25	2.4c	1.8b	0.27ab	0.23b	0.03a	0.01a	15b	3.5ab*
0	2.5c	1.9b	0.18a	0.08a	0.03a	0.01a	9a	1.8a*

Means followed by same letters within one column are not significantly different at $P \leq 0.05$.
* denotes a significant effect ($P \leq 0.05$) of measurement time (initial versus final) for that particular property

Leaching caused a reduction in EC in residues by one order of magnitude (Tables II and III) and a reduction in exchangeable Na by more than one-half (Table V). This is because Na in solution (in excess of the cation exchange capacity of the residue) was effectively removed from the residue by leaching. As expected, the leachate cation content was overwhelmingly dominated by Na^+ and concentrations were greater from mud than sand (Table VI). There was also a significant reduction in ESP (Table III) since Na^+ is held less strongly to cation exchange sites than divalent cations such as Ca^{2+} and Mg^{2+} and is therefore preferentially leached from the residue. The reduction in calculated ECEC after leaching (Table III) occurred because the ECEC measured in residues before leaching is an overestimate of the cation exchange capacity due to the large amounts of cations present in soil solution (that are extracted with ammonium acetate). Indeed, when cations in saturation paste extracts were subtracted from ammonium acetate-extractable cations in residues (prior to leaching), calculated ECEC values decreased from 11.7-37.7 $cmol_c$ kg^{-1} (Table III) down to 4-20 $cmol_c$ kg^{-1}.

TABLE VI: EFFECT OF ADDITION OF INCREASING PROPORTIONS OF RESIDUE MUD TO RESIDUE SAND ON MEAN IONIC COMPOSITION OF LEACHATES OVER THE 14-WEEK LEACHING PERIOD

Mud addition (% v/v)	pH	EC (dS m^{-1})	Mean concentration (mmol$_c$ L^{-1})			
			Na^+	Ca^{2+}	K^+	Mg^{2+}
100	10.3a	10.5b	132.7c	0.09a	0.21a	0.11a
25	10.4a	9.2b	122.3bc	0.07a	0.19a	0.19a
50	10.4a	9.0b	115.3b	0.06a	0.20a	0.21a
25	10.5a	7.0ab	78.4a	0.05a	0.11a	0.07a
0	10.6a	4.9a	58.1a	0.03a	0.10a	0.06a

Means followed by same letters within one column are not significantly different at $P \leq 0.05$.

The soluble and residual alkalinity present in residues can be quantified by measuring their short-term and long-term acid neutralizing capacities [8], [11]. That residue mud had values for both measurements that were an order of magnitude greater than those for sand indicates a much greater content of reactive components in mud (Table IV). The large reduction in short-term acid neutralizing capacity

following leaching was expected since, as already noted, HCO_3^-/CO_3^{2-} (soluble alkalinity) leaches with Na^+. The large reduction in residual acid neutralizing capacity is presumably due to a reduction in solid state/easily soluble alkalinity which is probably associated with dissolution of the amorphous mineral component (which is generally more soluble than crystalline minerals) (e.g. amorphous precursors of tricalcium aluminate and sodalite) [10] and subsequent leaching of the liberated alkalinity during the leaching period. The nature of the amorphous mineral component in bauxite residues and its relationship with the residual alkalinity present deserves future study. The very high residual alkalinity in the mud will make revegetation a challenge because of a continual background supply of alkalinity into solution. Nevertheless, in the long-term (e.g. > 10y) there is likely to be a decrease in EC, ESP and pH due to natural weathering and leaching [20], [21].

C. Physical Properties

The high microporosity (>40%) and large quantity of water held at field capacity by mud can result in surface layers becoming waterlogged during wet periods of the year [19], [22]. Although sand had a much greater macroporosity than mud, addition of sand to mud only had a substantial effect on macroporosity when the mixture contained 75% sand (Table 7). Indeed, additions of small amounts of sand (e.g. 25%) tended to reduce total porosity and had no significant effect on pore size distribution. This is attributable to the ability of small mud particles to fill macropore voids between sand particles. Additions of sand to mud at 25% are common practice [23] but results presented here suggest they have minimal effect on total porosity and pore size distribution of the medium.

TABLE VII: EFFECT OF ADDITION OF INCREASING PROPORTIONS OF RESIDUE MUD TO RESIDUE SAND ON PHYSICAL PROPERTIES

Mud addition (% v/v)	Total Porosity (m^3 m^{-3})	Pore size distribution (%)			Available Water (kg m^{-3})
		Micropores (<0.20 μm)	Mesopores (0.20-29 μm)	Macropore (>29 μm)	
100	0.55b	42.0d	58.0a	0a	357d
75	0.47ab	40.7d	59.3a	0a	305c
50	0.43a	36.1c	61.3a	2.6b	264b
25	0.41a	23.8b	60.5a	15.7c	251ab
0	0.42a	12.2a	55.0a	32.8d	230a

Means followed by same letters in one column are not significant difference at $P \leq 0.05$.

The available water holding capacity was much less for the sand than mud and the lack of available water during dry periods will potentially limit plant growth in residue sand. The addition of 25% mud to sand (i.e 75% sand) significantly increased available water (Table VII). Nevertheless, it also increased exchangeable Na, ESP, EC and alkalinity (Tables III, IV and V) so that whether addition of mud to sand is a viable strategy will depend on whether physical or chemical constraints are most limiting to plant growth.

D. Germination Index

Prior to leaching, germination was considerably less in mud than sand and after leaching, there was still significant inhibition of germination in mud (and mixtures containing a substantial amount of mud) (Table III). The limiting factors

in mud are the greater ESP, inorganic C and short-term and residual acid neutralizing capacity than sand. As already noted, a reduction in ESP can be achieved by adding gypsum followed by leaching. Indeed, although germination percentage was satisfactory (>80%) in all residues after leaching (Table III), plants are unlikely to grow satisfactorily in these materials (ESP of 20-82%). Patterns for seed germination versus subsequent growth of plants in bauxite residues are often dissimilar [24] since longer-term physiological effects of salinity/sodicity on plant growth are not assayed in short-term germination tests.

IV. CONCLUSIONS

The sand fraction of bauxite residue from the Darling Range deposit has a high content of unreactive quartz, a low content of sodalite and soluble and residual alkalinity and a much lower soluble and exchangeable Na content than the residue mud. These properties mean that after leaching, chemical constraints to plant growth in sand are minimal (other than nutrient deficiencies that can be corrected by regular fertilizer applications). Low water retention (due to the course nature of the sand) is a potential limitation to revegetation. Addition of fine textured mud to the sand is a possible option. Indeed, a 25% addition of mud to sand did increase available water. It did, however, have the potentially detrimental effects of causing increases in exchangeable Na, ESP and short-term and residual acid neutralizing capacity. Thus, if such an option were to be considered, more consideration would need to be focussed on the management of chemical constraints.

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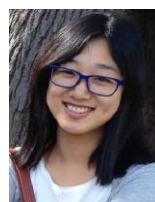
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