

# Mill mud and mill mud products: efficacy as soil amendments and assessment of environmental risk

# Final Report: Reef Water Quality Science Program Project 12C

Soil Processes, Science Division

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Great state. Great opportunity.

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## **Key Results and Implications**

- Fifty per cent of the total nitrogen in mill mud and mud ash is available for uptake by the first crop following application.
- It is not possible to predict the availability of residual nitrogen from mill mud/mud ash to crops subsequent to the first crop grown after application.
- There is no difference in the availability of nitrogen from incorporated or surface broadcast mill mud/mud ash.
- There are unlikely to be any benefits from mill mud/mud ash application at 100 wet tonnes/ha on soil 'health' (key chemical and physical properties) except in the case of light-textured soils.
- Either BSES-P or Colwell-P content of mill mud/mud ash can be used to indicate the availability of phosphorus in the products. However, P fertiliser requirements from the SIX EASY STEPS are based on BSES-P, so it is recommended that BSES-P content of the mill mud/mud ash is used to assess its P availability.
- Equations have been developed to calculate BSES-P and dissolved inorganic phosphorus in the soil solution in soils receiving mill mud/mud ash applications.
- At a mill mud application rate of 100 wet tonnes/ha to low P sorbing soils, BSES-P and the concentration of dissolved inorganic phosphorus in the soil solution may present an environmental risk to water quality by runoff, drainage or off-site sediment movement.
- Guidelines in industry Best Management Practices for discounting nitrogen and phosphorus inputs from mill by-products need to be updated.
- SafeGauge for Nutrients will be updated to take account of the nitrogen applied in mill mud/mud ash in assessing the risk of off-site nitrogen movement.
- SafeGauge for Nutrients will be updated to assess the risk of off-site movement of phosphorus from mill mud application.

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## 1 Background

Approximately 1.5 million wet tonnes of mill mud and 200,000 wet tonnes of mill ash are produced annually in Queensland. Taken together, these mill by-products contain approximately 5600 tonnes of total nitrogen (N) and 3400 tonnes total phosphorus (P). Mill mud and mill ash are extremely variable in composition; in a crushing season, the intra-mill variability is as great as inter-mill variability (Bloesch and Barry, 2010).

Bloesch and Barry (2010) reviewed research that had been undertaken on mill mud/mud ash and identified that there is very limited information available on the rates of release of bioavailable N, P and potassium (K) from mill mud and mill ash when applied to soil, and on the beneficial effects on soil properties of organic carbon added in these products.

The objectives of this project were to:

- Characterise the immediate bioavailability, and residual value, of N and P from mill mud and mud ash;
- Assess the efficacy of mill mud and mill mud products for improving soil health (soil chemical, physical and biological functions) by acting as carbon (C) sources and liming agents;
- Identify appropriate analytical methods for monitoring N and P bioavailability in batches of mill mud and mud ash over time and from different sources;
- Develop guidelines for application of mill mud products to land and advise policy implications.

To achieve these objectives, two glasshouse trials and two laboratory incubation experiments were used to monitor the bioavailability of N and P in mill mud and mud ash over time and to assess the effects of mill by-products on soil properties. These results were used to develop algorithms for updating risk assessment of mill mud/mud ash application in *SafeGauge for Nutrients*.

## 2 Pot Trials

Two pot trials were undertaken to characterise the immediate bioavailability, and residual value, of nitrogen (N) and phosphorus (P) from mill mud and mud ash.

## 2.1 Pot Trial 1

## 2.1.1 Soil

A yellow Kandosol (0–20 cm) from Bundaberg was used as the test soil.

## 2.1.2 Mill by-products

Mill mud and mud ash products were sourced from Millaquin and Bingera mills and their chemical composition is presented in Table 1.

## 2.1.3 Treatments

There were 20 treatments with three replicates per treatment and three destructive harvests. Pots were randomly positioned in the shade-house and randomly assigned a harvest number.

- 1 Mill mud 50 wet tonnes/ha surface
- 2 Mill mud 100 wet tonnes/ha surface
- 3 Mill mud 200 wet tonnes/ha surface
- 4 Mill mud 50 wet tonnes/ha incorporated
- 5 Mill mud wet 100 tonnes/ha incorporated
- 6 Mill mud 200 wet tonnes/ha incorporated
- 7 Mud/ash 50 wet tonnes/ha surface
- 8 Mud/ash 100 wet tonnes/ha surface
- 9 Mud/ash 200 wet tonnes/ha surface
- 10 Mud/ash 50 wet tonnes/ha incorporated
- 11 Mud/ash wet 100 tonnes/ha incorporated
- 12 Mud/ash 200 wet tonnes/ha incorporated
- 13 Urea (N equivalent to 50 wet tonnes/ha mill mud\*)
- 14 Urea (N equivalent to 100 wet tonnes/ha mill mud\*)
- 15 Urea (N equivalent to 200 wet tonnes/ha mill mud\*)
- 16 Urea (equivalent to 50 wet tonnes/ha mud/ash\*)
- 17 Urea (equivalent to 100 wet tonnes/ha mud/ash\*)
- 18 Urea (equivalent to 200 wet tonnes/ha mud/ash\*)

- 19 No basal N (basal P applied)
- 20 No basal N, No basal P

(\* N rate was based on the assumption that 30% of the total N in mill mud/mud ash was immediately bioavailable.)

## 2.1.4 Procedure

Mill by-products were either mixed through 7 kg of air dry <5 mm soil (incorporated) or applied on the soil surface after the soil had been added to 30 cm diameter tapered plastic pots. The surface area of soil in the pot was 398 cm<sup>2</sup> and this area was used to calculate rates of amendment addition to the pots. Urea was applied as a sub-surface band in all urea treatments. Pots were free-draining with saucers and any leachate was returned to the soil surface. Pots were maintained at field capacity throughout the trial. Basal K solution was added to all pots at planting and basal P (1 mg CaH<sub>2</sub>PO<sub>4</sub>.2H<sub>2</sub>O/pot) was added to treatments 13, 14, 15, 16, 17, 18 and 19.

## 2.1.5 Timeline

Three germinated one-eyed setts were planted in each pot on 1-2 June 2010. Harvests were taken on 17 August (78 days), 21 September (113 days) and 20 October 2010 (142 days) by cutting plants at ground level, weighing, then drying at 60°C for 48 hours, re-weighing, grinding and analysing for total N and total P. Soil was sub-sampled from the harvested pots, dried at 40°C for 48 hours, ground <2 mm and analysed.

## 2.1.6 Analytical methods

Soil analyses followed the methods specified in Rayment and Lyons (2011): pH (1:5 water), Method 4A1; electrical conductivity [EC (1:5)], Method 3A1; chloride (1:5), Method 5A2a; total organic carbon, Method 6B2b; Colwell-P, Method 9B2; BSES-P, Method 9G20; exchangeable K, Method 15A1; effective cation exchange capacity (ECEC), Method 15J1; KCI extractable ammonium-N and nitrate-N, Method 7C2b; total N, Method 7A5; Total P, Method 9A1; PBI<sub>Col</sub>, Method 9I2b; and total K, Method 17A1. Clay content was determined according to Thorburn and Shaw (1987) and HCI-P according to Guppy et al. (2000).

Analyses of mill by-products followed the same relevant soil analytical procedures.

Total N and P were determined on harvested plant material using micro Kjeldahl digest by block digestion. Nitrogen was determined by a modification of the method of Searle (1974) and P by a modification of the method of Murphy and Riley (1962) using an automated colorimetric procedure on a segmented flow analyser.

## 2.2 Pot Trial 2

## 2.2.1 Soils

Surface samples (0–20 cm) of two soils from the Mackay region (a loamy sand and a clay) were used as the test soils.

## 2.2.2 Mill by-products

The mill mud sourced from Millaquin for Pot Trial 1 was used in this experiment. Its chemical composition is presented in Table 1.

## 2.2.3 Treatments

There were 12 treatments per soil with five replicates per treatment and one harvest. Pots were randomly positioned in the shade-house.

Treatments were:

- 1. Mill mud @ 50 wet tonnes/ha, applied to surface
- 2. Mill mud @ 100 wet tonnes/ha, applied to surface
- 3. Mill mud @ 200 wet tonnes/ha, applied to surface
- 4. Mill mud @ 50 wet tonnes/ha, incorporated
- 5. Mill mud @ 100 wet tonnes/ha, incorporated
- 6. Mill mud @ 200 wet tonnes/ha, incorporated
- 7. Urea N equivalent to mill mud @ 50 wet tonnes/ha\*
- 8. Urea N equivalent to mill mud @ 100 wet tonnes/ha\*
- 9. Urea N equivalent to mill mud @ 200 wet tonnes/ha\*
- 10. Urea equivalent to 100 kg N/ha
- 11. No basal N (basal P applied)
- 12. No basal N, No basal P

(\* N rate was based on the assumption that 30% of the total N in mill mud was immediately bioavailable)

## 2.2.4 Procedure

Mill mud was either mixed through 7 kg of air dry <5 mm soil (incorporated) or applied on the soil surface after the soil had been added to 30 cm diameter plastic pots. Urea was applied as a subsurface band in all urea treatments. Pots were free-draining with saucers and any leachate was returned to the soil surface. Pots were maintained at field capacity throughout the trial. Basal K solution was added to all pots at planting and basal P (1 mg CaH<sub>2</sub>PO<sub>4</sub>.2H<sub>2</sub>O/pot) was added to treatments 7, 8, 9, 10 and 11.

## 2.2.5 Timeline

Three germinated one-eyed setts were planted in each pot on 11 July 2012, and plants were harvested on 7–8 November 2012 (120 days) by cutting at ground level. Plants were weighed, dried at 60°C for 48 hours, re-weighed, ground and analysed for total N and total P.

## 2.2.6 Analytical methods

See Section 2.1.6 above.

## **3 Pot Trial Results**

## 3.1 Pot Trial 1

## 3.1.1 Soil

The soil (0–20cm) had the following properties: pH (1:5 water), 5.9; EC, 0.04dS/m; total organic C, 0.7%; Colwell-P, 33 mg/kg; BSES-P, 40 mg/kg; PBI<sub>Col</sub>, 25; and exchangeable K, 0.06 cmol<sub>o</sub>/kg.

## 3.1.2 Mill by-products

The mill mud and mud ash products used in this project were slightly acidic, with EC and chloride levels high by soil standards. Total organic C was typically high and the C/N ratios of 26 and 30, respectively, are indicative of initial N immobilisation (tie-up of mineral N) until these ratios decrease because of microbial oxidation of the carbon. The mill mud had high initial ammonium-N content and the mud ash had high initial nitrate-N content; these concentrations are transitory and dependent on mill mud/mud ash age and storage conditions. In both products, mineral N (ammonium-N plus nitrate-N) comprised less than 0.5% of total N.

When compared to the mean values reported by Bloesch and Barry (2010) for a large number of mill mud samples from various Queensland mills, the Millaquin mill mud was slightly more acidic (6.7 cf. 7.2), similar in organic C content (33.5% cf. 32.7%), slightly lower in N content (1.28% cf. 1.38%), and higher in total P (1.51% cf. 1.36%).

Note that 95% of total P in the mill mud was extractable by the HCI-P method, and the corresponding figure for mud ash was 100%. This indicates that there was a negligible amount of organic P in the mill mud and none in the mud ash; the entire quantity of P in the mill mud and the mud ash was in the form of acid-extractable calcium phosphates. BSES-P extracted 86% and 77% of the total P in the mill mud and mud ash, respectively; Colwell-P extracted 65% of the total P in the mill mud and 63% of the total P in mud ash. These extractability figures are much higher than those reported in Bloesch and Barry (2010) for a range of mill muds and mud ashes (63% BSES-P; 53% Colwell-P). Exchangeable K comprised 69% of total K in the mill mud and 50% of total K in the mud ash, and these extractabilities are considerably higher than the 10% reported by Bloesch and Barry (2010) for a suite of the products. These contrasts in extractable P and K are explicable if both the mill mud and mud ash used in this project had a higher than normal content of soil. **Table 1. Chemical composition on a dry weight basis (600C) of mill mud and mud ash used in the pot trials.** 

		pH (w)	) EC CI Exchangeable Cations			Total Org C	Total N	2M KCI extr.				
					Са	Mg	Na	K	Č		NH4-N	NO3-N
			(dS/m)	(mg/kg)		cm	ol(+)/ kg		(%)	(%)	(mg/kg)	(mg/kg)
Millaguin mill mud	Mean	6.7	1.44	204	44.4	24.4	0.10	2.44	22.5	1.00	44	12
willaquin mili mud		6.7	1.41	301	44.4	34.4	0.18	3.44	33.5	1.28	44	13
	se mean	0.0	0.00	1	0.9	1.2	0.00	0.11	1.0	0.00	4	1
Bingera mud ash	Mean	6.7	1.80	544	39.0	23.2	0.97	7.95	25.4	0.84	2	36
	se mean	0	0.08	8	1.0	0.8	0.04	0.22	0.0	0.01	0	0
		Colwell	BSES	HCI-P	Total P	Total K	Oven	"As is"				
		Р	Р				moisture	moisture				
		(mg/kg)	(mg/kg)	(mg/kg)	(%)	(%)	%	%				
Millaguin mill mud	Mean	9825	12950	14400	1.51	0.19	250.5	71.5				
	se mean	124	247	85	0.04	0.01	1.8	0.4				
Bingera mud ash	Mean	5195	6325	8220	0.82	0.62	324	76.5				
	se mean	244	46	42	0.01	0.01	0.7	0.4				

## 3.1.3 Crop dry matter yield

Crop yield (converted from fresh weight g/plant to fresh weight tonnes/ha on an area basis) at Harvest 3 is presented in Fig. 1. There was no significant (P=0.05) response to the application of basal P (cf. treatments 19 and 20). Mud ash at 50 wet tonnes/ha did not increase yield over the nil N treatment (cf. treatments 7, 10 and 19). All other mud ash and mill mud treatments gave higher yields than the nil N treatment, and there was a trend (sometimes not significant) for yield to increase with increasing rate of application. There was no significant effect of placement (surface application vs incorporation) on yield for either product except for the mud ash at 100 wet tonnes/ha when surface application exceeded incorporated product.

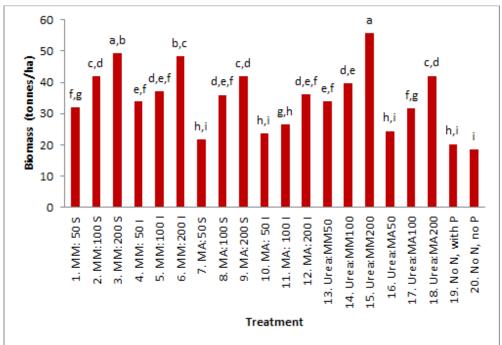


Figure 1. Fresh weight of tops at Harvest 3, Pot Trial 1, for all treatments. Treatments labelled with the same letter are not significantly different at P=0.05

## 3.1.4 Crop N uptake and potentially mineralisable soil N

Crop N uptake (expressed as mg N/kg soil) was calculated for each treatment and related to total applied N (mg N/kg soil) to determine the percentage recovery of applied N (Fig. 2a, 2b). Note that N uptake values for the urea and mill product treatments include N uptake from mineralisation of soil organic matter (viz. control treatment). Regression lines fitted to the different products indicated that 25.3% of urea-N was recovered at Harvest 1, increasing to 38.5% at Harvest 3. Corresponding recoveries for mill mud ranged from 2.3% (incorporated) to 5.1% (surface) at Harvest 1, with both placements having the same recovery of 7.1% at Harvest 3. Mud ash recoveries ranged from 2.5% (incorporated) to 8.2% (surface) at Harvest 1, and were identical to the mill mud recovery of 7.1% at Harvest 3.



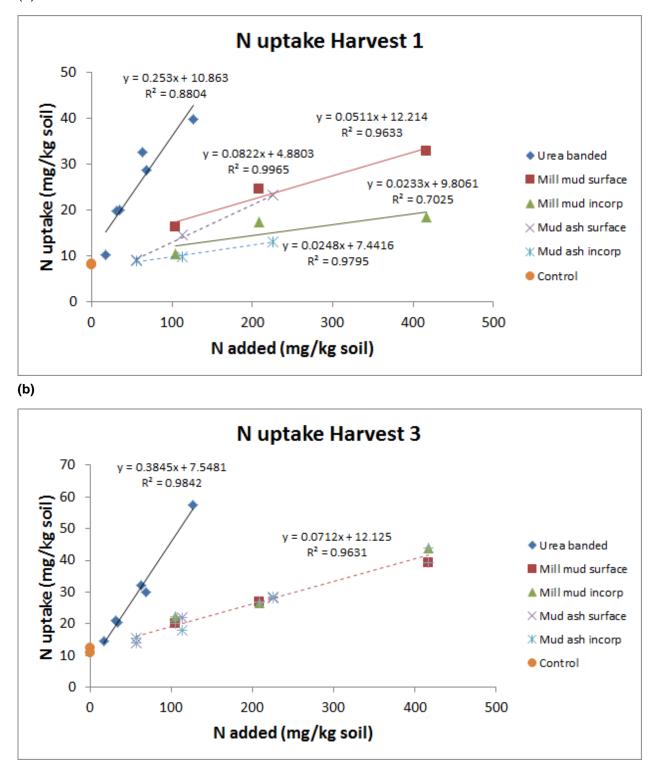


Figure 2. Nitrogen uptake in tops related to total applied N for (a) Harvest 1 and (b) Harvest 3 in Pot Trial 1.

To assess the quantity of mineral N (ammonium-N plus nitrate-N) that could potentially be produced by mineralisation of soil organic matter from the treated soils, soil samples were incubated for seven days at 30°C at a soil moisture content equivalent to 80% field capacity; mineral-N was determined on Day zero and Day seven. The quantity of mineral-N produced during this seven day period is termed 'Potentially Mineralisable N' (PMN) and the values are plotted in Fig. 3. This measurement is used as a benchmark index for comparing the rate and quantity of mineral N produced from the breakdown of soil organic matter across soils.

There were no significant (P=0.05) differences in PMN between harvests for any particular treatment. The PMN values for the two urea treatments were not significantly different from the unamended control soil, although PMN for the urea treatment equivalent to the mill mud N application tended to be higher than that of the mud ash N equivalent because more N was applied in the former treatment. Mill mud and mud ash treatments were not significantly (P=0.05) different, although the mean PMN for the mill mud over all three harvests was higher than that of the mud ash.

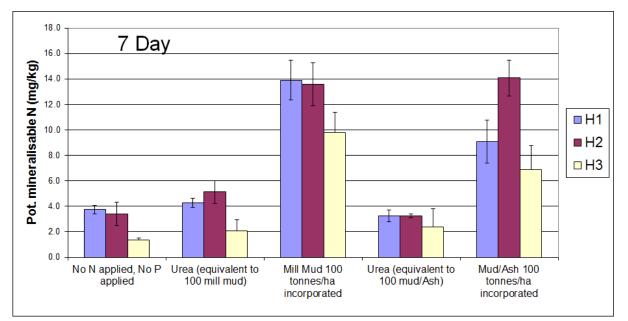


Figure 3. Potentially mineralisable N after seven days for selected treatments at the three harvests in Pot Trial 1. Mean standard error bars are indicated on the plot.

## 3.1.5 Crop P uptake and soil P tests

As there were no significant (P=0.05) trends for either BSES-P or Colwell-P to change with time in any incorporated amendment treatments (data not presented), crop P uptake at Harvest 3 is plotted against soil P tests post-Harvest 1 for all incorporated mill mud/mud ash treatments (Fig. 4). There were close linear relationships when either BSES-P or Colwell-P was related to crop P uptake. These results indicate that either soil test is useful for assessing available soil P in mill mud and mud ash as well as assessing the availability of P in soils amended with the products. However, P fertiliser requirements from the SIX EASY STEPS are based on BSES-P, so it is recommended that BSES-P content of the mill mud/mud ash is used to assess its P availability.

The lack of a yield response to basal P applied to the control soil (Fig. 1) indicates that the crop P uptake recorded for both products is luxury (non-essential) uptake.

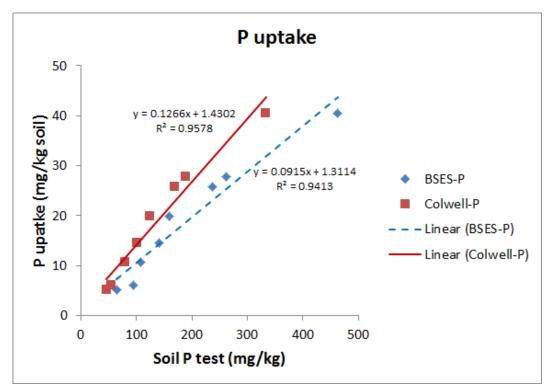


Figure 4. Crop P uptake at Harvest 3, Pot Trial 1, as a function of BSES-P and Colwell-P across incorporated mill mud/mud ash treatments.

## 3.1.6 Soil P tests and soil solution P

Soil solution P concentration (SSP), which is related to dissolved inorganic P in runoff water (Dougherty et al., 2011), can be estimated from [P Buffer Index (PBI)/Colwell-P], x, by the following equation (Moody, 2011):

SSP= 0.0512e1.5363x (R2= 0.80).....Equation 1

A PBI value of 100 was used to develop this equation, and the estimated soil solution P concentrations in equilibrium with the various rates of mill mud and mud ash are given in Table 2.

Table 2. Estimated soil solution P concentrations, BSES-P and Colwell-P resulting from various rates of mill mud and mud ash incorporated into a soil.

Treatment	Soil solution P concentration (mg P/L)	BSES-P (mg/kg)	Colwell-P (mg/kg)
No N applied, No P applied	1.04	64	46
No N applied, P applied	1.25	94	54
Mill Mud 50 tonnes/ha incorporated	7.12	160	124
Mill Mud 100 tonnes/ha incorporated	989	261	189
Mill Mud 200 tonnes/ha incorporated	>>1000	463	333
Mud/Ash 50 tonnes/ha incorporated	0.44	108	79
Mud/Ash 100 tonnes/ha incorporated	3.82	140	102
Mud/Ash 200 tonnes/ha incorporated	285	237	169

## 3.2 Pot Trial 2

## 3.2.1 Soils

Key properties of the two soils are presented in Table 3. The high clay content of the clay soil resulted in much higher ECEC and PBI values than were present in the lighter textured soil. The clay soil had higher organic carbon content than the loamy sand.

Soil	pH (1:5 water)	EC (1:5) (dS/m)	Organic C (W-B) (%)	BSES-P (mg/kg)	Colwell-P (mg/kg)	PBI	ECEC (cmol <sub>c</sub> /kg)	Clay (%)
Clay	5.3	0.16	1.7	28	57	270	17	51
Loamy Sand	5.7	0.12	1.1	76	36	42	3.8	3

#### Table 3. Selected properties of the clay soil and loamy sand soil used in Pot Trial 2.

#### 3.2.2 Crop dry matter yield

Dry matter yields are presented in Fig. 5 and Fig. 6. There was a significant (P<0.05) response to N applied to the loamy sand, but not to the clay (cf. Nil P and Nil N treatments). For the loamy sand soil there was a trend for yield to increase with increasing application rate of mill mud, either surface applied or incorporated, but this response was only significant (P<0.05) for the 200 wet tonnes/ha treatment (Fig. 5). Yields at the highest application rate of mill mud were not significantly different from the yield for 100kg N/ha applied as banded urea.

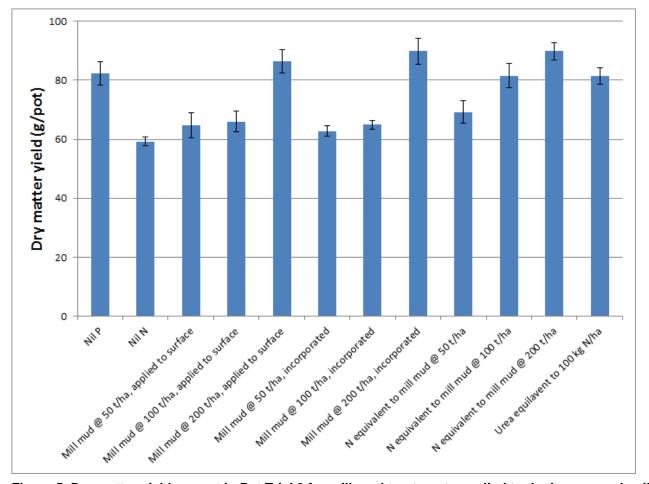


Figure 5. Dry matter yield per pot in Pot Trial 2 for mill mud treatments applied to the loamy sand soil. N equivalent treatments were applied as sub-surface urea.

For the clay soil, there was also a trend for dry matter yield to increase with increasing mill mud rate, but this increase was not significant (P=0.05) except for the 100 and 200 wet tonnes/ha incorporated product treatments. (Fig. 6).

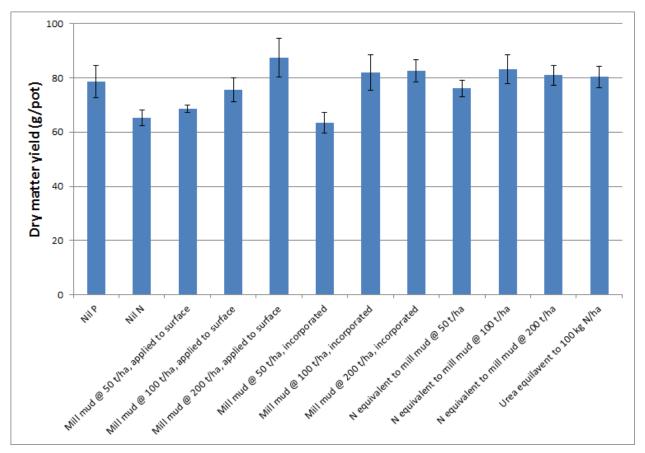
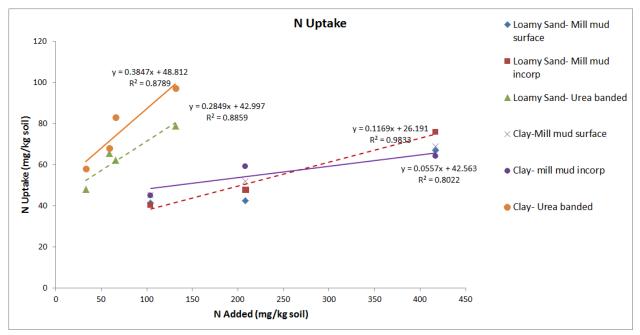


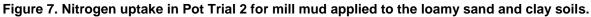
Figure 6. Dry matter yield in Pot Trial 2 for mill mud treatments applied to the clay soil.

## 3.2.3 Crop N uptake

Crop N uptake (expressed as mg N/kg soil) was calculated for each treatment and related to total applied N (mg N/kg soil) to determine the percentage recovery of applied N for both soil types (Fig. 7). Note that N uptake values for the urea and mill mud treatments include N uptake from mineralisation of soil organic matter.

Regression lines fitted to the banded urea treatment indicate that 28% and 38% of the applied urea N was recovered in the crop tops in the loamy sand and clay soils, respectively. There was little difference in recoveries of N from surface application or incorporation of mill mud in either soil, so N uptake data was pooled and regressions calculated for each soil. Recovery of N from the mill mud was 12% in the loamy sand and 6% in the clay soil.





## 3.2.4 Crop P uptake and soil P tests

There were weak (not significant at P=0.05) trends in both soils for P uptake to increase in response to increasing BSES-P (Fig. 8). This result was unexpected because there were strong linear trends for BSES-P to increase in both soils in response to increasing mill mud application rate (Fig. 9).

#### 3.2.5 Soil P tests and soil solution P

Soil solution P concentrations (SSP) were calculated from [P Buffer Index (PBI)/Colwell-P] for the two soils using Equation 1 above (Table 4).

Table 4. Estimated soil solution P concentrations, BSES-P and Colwell-P in equilibrium with various rates of mill mud incorporated into the clay and loamy sand soils.

Treatment		Clay		Loamy Sand		
	Soil sol'n P (mg/L)	BSES-P (mg/kg)	Colwell-P (mg/kg)	Soil sol'n P (mg/L)	BSES-P (mg/kg)	Colwell-P (mg/kg)
Nil P	0.072	22	61	0.18	34	53
Mill mud @ 50 tonnes/ha	0.097	43	113	8.58	140	149
Mill mud @ 100 tonnes/ha	0.152	110	191	139.6	216	256
Mill mud @ 200 tonnes/ha	0.239	214	271	>500	488	472

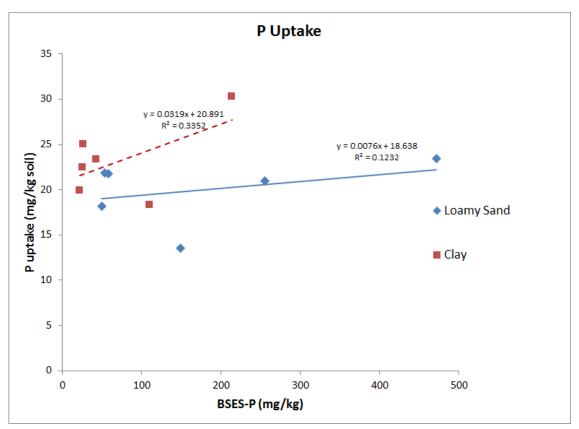


Figure 8. Phosphorus uptake in Pot Trial 2 related to BSES-P resulting from rates of mill mud incorporated into the loamy sand and clay soils.

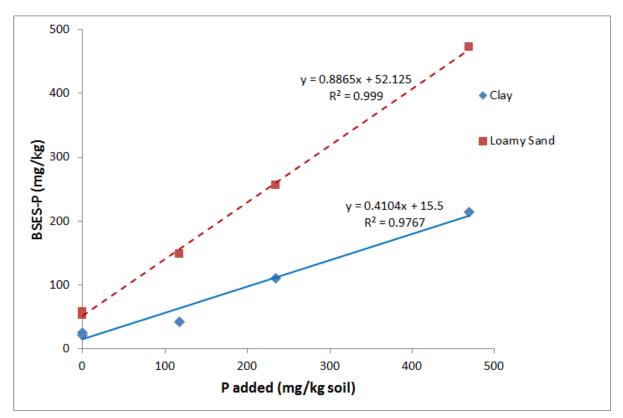


Figure 9. Increase in BSES-P with increasing P application from incorporated mill mud.

## **4 Discussion: Pot Trials**

## 4.1 N availability

In Pot Trial 1, crop tops recovered 38% of the N applied as urea and 7% of the total N applied in mill mud or mud ash 140 days after application to a light-textured soil from Bundaberg. In Pot Trial 2, the crop tops recovered 28% and 38% of the N applied as urea 120 days after application to a loamy sand soil and a clay soil, respectively; both soils were from Mackay. Crop N recovery of the total N applied as mill mud (same product as that used in Pot Trial 1) was 12% (loamy sand) and 6% (clay). Nitrogen mineralised from soil organic matter during the trials amounted to 12 mg N/kg soil in Pot Trial 1, and 35 and 43 mg N/kg soil for the loamy sand and clay soil, respectively, in Pot Trial 2. These differing rates of soil N mineralisation (approximately equivalent to 12, 35 and 43 kg N/ha.10 cm) roughly reflect the differing soil organic C contents of the soils (0.7%, Pot Trial 1 soil; 1.1%, Pot Trial 2 loamy sand; and 1.7%, Pot Trial 2 clay), and highlight the significant, and variable, quantities of N from this source.

Considering the crop N uptakes across both trials, it appears that approximately 35% of the bandapplied urea N was recovered in the crop tops during a growing period of 120-140 days. The corresponding recovery of total N applied as mill mud or mud ash was approximately 10%. The pot experiments were carried out under controlled conditions and it is considered that N losses (e.g. leaching, denitrification) would have been minimal. Nitrogen applied as urea (and therefore almost immediately available as ammonium-N) that was not accounted for in the crop tops (i.e. 65%) was presumably in the root mass and also incorporated into the soil organic matter via the soil microbial biomass. This observation is supported by the trends in seven day potentially mineralisable N (PMN) in Pot Trial 1; additional N as urea provided only a slight, although non-significant (P=0.05) increase in PMN after 140 days indicating that the N of urea origin had become part of the larger soil organic N pool and was no more readily available than N derived from mineralisation of soil organic matter.

Only about 10% of the total N applied as mill mud or mill ash was recovered in the crop tops under ideal growing conditions, leaving a deficit of 90% unaccounted for. Similar to urea, some of the N mineralised from the mill mud/mud ash would be incorporated into the root mass, but the seven day PMN figures indicate that there is a significant further amount of N that can be mineralised from mill mud/mud ash over and above that occurring from the background (control) mineralisation of soil organic matter. As a very rough estimate of the mineralisation of mill mud/mud ash N that occurs post-140 days, it is assumed that an additional 25% of the total N applied is released in the first crop season to bring the cumulative release of total N from mill mud products up to 35%, as was observed for the urea-N.

Estimating the amount of N released from mill mud/mud ash sources to subsequent crops after the crop receiving the application is problematic because the bioavailability of this residual N would very likely be the same as N mineralising from the soil organic matter. The best approach for taking account of mill mud/mud ash N contribution to subsequent crops would be direct assessment of the soil's nitrogen mineralisation potential at the time of subsequent crop emergence by soil analysis or evaluating crop N status.

## 4.2 P availability

The close correlations between soil P tests (Colwell-P and BSES-P) and crop P uptake in Pot Trial 1, and to a lesser extent in Pot Trial 2, indicate that either BSES-P or Colwell-P can be used to

assess P availability in amended soils. The large proportion of total P in mill mud and mud ash that is BSES- and Colwell-extractable demonstrates these products are excellent sources of immediately available P. There were strong correlations between application rates of mill mud/mud ash and BSES-P and Colwell-P in all three soils used in the pot trials, so these soil P tests reflect the previous history of mill mud/mud ash applications.

It is evident from both pot trials that an application rate of 50 wet tonnes/ha of either mill mud or mud ash causes soil extractable P to exceed (greatly so when the background soil already has adequate available P for maximum crop yield) the levels necessary for maximum yield according to the SIX EASY STEPS guidelines. There is a perception that off-site P movement is only of concern when sediment (with sorbed P) moves off-site, however, this is completely erroneous. The soil solution P concentrations [i.e. Filterable Reactive Phosphate (FRP)] when 50 wet tonnes/ha of either mill mud or mud ash was incorporated into the soil exceeded the ANZECC/ARMCANZ (2000) water quality guidelines (0.004-0.020 mg FRP/L) by a factor ranging from 5 (clay soil, Pot Trial 2) to 1000 (Pot Trial 1 soil; loamy sand soil, Pot Trial 2). This finding raises concerns about the potentially detrimental impacts of mill mud/mud ash application on surface water quality, particularly runoff from cane blocks that have received multiple applications. The risk of off-site FRP movement as runoff and/or drainage to groundwater is particularly high where mill by-products are applied to soils of low P sorbing capacity (i.e. PBI<35) such as sandy soils.

## **5 Incubation Experiments**

The objectives of the incubation experiments were to assess the efficacy of mill mud and mill mud products for improving soil health (soil chemical, physical and biological functions) by acting as carbon sources and liming agents.

## 5.1 Incubation Experiment 1

## 5.1.1 Treatments

The following amendments were incorporated into the soils: mill mud ex Millaquin, mill ash ex Bingera, soybean residues, cane trash, soybean residues + cane trash, cane biochar ex T McShane, mill mud + biochar, control.

Cane trash and soybean trash were chopped into <1 cm pieces. Biochar was sieved <2 mm.

## 5.1.2 Rate

Materials were applied at rates equivalent to 10 tonnes total C/ha (approximately 100 wet tonnes/ha mill mud).

## 5.1.3 Soils

The clay soil and loamy sand soil from Pot Trial 2 were used in this experiment.

## 5.1.4 Timeline

The experiment commenced on 31 July 2012, and was terminated on 22 October 2012.

## 5.1.5 Procedure

Amendments were thoroughly mixed through bulk soil samples at the appropriate rates and amended soil then packed to a bulk density of 1.3 g/cm<sup>3</sup> in plastic tubes capped at one end. There were three replicates per treatment. The tubes were 8.6 cm in diameter and 10 cm long. Deionised water was added to the tubes to bring the soil to field capacity by weight. Tubes were then loosely capped and incubated at 25°C in an incubator. Tubes were removed every second day and watered to weight.

Soil solution samples (not reported) were taken under vacuum through micro-fibres at 7, 17, 24, 31, 59 and 84 days after set-up. Immediately following soil solution extraction at 17, 31 and 84 days, three tubes per treatment were destructively sampled, the soil was mixed, sub-sampled, and extracted for ammonium-N and nitrate-N using 2*M* KCI (results not reported).

## 5.1.6 Analytical methods

Soil analyses followed the methods specified in Rayment and Lyons (2011): pH (1:5 water), Method 4A1; total organic carbon, Method 6B2b; ECEC, Method 15J1; total N, Method 7A5; and PBI<sub>Col</sub>, Method 9I2b. Aggregate stability was assessed as the proportion by weight of the whole <2 mm soil retained on a 53 um sieve following end-over-end shaking for 16 hours in a 1:3 soil:water suspension with chemical dispersant. Particulate organic C (POC) was determined by analysing the >53 um soil fraction for total organic C and calculating as mg C/kg whole soil.

## 5.2 Incubation Experiment 2

At the conclusion of Pot Trial 2 (120 days after incorporation of amendments), soil was removed from each pot, coarse root material removed, and the soil was thoroughly mixed and sub-sampled for analysis using the methods described in Section 5.1.6 above.

## **6 Incubation Experiment Results**

## 6.1 Incubation Experiment 1

## 6.1.1 Chemical properties

Total organic C analysis 84 days after amendment addition was unable to separate the amended treatments from the controls except for the biochar treatment (Table 5). Likewise, total N analyses were unable to discriminate between treatments even though there was a wide variation in C/N ratio of the applied materials. The particulate organic C (POC) was more discriminatory than total organic C in the clay soil, and indicated a significant (P<0.05) increase in POC in soybean, cane trash, soybean + cane trash and biochar treatments. There was a trend in the loamy sand soil for POC to be higher in these treatments than in the control soil, but variability within treatments prevented these trends being significant.

For both soils, effective cation exchange capacity (ECEC) showed a significant (P<0.05) increase over the control soil for most amendment treatments.

## 6.1.2 Aggregate stability

There were significant (P<0.05) increases in aggregate stability in the loamy sand due to mill mud and biochar incorporation, but no other amendments impacted on aggregate stability in this soil and there were no effects at all in the clay soil (Table 5).

Table 5. Effect of various amendments applied at 10 tonnes C/ha on key soil chemical and physical properties of two soils, 84 days after incorporation. Values with the superscript are significantly (P=0.05) higher than other values.

		Total org C (%)	Total N (%)	ECEC (cmol <sub>c</sub> /kg)	Aggreg. (> 53 um)	POC (mg/kg whole soil)
Loamy	Control	1.4	0.1	4.0	39.6	7.4
Sand	Mill Mud	1.5	0.1	7.5 <sup>a</sup>	41.5 <sup>ª</sup>	8.1
	Mill Ash	1.6	0.1	6.3 <sup>a</sup>	39.9	8.6
	Soybean	1.6	0.1	7.0 <sup>a</sup>	40.6	9.2
	Cane Trash	1.7	0.1	4.5	40.9	9.5
	Soybean+Cane	1.7	0.1	5.4 <sup>a</sup>	41.8	8.5
	BioChar	1.9 <sup>ª</sup>	0.1	5.7 <sup>a</sup>	42.5 <sup>a</sup>	11.4
Clay	Control	1.8	0.1	18.8	24.3	2.4
	Mill Mud	2.0	0.2	22.8ª	24.8	3.6
	Mill Ash	2.0	0.2	21.6ª	25.0	4.0
	Soybean	2.1	0.2	22.1 <sup>a</sup>	25.5	4.9 <sup>a</sup>
	Cane Trash	2.0	0.2	19.0	25.3	4.3 <sup>a</sup>
	Soybean+Cane	2.0	0.2	20.1	25.5	4.8 <sup>a</sup>
	BioChar	2.3 <sup>a</sup>	0.2	20.4	25.2	7.2 <sup>ª</sup>

## 6.2 Incubation Experiment 2

## 6.2.1 Physical properties

The Dispersion Ratio (which is related to aggregate stability under disruptive shaking without chemical dispersion) was not affected by mill mud application in either soil (Table 6). There was also no significant effect of mill mud application on water content of either soil at field capacity (0.1 bar water content) or wilting point (15 bar water content).

Table 6. Effect of mill mud at 100 wet tonnes/ha on key physical properties of two soils, 120 days after incorporation. Standard errors of the means are in parentheses.

	Treatment	15 bar Water (%)	0.1 bar Water (%)	Dispersion Ratio
Loamy Sand	Control	3.4 (0.2)	6.9 (0.0)	0.72 (0.02)
	Mill mud @ 100 wet tonnes/ha	3.8 (0.2)	7.1 (0.2)	0.72 (0.01)
	Urea @100 kg N/ha	3.7 (0.1)	7.1 (0.4)	0.72 (0.02)
Clay	Control	17.3 (0.3)	24.8 (0.2)	0.59 (0.02)
	Mill mud @ 100 wet tonnes/ha	17.3 (0.2)	24.3 (0.5)	0.62 (0.03)
	Urea @100 kg N/ha	17.2 (0.3)	24.1 (0.5)	0.59 (0.02)

## 6.2.2 Chemical properties

Mill mud application to the loamy sand soil significantly (P<0.05) increased soil pH to a value similar to the pH of the mill mud, but there was no effect on pH of the clay soil (Table 7). There were no significant effects of mill mud on PBI in either soil. Effective cation exchange capacity (ECEC) was significantly (P<0.05) increased by mill mud incorporation in the loamy sand, and ECEC increased with increasing rate of application (Table 7). However, there was no effect of mill mud on ECEC of the clay soil.

As was observed in the pot trial soil analyses, total organic C (TOC) was not significantly (P=0.05) increased 120 days after mill mud application to either soil, although there was a trend in the loamy sand for TOC to consistently increase with rate (Table 7).

Table 7. Effect of rates of mill mud on key chemical properties of two soils, 120 days after incorporation. Standard errors of the means are in parentheses.

	Treatment	pH (1:5 water)	PBI	ECEC (cmol <sub>c</sub> /kg)	Total Org C (%)
Loamy	Control	5.9 (0.0)	36 (0.4)	5.2 (0.13)	2.0 (0.13)
Sand	Urea @ 100 kg N/ha	5.9 (0.02)	36 (0.7)	4.9 (0.09)	2.0 (0.04)
	Mill mud @ 50 wet tonnes/ha	6.4 (0.02)	48 (1.9)	6.3 (0.15)	1.9 (0.12)
	Mill mud @ 100 wet tonnes/ha	6.8 (0.02)	59 (6.5)	7.1 (0.11)	2.1 (0.09)
	Mill mud @ 200 wet tonnes/ha	7.1 (0.2)	107 (15.5)	9.9 (1.1)	2.3 (0.28)
Clay	Control	5.6 (0.04)	212 (4.9)	21.2 (0.17)	1.8 (0.05)
	Urea @ 100 kg N/ha	5.6 (0.1)	199 (8.4)	21.8 (0.32)	1.9 (0.04)
	Mill mud @ 50 wet tonnes/ha	5.4 (0.0)	191 (9.5)	21.2 (0.18)	1.7 (0.08)
	Mill mud @ 100 wet tonnes/ha	5.7 (0.1)	191 (4.9)	22.5 (0.68)	2.1 (0.11)
	Mill mud @ 200 wet tonnes/ha	5.9 (0.0)	191 (9.5)	23.4 (0.37)	2.0 (0.06)

## **7 Discussion: Incubation Experiments**

## 7.1 Effect of mill mud/mud ash on soil physical properties

At the application rate of 100 wet tonnes mill mud or mud ash/ha, there was no effect on soil water holding capacity or water stable aggregation in the <2mm soil particle size of two soils of contrasting texture (Incubation Experiment 2), although there was an increase in stable micro-aggregates in the loamy sand soil, but not the clay soil, in Incubation Experiment 1. These results suggest that rates in excess of 100 wet tonnes/ha of mill mud/mud ash may be necessary if the aim is to improve soil physical properties; however, such improvements are only likely to occur in soils with light-textured (sandy) topsoils (e.g. Kandosols, some Dermosols, some Kurosols), if at all. From a nutrient application/environmental viewpoint, such rates are unacceptably high (see Section 8.2.2).

## 7.2 Effect of mill mud/mud ash on soil chemical properties

Mill mud application to the light-textured loamy sand soil increased soil pH and effective cation exchange capacity (ECEC), with the effect increasing with rate of application (Table 7). There were no such effects on the heavier-textured clay soil. The liming capacity of the mill mud (due to its neutral pH and content of functional organic groups such as carboxylates) is apparently limited and was only effective in the loamy sand soil which has a low pH buffer capacity. The higher pH buffer

capacity of the clay soil resulted in no significant effect (P=0.05) of the mill mud on soil pH except for a small increase at the highest rate of application. Mill mud has a substantial ECEC (approximately 57-64cmol<sub>o</sub>/kg; Bloesch and Barry, 2010) that is pH dependent, and as soil pH increases, so does ECEC. This pH effect is evident in the loamy sand soil, with ECEC increasing by 40% as soil pH changed from 5.9 to 6.8 as a result of the application of 100 wet tonnes mill mud /ha. The clay soil has a substantial ECEC because of its clay content and mineralogy, and there was no effect of mill mud on ECEC in this soil. In summary, mill mud at an application rate of 100 wet tonnes/ha is unlikely to have any impact on the soil pH or ECEC of heavy-textured surface soils. On the other hand, mill mud application to light-textured surface soils will increase both soil pH and ECEC, although the duration of this effect will depend on how quickly the carbon in the mill mud is oxidised or incorporated into the soil microbial biomass. These conclusions are based on results from only two soils, and assessment of mill mud application on the properties of a wider range of soil types, including sodic soils, is required.

## 8 Decision Support

The objectives of the decision support component of the project were to:

- Identify appropriate analytical methods for monitoring N and P bioavailability in batches of mill mud and mill mud products over time and from different sources.
- Assess the environmental risk associated with the application of mill mud and mill mud products to different soil types in different regions.
- Develop guidelines for application of mill mud products to land and advise policy implications.

## 8.1 Analytical methods for assessing nutrient bioavailability

## 8.1.1 Nitrogen

A proportion of the total N content of mill mud/ mud ash can be used as an indicator of the potential of the material to supply N to the current crop. The results above show that 35% of the total N applied as urea is recovered in plant tops after 120-150 days. Limited data on N content of above-ground biomass and excavated cane stools at stalk elongation (D Sparkes, QDAFF 2014) indicate that N in the tops comprises 70% of N in the entire plant (i.e. above-ground biomass plus stool). It can be inferred, therefore, that the current crop recovers approximately 50% of applied N that is immediately bioavailable (e.g. urea-N) under conditions of negligible N loss. This 50% recovery of applied N is suggested as the upper limit for potential recovery of the total N applied in mill by-products, i.e.:

## N available to current crop (kg N/ha) = 0.50\*Total N (kg/ha) applied as mill mud/mud ash

## 8.1.2 Phosphorus

The quantity of BSES-extractable P applied in the mill mud/mud ash is correlated with crop P uptake (Fig. 4, Fig. 8). However, incorporating the mill mud/mud ash in the soil exposes the mill mud/mud ash P to soil fixation processes that can be assessed by determining the P buffer index (PBI) of the receiving soil; as PBI increases, P fixation increases, and the availability of the applied P decreases. The three soils used in the pot trials and incubation studies of this project had different PBIs and Table 8 indicates the BSES-P extractability (i.e., BSES-P Amended soil/BSES-P Mill mud) at different PBIs.

	BSES-P extractability				
PBI	(%)				
25	107				
42	88				
270	41				

Table 8. Effect of PBI of the receiving soil on the extractability of BSES-P from mill mud/mud ash sources.

A linear regression equation was fitted to the relationship between the PBI of the receiving soil and the extractability of the mill mud/mud ash BSES-P incorporated into the soil (Fig. 10).

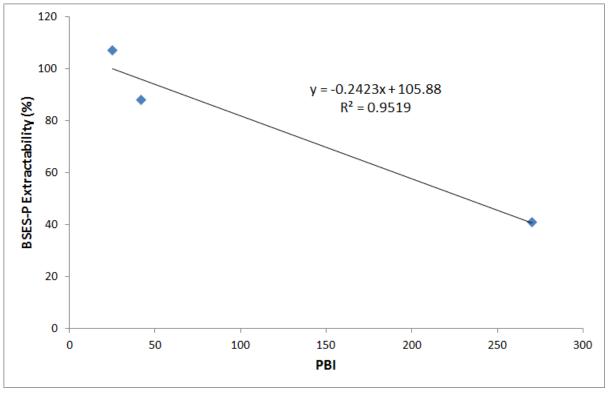


Figure 10. The relationship between the PBI of the receiving soil and the extractability of the mill mud/mud ash BSES-P incorporated into the soil. Data from the three soils used in the current study.

From the regression equation in Fig. 10:

## BSES-P in amended soil after incorporation of mill mud/mud ash =

## BSES-P Receiving soil + BSES-P Mill mud \*[(106 - 0.24 PBI Receiving soil)/100]

where BSES-P <sub>Receiving soil</sub> and BSES-P <sub>Mill mud</sub> are mg P/kg soil. BSES-P <sub>Mill mud</sub> is calculated from the total amount of BSES-P applied/ha as mill mud/mud ash divided by the weight of the receiving soil (e.g. 10<sup>6</sup> kg soil equates to 1 ha.10 cm of soil of bulk density 1 g/cm<sup>3</sup> assuming mill mud/mud ash incorporation to a depth of 10 cm).

## 8.2 Decision support tools for assessing environmental risk

Application of mill mud/mud ash by either incorporation or broadcasting will have environmental implications because of the ready bioavailability of both N and P in the products. From the above results, the following parameters can be used to calculate this bioavailability:

Nitrogen - total N applied (kg/ha) in the mill mud/mud ash

Phosphorus - BSES-P and PBI of the receiving soil

- BSES-P (dry weight basis) of mill mud/mud ash
- moisture content (60°C) of mill mud/mud ash
- depth of incorporation of mill mud/mud ash.

Note: Calculations are not applicable to broadcast or strip-applied product that is not incorporated into the soil. This is because of the limited and non-uniform contact that occurs between the product and the soil when product is broadcast.

## 8.2.1 SafeGauge for Nutrients update

SafeGauge for Nutrients will be updated so that these nutrient inputs are calculated from userentered data and added to the N (Table 9) and P (Table 10) budgets used to assess risk indices.

Table 9. Look-up table for estimating N inputs to the current crop from mill mud/mud ash application. <sup>A</sup>'Low' and 'Mean' refer to the values reported in Bloesch and Barry (2010) for a range of mill muds from Queensland mills.

Application rate (wet tonnes/ha)	Water content (60°C)	'Low' Total N (%)	'Mean' Total N (%)	'Low value' <sup>A</sup> N addition (kg N/ha)	'Mean value' <sup>A</sup> N addition (kg/ha)
50	75	0.84	1.48	53	93
100	75	0.84	1.48	105	185
150	75	0.84	1.48	158	278

The amount of N available to the crop following mill mud application at 150 wet tonnes/ha varies from 158kg N/ha to 278kg N/ha, depending on the assumed total N content of the product (Table 9). The current discount that is made in the SIX EASY STEPS for this mill mud application rate is 80 kgN/ha in the current crop and reduced amounts for the following two crops, with a cumulative discount of 140 kg N/ha. It is therefore recommended that the 'low value' N addition of 105 kg N/ha is used as the discount for mill mud N contribution to the current crop. Where the total N content of the applied mill mud/mud ash is actually known, then the appropriate N discount can be calculated from the equation in Section 8.1.1. Based on the findings of this project, discounts for N supplied

from the mill mud/mud ash are only applicable to the plant crop following mud application to the fallow, or the ratoon crop receiving the application.

Table 10. Look-up table for estimating additional BSES-P in the amended soil following application and incorporation to 10 cm of mill mud. It is assumed that the mill mud (dried at 60°C) has a BSES-P concentration of 4665 mg P/kg [mean value reported in Bloesch and Barry (2010) for a range of mill muds from Queensland mills]. Superscript indicates that this additional BSES-P concentration is in equilibrium with a soil solution P concentration that exceeds ANZECC water quality guidelines by at least ten-fold.

		Receiving soil PBI		
Application rate (wet tonnes/ha)	Water content (60°C) (%)	Low PBI (35)	Medium PBI (140)	High PBI (280)
50	75	+57 <sup>A</sup>	+42	+23
100	75	+114 <sup>A</sup>	+84	+45
150	75	+171 <sup>A</sup>	+127 <sup>A</sup>	+68

<sup>A</sup>FRP in soil solution>10 times ANZECC water quality guidelines

The BSES-P concentration in the receiving soil following amendment with mill mud can be estimated by adding the BSES-P values in Table 10 to the BSES-P of the soil prior to amendment. It is apparent that even if mill mud at 100 wet tonnes/ha is added to a soil with zero BSES-P, the soil solution of an amended low PBI soil will far exceed ANZECC water quality guidelines for filterable reactive phosphate.

## 8.2.2 Mill mud as a nutrient source

Table 11 indicates the quantities of N and P applied at different rates of mill mud in comparison to the typical nutrient removal rates of sugarcane crops. There is a gross imbalance in the N/P inputs, with an application of 50 wet tonnes/ha of mill mud providing sufficient N for one crop but sufficient P for five crops. Mill mud should therefore be viewed by industry as a P source, and the repeat frequency of mill mud applications to a block based on the cumulative P loading to the soil. As indicated in Section 4.2 above, it is the P added in the mill mud that poses an environmental risk.

Nutrient	Nutrient removal in cane/tops/trash <sup>a</sup> (kg/ha)		Nutrient input from mill mud <sup>в</sup> (kg/ha)		
	Annual	5 year crop cycle	50 wet tonnes/ha	100 wet tonnes/ha	
N	136	680	185	370	
Р	21	105	114	228	

 Table 11. Nutrient (N and P) inputs from mill mud compared to the nutrient removal rates by sugarcane crops

<sup>A</sup>Chapman et al. (1981); <sup>B</sup>Barry et al. (1998)

## 8.3 Guidelines for application of mill by-products

- There was little difference between mill mud/mud ash that was broadcast or incorporated in efficacy of nutrient supply. However, incorporation will reduce the risk of nutrient loss by runoff and may reduce potential denitrification losses.
- Application of mill mud/mud ash at rates >100 wet tonnes/ha will cause unacceptable concentrations of inorganic phosphorus in the soil solution, resulting in high FRP concentration in runoff water. Low P sorbing soils (PBI<35) are particularly high risk for producing runoff and/or drainage water with unacceptable FRP concentrations following mill mud/mud ash application.
- Mill mud/mud ash applications to improve soil 'health' by improving soil chemical and/or physical characteristics will need to be at/higher than 100 wet tonnes/ha. In any case, soil 'health' benefits are only likely to accrue in soils with light-textured (sandy) topsoil.
- Strip application of mill mud/mud ash over the crop row doubles the application rate per unit area of fertilised soil, i.e. 50 wet tonnes/ha applied only over the crop row actually results in an application of 100 wet tonnes/ha of crop row, assuming the row comprises 50% of the area of the block.

## **9 Further Work**

- After the initial crop following mill mud/ash application, the inability to discriminate residual N of mill by-product origin from background soil organic nitrogen highlights the necessity to have reliable prognostic indices for estimating the amount of nitrogen likely to be mineralised from the soil organic matter pool during the rest of the crop cycle.
- The uncertainty in estimating the amount of nitrogen released to the crop from residual mill by-product nitrogen and from the soil organic matter could be addressed by developing an 'on the go' assessment of plant/ratoon crop nitrogen status before the 'out of hand' growth stage. This assessment could be used to inform side-dress fertiliser application rates.

Given the risk of off-site dissolved and particulate phosphorus movement from soils with a
history of mill by-product applications, it is timely to undertake a survey of the phosphorus
status of cane soils to identify high risk areas for targeted remedial action. Dissolved and
particulate phosphorus are both drivers of algal blooms in freshwater and marine
ecosystems, yet the significance of dissolved inorganic phosphorus to Reef water quality
has not been sufficiently explored and addressed to date.

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## Attachment 1

Paper presented at the 2014 Conference, Australian Society of Sugar Cane Technologists, Gold Coast, 29 April-1 May 2014.

#### Ag 12. EFFECTS OF THE APPLICATION OF MILL MUD/MUD ASH ON SOIL PROPERTIES AND AVAILABLE SOIL NITROGEN AND PHOSPHORUS

By

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#### Abstract

Mill mud and mud ash are valuable sources of nutrients (nitrogen, phosphorus, potassium) as well as having high contents of organic carbon. Pot experiments and an incubation study were carried out using three cane soils of differing textures to determine the rate of release of nitrogen (N) and phosphorus (P) from the products for uptake by sugarcane, and to determine the effects of mill mud on key soil chemical and physical properties. Approximately 10% of the total N applied in the mill by-products was recovered in sugarcane tops grown for 120-142 days, and using the availability of urea-N as a benchmark, it was estimated that a further 25% of the total N applied in the mill by-products would be available for crop uptake in the first season. Phosphorus availability in the products could be measured using BSES-P, and an equation was developed for calculating soil BSES-P after amendment with the mill by-products. Application rates in excess of 100 wet tonnes/ha amended soil were required for measurable changes in soil cation exchange capacity and aggregate stability, and these changes only occurred in the light-textured (3% clay) soil. However, these application rates resulted in unacceptably high concentrations of dissolved inorganic P in the soil solution that posed a risk to water quality.