

SUGAR MILL WASTES CAN BE IMPORTANT SOIL AMENDMENTS

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Abstract

The effects of additions of three wastes from sugar mills on the properties of an acid soil were investigated. The wastes used were boiler ash and filter cake from a conventional milling operation and fly ash from a new milling process, where filter cake is burnt in the boilers. Additions of each of the wastes at two sites raised soil pH and increased maize yields in a pot experiment; the effect being greater at the higher rate. Concentrations of exchangeable Al and total and monomeric Al in soil solution were reduced by additions of each of the wastes. Thus, the increase in pH caused precipitation of potentially phytotoxic soluble and exchangeable Al as hydroxy-Al polymers. Waste additions increased the basal respiration rate and the activities of the soil enzymes arylsulphatase and acid phosphatase. Additions of filter cake increased the organic C content of the soil and the amount of N mineralised during incubation. It was concluded that all of the waste materials had a liming effect and at high rates they increased the microbial activity in the soil. In particular, filter cake was found to be a valuable organic amendment that should be recycled back onto sugarcane fields to increase their fertility and organic matter status.

Keywords: Filter cake, fly ash, boiler ash, soil acidity, soil microbial activity

Introduction

Filter cake (also known as filter press mud) is a waste produced during clarification of juice, extracted from the cane, following milling. Lime is added to the heated juice and the suspended organic matter is then filtered in filter presses and collected as a solid cake. The filter cake contains substantial amounts of CaCO₃, and with the filtered impurities, it constitutes a valuable fertilizer/liming material as well as an organic amendment (Alexander, 1971; Roth, 1971; Moberly and Meyer, 1978). It is often applied as an amendment to sugarcane soils immediately surrounding the mills. Despite this, large stock-piles of filter cake often accumulate at mill sites and this is generally considered a waste disposal problem. Most mills use their fibre wastes from milling (bagasse) as a fuel to fire their boilers and thus produce their own energy. At times when there is a shortage of bagasse, coal can be used as an additional fuel. The "boiler ash" produced is normally stockpiled and / or dumped. The drive towards greater energy efficiency in the mills has led to further extraction of sugar from filter cake followed by recycling of dried filter cake to fire the boilers along with bagasse. Thus, the two main organic (combustible) wastes of milling are burnt to provide energy. However, from an environmental viewpoint this also means that no organic wastes are produced that can be returned to sugarcane soils, the only waste product produced is "fly ash" from the boilers.

Loss of soil organic matter is a major contributor to soil degradation under sugarcane monoculture (Meyer *et al.*, 1996). A loss of soil organic matter generally leads to a decrease in soil biological activity and degradation of soil physical conditions (Haynes and Beare, 1996). For this reason, use

of filter cake as an organic amendment for sugarcane soils has been actively promoted by SASEX for many years. Ash can also be a valuable amendment (Hackett *et al.*, 1999; Raison, 1979). If combustion of plant material is nearly complete most of the carbon (C), hydrogen (H), oxygen (O), nitrogen (N) and organic sulphur (S) and phosphorus (P) are volatilized (Zhang *et al.*, 2001), while many of the cationic constituent elements are rendered water-soluble and hence, immediately available for plant uptake (Raison, 1979). The most beneficial aspect of ash additions to soils is their high pH and therefore their liming capacity (Lerner and Utzinger, 1986; Vance, 1996).

It is clear that all three waste products from sugar mills (filter cake, boiler ash and fly ash) could be used as liming materials to raise soil pH and add nutrients to the soil. Filter cake has a significant organic matter content and will add basic cations plus C, N, S and P in organic forms while the ash will add predominantly basic cations and anions such as carbonate and silicate. Liming will ameliorate soil acidity by precipitating exchangeable and soluble Al and will therefore promote crop growth (particularly of acid sensitive plants). Liming could also increase soil microbial activity (Carter, 1986; Curtin *et al.*, 1998) and additions of organic matter in the filter cake should magnify such an effect.

The purpose of this study was to compare the effects of waste applications from conventional milling (i.e. boiler ash and filter cake) with those from new milling operations (i.e. fly ash) on the properties of two acid soils from the sugar belt and on plant growth in them. Using a simple laboratory incubation/pot experiment, it was hoped to demonstrate the value of recycling these waste products back onto sugarcane soils. On one of the soils, the effects of waste applications on phytotoxic Al concentrations in soil solution and on soil microbial and enzyme activity was investigated in detail.

Methods and Materials

Experimental design

Soils were collected from two sites on the coastal lowlands of KwaZulu-Natal. The two soils were chosen to be representative of the main groups of soils that are prevalent in the sugar belt. Samples (0-10 cm) were taken from areas under undisturbed veld in the Upper Tongaat area. The first soil was of the Cartref form (Gleyic Luvisol, FAO; Inceptisol, USDA) and it had a clay content of 13%. The second was of the Shortlands form (Chromic Luvisol, FAO; Oxisol, USDA) with a clay content of 36%. The soils were analyzed for available nutrients at the SASEX Fertiliser Advisory Service (FAS) laboratory (Meyer *et al.*, 1997; Beater, 1962) and the results are presented in Table 1.

Table 1. Some selected chemical properties of the two soils used.

Soil type	Truog-extractable P	CaCl ₂ -extractable Al (mg kg ⁻¹)	Exchangeable cations (mg kg ⁻¹)				pH (H ₂ O)
			K	Ca	Mg	Na	
Cartref	9	11	94	43	22	12	4.6
Shortlands	14	5	112	302	111	38	5.1

The filter cake and boiler ash originated from the Maidstone mill (Tongaats) while the fly ash came from the Union Co-op mill (Dalton) where both bagasse and filter cake are burnt. These materials were analysed by FAS (Meyer *et al.*, 1997; Beater, 1962) and their chemical properties are shown in Table 2.

Table 2. Some chemical properties of the waste materials.

Waste	pH (H ₂ O)	Total element content (%)						Citric soluble P (mg kg ⁻¹)	Water extractable Si (mg kg ⁻¹)
		N	P	K	Ca	Mg	Si		
Filter cake	6.85	1.68	1.13	0.31	1.1	0.40	3.2	1.04	124
Fly ash	7.69	0.01	0.71	0.42	2.3	0.73	8.4	0.49	375
Boiler ash	7.45	0.13	0.46	0.47	4.4	0.55	8.3	0.21	152

The experiment involved two parts. First, the two soils were incubated with the three wastes at two rates and half of the samples were kept for chemical/biochemical analysis. Secondly, the other half of the samples were used in a pot experiment in which maize was grown. Due to time constraints, detailed chemical and biochemical analyses were carried out only on the Cartref samples. Soil pH and maize dry matter yields were, however, recorded for both soils.

The soils were air-dried and sieved (< 2 mm) and the waste materials were dried and ground (<0.5 mm). The incubation experiment had three sources of waste (filter cake, boiler ash and fly ash) and a control. These wastes were added to the two soils (six replicates per treatment) at two rates (10 and 20 mg g⁻¹) which are equivalent to about 10 and 20 Mg ha⁻¹ to a depth of 10 cm respectively. The rates of addition were relatively high but were chosen to match those used by Mokolobate (2000). Amendments were thoroughly mixed with the soil samples (1kg), placed in 2L plastic containers (fitted with perforated lids), rewetted to 70% of the water holding capacity and incubated at 25 °C for eight weeks. Containers were opened each week and water was added when necessary to maintain the soil at a predetermined soil water content.

At the end of the incubation, three replications were removed for chemical and biochemical analysis while the other three were placed in one-litre plastic pots and transferred to the glasshouse for use in a pot experiment. Twenty-four hours prior to sampling for chemical analysis, soils were rewetted to 100% water holding capacity and solutions were extracted from subsamples of the Cartref soil by centrifugation (Eikhatib *et al.*, 1987). The soils were then allowed to dry to 70% of the water holding capacity and subsamples of the Cartref soil were sieved (< 2 mm) and stored at 1 °C for subsequent biochemical analysis. The remainder of the Cartref soil was air-dried and sieved (< 2 mm). A subsample was finely ground (< 150 : M) for organic C analysis.

Pot experiment

For the glasshouse experiment, three replications of treatments for both soils were transferred into 2 litre pots and planted with maize (*Zea Mays* L. cv. Star 7711). Basal dressings of N, P, K, Mg, Mn, Mo, Zn, Cu and B at rates of 220, 25, 200, 100, 10, 10, 10, 10 and 4 mg kg⁻¹ respectively were applied to each of the pots. Six seedlings were planted per pot and were later thinned to three. The plants were placed in the glasshouse at a temperature ranging between 25 and 26 °C. The maize was allowed to grow for 9 weeks, until clear treatment effects could be seen but before the plants became root bound. Plants were harvested and the herbage was oven dried (at 70 °C) and weighed.

Soil analysis

Soil pH was measured in both soils with a glass electrode, using a 1:2.5 soil : water ratio. Other chemical and biochemical properties were measured only on the Cartref soil. Exchangeable aluminium (Al) was extracted with 1 M KCl (1:2.5 soil : extractant ratio for 1 hour) and Al was determined by atomic absorption spectrophotometry. Monomeric Al (Al_{mono}) in soil solution was measured in the filtrate (0.05 : M millipore filter extract) by the pyrocatechol violet (PCV) method (Kerven *et al.*, 1989), and total soluble Al (Al_T) was measured by a modified PCV method using LaCl₃ - Fe reagent after the solution had been passed through a 0.22 : M filter (Menzies *et al.*,

1992). We accept that the PCV reagent will tend to overestimate Al_{mono} because it will react with a small amount of Al present in Al-organic matter complexes (Parfitt *et al.*, 1995).

Organic C was determined by the Walkley-Black oxidation procedure (Walkley, 1947). Basal respiration was determined by placing 30 g soil into 50 ml beakers and incubating the samples in the dark at 25 °C in one litre airtight sealed jars along with 10 ml of 0.5 M NaOH solution. The CO_2 -C evolved was measured after 10 days by titration (Anderson, 1982). Exchangeable inorganic soil N (NH_4^+ and NO_3^- -N) was determined by distillation (Keeney and Nelson, 1982) at the beginning and end of incubation after extraction with 2 M KCl using a 1:5 soil : extractant ratio. The assays of various enzyme activities were based on the release and quantitative determination of the product in a reaction mixture. Samples were incubated with a suitable substrate and buffer solution. Assays were performed to determine the activity of acid phosphatase (EC 3.1.3.2), alkaline phosphatase (EC 3.1.3.1) and aryl-sulphatase (EC 3.1.6.1) as described by Tabatabai (1994). Enzyme activity was expressed as : mol product released g^{-1} soil h^{-1} .

Statistical analysis

Experimental results were analyzed by Analysis of Variance using the Genstat V package. Least significant differences (LSD) were calculated at the 5% level.

Results and Discussion

It is evident that all three waste materials functioned as liming agents when added to acid soils (Figure 1). Boiler ash was the most effective in this regard but both fly ash and filter cake also had substantial liming ability. Although boiler ash had a lower initial pH than fly ash (Table 1) it was more effective in raising soil pH. This is presumably related to its much higher basic cation content (especially Ca) (Table 1). The acid-neutralizing ability of ash results from oxides and carbonates of basic cations that remain after incineration (Vance, 1996). Filter cake also induced a pH increase and the dominant mechanism involved was probably simply its relatively high $CaCO_3$ content. A sample of filter cake from the same mill as that used in this study was shown by Mokolobate and Haynes (2002) to have a $CaCO_3$ content of 12% and it acted as a liming material.

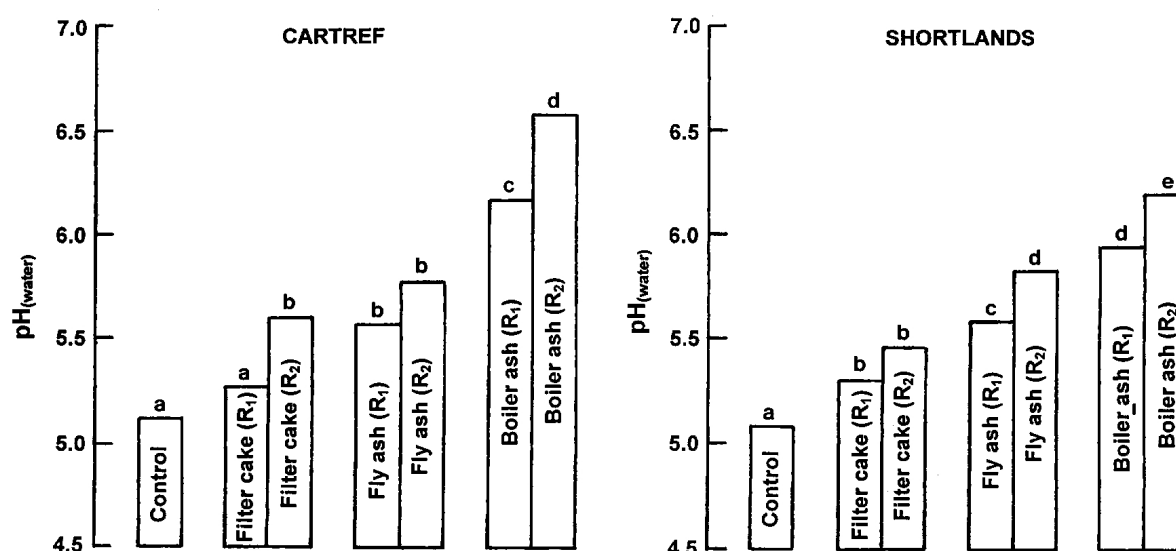


Figure 1. Effects of incubation of wastes added to samples of Cartref and Shortlands soils on pH. Rates of waste addition were 10 (R₁) and 20 (R₂) $mg\ g^{-1}$. Means associated with the same letter are not significantly different ($P \leq 0.05$).

In addition to raising soil pH, the amendments will also add significant amounts of nutrients to the soils. As shown in Table 1, both ashes contained substantial amounts of K, Ca, Mg and Si. The filter cake had a relatively high N content and also contained significant quantities of P and Ca.

As expected, concomitant with the increase in pH induced by addition of these wastes was a marked decrease in exchangeable Al (Figure 2). This was accompanied by a decrease in both Al_T and Al_{mono} in soil solution (Figure 3). Thus, the liming effect of the wastes caused precipitation of exchangeable and soluble Al^{3+} species as hydroxy-Al species.

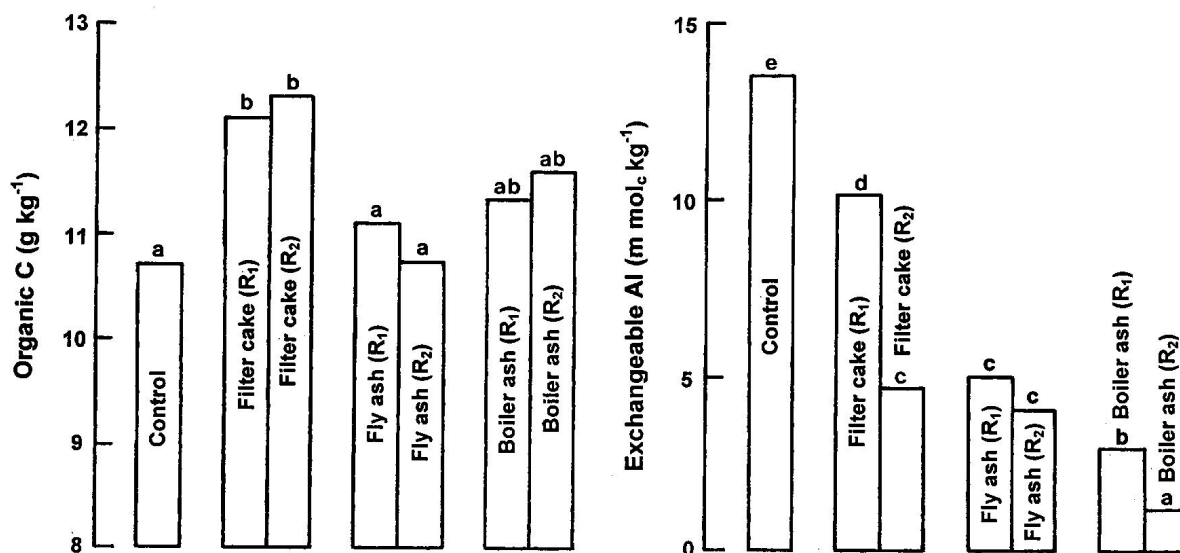


Figure 2. Effects of incubation of wastes added to samples of Cartref soil on concentrations of organic C and exchangeable Al.

For explanation of terms see Figure 1.

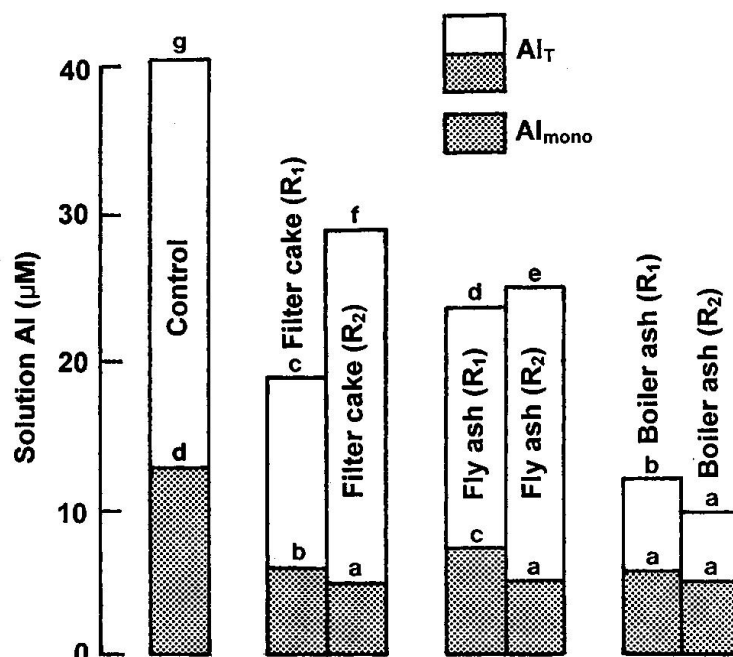


Figure 3. Effects of incubation of wastes added to samples of Cartref soil on concentrations of total (Al_T) and monomeric (Al_{mono}) Al in soil solution.

For explanation of terms see Figure 1.

The Al_T concentration in soil solution ranged from 10 to 41 : M (Figure 3) which is in the range of 10 - 350 : M typically found in soil solutions extracted from acid soils (Bruce *et al.*, 1988; Parfitt *et al.*, 1995). The proportion of Al_T present as Al_{mono} in the unamended soil was 33% of which is also in agreement with the results of others (Berek *et al.*, 1995; Slattery and Morrison, 1995). It is the activity of Al^{3+} and/or monomeric hydroxy- Al species (eg. $AlOH^{2+}$ and $Al(OH)_2^+$) that are most negatively correlated with depressed yields due to Al toxicity (Haynes and Mokolobate, 2001). Measured values of Al_{mono} were reduced from 13 : M in the control soil to below 7.5 : M (mean 5.6 : M) in amended soils (Figure 3). Such a reduction could well be of considerable significance. For example, the critical soil solution Al_{mono} concentration above which maize growth is limited by Al toxicity is in the range of 3-8 : M (Harper *et al.*, 1995; Diatloff *et al.*, 1998).

Indeed, the observed increases in plant growth in response to addition of amendments (Figure 4) were presumably principally lime responses. That is, the decrease in exchangeable Al and Al_{mono} in soil solution resulted in amelioration of Al toxicity and increased growth. Plant growth is often limited by Al toxicity in acid soils and this is characterized by a marked reduction in root and shoot growth. Aluminium toxicity disrupts mineral nutrition and limits the ability of plants to absorb and translocate P, Ca and other nutrients (Kochian, 1995). Since Ca and Mg was not applied in the basal dressing, the observed yield responses could well be partially attributable to Ca and Mg additions in the waste materials. Thus, yield responses may be the result of an interaction between increased pH and increased available Ca and Mg especially in the Cartref soil which was deficient in these nutrients.

Although in comparison to maize, sugarcane is relatively acid tolerant (Hetherington *et al.*, 1988), lime applications are required in order to maintain soil pH at acceptable levels. Indeed, large applications of nitrogenous fertilisers made to sugarcane soils tend to result in acidification and the pH of South African sugarcane soils is tending to gradually decrease (Meyer *et al.*, 1996). Thus both boiler and fly ash represent valuable amendments to sugarcane soils.

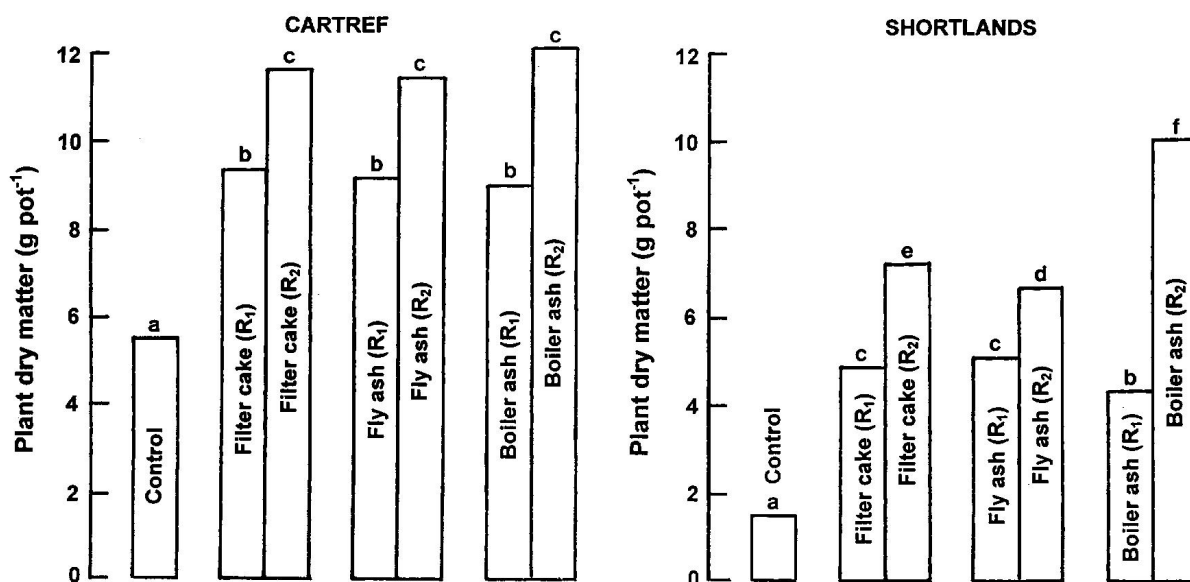


Figure 4. Effects of incubation of wastes added to samples of Cartref and Shortlands soils on subsequent growth of maize plants after nine weeks.

For explanation of terms see Figure 1.

The increase in soil organic C content caused by additions of filter cake (Figure 2) was expected since it had a substantial organic matter content (Table 1). The tendency for elevated organic C contents in ash-amended soils is presumably due to C of a charcoal-like nature (i.e. C in ash) being

measured by the dichromate oxidation procedure used (Skjemstad *et al.*, 1999). Charcoal C is considered as biologically inert inactive C whilst the C from filter cake will provide a substrate for soil microbial activity.

Despite the fact that the lower rate of waste decreased or had no effect on basal respiration (Figure 5), the higher rate markedly increased respiration rates. Thus, microbial activity was only increased at the higher rate of addition. This increased activity is probably primarily attributable to the increase in soil pH but additions of Ca, Mg, K and P in the ash could also have increased the soil fertility status and microbial activity. As noted above, in the case of filter cake, the addition of a C source may have been an additional factor. Nonetheless, CO₂ evolution rates from filter cake treatments were, in fact, less than those from the boiler ash treatments suggesting that an elevation in pH was having a dominant effect. Other workers have shown that liming can increase microbial activity due to activation and/or proliferation of microbial species already present in the soil but which were relatively inactive before liming (Carter, 1986; Haynes and Swift, 1988; Neale *et al.*, 1997) so that raising soil pH and precipitating soluble and exchangeable Al induces increased microbial activity.

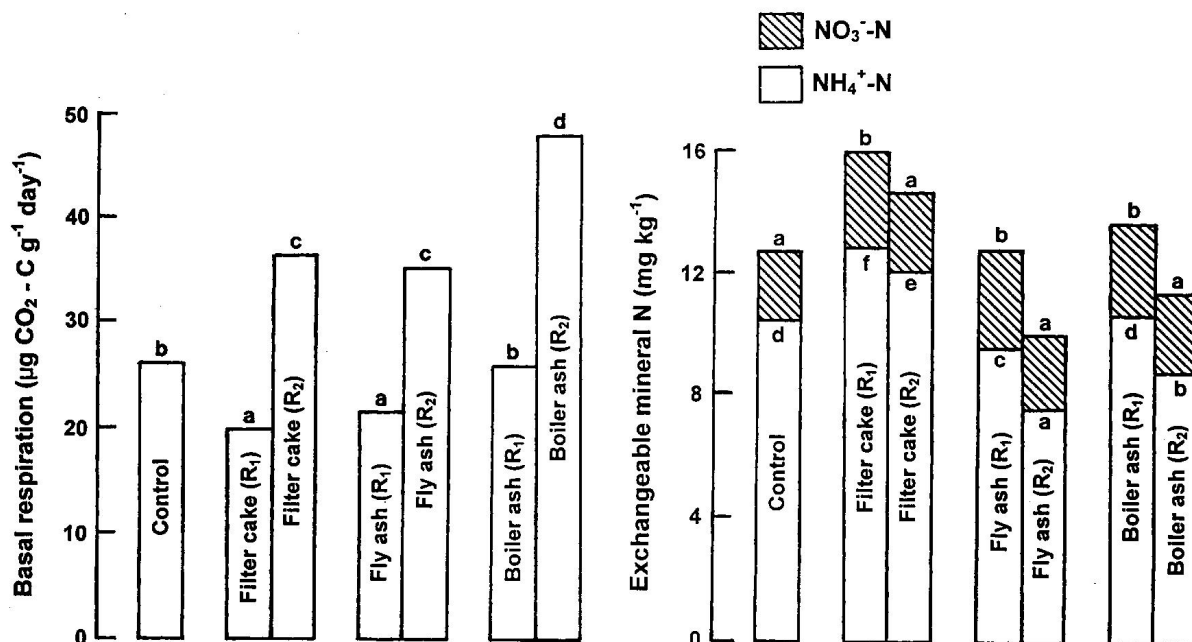


Figure 5. Effects of incubation of wastes added to samples of Cartref soil on basal respiration rate and accumulation of NH₄⁺- and NO₃⁻-N in the soil over a 10-day aerobic incubation. For explanation of terms see Figure 1.

While the basal respiration rate was only increased at the high rate of waste addition, the activities of both arylsulphatase and acid phosphatase were significantly increased by both the 10 and 20 mg g⁻¹ application rates (Figure 6). Indeed, for arylsulphatase the effect was very marked. Such results demonstrate that the activities of exocellular enzymes involved in specific nutrient transformations in soils are not necessarily directly related to soil microbial activity. This is because exocellular enzymes can be released in response to deficiencies of specific nutrients, negative feedback mechanisms can occur, they can be protected by sorption onto soil colloids and their activity can be influenced greatly by environmental factors (e.g. soil pH, ionic strength) (Tabatabai, 1994).

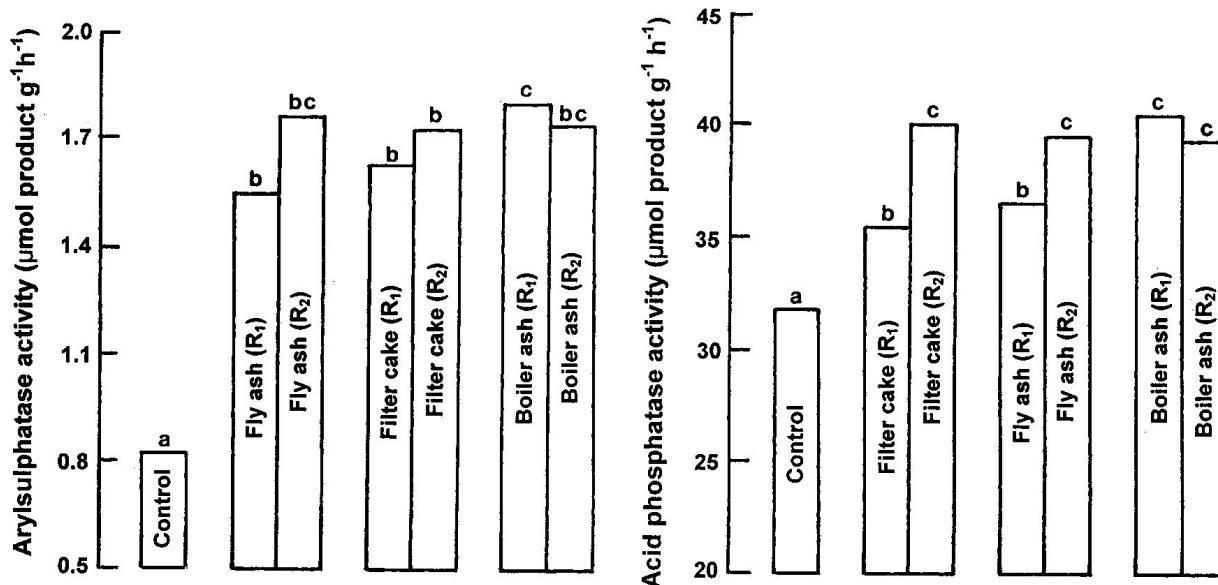


Figure 6. Effects of incubation of wastes added to samples of Cartref soil on arylsulphatase and acid phosphatase activities.

For explanation of terms see Figure 1.

The greater accumulation of mineral N in the soil in the filter cake treatment when compared with the control (Figure 5) was expected and was attributable to mineralisation of organic N present in the filter cake. However, the decreased accumulation of mineral N with increasing rates of addition of each of the wastes is surprising. The decrease was associated with an increase in CO₂ evolution and thus microbial activity. This suggests that relatively greater microbial immobilisation of N occurred at the high rates of waste addition. There is little C or N in ash so the effect does not appear to be related directly to C and N inputs to the soil. A possible explanation is that the soil was sampled from under undisturbed, unfertilised grassveld. Such plants grow under N - limited conditions, thus they have a low N content and their litter has a wide C / N ratio. Thus, as microbial activity is stimulated by large additions of wastes, there is greater immobilisation of N by the microflora decomposing the plant residues held in the soil, and mineral N accumulation is therefore decreased.

Conclusions

Results of this study show that boiler and fly ash are effective at raising soil pH and land application of these wastes could serve as liming materials. As well as raising pH, applications of ash result in additions of basic cations, P and Si and thus an improvement in soil fertility. It should be noted that the coal constituent of some boiler ashes could have the potential to cause accumulation of toxic levels of certain elements (e.g. B, Mo, S and Se) and this needs to be monitored.

In addition to having a liming effect and adding nutrients, filter cake is an organic material and its application will, as shown here, tend to raise soil organic matter levels. Since loss of soil organic matter is a common problem in sugarcane soils, this is an important attribute of the waste. Intensive efforts need to be continued to recycle this waste back to sugarcane land.

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