

Biosolids Soil Application: Agronomic and Environmental Implications 2014

Guest Editors: Silvana Torri, Rodrigo Studart Corrêa,
Giancarlo Renella, Alejandro Vadecantos, and Leonid Perelomov





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Applied and Environmental Soil Science

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Editorial

Biosolids Soil Application: Agronomic and Environmental Implications 2014

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World production of biosolids is continuously increasing due to global urbanization of society and the increase in wastewater treatment coverage [1, 2]. Nowadays, the accumulation of biosolids poses a growing environmental problem.

With the recent banning of ocean disposal and new stringent landfilling regulations, much more biosolids are now beneficially reused. Agriculture use of biosolids is still considered the main feasible disposal option, being both a cost-effective practice and a way to close the nutrient loop in agriculture and remediated soils. On the other hand, due to the ever increasing transportation, disposal costs, and increasing public pressure, efforts are being made to reduce the quantity of biosolids for disposal [3]. However, the demographic dynamics and technoeconomical problems in some developing countries lead to uncontrolled use of raw sewage in land application. This is mainly due to the fact that, in several areas around the world, increasing urbanization may lead to wastewater production in the order of 4.106 L per day, of which only a minor proportion can be effectively treated. Therefore, sewage systems often flow together with industrial effluents, enriching the biosolids with heavy metals, organic xenobiotics, endocrine disruptors, and hormones [4]. Land disposal of such material may cause detrimental effects on soil quality, crop production, and animal or human health.

In many countries, water shortages result in the use of treated municipal wastewater for agriculture irrigation.

Moreover, this practice is considered a valuable source for recycling and reuse of nutrients and water [5]. However, the use of sewage effluents for irrigation may cause soil pollution by the accumulation of hazardous elements [6]. Consequently, there is a considerable interest in the effects of this practice on the quality of crops intended for human consumption.

S. Shanmugam and L. K. Abbott reviewed the potential for applying modified biosolids with soil amendments such as lime, clay, and biochar. The potential benefits of combining biosolids with other amendments can include reduction in microbial processes, leading to slow biosolids-borne nutrients. Co-application of biosolids with clay and biochar may lead to the retention of N, decreasing N leaching through various mechanisms. Further investigation of the influence of amendments on modified biosolids products would enable a better understanding of soil nutrient cycling over the short and long term following its land application.

Progressive soil pollution with heavy metals also prevents the optimal use of other organic matter sources such as manure because this may increase soil metal mobility due to manure-induced soil alkalinisation. This can in turn increase metal solubilization and availability to crops caused by increased organic matter solubilization and soil salinity. The complex interplay of the environmental factors that can increase heavy metal toxicity was well illustrated by the work

of A. R. A. Usman who shows how the increase of soil salinity values can increase the Cd toxicity to soil microorganisms in contaminated soils and its availability to crops such as barley.

N mineralization of bioresources was studied by S. W. Ives et al., who have incubated for 56 days at 12.5°C soils samples treated with anaerobically digested biosolids, limed biosolids, poppy seed waste, and poppy mulch. The bioresources have, respectively, released 25%, 45%, 36%, and 8% of their total applied N as plant available nitrogen by the end of the incubation period. Mineralization rates were not related to bioresources C:N ratios; the poppy mulch showed a significant negative priming effect over the incubation period. Based on N contents released through mineralization, the incorporation of bioresources in soils of temperate regions may not match crop N demands.

The effects of composted biosolids on foliar concentrations of N, P, and K in *Salix miyabeana* (SX 64) were studied by A. K. Quaye et al. The authors have found that composted biosolids increased soil P supply more than commercial fertilizer used as control and that foliar nutrient concentrations reached or even exceeded values reported for optimal growth of field grown *Salix* species. Foliar N and K concentrations were significantly higher at the beginning of the growing season but decreased as the season progressed. On the other hand, foliar P concentrations increased towards the end of the growing season. The significant effect of the organic amendment on soil P supply and its comparable effect on foliar concentration make composted biosolids a cost-effective option relative to commercial fertilizers, besides the environmental benefits of land application of organic wastes.

S. Shanmugam and L. K. Abbott have investigated the residual effect of biosolids soil application (50 t ha^{-1}) on ryegrass growth and on soil microbial biomass in a glasshouse experiment. Results have showed that the residual effect of biosolids on plant growth lasted for five ryegrass cycles. Soil microbial biomass N was not significantly influenced by biosolids application. The residual concentration of mineral N remaining in soil was also negligible and did not pose risk to the environment.

R. C. Nogueiro et al. have compared the effectiveness of three P extractants considering the (i) availability of P in soils amended with sewage sludge for 13 consecutive years and correspondent maize P-uptake and (ii) availability of P in soils amended with a single application of sewage sludge and composted sewage sludge and correspondent sugarcane P-uptake. They have found no significant correlations between P levels extracted from soils and P concentrations in diagnostic leaves. Resin extracted larger amounts of P in the short term experiment, while acidic extractants yielded larger amounts in the long-term experiment.

Another evidence of the fertilizer value of biosolids in agronomy is described in the work of L. A. J. Teixeira et al. The application of biosolids may release higher agronomic efficiency on fruit production of banana plants than the equivalent application rates of mineral fertilizers. These results suggest that other benefits beyond nutrient inputs, such as an improvement on soil physical properties, a slower release of nutrients through the mineralization of organic

matter added with the sludge, and triggering of soil microbial activity, are taking place due to biosolids application.

In the paper "Surface-Applied Biosolids Enhance Soil Organic Carbon and Nitrogen Stocks but Have Contrasting Effects on Soil Physical Quality," mid- to long-term impacts of land-applied biosolids were studied by V. L. Jin. This study evaluates the effects of surface-broadcast biosolids application rate and duration on soil organic carbon stocks, soil aggregate stability, and selected soil hydraulic properties in a municipally operated, no-till forage production system. Total SOC stocks increased nonlinearly with application rate in perennial grass fields treated for 8 years with 0, 20, 40, or 60 Mg of Class B biosolids $\text{ha}^{-1} \text{yr}^{-1}$ (midterm treatments). Soil organic C stocks in long-term treatment fields receiving 20 years of $20 \text{ Mg ha}^{-1} \text{yr}^{-1}$ were 36% higher than those in midterm fields treated at the same rate. Surface-applied biosolids had contrasting effects on soil physical properties. Soil bulk density was little affected by biosolids applications, but applications were associated with decreased water-stable soil aggregates, increased soil water retention, and increased available water-holding capacity. This study contrasts the potential for C storage in soils treated with surface-applied biosolids with application effects on soil physical properties, underscoring the importance of site-specific management decisions for the beneficial reuse of biosolids in agricultural settings.

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Research Article

Surface-Applied Biosolids Enhance Soil Organic Carbon and Nitrogen Stocks but Have Contrasting Effects on Soil Physical Quality

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Mid- to long-term impacts of land applying biosolids will depend on application rate, duration, and method; biosolids composition; and site-specific characteristics (e.g., climate, soils). This study evaluates the effects of surface-broadcast biosolids application rate and duration on soil organic carbon (SOC) stocks, soil aggregate stability, and selected soil hydraulic properties in a municipally operated, no-till forage production system. Total SOC stocks (0–45 cm soil) increased nonlinearly with application rate in perennial grass fields treated for 8 years with 0, 20, 40, or 60 Mg of Class B biosolids ($\text{DM ha}^{-1} \text{yr}^{-1}$) (midterm treatments). Soil organic C stocks in long-term treatment fields receiving 20 years of $20 \text{ Mg ha}^{-1} \text{yr}^{-1}$ were 36% higher than those in midterm fields treated at the same rate. Surface-applying biosolids had contrasting effects on soil physical properties. Soil bulk density was little affected by biosolids applications, but applications were associated with decreased water-stable soil aggregates, increased soil water retention, and increased available water-holding capacity. This study contrasts the potential for C storage in soils treated with surface-applied biosolids with application effects on soil physical properties, underscoring the importance of site-specific management decisions for the beneficial reuse of biosolids in agricultural settings.

1. Introduction

Beneficial use of biosolids is a key management tool for publicly owned treatment works (POTWs) for waste utilization and is more highly regulated than land application of animal wastes [1–3]. When applied in compliance with regulations limiting trace metals inputs and at agronomically appropriate rates, land-applied biosolids are effective soil conditioners and can supplement or replace commercial inorganic fertilizers while improving soil water conservation [4]. In addition to using biosolids amendments to restore or build soil quality, organic waste applications could promote the mitigation of atmospheric carbon dioxide levels by enhancing soil organic carbon (SOC) storage [5–7].

The quantity and frequency of inputs, method and timing of application, biosolids composition, and site edaphic factors (e.g., soils, climate) as well as other associated management practices (e.g., tillage, irrigation) affect the transfer of biosolids-C into SOC pools [7–12]. Several literature reviews show that inputs of biosolids-derived organic matter can lead to both short-term and longer-term increases in SOC [4, 7–9, 13]. Further, organic matter increases associated with land applying biosolids are often linked to improvements in soil physical quality, including decreased soil bulk density, increased soil aggregate stability, enhanced soil moisture retention, and lower soil erosion potential [5, 9–11, 14].

Soil responses to land application in no-till systems using surface-applied biosolids may differ from soil responses in

cultivated management systems that use tillage to incorporate biosolids into surface soils. The available literature on surface-applied biosolids, particularly under field conditions, is limited relative to the number of published studies on incorporated biosolids. The objectives of this study were to evaluate the effect of application rate and duration on SOC stocks, soil aggregate stability, and selected soil hydraulic properties at a municipal biosolids recycling facility that surface-applies Class B biosolids on large-scale, no-till perennial forage production fields. Because the impact of biosolids applications on SOC and soil physical properties can be highly variable both temporally and spatially [11, 15, 16], studies assessing the mid- and long-term effects of repeated biosolids applications, such as the one conducted here, are critical to evaluate treatment effects and system sustainability in large-scale operational settings [5].

2. Materials and Methods

2.1. Site and Biosolids Descriptions. This site is a publicly owned, municipally operated 485 ha, zero-discharge facility in Travis County, Texas, USA. Soils are silt loams and silty clay loams (fine-silty, mixed, superactive, thermic Cumulic Haplustolls) and are very deep (>2 m to groundwater) and well drained, with slopes <1% [21]. Study site and management descriptions were reported previously [22] and were recently updated [17].

Biosolids are land-applied as anaerobically digested, dewatered Class B biosolids. Biosolids originate from sewage solids pumped to this recycling center from two major municipal wastewater treatment facilities servicing almost 95% of the city population (~900,000). Solids are separated from the wastewater and thickened using a cationic, petroleum-based dewatering polymer before and after anaerobic digestion. Thickened digested biosolids are belt-pressed to further reduce water content and then stockpiled on-site until land application. Biosolids are analyzed monthly by the POTW, which provided values for selected biosolids properties for 12 consecutive months (ranges and means from [17], Table 1). Total organic C was measured in grab-samples of stockpiled biosolids (described in the next subsection). Regulated metal loadings were well below the 40 CFR Part 503 ceiling limits (data not shown) [3].

All biosolids applications were managed by the municipal recycling facility and integrated with other facility operations. Biosolids were surface-applied with manure spreaders to ~220 ha of no-till perennial forage production fields of coastal bermudagrass (*Cynodon dactylon* L.), which included long-term (20 yr) and midterm (8 yr) application fields. All rates are reported in unit dry mass (DM). Forage was mechanically harvested multiple times per season, depending on productivity, and no grazing was used at this site. The long-term application field (14 ha) received 20 Mg biosolids $\text{ha}^{-1} \text{yr}^{-1}$ from 1985 to 2005. Application rates were reduced to 5 Mg $\text{ha}^{-1} \text{yr}^{-1}$ every 2 years in 2007 to comply with the facility's nutrient management plan. A single application at this lower rate occurred before soil sampling in July 2009 (see next section). Midterm application fields were established in

2002 and consisted of 20 ha, 36 ha, and 49 ha parcels that received 20, 40, and 60 Mg $\text{ha}^{-1} \text{yr}^{-1}$, respectively. Each parcel was split into two fields receiving the same application rate. Under municipal operating protocols, biosolids were applied one field at a time. Depending on field size, experimental application rate, the rate of sludge inputs from the municipality, and other operational constraints, biosolids were applied continuously to a given field until the target experimental annual treatment rate was fully applied (e.g., as short as 2–3 weeks or as long as 6–8 weeks). Surface applications would then proceed to the next field until all fields had been applied for the year. Biosolids were then stockpiled until the following year when surface applications could be resumed. Adjacent to the midterm 20 Mg $\text{ha}^{-1} \text{yr}^{-1}$ area, a 2 ha coastal bermudagrass control field has no recorded history of any nutrient inputs from biosolids, animal or green manures, or commercial fertilizer.

2.2. Soil Sampling and Analyses. Soil cores (6.35 cm diameter, ~50 cm deep) were collected in July 2009 from treated fields (unamended control; 20 dry Mg biosolids $\text{ha}^{-1} \text{yr}^{-1}$ for 20 yr; 20, 40, and 60 Mg $\text{ha}^{-1} \text{yr}^{-1}$ for 8 yr) using a truck-mounted hydraulic soil probe. Soils were collected in fields that were not undergoing active application and where biosolids had not been applied for at least 30 days before sampling to comply with POTW safety requirements and EPA 40 CFR Part 503 reentry guidelines [3], respectively. Compliance with municipal operational guidelines and federal safety guidelines resulted in sampling soils from only one field per treatment level. Paired soil cores were collected at four or five locations at 50 m intervals along a transect that spanned the center of each treatment field. No cores were sampled within 50 m of the field edge due to possible edge effects (i.e., increased soil compaction due to higher equipment traffic). Visible biosolids and plant material were removed from the soil surface prior to sampling. Each core was collected in a clear polyethylene tube liner, transported to the laboratory, and then extruded and separated into the following depth increments: 0–5, 5–10, 10–15, 15–25, 25–35, and 35–45 cm. Selected soil properties from July 2009 are presented for 0–30 cm soils collected separately from those used in this study (Table 2).

For each set of paired soil cores in all treatments, one soil core was used to determine soil bulk density (D_b , Mg m^{-3}) from samples dried at 105°C for each depth increment. Soils from the second core were air-dried and passed through a 2 mm mesh sieve for all other soil physical and chemical analyses. Soil organic C and total N were measured by dry combustion (Vario Max CHN, Elementar, Hanau, Germany). Soils were combusted at 600°C to avoid release of soil inorganic C, which constituted approximately 13% of total C in surface soils to as much as 80% in deeper soils (data not shown). Soil stocks of SOC and total N (0–45 cm; Mg C or N ha^{-1}) were calculated by depth increment using measured bulk densities and summing over all increments. Dry combustion at 600°C was also used to measure total organic C in biosolids grab-samples.

TABLE 1: Minimum, maximum, and mean concentrations for selected biosolids constituents and total loading of dry matter (DM) and by constituent based on treatment rate, application area, and years of application (adapted from [17]).

Biosolids application rate ($\text{Mg ha}^{-1} \text{yr}^{-1}$)			20	40	60	20		
Application duration (yr)			8	8	8	20^{\dagger}		
Treatment area (ha)			20	36	49	14		
Biosolids constituent [‡]	Unit	Min	Max	Mean	Total loading, Mg (DM) ha^{-1}			
Total solids	%	15.2	19.3	17.6	160	320	480	400
Total organic C	kg Mg^{-1}	296.1	321.6	307.9	49.3	98.5	147.8	123.1
Total Kjeldahl N	kg Mg^{-1}	27.8	67.9	52.1	8.3	16.7	25.0	20.8
Total P	kg Mg^{-1}	13.4	29.1	16.1	2.6	5.2	7.7	6.4
K	kg Mg^{-1}	0.2	3.4	1.5	0.2	0.5	0.7	0.6
pH	—	8.2	8.7	8.5	—	—	—	—
Electrical conductivity	mS cm^{-1}	2.7	3.9	3.5	—	—	—	—

[†]Annual applications in this treatment reduced to biennial applications of $5 \text{ Mg ha}^{-1} \text{ yr}^{-1}$ in 2007.

[‡]Environmental Protection Agency (EPA) or Standard Methods (SM) protocols used to quantify each constituent, in order listed: SW846 9060A; EPA 351.2; SM 4500-P BE; EPA 6020; SM 4500-H; EPA 9045D; SM 2540G.

TABLE 2: Means (se) for selected soil properties in biosolids-amended soils (0–30 cm).

Application rate ($\text{Mg ha}^{-1} \text{yr}^{-1}$)	20	40	60	20
Application duration (yr)	8	8	8	20^{\dagger}
Sand ^b (%)	21.3 (2.3)	22.2 (2.9)	24.4 (1.6)	25.5 (2.1)
Silt (%)	55.0 (1.0)	56.1 (1.3)	53.5 (1.7)	52.7 (0.6)
Clay (%)	23.7 (2.0)	21.7 (2.1)	22.1 (1.3)	21.8 (2.0)
pH	7.50 (0.03)	7.60 (0.04)	7.62 (0.03)	7.49 (0.03)
Electrical conductivity (mS cm^{-1})	0.69 (0.03)	0.70 (0.04)	0.74 (0.12)	0.54 (0.05)
Extractable NO_3^- (mg N kg^{-1})	25.4 (6.8)	31.8 (3.6)	39.5 (8.5)	14.3 (1.2)
Water-soluble PO_4^{3-} (mg P kg^{-1})	55.0 (21.1)	22.2 (2.3)	28.6 (2.2)	90.4 (7.5)
Particulate organic matter (mg g^{-1})	10.9 (1.0)	8.6 (0.4)	9.2 (1.1)	12.9 (1.0)

^aAnnual applications in this treatment reduced to biennial applications of $5 \text{ Mg ha}^{-1} \text{ yr}^{-1}$ in 2007. ^bParticle size distributions (sand, silt, and clay) were determined by hydrometer [18]; pH and electrical conductivity were determined by electrode in 1:1 water solution [19], EPA 9045D; 2M KCl-extractable NO_3^- and water-extractable PO_4^{3-} were measured in 1:10 soil extracts using continuous flow colorimetry (EPA 353.2, EPA 365.5); and particulate organic matter (0.5–2.0 mm) was measured by weight loss on ignition [20].

Water-stable aggregates were determined using the single sieve method [23] using soil aggregates in the 1–2 mm size range for 0–5, 5–10, 10–15, 15–25, and 25–35 cm depth increments. Aggregates in this size class were isolated by retaining 2 mm sieved air-dried soil passed through a 1 mm mesh sieve. Air-dried aggregates were prewetted with a fine mist for 1 hr using a commercial humidifier prior to direct immersion at a rate of 35 times per min for 3 min. The fraction of initial soil mass remaining was considered water-stable, and values were converted to a mass-per-area basis using soil bulk densities for each soil depth and summed for total water stable aggregates in the entire 0–35 cm soil depth.

Soil water characteristic curves were developed for 0–5, 5–10, 10–15, and 15–25 cm soil increments to evaluate management impacts on near-surface soil hydraulic properties. Air-dried soils from 3 to 5 replicate cores were wetted to a range of different volumetric water contents (~0.04 to $0.20 \text{ m}^3 \text{ m}^{-3}$), sealed for a 24–36 hr equilibration period, and then measured for soil matric potential using a chilled-mirror dewpoint psychrometer (WP4-T, Decagon Devices, Pullman, WA) calibrated with a certified 0.1 mol kg^{-1} KCl salt solution. Nonlinear equations were fitted to measured data using

a power function. Fitted equations were used to estimate soil volumetric water content ($\text{m}^3 \text{ m}^{-3}$) at permanent wilting point (PWP) and field capacity (FC) for soil matric potential values of -1.5 MPa and -0.033 MPa , respectively. Available water capacity (AWC; $\text{m}^3 \text{ m}^{-3}$) was defined as the difference in volumetric water content between FC and PWP.

2.3. Statistical Analyses. The absence of field replication in this municipally designed land-application operation precluded the use of analyses of variance to evaluate results. Instead, effects of biosolids application rate (0, 20, 40, and $60 \text{ Mg dry biosolids ha}^{-1} \text{ yr}^{-1}$ for 8 years) on measured soil physical and chemical properties were evaluated using regression analyses. Linear regressions were fit to data unless significant lack-of-fit indicated that a nonlinear regression was necessary. The effect of biosolids application duration was assessed for differences between 8 yr and 20 yr of application at $20 \text{ Mg dry biosolids ha}^{-1} \text{ yr}^{-1}$ using two-tailed *t*-tests for groups with unequal variances. Means were considered different at the 0.05 significance level. To explore potential relationships between D_b and SOC with WSA and AWC,

TABLE 3: Soil bulk density (D_b , Mg m^{-3}) and moisture properties estimated from soil water characteristic curves fitted to power functions ($y = ax^b$) for individual replicate cores (n) per treatment. Ranges in coefficients of variation (R^2) for curve fits and means (se) shown for soil water content ($\text{m}^3 \text{m}^{-3}$) at permanent wilting point (θ_{PWP}), field capacity (θ_{FC}), and available water capacity (AWC) in 0–5, 5–10, 10–15, and 15–25 cm soils.

Soil depth cm	Rate $\text{Mg ha}^{-1} \text{yr}^{-1}$	Duration Years	D_b Mg m^{-3}	n	R^2	θ_{PWP} $\text{m}^3 \text{m}^{-3}$	θ_{FC} $\text{m}^3 \text{m}^{-3}$	AWC $\text{m}^3 \text{m}^{-3}$
0–5	0	0	1.06 (0.06)	5	0.846–0.987	0.099 (0.006)	0.223 (0.021)	0.124 (0.017)
	20	8	1.21 (0.04)	4	0.908–0.990	0.121 (0.009)	0.327 (0.025)	0.207 (0.019)
	40	8	0.94 (0.08)	4	0.918–0.989	0.126 (0.018)	0.391 (0.049)	0.265 (0.031)
	60	8	1.01 (0.05)	2	0.811–0.934	0.281 (0.054)	0.907 (0.032)	0.625 (0.086)
5–10	20	20	0.93 (0.01)	4	0.816–0.991	0.129 (0.008)	0.353 (0.023)	0.224 (0.017)
	0	0	1.23 (0.03)	5	0.864–0.988	0.118 (0.002)	0.235 (0.012)	0.117 (0.011)
	20	8	1.25 (0.01)	4	0.972–0.994	0.100 (0.004)	0.270 (0.019)	0.169 (0.015)
	40	8	1.32 (0.02)	4	0.954–0.976	0.110 (0.010)	0.318 (0.021)	0.208 (0.015)
10–15	60	8	1.36 (0.05)	2	0.903–0.973	0.155 (0.015)	0.535 (0.031)	0.380 (0.016)
	20	20	1.20 (0.01)	4	0.885–0.995	0.125 (0.003)	0.333 (0.009)	0.208 (0.006)
	0	0	1.30 (0.05)	4	0.904–0.968	0.109 (0.007)	0.223 (0.025)	0.114 (0.018)
	20	8	1.36 (0.07)	3	0.970–0.994	0.103 (0.006)	0.292 (0.009)	0.189 (0.009)
15–25	40	8	1.32 (0.04)	4	0.963–0.996	0.102 (0.010)	0.289 (0.018)	0.187 (0.012)
	60	8	1.32 (0.05)	2	0.960–0.971	0.113 (0.005)	0.431 (0.011)	0.318 (0.016)
	20	20	1.29 (0.04)	4	0.971–0.992	0.116 (0.004)	0.281 (0.019)	0.165 (0.016)
	0	0	1.34 (0.03)	5	0.876–0.979	0.107 (0.002)	0.229 (0.009)	0.121 (0.007)
20	20	8	1.31 (0.05)	4	0.961–0.993	0.103 (0.005)	0.295 (0.025)	0.192 (0.021)
	40	8	1.35 (0.04)	4	0.944–0.986	0.100 (0.009)	0.314 (0.022)	0.214 (0.022)
	60	8	1.33 (0.05)	2	0.940–0.986	0.107 (0.014)	0.506 (0.019)	0.399 (0.017)
	20	20	1.29 (0.05)	4	0.968–0.994	0.113 (0.002)	0.278 (0.010)	0.166 (0.010)

percent values for SOC (soil concentration) and WSA (% mass remaining) were used instead of stocks per unit area to avoid autocorrelations between these metrics and D_b . Statistical tests were conducted using SAS 9.3 (SAS Inc., Cary, NC).

3. Results and Discussion

3.1. Soil Organic Carbon and Total Nitrogen Stocks. To assess the extent to which surface-applied biosolids affected SOC and total N with soil depth, SOC and total N stocks were evaluated to a 45 cm soil depth in this study, deeper than most published studies on surface-applied biosolids. Here, surface application rates were positively correlated with SOC and total N in the top 15 cm soil layer, somewhat deeper than other studies reporting that SOC and N gains are limited to the top <10 cm of soils [24–27]. The current results underscore the fact that the effects of application rate and duration on SOC and total N stocks can be highly variable and depend on biosolids-specific composition as well as site-specific factors (e.g., soils, management) [5, 7, 28].

When examined for the entire profile depth sampled (0–45 cm), soil stocks of SOC increased nonlinearly with application rate after 8 yr of continuous annual applications (Figure 1(a)). A previous study using laboratory incubations to evaluate C and N mineralization responses in these soils reported similar increases in SOC (32 to 92%) in 0–10 cm soils treated with biosolids relative to untreated control soils [22].

Other field studies on repeated surface-applied biosolids also have found increases in SOC [4, 12, 24–27, 29] and found that increases are positively correlated with application rate [4, 25]. Site-specific increases in SOC have been reported for application rates as low as 2 to 7 $\text{Mg ha}^{-1} \text{yr}^{-1}$ [24, 30]. Field studies following a single application of surface-applied biosolids also report increases in SOC [8, 30–33].

Long-term applications of 20 $\text{Mg biosolids ha}^{-1} \text{yr}^{-1}$ over 20 yr resulted in a 36% increase in SOC stocks compared to the 8 yr treatment at the same rate (Figure 1(a)). In contrast, Gaskin et al. [26] reported that SOC levels were not different between bermudagrass fields receiving repeated surface applications for <6 yr or for >6 yr, though SOC levels in both treatments were higher than unamended fields. Long-term gains in SOC have been reported in other studies which found that increased SOC or SOM levels can persist up to 27 yr after biosolids applications have ceased [24, 25, 29, 34–36].

Soil total N stocks (0–45 cm) followed similar trends to SOC, increasing nonlinearly with biosolids application rate (Figure 1(b)). In soils treated with 20 yr of biosolids applications at the lowest rate, total soil N stocks were 32% greater in long-term treated fields compared to midterm fields. In midterm fields, increasing application rates resulted in greater relative increases in total N stocks compared to SOC stocks. Higher enrichment in total N compared to SOC following biosolids application is well documented [5, 22, 25, 32, 33, 37] and could increase the risk of potential N losses

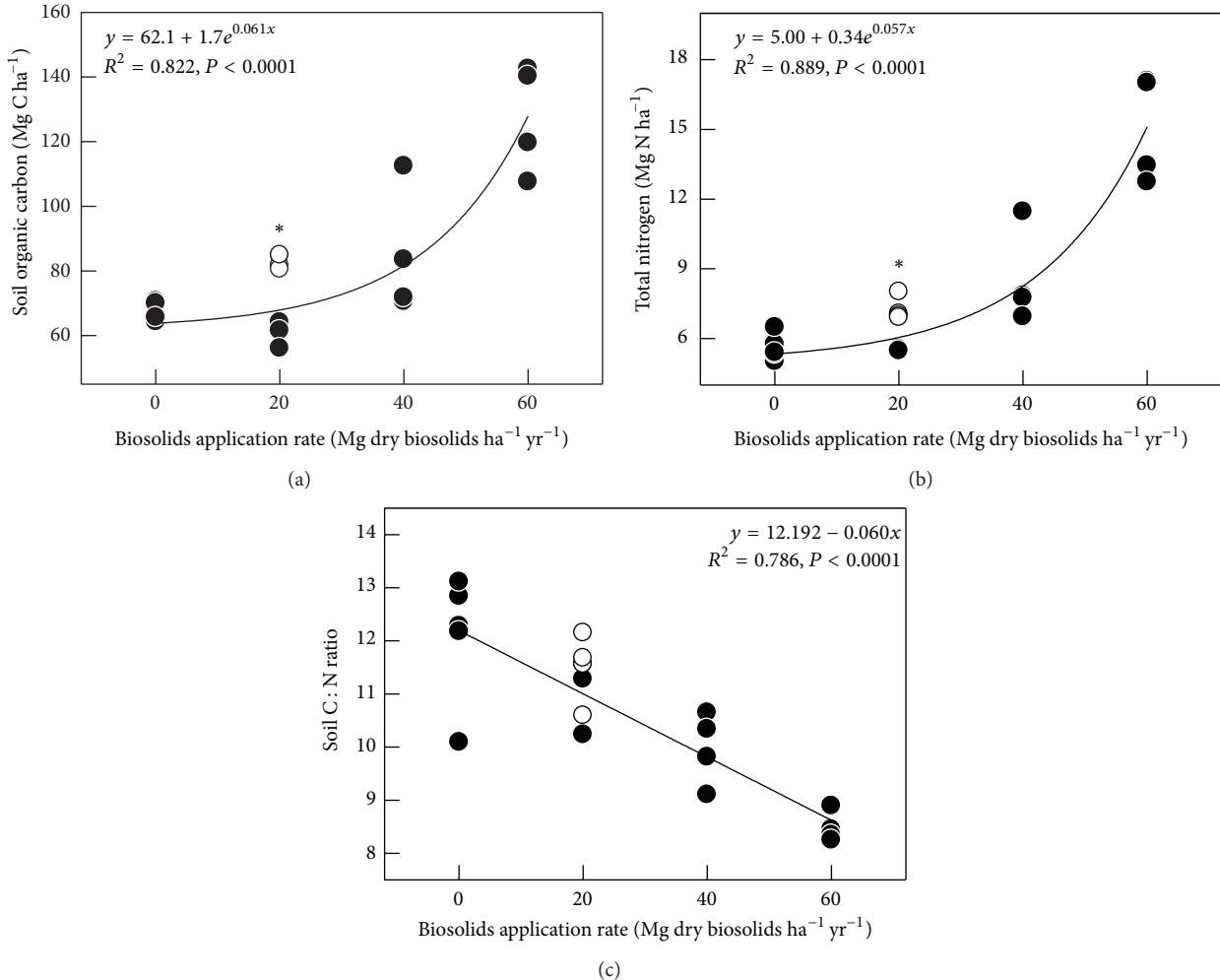


FIGURE 1: Effect of biosolids application for 8 yr (midterm; solid symbols) at four treatment rates on (a) soil organic carbon stocks (Mg C ha^{-1} , 0–45 cm); (b) total nitrogen stocks (Mg N ha^{-1} , 0–45 cm); and (c) carbon-to-nitrogen ratio (0–45 cm). Open symbols show long-term applications of $20 \text{ Mg biosolids ha}^{-1} \text{ yr}^{-1}$ for 20 yr (not included in regression shown). The asterisk indicates significant difference between midterm and long-term treatments ($P < 0.05$).

by increasing direct N inputs and by stimulating organic N mineralization [5, 38]. Using laboratory incubations from soils at this site, Jin et al. [22] found that biosolids applications resulted in more volatile fluctuations in potential N mineralization and immobilization in surface soils (0–10 cm) amended at the two higher application rates but not in midterm or long-term soils applied at the lowest rate relative to control soils. A more recent study, however, reported elevated soil nitrate-N concentrations throughout the whole soil profile (0–110 cm) in all biosolids-treated fields compared to unamended control [17]. Further, nitrate-N concentrations were $>25 \text{ mg N kg}^{-1}$ in subsoils deeper than 50 cm and were potentially below the predominant rooting zone, indicating an increased risk of N leaching losses [17]. In contrast to these findings, a recent literature review reported that increases in soil organic N with repeated organic matter inputs can enhance long-term availability of N to crops, often without resulting in greater nitrate leaching to groundwater [5].

Mean values for soil C : N decreased linearly with increasing biosolids application rates, from 12.1 ± 0.5 in unamended

control soils to 11.1 ± 0.3 , 10.0 ± 0.3 , and 8.5 ± 0.1 for 20, 40, and $60 \text{ Mg ha}^{-1} \text{ yr}^{-1}$, respectively (Figure 1(c)). Soils under long-term application had a C : N of 11.5 ± 0.3 , similar to control soils. Measured soil C : N values were within the range of reported values following biosolids applications [6, 24, 33, 37, 39] and followed expected decreases in C : N values associated with increasing rates or repeated applications of biosolids [6].

3.2. Soil Bulk Density. Soil bulk densities (D_b) for individual soil depth increments were generally similar across application rates, except in 5–10 cm soils where D_b tended to increase with biosolids application rate (Table 3, Figures 3(a) and 3(b)). For the $20 \text{ Mg biosolids ha}^{-1} \text{ yr}^{-1}$ rate, long-term applications improved D_b (e.g., decreased D_b) in the two surface soil increments (0–5 cm, 5–10 cm) compared to the midterm treatment. When aggregated to 0–45 cm depth, however, D_b were similar between control and biosolids-amended soils. Earlier studies for soils from this site report contrasting effects of biosolids additions on D_b . Jin et al.

[22] reported no effects of biosolids applications on D_b in 0–10 cm soils. Haney et al. [17] found that D_b in 0–30 cm soils increased with application rate, presumably as a result of higher wheel traffic required to land-apply at greater biosolids rates, but not with application duration at the lowest rate [17]. Results from the current study and previous reports are consistent with the variability in soil responses found in other studies on surface-applied biosolids which have reported no effects or increased D_b [12, 32, 33, 40] or decreased D_b due to biosolids application [24, 27].

In contrast, decreases in D_b following the incorporation of biosolids into soils are reported commonly [4, 6, 9–11, 13, 16, 41–43]. The physical mixing of soils alone decreases D_b . Depending on application rate, the incorporation of biosolids can further contribute to lowering D_b by diluting soil mineral fractions with biosolids organic matter (OM) and subsequently increasing soil microporosity [9, 10, 13]. Surface broadcasting biosolids at this no-till perennial forage production site limited the impact of physically disturbing and homogenizing biosolids with surface soils. Changes to D_b , therefore, could be attributed to wheel traffic and other management practices associated with forage production. The absence of D_b changes with application rate observed here could be a result of the breakdown of biosolids into fine powder over time (and, in this case, with increased wheel traffic), which would limit changes to D_b compared to more discrete biosolids aggregates [40]. Improvements in D_b in the long-term field relative to the midterm field also suggest that biosolids applications at the lowest rate can result in improvements in this specific soil physical quality metric.

3.3. Soil Water-Stable Aggregates. Water-stable aggregates (WSA) as a percent of whole soil decreased with increasing application rate in midterm applied fields at all measured soil depths relative to unamended controls. When converted to a mass-per-area basis, soil WSA decreased linearly with application rate at each soil depth and for the full 0–35 cm profile (Figure 2). Soil WSA was also lower in long-term applied fields compared to the midterm fields at the $20 \text{ Mg ha}^{-1} \text{ yr}^{-1}$ application rate, but in the 0–5 cm soil increment only (Figure 2(a)). Long-term decreases in soil WSA in near-surface soils also resulted in overall decreases in WSA observed for the full 0–35 cm profile (Figure 2(f)).

Organic amendments such as biosolids tend to increase soil physical fertility primarily by improving aggregate stability [5]. Measurement of water-stable aggregates specifically addresses the potential for soil loss by water erosion and the potential to maintain soil structure which affects the transmission and retention of water. While organic amendments can increase soil aggregate stability by 1- to 10-fold, stability responses vary both spatially and temporally [4, 9, 11, 13, 15, 44]. Stability will also be affected by application rate and frequency, though aggregate stability is not always positively correlated with application rate [11]. Greater plant growth and soil biological activity associated with organic matter additions are the key to increasing aggregate formation and stability. Organic inputs from aboveground (e.g., plant litter) and belowground (e.g., root litter and exudates, microbial

turnover and exudates) are essential for aggregate cohesion or water repellency [6, 11, 25, 31–33, 39, 41]. Despite reported increases in microbial activity [22] and greater forage yields with increasing biosolids additions (J. Slagle, personal communication), WSA at all soil depths and for whole soils (0–35 cm) decreased with application rate and duration, in contrast to the majority of studies finding increases in aggregate stability after biosolids additions.

Both no change and variable changes in aggregate stability have been reported in surface-applied and incorporated biosolids studies [13, 16, 40, 43]. Only few studies have reported decreases in aggregate stability following additions of biosolids, animal manure, or other organic amendments [44–46]. Degradation of aggregates and decreased stability may be due to biosolids composition, specifically the increase in compounds directly or indirectly involved in the dispersion of fine soil particles. Dispersive agents in biosolids include humic substances [10, 45], anionic and nonionic surfactants [37], and high amounts of Na^+ , K^+ , and/or organic P [14, 44]. While the composition of organic compounds in biosolids used here is beyond the scope of this study, a recent study reported that extractable K in these soils is not affected by biosolids rate or duration but that mineralization of organic P likely has contributed to high ortho-phosphate levels in the top 30 cm of soil [17]. In addition, soils at this site are calcareous, and the presence of calcium carbonates is expected to contribute to flocculation and aggregation [14]. Increases in biosolids-derived organic P, however, could bind to clay minerals and increase clay dispersion [44] or reduce the activities of Al^{3+} or Ca^{2+} which are known aggregate bonding agents [14].

3.4. Soil Water Characteristics and Moisture Retention. Soil volumetric water contents tended to be higher at both permanent wilting point (PWP; -1.5 MPa) and field capacity (FC; -0.033 MPa) in biosolids-amended soils relative to unamended controls (Table 3). Values generally were within the range reported for fine-textured soils [4, 9, 40]. Despite relatively high R^2 values for soil moisture characteristic curves fitted to soils treated with the $60 \text{ Mg ha}^{-1} \text{ yr}^{-1}$ application rate, estimates of volumetric water content at FC were high at all soil depths, possibly due to the limited number of replicate cores used and/or high SOC levels. Available water capacity (AWC) also increased with biosolids application rate and, to a lesser extent, with increased duration of application. Changes in soil water characteristics in biosolids-amended soils were detectable to 35 cm depth, with the largest changes occurring in 0–10 cm surface soils.

Other studies have also found that surface-applied biosolids increase soil water retention at FC and PWP [27, 30, 37, 40]. Increases in soil surface area after OM addition may contribute to greater water retention at PWP [10] while increased microporosity enhanced water retention at FC [9]. Increased soil water retention at PWP and/or FC has been found to be positively correlated with application rate in some cases [10, 30, 47] but not others [40, 48]. Changes in soil water retention, however, are soil-specific and are typically greater for coarser-textured soils compared to finer-textured soils such as those in the current study [9, 10, 30, 37, 40].

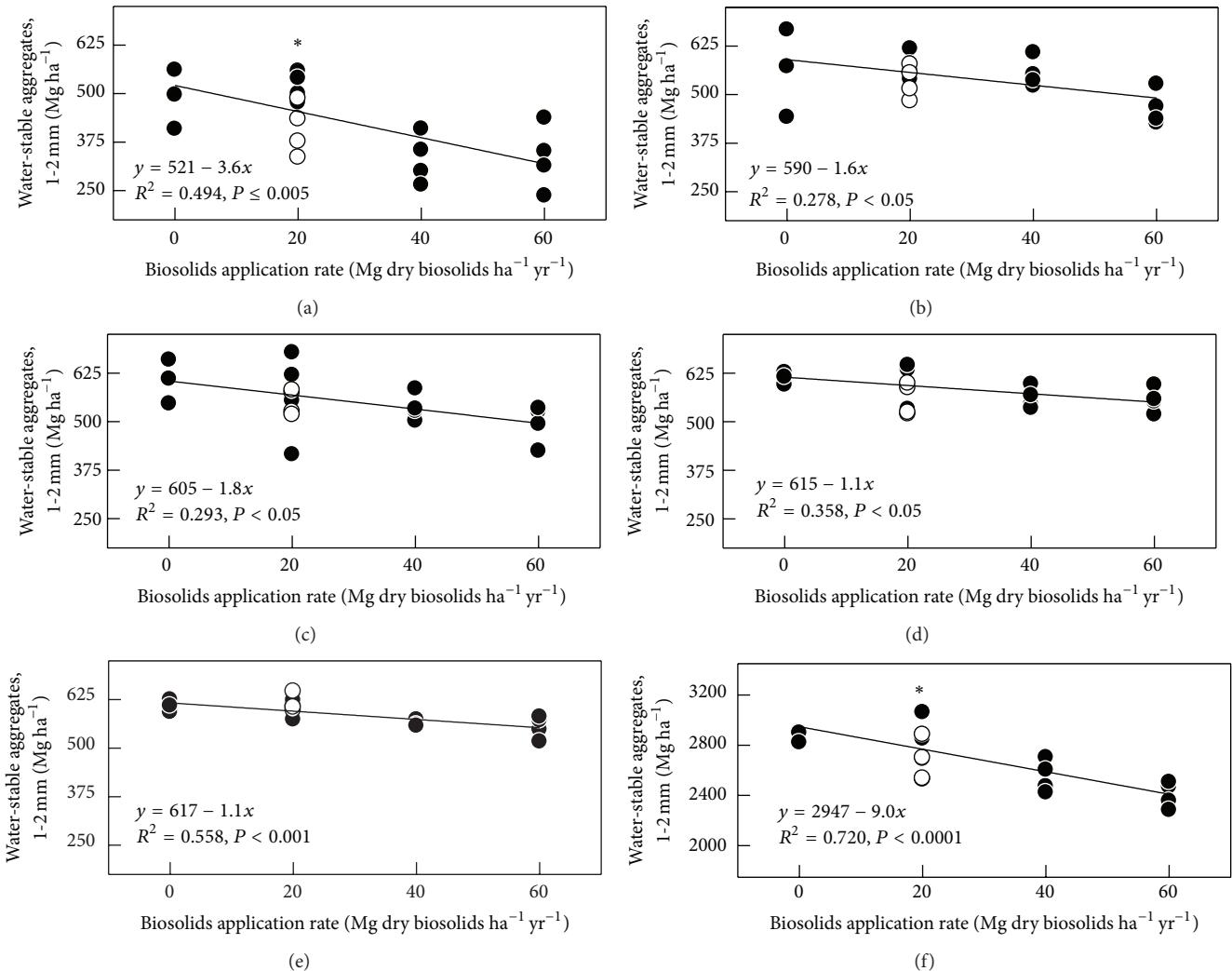


FIGURE 2: Effect of biosolids application for 8 yr (midterm; solid symbols) at four treatment rates on soil water-stable aggregates (1-2 mm; Mg ha^{-1}) in (a) 0–5 cm; (b) 5–10 cm; (c) 10–15 cm; (d) 15–25 cm; (e) 25–35 cm; and (f) whole soil, 0–35 cm. Open symbols show long-term applications of $20 \text{ Mg biosolids ha}^{-1} \text{ yr}^{-1}$ for 20 yr (not included in regression shown). The asterisk indicates significant difference between midterm and long-term treatments ($P < 0.05$).

In the review by Kladivko and Nelson [40], small but nonsignificant increases in AWC occurred with surface-applied biosolids additions, though a more recent review of biosolids recycling studies in Washington, USA, showed variable effects of biosolids on AWC for a range of different textured soils [4]. Improvements in water retention with no increases in AWC have also been measured in studies of incorporated biosolids [8–10]. These studies report AWC on a gravimetric basis, however, and changes in AWC are generally assumed to be nonsignificant on a volumetric basis [9]. This contrasts with findings in this study that, even if AWC values from the highest application rate are omitted, biosolids additions enhanced volumetric AWC which also increased with biosolids application rate and duration.

3.5. Relationships between SOC and Soil Physical Properties.

In general, biosolids applications tend to decrease D_b , increase total porosity, and increase soil moisture retention

primarily through addition of biosolids-derived organic matter [5, 6, 10, 11, 13, 42, 47]. In addition to enhancing organic C inputs to the soil, added organic matter improves D_b (e.g., decreases D_b) by increasing the proportional distribution of micropores involved in water storage ($0.5\text{--}50 \mu\text{m}$) and transmission ($50\text{--}500 \mu\text{m}$) [10, 13]. Increases in microporosity subsequently can lead to increased soil water retention and improvement of other soil hydraulic properties. In this system, however, improvements in soil physical properties and SOC due to biosolids application could be offset by increased wheel traffic required to apply higher rates.

In this study, expected decreases in soil D_b and increases in WSA (% mass remaining) associated with greater SOC concentrations (%C) were not observed (Figures 3(a)–3(c)), but increased SOC was linked to greater soil AWC (Figure 3(d)). Soil D_b was generally unchanged by biosolids application rate in the midterm, but long-term improvements in D_b were not associated with greater WSA or AWC in either surface soil increment (0–5 cm,

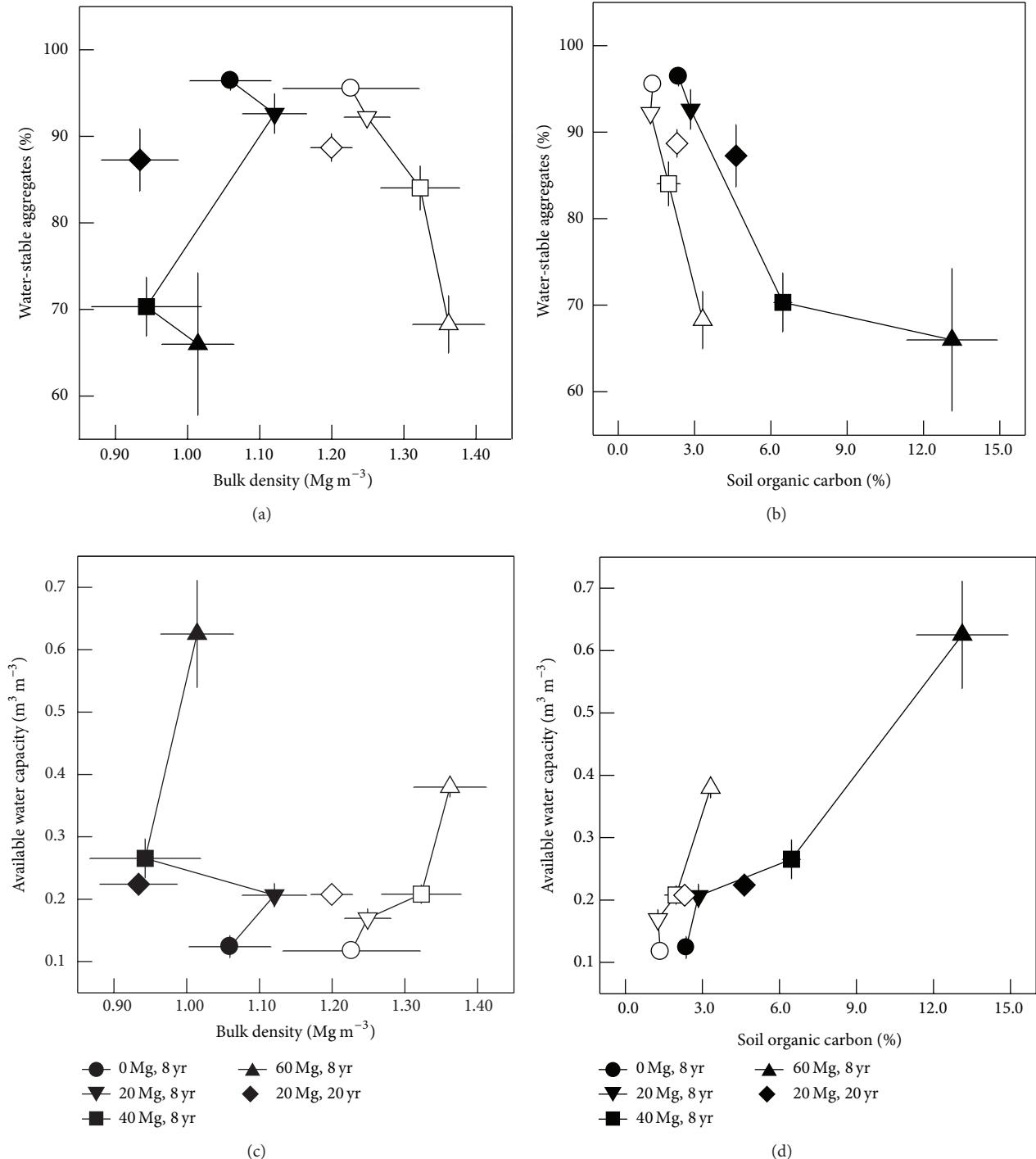


FIGURE 3: Effect of biosolids application rate and duration on relationships between (a) soil water-stable aggregates (% mass remaining) and soil bulk density (Mg m^{-3}), (b) soil available water capacity ($\text{m}^3 \text{m}^{-3}$) and soil bulk density, (c) soil water-stable aggregates and soil organic carbon (%C), and (d) soil available water capacity and soil organic carbon. Closed symbols show 0–5 cm soils; open symbols show 5–10 cm soils. Means (se) are shown, and symbols occasionally obscure se bars.

5–10 cm). Further, neither midterm nor long-term increases in SOC ameliorated biosolids management impacts on WSA, which generally declined in all biosolids-amended soils.

4. Conclusions

Land applying biosolids can serve multiple functions in addition to the beneficial reuse of wastes, including replacement

of commercial fertilizers for crop production, improvement of soil condition, and storage of organic carbon in managed soils. In this study, we demonstrated that surface applications of biosolids can enhance SOC levels and improve soil hydraulic properties. Measured increases in SOC with application rate, however, were not associated with improvements in soil bulk density or soil aggregate stability reported in many other studies. Even under long-term application at the lowest rate where SOC increased and D_b improved, degradation of soil WSA still occurred. These results demonstrate the potential for carbon storage in soils treated with surface-applied biosolids, but the contrasting effects of biosolids applications on soil physical properties underscore the importance of site-specific management decisions to minimize costs and maximize benefits associated with recycling biosolids in agricultural settings.

Disclaimer

Mentioning of trade names or commercial products in this paper does not imply recommendation or endorsement by the U.S. Department of Agriculture.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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Research Article

Residual Effects of Lime- and Clay-Amended Biosolids Applied to Coarse-Textured Pasture Soil

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This study investigated whether there was residual effect of application of lime- and clay-amended biosolids (LaBC[®]) on ryegrass growth and soil microbial biomass in a coarse-textured, acid pasture soil. Reapplied LaBC[®] increased fertiliser-use efficiency and plant growth in this glasshouse experiment. Soil management history was established with a single application of LaBC[®] (50 t ha⁻¹ wet weight equivalent) with or without inorganic fertiliser (NPK) prior to growing annual ryegrass for 5 cycles. In cycle 6 there was no residual nutrient effect of the original application of LaBC[®] but there was a residual liming effect of the previously applied LaBC[®]. A nutrient effect of reapplied LaBC[®] in plant growth cycle 6, had little residual benefit in cycle 7. The residual concentration of inorganic N remaining in this coarse-textured acid soil after a single application of LaBC[®] was negligible and did not appear to be a risk to the environment when applied at 50 t ha⁻¹ wet weight equivalent.

1. Introduction

Soils on the Swan Coastal Plain in Western Australia (WA) are generally coarse-textured and nutrient deficient and need remedial measures to address soil conditions such as acidity [1]. In addition, these soils are characterised by water repellence that leads to poor water-infiltration and poor nutrient and water retention capabilities in soil that eventually can cause problems for the quality of nearby waterways [2]. Appropriate soil management and nutrient application practices are needed to provide better conditions for plant growth and to reduce the potential for nutrient leaching into waterways on the Swan Coastal Plain [3, 4]. It has been suggested that lime-amended biosolids further blended with clay (called LaBC[®]) could be incorporated as a soil amendment to remediate these sandy soils [5]. However, the nutrient release pattern for plant available N from LaBC[®] and considerations for a potential long-term nutrient release need to be monitored to ensure safe recycling of nutrients in this acid sandy soil [6].

Amending soil with biosolids can increase plant growth in degraded pastures associated with improvements in soil

fertility [7–9]. Pasture productivity can be increased with application of both organic and inorganic sources of nutrients in such soils [10–13]. Liming, alone or in the form of alkaline stabilised sewage sludge (biosolid), has been used to alleviate soil acidity [14–16]. Various forms of biosolids are available for use in agriculture, including lime-stabilised biosolids [17, 18] and a modified clay-amended biosolid product (LaBC[®]) which was created for use on acid sandy soils of the Swan Coastal Plain in WA [5, 19]. LaBC[®] is similar to a biosolid product that was developed by mixing biosolids with river-sediments for application to native grasses in Illinois, USA [20].

Soil management practices that include amending soils with organic or inorganic fertilisers can release residual nutrients (N and P) for some time after their application [21, 22]. Several studies have demonstrated residual effects of applied amendments such as biosolids [23–26], inorganic fertilisers [27, 28], and lime [29–31] that increased soil macronutrients and microbial dynamics in a range of degraded and agricultural lands. A recent study in a sandy-loam soil demonstrated a differential effect on N availability for a repeated application of fertiliser and/or organic manures

TABLE 1: Pretreatment details for the establishment of a pasture soil history during 5 cycles of growth of annual ryegrass in a coarse-textured acid soil under glasshouse conditions.

Initial soil treatments	Fertiliser applied in 5 previous cycles of annual ryegrass by the Water Corporation of Western Australia
Control	Not applied
^a LaBC®	Not applied
^b Fertiliser	Applied
LaBC® + fertiliser	Applied

^aLaBC® = 50 t ha⁻¹ of wet weight equivalent applied once only in the first cycle.

^bFertiliser = macronutrient fertiliser applied according to district practice at each cycle.

in a 17-year experimental site [22]. Long-term N application stimulated gross nitrification more than 5.3 times and soil organic matter was 2.7 times greater in fertiliser applied treatments. A 2-year study concluded that sewage sludge used as an organic amendment was not a complete substitute for mineral fertilisers as inorganic nutrient inputs resulted in greater ryegrass growth on a land-fill site restored with London clay [32]. The N release pattern for the supply of plant available nutrients from biosolids or fertilisers needs to be independently assessed according to the rate applied and soil conditions, because a generalised approach for estimating nutrient supply has several limitations when budgeting nutrient release [33].

In our previous study, clay addition to lime-amended biosolids (LaBC®) increased microbial biomass in an acid sandy soil, which was associated with an increase in soil pH when the amendments were incubated for 30 weeks [19]. It was also found that N was released over a longer period when clay was added to the biosolid with lime (as LaBC®) rather than when either clay or lime was added independently [19]. In addition, a long-term effect of LaBC® and its potential for supplying plant nutrients has been investigated using ryegrass at both field and glasshouse scales [6]. However, the residual effect of LaBC® on soil fertility, especially in relation to microbial activities, is not known. Therefore, in order to study the residual effects of LaBC® on soil fertility, LaBC® was initially applied once as a basal amendment with initial and repeated applications of inorganic fertiliser containing N, P, K, Ca, and Mg before sowing 5 sequential cycles of ryegrass. This established a fertiliser history in the glasshouse experiment (Table 1). Subsequently, the residual concentration of N remaining in the soil after these 5 growth cycles was assessed after a single reapplication of LaBC® in a sixth growth cycle of ryegrass (Figure 1). The residual effectiveness of this reapplied LaBC® was also determined in a seventh growth cycle. Ryegrass biomass, soil inorganic N, pH, and soil microbial biomass N were assessed after the sixth and seventh cycle of ryegrass.

The aim was to determine whether there was a residual effect of LaBC® application or an effect of reapplication of LaBC® to soil that is with a history of LaBC® application. This was assessed with and without the application

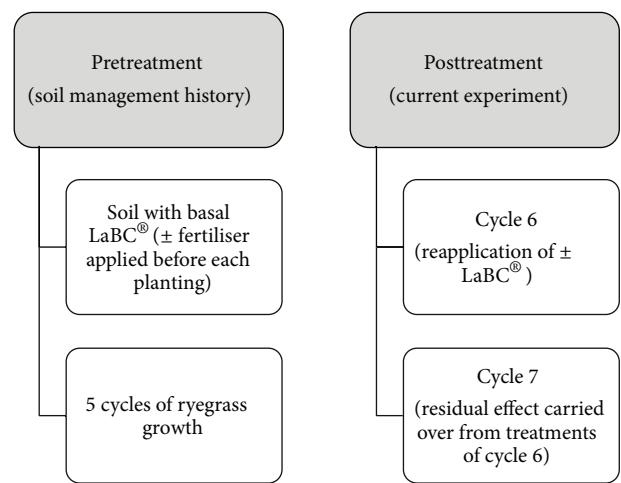


FIGURE 1: Steps involved in determining the residual effect of LaBC® in either the presence or absence of added fertilizer. Dry LaBC® = 50 t ha⁻¹ of wet weight equivalent; fertiliser = macronutrient fertilisers (N, P, K, Ca, and Mg) applied according to district practice.

of basal inorganic fertiliser to determine whether there is potential environmental risk from application of LaBC®. It was hypothesised that (i) reapplication of LaBC® would have an immediate effect of increasing soil pH regardless of prior history of LaBC® and fertiliser application and (ii) application of LaBC® to soil with a fertiliser history would release more N and increase ryegrass growth to a greater extent than an application of LaBC® to soil without a fertiliser history.

2. Materials and Methods

2.1. Experimental Design

2.1.1. Pretreatment of Soil with LaBC® and Fertilizer for Establishing Soil Management History. Soil used for this experiment was taken from pots that had been pretreated under controlled glasshouse conditions in the following way by the Water Corporation of Western Australia (WA). Prior to establishing the fertiliser history, a coarse-textured acidic sandy soil had been collected from a depth of 0–10 cm after removing coarse surface litter. The field was adjacent to a site with natural woodland vegetation at Bullsbrook (31° 40' 0" South, 116° 0' 0" East), WA, and represented the Swan Coastal Plain-Bassendean sands of the Ellen Brook catchment (pale deep, bleached-Orthic Tenosol [34]). The soil had been air-dried and passed through a 4 mm sieve. It was dominated by sand (98%; 20 µm–2 mm) with a small silt fraction (2%; <20 µm). Soil chemical properties were pH (CaCl₂) 5.1, total carbon 1.3%, total nitrogen 0.1%, total oxidisable carbon 0.15 mg g⁻¹, and inorganic nitrogen 0.8 µg g⁻¹.

Free draining pots (175 mm in diameter, 2.8 L) had been filled with 5 kg of soil, packed to a bulk density of 1.6 g cm⁻³ (Water Corporation of WA). The pots had been placed inside a bucket to simulate leaching after irrigation under controlled glasshouse conditions. Dry LaBC® (50 t ha⁻¹ wet weight equivalent, 2 to 4 mm) had been applied in cycle 1,

TABLE 2: Single basal and reapplication of LaBC® during 7 cycles of ryegrass grown in pots of coarse-textured, acid soil under glasshouse conditions to determine the residual effects of LaBC®.

Ryegrass cycles 1 to 5	Ryegrass cycle 6	Ryegrass cycle 7
History of single basal application of LaBC® +/- fertiliser	LaBC® reapplied to soil from half of each pot after cycle 5 (+LaBC®) Remaining soil was untreated (-LaBC®)	No LaBC® added in this cycle (to all prior treatments)
Control	+/-LaBC®	-LaBC®
^a LaBC®	+/-LaBC®	-LaBC®
^b Fertiliser	+/-LaBC®	-LaBC®
LaBC® + fertiliser	+/-LaBC®	-LaBC®

^aLaBC® = 50 t ha⁻¹ of wet weight equivalent applied in cycle 6.

^bFertiliser = macronutrient fertiliser applied according to district practice at each cycle.

and macronutrient fertiliser treatments (district practice) had been applied in each cycle according to the treatment schedule (Table 1). The amendments had been fully incorporated through the soil prior to planting ryegrass seeds. Macronutrient fertiliser had been applied on the soil surface prior to planting ryegrass where applicable in each of the 5 cycles (Table 1). The macronutrients had been applied at district farming practice rates equivalent to Flexi N (11.88 kg ha⁻¹), triple superphosphate (20.03 kg ha⁻¹), K₂SO₄ (54.51 kg ha⁻¹), CaCl₂(30.67 kg ha⁻¹), and MgSO₄ (26.62 kg ha⁻¹). Each treatment had been replicated 3 times and pots watered with deionised water had been maintained at 60% field capacity throughout this pretreatment experimental phase.

For the pretreatment conducted by the Water Corporation of WA, seeds of annual ryegrass (*Lolium multiflorum*, cultivar Winter Star) had been pregerminated in Petri dishes and 12 seedlings of uniform size transplanted into each pot as 1-week-old seedlings. Plants had been grown for six weeks after transplanting and the plant biomass was harvested at the end of each growth cycle. Following each harvest, pots had been returned to the glasshouse with a simulated fallow of approximately 2 weeks without irrigation. Following this 2-week period, all contents had been emptied from pots and passed through a sieve (4 mm) to remove coarse root debris. After each fallow period, the pots had been planted with new set of 1-week-old seedlings and macronutrient fertilisers were applied to the soil surface according to treatment schedule (Table 1). The same method had been repeated in total for five cycles of ryegrass growth and harvests. The entire process of establishing the soil history is summarised in the flow diagram (Figure 1).

2.1.2. Reapplication of LaBC® (+/-) to Soil for Cycles 6 and 7 of Ryegrass. Soil from each pot, which had been exposed to pretreatment cycles 1 to 5, was divided and placed into 2 equal smaller pots (115 mm in diameter, 1.5 L) prior to reapplication of treatments (Figure 1). LaBC® was reapplied at the start of cycle 6 (Table 2).

For cycles 6 and 7, pots were filled with 1.65 kg of soil collected from the pretreated soil and packed to a bulk density of 1.6 g cm⁻³. Dry LaBC® (2 to 4 mm fraction) was applied according to the treatment schedule (Table 2) and fully incorporated throughout the soil prior to planting. Pots

were irrigated with deionised water and maintained at 60% field capacity level throughout the experiment. Plants were sown with ryegrass and harvested after 6 weeks in cycles 6 and 7 as described above for the 5 pretreatment cycles. Each treatment was replicated 3 times.

2.2. Characteristics of LaBC®. The LaBC® blend was air dried at 40°C and homogenised using a mechanical crusher. The amendment was then passed through a sieve that captured particles between 2 and 4 mm before application. Basic characteristics of the amendment were analysed using standard procedures [35]. The pH (CaCl₂) was 8.2. Oxidisable C and carbonate contents were measured on 0.5 M K₂SO₄ extracts (sample-solution ratio of 1: 4) using an autosampler (Shimadzu, ASI-5000 A) and TOC analyser (Shimadzu, TOC 5000 A) based on wet combustion method [36]. LaBC® had 3% total C, 0.3% total N, 2.4 mg g⁻¹ oxidisable C, 0.9 mg g⁻¹ carbonate, 46 µg g⁻¹ total inorganic N, and traces of micronutrients such as Zn and Cu (mg kg⁻¹, dry weight basis).

2.3. Sampling and Analyses. Plants were harvested after 6 weeks in cycles 6 and 7. Shoots were cut just above the soil surface and their base was washed with deionised water to remove any adhering soil particles. Soil from each pot was then transferred to separate trays to collect root biomass; larger roots were gently removed by hand whereas the finer roots were removed by sieving through a 2 mm sieve. The soil was returned into the respective pots after removing the roots and root fragments. All plant samples were dried to a constant weight at 40°C for 7 days in a hot air oven and weighed to record dry weight biomass.

Soil subsamples (approximately 50 g) were collected at the end of each harvest, after removing the finer root fragments and homogenising the soil from each pot. Soil pH (1: 5 v/v ratio of soil/0.01 M CaCl₂ suspension) was measured after shaking for 1 hour continuously in a mechanical end-over-end shaker [35].

Soil inorganic N (NO₃⁻-N + NH₄⁺-N) was determined using fresh soil (10 g dry weight equivalent) extracts of 0.5 M K₂SO₄ (40 mL) after shaking in a mechanical end-over-end shaker for 1 hour and microbial biomass N (MBN)

TABLE 3: Shoot dry biomass (g pot^{-1}) in ryegrass cycles 6 and 7 with reapplied LaBC[®] (+/-) as influenced by residual effects of soil amendments from previous management history.

Soil management history (cycles 1 to 5)	Shoot dry biomass (g pot^{-1})			
	Ryegrass cycle 6		Ryegrass cycle 7	
	-LaBC [®]	+LaBC [®] (50 t ha^{-1})	Current treatment (+/-LaBC [®])	Treatment -LaBC [®] from cycle 6
Control	$0.03 \pm 0.03^{\text{c}}$	$0.33 \pm 0.03^{\text{b}}$		$0.11 \pm 0.02^{\text{d}}$
^a LaBC [®]	$0.10 \pm 0.00^{\text{c}}$	$0.39 \pm 0.01^{\text{b}}$		$0.09 \pm 0.01^{\text{d}}$
^b Fertiliser	$0.28 \pm 0.04^{\text{b}}$	$0.40 \pm 0.06^{\text{b}}$		$0.15 \pm 0.00^{\text{d}}$
LaBC [®] + fertiliser	$0.33 \pm 0.03^{\text{b}}$	$0.60 \pm 0.06^{\text{a}}$		$0.11 \pm 0.02^{\text{d}}$

^aLaBC[®] = 50 t ha^{-1} of wet weight equivalent.

^bFertiliser = macronutrient fertilisers (N, P, K, Ca, and Mg) applied according to district practice.

Values are mean \pm SE ($n = 3$). The letters followed by mean \pm SE were based on LSD ($P < 0.05$).

The main and interaction effects of treatments with the same letters, within the same column and between columns within each cycle, respectively, were not significant ($P > 0.05$).

was determined using the same procedures in our previous experiments [19].

2.4. Statistical Analyses. Cycle 6 was set up in a complete randomised block design (4 pretreatment histories \times 2 LaBC[®] posttreatments) with three replicates. Cycle 7 was the same as cycle 6. Statistical analyses were performed using Genstat version 12. Two-way analysis of variance (ANOVA) was used to compare the residual effect of the pretreatment soil management history on posttreatment of LaBC[®] and the least significant difference (LSD) was determined at $P \leq 0.05$.

3. Results

3.1. Shoot Dry Biomass (g pot^{-1}). Shoot biomass was significantly influenced ($P < 0.05$) only by fertiliser from the basal history but not by the previously applied LaBC[®], after 5 cycles of ryegrass planting (Table 3). When LaBC[®] was reapplied at 50 t ha^{-1} in cycle 6, shoot biomass was increased (by $>100\%$) compared to non-reapplied LaBC[®]. A residual effect of reapplied LaBC[®] from cycle 6 was carried over to cycle 7.

3.2. Root Dry Biomass (g pot^{-1}). Root biomass was significantly influenced ($P < 0.05$) only by fertiliser from the basal history but not by the previously applied LaBC[®], after 5 cycles of ryegrass planting (Table 4). It was increased 3-fold by the fertiliser from the prior history. When LaBC[®] was reapplied at 50 t ha^{-1} in cycle 6, it did not have a significant effect regardless of the soil history. However, a residual effect of reapplied LaBC[®] from cycle 6 was carried over to cycle 7.

3.3. Root:Shoot. Root:shoot was significantly increased ($P < 0.05$) by pretreatment of soil with both LaBC[®] and fertiliser and in combination when assessed in cycle 6 (Table 5). A residual effect of fertiliser but not LaBC[®] lasted through to cycle 7. When LaBC[®] was reapplied at 50 t ha^{-1} in cycle 6, it did not have a significant ($P > 0.05$) effect on

root:shoot regardless of the soil history and the same pattern was also observed at cycle 7 (Table 5). Overall, the residual effect of fertiliser history significantly ($P < 0.05$) influenced the root:shoot but only in the absence of reapplied LaBC[®].

3.4. Shoot N Concentration (%). Shoot N concentration was not influenced ($P > 0.05$) by LaBC[®] and/or fertiliser applied to the pretreated soil when assessed in cycle 6 (Table 6). When LaBC[®] was reapplied at 50 t ha^{-1} in cycle 6, it did not have a significant ($P > 0.05$) effect regardless of the soil history and no residual effect of reapplied LaBC[®] was carried over to cycle 7.

3.5. Soil pH. Soil pH (CaCl_2) was significantly influenced ($P < 0.05$) by LaBC[®] applied to the pretreated soil when assessed in cycle 6 (Table 7). When LaBC[®] was reapplied at 50 t ha^{-1} in cycle 6, there was a further significant ($P < 0.05$) increase (by 1-2 units) in soil pH regardless of the soil history. The residual effect of reapplied LaBC[®] from cycle 6 was carried over to cycle 7 and the pH increased by 0.5 units.

3.6. Soil Total Inorganic N. Soil total inorganic N was significantly ($P < 0.05$) increased by fertiliser only application with or without reapplied LaBC[®] in cycle 6 but not when LaBC[®] was reapplied without fertiliser (Table 8). There was no residual N effect of reapplied LaBC[®] carried from cycle 6 on to cycle 7.

3.7. Soil Microbial Biomass N. Soil microbial biomass N was not significantly influenced ($P > 0.05$) by LaBC[®] and/or fertiliser applied to the pretreated soil when assessed in cycle 6 (Table 9). In cycle 6, when LaBC[®] was reapplied, there was no influence on soil MBN regardless of soil history, and there was no residual effect in cycle 7 (Table 9).

4. Discussion

In the absence of reapplied LaBC[®], there was no residual nutrient effect of a single basal application of LaBC[®].

TABLE 4: Root dry biomass (g pot^{-1}) in ryegrass cycles 6 and 7 with reapplied LaBC[®] (+/-) as influenced by residual effects of soil amendments from previous management history.

Soil management history (cycles 1 to 5)	Root dry biomass (g pot^{-1})			
	Ryegrass cycle 6		Ryegrass cycle 7	
	-LaBC [®]	+LaBC [®] (50 t ha^{-1})	Current treatment (+/-LaBC [®])	Treatment -LaBC [®] from cycle 6
Control	$0.23 \pm 0.03^{\text{d}}$	$0.53 \pm 0.13^{\text{cd}}$	$0.10 \pm 0.02^{\text{c}}$	$0.50 \pm 0.10^{\text{b}}$
^a LaBC [®]	$0.34 \pm 0.08^{\text{cd}}$	$0.58 \pm 0.06^{\text{cd}}$	$0.31 \pm 0.09^{\text{bc}}$	$0.45 \pm 0.06^{\text{b}}$
^b Fertiliser	$1.08 \pm 0.22^{\text{a}}$	$0.57 \pm 0.15^{\text{cd}}$	$1.03 \pm 0.20^{\text{a}}$	$0.59 \pm 0.14^{\text{b}}$
LaBC [®] + fertiliser	$1.03 \pm 0.12^{\text{ab}}$	$0.67 \pm 0.09^{\text{bc}}$	$0.95 \pm 0.11^{\text{a}}$	$0.52 \pm 0.09^{\text{b}}$

^aLaBC[®] = 50 t ha^{-1} of wet weight equivalent.

^bFertiliser = macronutrient fertilisers (N, P, K, Ca, and Mg) applied according to district practice.

Values are mean \pm SE ($n = 3$). The letters followed by mean \pm SE were based on LSD ($P < 0.05$).

The main and interaction effects of treatments with the same letters, within the same column and between columns within each cycle, respectively, were not significant ($P > 0.05$).

TABLE 5: Root : shoot in ryegrass cycles 6 and 7 with reapplied LaBC[®] (+/-) as influenced by residual effects of soil amendments from previous management history.

Soil management history (cycles 1 to 5)	Root: shoot			
	Ryegrass cycle 6		Ryegrass cycle 7	
	-LaBC [®]	+LaBC [®] (50 t ha^{-1})	Current treatment (+/-LaBC [®])	Treatment -LaBC [®] from cycle 6
Control	$0.67 \pm 0.67^{\text{d}}$	$1.56 \pm 0.22^{\text{bcd}}$	$0.89 \pm 0.21^{\text{b}}$	$1.38 \pm 0.32^{\text{b}}$
^a LaBC [®]	$3.33 \pm 0.88^{\text{ab}}$	$1.50 \pm 0.14^{\text{bcd}}$	$3.22 \pm 0.75^{\text{b}}$	$1.48 \pm 0.13^{\text{b}}$
^b Fertiliser	$4.28 \pm 1.62^{\text{a}}$	$1.45 \pm 0.37^{\text{bcd}}$	$6.64 \pm 1.13^{\text{a}}$	$1.29 \pm 0.36^{\text{b}}$
LaBC [®] + fertiliser	$3.14 \pm 0.43^{\text{abc}}$	$1.13 \pm 0.16^{\text{cd}}$	$8.97 \pm 1.63^{\text{a}}$	$1.34 \pm 0.31^{\text{b}}$

^aLaBC[®] = 50 t ha^{-1} of wet weight equivalent.

^bFertiliser = macronutrient fertilisers (N, P, K, Ca, and Mg) applied according to district practice.

Values are mean \pm SE ($n = 3$). The letters followed by mean \pm SE were based on LSD ($P < 0.05$).

The main and interaction effects of treatments with the same letters, within the same column and between columns within each cycle, respectively, were not significant ($P > 0.05$), based on 2-way ANOVA.

TABLE 6: Shoot N concentration (%) in ryegrass cycles 6 and 7 with reapplied LaBC[®] (+/-) as influenced by residual effects of soil amendments from previous management history.

Soil management history (cycles 1 to 5)	Shoot N concentration (%)			
	Ryegrass cycle 6		Ryegrass cycle 7	
	-LaBC [®]	+LaBC [®] (50 t ha^{-1})	Current treatment (+/-LaBC [®])	Treatment -LaBC [®] from cycle 6
Control	$1.79 \pm 0.12^{\text{cd}}$	$2.11 \pm 0.05^{\text{abc}}$	$1.46 \pm 0.13^{\text{a}}$	$1.33 \pm 0.06^{\text{a}}$
^a LaBC [®]	$1.78 \pm 0.04^{\text{cd}}$	$2.18 \pm 0.13^{\text{ab}}$	$1.58 \pm 0.06^{\text{a}}$	$1.52 \pm 0.03^{\text{a}}$
^b Fertiliser	$1.86 \pm 0.1^{\text{bcd}}$	$2.28 \pm 0.22^{\text{a}}$	$1.49 \pm 0.03^{\text{a}}$	$1.44 \pm 0.04^{\text{a}}$
LaBC [®] + fertiliser	$1.75 \pm 0.1^{\text{d}}$	$2.35 \pm 0.07^{\text{a}}$	$1.46 \pm 0.08^{\text{a}}$	$1.54 \pm 0.01^{\text{a}}$

^aLaBC[®] = 50 t ha^{-1} of wet weight equivalent.

^bFertiliser = macronutrient fertilisers (N, P, K, Ca, and Mg) applied according to district practice.

Values are mean \pm SE ($n = 3$). The letters followed by mean \pm SE were based on LSD ($P < 0.05$).

The main and interaction effects of treatments with the same letters, within the same column and between columns within each cycle, respectively, were not significant ($P > 0.05$), based on 2-way ANOVA.

TABLE 7: Soil pH in ryegrass cycles 6 and 7 with reapplied LaBC® (+/-) as influenced by residual effects of soil amendments from previous management history.

Soil management history (cycles 1 to 5)	Soil pH			
	Ryegrass cycle 6		Ryegrass cycle 7	
	-LaBC®	+LaBC® (50 t ha ⁻¹)	Current treatment (+/-LaBC®)	Treatment -LaBC® from cycle 6
Control	5.0 ± 0.03 ^d	6.6 ± 0.06 ^b	4.9 ± 0 ^e	7.1 ± 0.09 ^b
^a LaBC®	5.4 ± 0.00 ^c	6.8 ± 0.06 ^a	5.4 ± 0.03 ^c	7.4 ± 0.07 ^a
^b Fertiliser	4.7 ± 0.03 ^e	6.5 ± 0.12 ^b	4.6 ± 0.03 ^f	7.1 ± 0.06 ^b
LaBC® + fertiliser	5.3 ± 0.07 ^c	6.9 ± 0.09 ^a	5.2 ± 0.06 ^d	7.2 ± 0.06 ^b

^aLaBC® = 50 t ha⁻¹ of wet weight equivalent.

^bFertiliser = macronutrient fertilisers (N, P, K, Ca, and Mg) applied according to district practice.

Values are mean ± SE (*n* = 3). The letters followed by mean ± SE were based on LSD (*P* < 0.05).

The main and interaction effects of treatments with the same letters, within the same column and between columns within each cycle, respectively, were not significant (*P* > 0.05), based on 2-way ANOVA.

TABLE 8: Total inorganic N (μg g⁻¹ soil) in ryegrass cycles 6 and 7 with reapplied LaBC® (+/-) as influenced by residual effects of soil amendments from previous management history.

Soil management history (cycles 1 to 5)	Total inorganic N (μg g ⁻¹ soil)			
	Ryegrass cycle 6		Ryegrass cycle 7	
	-LaBC®	+LaBC® (50 t ha ⁻¹)	Current treatment (+/-LaBC®)	Treatment -LaBC® from cycle 6
Control	0 ± 0 ^d	0.68 ± 0.33 ^{cd}	0.20 ± 0.06 ^b	0.33 ± 0.17 ^{ab}
^a LaBC®	0.25 ± 0.25 ^{cd}	0.92 ± 0.04 ^{bc}	0.20 ± 0.15 ^b	0.60 ± 0 ^a
^b Fertiliser	0.09 ± 0.09 ^d	1.70 ± 0.37 ^a	0.37 ± 0.09 ^{ab}	0.63 ± 0.07 ^a
LaBC® + fertiliser	0.29 ± 0.14 ^d	1.50 ± 0.43 ^{ab}	0.37 ± 0.12 ^{ab}	0.43 ± 0.15 ^{ab}

^aLaBC® = 50 t ha⁻¹ of wet weight equivalent.

^bFertiliser = macronutrient fertilisers (N, P, K, Ca, and Mg) applied according to district practice.

Values are mean ± SE (*n* = 3). The letters followed by mean ± SE were based on LSD (*P* < 0.05).

The main and interaction effects of treatments with the same letters, within the same column and between columns within each cycle, respectively, were not significant (*P* > 0.05), based on 2-way ANOVA.

TABLE 9: Microbial biomass N (μg g⁻¹ soil) in ryegrass cycles 6 and 7 with reapplied LaBC® (+/-) as influenced by residual effects of soil amendments from previous management history.

Soil management history (cycles 1 to 5)	MBN (μg g ⁻¹ soil)			
	Ryegrass cycle 6		Ryegrass cycle 7	
	-LaBC®	+LaBC® (50 t ha ⁻¹)	Current treatment (+/-LaBC®)	Treatment -LaBC® from cycle 6
Control	7.60 ± 7.60 ^a	0 ± 0 ^a	7.67 ± 5.55 ^{ab}	3.10 ± 1.84 ^b
^a LaBC®	3.20 ± 3.20 ^a	3.90 ± 3.90 ^a	2.54 ± 2.54 ^b	7.20 ± 3.69 ^b
^b Fertiliser	7.63 ± 6.83 ^a	15.85 ± 14.77 ^a	7.61 ± 5.08 ^{ab}	22.94 ± 9.33 ^a
LaBC® + fertiliser	4.42 ± 2.17 ^a	09.77 ± 4.89 ^a	10.70 ± 2.74 ^{ab}	10.85 ± 6.13 ^{ab}

^aLaBC® = 50 t ha⁻¹ of wet weight equivalent.

^bFertiliser = macronutrient fertilisers (N, P, K, Ca, and Mg) applied according to district practice.

Values are mean ± SE (*n* = 3). The letters followed by mean ± SE were based on LSD (*P* < 0.05).

The main and interaction effects of treatments with the same letters, within the same column and between columns within each cycle, respectively, were not significant (*P* > 0.05), based on 2-way ANOVA.

However, soil pH was maintained at a significantly higher level, indicating a residual liming effect of the previously applied LaBC® in this acid soil. Furthermore, increased ryegrass shoot and root weight were associated with a residual effect of fertiliser history in addition to the direct nutrient release from reapplied LaBC®. The effect of reapplied LaBC® was generally confined to plant growth cycle. Where there were benefits of LaBC®, there was no corresponding change in microbial biomass N.

Combined application of organic amendments and inorganic mineral fertilisers is a balanced approach while budgeting plant nutrient management and carbon sequestration practices [37–41]. Inorganic soluble N fertilisation effects on vegetation cover, plant biomass, and yield [42, 43] can be immediately noticeable, significantly higher, and long-lasting [39, 44–48]. For example, application of inorganic N (37 kg ha^{-1}) and P (94 kg ha^{-1}) fertilisers increased growth of both shoot and root biomass and increased the root : shoot ratio of cool-season wheatgrass (*Agropyron* sp.) when grown in a low fertile strip mine soil under glasshouse and field conditions [49]. In a 3-year study [46], a single application of inorganic fertilisers (100 kg N and 35 kg P ha^{-1}) on a degraded semiarid ecosystem continuously supported a significantly higher native plant cover with shrubs and grasses (to 29%) after 24 months compared with a single application of screened and unscreened biosolid and municipal waste composts (each at 40 t ha^{-1} , supplied a range of nutrients $10\text{--}54 \text{ kg N}$ and $6\text{--}68 \text{ kg P ha}^{-1}$). The root biomass and root length did not differ significantly between treatments, but the root : shoot was significantly lower (2.1) with inorganic fertiliser application associated with increased shoot biomass. However, the effect was less pronounced after 3 years. Similarly, in the evaluation of residual effects of the biosolid product LaBC® and fertiliser in our study, residual effects of LaBC® were minimal compared with the residual effects of fertiliser.

LaBC® had a residual effect on increasing soil pH, primarily due to the incorporation of lime into this biosolids byproduct. In contrast, continuous application of mineral fertilisers, especially inorganic N, can acidify the soil environment [50] as was demonstrated in cycles 6 and 7 of our study where there was a decrease in soil pH (CaCl_2) (to 4.6) associated with inorganic fertiliser application. An increase in soil pH can potentially increase N mineralisation and organic substrate availability thereby increasing microbial biomass and its activity [51]. Similar liming effects have been shown in an incubation study over 56 days [31].

In contrast with the residual liming effect, inorganic N released from LaBC® was not sufficient to carry over to a sixth cycle of ryegrass growth after a single application. Soil N mineralisation is considered to be a crucial factor while determining agronomical values of organic amendments [52]. Soil inorganic N was significantly higher only when LaBC® was reapplied in soils that had received either mineral fertiliser alone or mineral fertiliser in combination with LaBC® application in the past. In addition to the residual effect of inorganic fertilisers, the added slow release nutrients from

reapplied LaBC® contributed to plant available nutrients only within one ryegrass growth cycle 6. This implies that LaBC® reapplied after previous basal application of LaBC® (50 t ha^{-1} wet weight equivalent) would contribute little towards the potential of N leaching into the immediate environment.

Increased soil microbial biomass and activities indicate improved biological components of soil fertility status after application of organic amendments [53, 54]. Nevertheless, in the acid sandy soil used in this study, reapplied LaBC® did not increase microbial biomass N although soil pH and mineralisation based on soil inorganic N were higher with reapplied LaBC®. In contrast, microbial biomass N changed over time with application of LaBC® in the absence of plants [19]; the presence of plants may have introduced competition for N at the level of LaBC® applied (50 t ha^{-1}).

5. Conclusions

- (i) As hypothesised, reapplication of a lime- and clay-amended biosolids product, LaBC®, had an immediate effect of increasing soil pH regardless of prior history of LaBC® and fertiliser application. The main benefit of LaBC® was to increase soil pH (CaCl_2) which was maintained at about 5.4 even after 6 cycles of ryegrass growth following a single application.
- (ii) The second hypothesis is that the application of LaBC® to soil with a fertiliser history would release more N and increase ryegrass growth to a greater extent than an application of LaBC® to soil without a fertiliser history; this was not supported.
- (iii) The release of inorganic N with reapplied LaBC® appeared to be confined to the current growth cycle, with little or no residual effect.
- (iv) Our investigation demonstrates that both reapplied and previously applied LaBC® at 50 t ha^{-1} wet weight equivalent are unlikely to pose a risk of leachable N in this sandy soil, based on the available soil inorganic N measured.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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Review Article

Potential for Recycling Nutrients from Biosolids Amended with Clay and Lime in Coarse-Textured Water Repellence, Acidic Soils of Western Australia

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Application of biosolids in soils is an efficient method of recycling nutrients from biosolids and it is considered even safer when it is modified after mixing and diluting with other suitable soil organic amendments. A variety of soil organic amendments, such as green manures and composts, are used for modifying and co-composting with biosolids. However, these may not be considered as appropriate biosolids disposal and remedial measures for soils with unique problems such as low soil pH, water repellence nature, and poor water and nutrient retention capacities due to soil textural issues. Historically, soil amendments such as lime, clay, and recently biochar are being applied for such problematic soils at Western Australia and these researches focused mostly on improvement in soil physical and chemical properties. However, studies with potential for applying modified biosolids with these amendments are not complete yet. This review focused on identifying such gaps in these studies from over 170 peer-reviewed key research and review articles published over decades to latest in these areas.

1. Introduction

Coarse-textured, sandy soils are common in Mediterranean regions of Australia, which extend from Geraldton north of Perth, across south-western Australia and southern-south Australia, and into the Wimmera, Mallee, and northern districts of Victoria [1]. The Swan Coastal Plain on the coast of south-western Australia, within this Mediterranean environment, experiences hot dry summer and cool, wet winter with annual rainfall ranges between 500 and 600 mm [2]. Low fertility and soil nutrient deficiency are common in this climatic zone as the soils are derived from weathered, ancient rocks and are low in soil organic matter [1, 3]. Deep sand occurs at more than 80 cm depth and the pale deep sand found on the Swan Coastal Plain is typically classified as bleached-Orthic Tenosol [4]. Soil acidity, water repellence, poor water-holding, nutrient leaching, and environmental

degradation are major issues that need improved soil management practices to increase productivity in the dry-land farming zones of this region [1, 5, 6].

Biosolids disposal is an international environmental issue as many developed countries are incinerating their biosolids or disposing them as landfills and ocean dumps [7]. Global production of biosolids exceeds 10 M t yr^{-1} and the production average is $27 \text{ kg of dry biosolids person}^{-1} \text{ year}^{-1}$ [8]. Several studies have focused on the advantage of recycling biosolids for beneficial agronomic effects on soil fertility from the organic matter and readily available and/or slow release plant nutrients available in the sludge [9–12]. Sludge addition usually increased plant growth both in field and in greenhouse experiments on different crop species [13–15]. On the contrary, research on the limitations of biosolids has demonstrated that excessively applied biosolids can release

heavy metal and the metal accumulation and cause stress and restrictive effects on soil microbes [16–24] and on plant root and shoot biomass [25].

Other studies have focused on managing excessive N and P nutrient release from biosolids and risk for nearby surface water quality [26–29]. For instance, the Western Australian State of the Environment Report identified that eutrophication led to nearly 30% of accidental fish kills associated with excessive P and N leaching into waterways through fertiliser application ($TN = 0.75 \text{ mg N L}^{-1}$, $TP = 0.03 \text{ mg P L}^{-1}$ trigger values indicated for the Swan-Canning estuaries as per guidelines of ANZECC and ARMCANZ) [30]. These studies show that no generalised application rate can be recommended; rather it is necessary to investigate sources of biosolids separately for a particular soil due to varying content of nutrients and metals present within biosolids [31–35].

1.1. Modified Biosolids Products. Traditionally, biosolids are applied to land in their original form for their nutritional value after treating raw sludge [15, 36, 37]. Plant nutrient imbalances as either deficiencies or phytotoxicities can occur when using these materials in unmodified forms [38]. This could outweigh the fertiliser value of sludge application. However, it is possible to rectify this problem by slightly modifying the physical and chemical properties of biosolids in addition to improving their formulation for safe disposal for specific soil conditions [38].

Several forms of modified biosolids are available for use in agriculture. The simple form of a biosolids mixture is lime-stabilised biosolids or LAB (lime-amended biosolids) [39, 40]. For example, a study conducted by Sloan and Basta [9] noticed that alkaline biosolids caused greater reductions in phytotoxic-Al when applied in strong acid soils than when applied with nonalkaline biosolids. In another study, Luo and Christie [39] found increased yield of barley when amending a mild to strong acid sandy loam ($\text{pH } 4.5$) soil with alkaline-stabilised biosolids at $33.5 \text{ t dry matter ha}^{-1}$. In this study, fly-ash, a liming material resulting from coal combustion, was used for alkaline stabilisation. The liming effect of the modified biosolids increased soil pH by about 1.2–1.5 units and as a consequence Al toxicity was reduced (from 4.0 to 0.1 cmol kg^{-1}) in barley crop that was grown under strong acid sandy soil conditions.

Manipulating soil pH using alkaline stabilised biosolids helped suppress the soilborne pathogenic nematode, *Meloidogyne incognita*, in addition to supplying plant nutrients in a loamy sand soil [41]. In this study, N-Viro soils, a commercial product developed by mixing biosolids with alkaline byproducts (such as coal ash, cement, and lime kiln dusts), was used as a soil amendment, further amended with urea fertiliser and kept for 5 days in incubation. It suppressed nematode growth which was directly associated with higher NH_3 production from urea and indirectly through increased soil pH by the alkaline biosolids.

Studies have also focused on mixing a variety of materials with biosolids to alter the nutrient or heavy metal availability that enhanced safe nutrient levels [42]. For example, there were benefits when biosolids were mixed with river sediments

and/or composted with other organic materials such as oats straw and cattle manures [43–46]. A greenhouse study showed a significant increase in plant growth when soil was amended with a mixture of sediment and biosolids compared with application of either sediment or biosolids alone [44]. In this case, heavy metals were within an accepted range in the plant tissues when grown with the mixture. However, the impact on soil microbial communities was not assessed in this study. A formula that contains a mixture of biosolids with river-sediments was investigated for recycling nutrients from biosolids using native grasses [46]. It was suggested that mixing of biosolids with river sediments has potential benefit in increasing organic and inorganic nutrient content of the sediments and in improving sediment quality; the reclamation of damaged soils was improved at a field scale for the combination of biosolids and sediments compared with either sediment or biosolids alone. This study confirmed that a mixture of river sediment improved soil texture and increased plant available nutrients, microbial biomass, and plant growth. The mixture altered microbial community composition, with relative increases in Gram-negative bacteria and decreases in Gram-positive bacteria, fungi, and actinomycetes [46].

1.2. Research on LaBC in Western Australia. LaBC is a slow release organic amendment [47]. It is a typical modified clay and lime amended biosolids product (LaBC: lime-amended BioClay) formulated for use in acid sandy soils of the Swan Coastal Plain in Western Australia [47]. It has been demonstrated that LaBC has lower chemical contaminant threshold concentrations than that required for unrestricted use (C1 classification) based on Western Australian Biosolids Guidelines [47, 48]. This is a consequence of the severe dilution of the biosolids in a lime and clay blended product. LaBC has a number of desirable soil-organic amendment qualities such as improving water and nutrient retention capacity of coarse textured soils. It has been shown to alter soil pH (CaCl_2 extract) from 5.0 to pH of 8.0 as the product-blend is strongly alkaline (pH of 10.0) due to the presence of lime [48]. In addition, LaBC is rich in clay and organic matter and rich with significant amounts of plant nutrients from the biosolids component [48]. The clay material blended in the LaBC is not hydrophobic and has potential to reduce the severity of hydrophobicity of sandy soils when applied at rates greater than 50 tonnes per hectare [47–49].

The suitability of LaBC as a soil organic amendment has been investigated at both laboratory and field scales to evaluate the safe recycling of biosolids in the environment [48–52]. The chemical effects of LaBC in providing agronomical benefits for crop growth have been extensively demonstrated under both field and laboratory conditions [47, 48, 53]. Furthermore, field experiments have been initiated to investigate nutrient leaching risks after its applications and to study the long-term effect of LaBC using ryegrass [47, 50, 53]. However, the overall effect of LaBC on soil microbiological fertility was not considered in these studies and this requires investigation before the product is recommended for wide application in the soils of Ellen Brook catchment [54].

2. Major Problems in Sandy Soils and Their Causes

2.1. Soil Acidity. In south-western Australia, soil acidification is widespread and results in part from inefficient use of nitrogen in agriculture that enhances natural acidification processes [40, 47, 55]. Soil acidification from pasture and cropping affects about 3 M ha of cropped land in the Mediterranean region of Australia [56]. Soil acidity (mostly pH less than 5.5) commonly develops in agricultural lands in association with building-up of organic matter, frequent application of mineral fertilisers, and leaching of nitrate [55]. Acidity also creates Al and Mn toxicity to plants [9, 39, 57] and inhibits root nodulation and N-fixation in legumes [1, 58–61]. Low soil pH reduces the growth and activities of many soil microorganisms and this eventually leads to lower plant productivity due to decreased organic matter decomposition and mineralisation [62, 63]. Historically, agricultural lime or other liming materials are applied at rates of 1–1.5 t ha⁻¹ every 7–10 years in Australia based on soil buffering capacity and properties of liming materials, towards increasing soil pH for favourable plant productivity [40, 55, 64].

2.2. Soil Water Repellence. Soil water repellence develops by the accumulation of plant and fungi derived hydrophobic organic coating on the sand grains [6, 65, 66]. Soil organic materials, in particular stable humic fraction, have been identified as main reason for water repellence on sand grains [67–69]. Water repellence in soils of south-western Australia occurs mostly in coarse-textured sandy soils and sandy duplex soils that have less than 5% clay on the surface [1, 70, 71]. Water repellent soils have been investigated worldwide [67], for example, in USA, Florida and California, in New Zealand, and in Australia, and the involvement of organic matter is considered a factor associated with water repellence in all of these regions [67, 69]. In Australia, more than 5 million ha of land has been characterised with water repellent sands, including those on the Swan Coastal Plain [6, 72, 73]. Several studies also reported accumulation of wax-like long chain fatty acids of either fungal or plant origin and other similar types of organic compounds of plant origin, in particular, from *Eucalyptus* spp. as causes of water repellence [66, 74–76]. The nonwetting nature of soil increases the risk of water and wind erosion. Some 2 million ha of sandy soils across southern Australia is affected by wind erosion and leads to severe productivity losses [1, 6, 66, 77]. Nonuniform wetting of soil, often associated with nonwetting properties, results in severe yield reduction in agricultural land due to poor germination of seeds and less plant establishment in these regions [6, 66, 69, 78, 79].

2.3. Nutrient Leaching. Excessive fertilisation can accelerate nutrient leaching in sandy agricultural soils and has potential to cause environmental problems such as eutrophication [80], soil acidity [81], and ground water pollution [82, 83]. For instance, commercial bags of garden and all-purpose fertilisers should not contain more than 2% and 1% of P, respectively, according to the Environment Protection regulations guidelines of Western Australia. Nutrient retention capacity of

sandy soils is generally very poor with excessive fertilisation in sandy soils increasing nutrient leaching [84] and free-draining sandy soils can threaten nearby water bodies.

For the Swan Coastal Plain in south-western Australia, nutrient leaching is a primary cause of ground water pollution [85, 86]. The contaminated groundwater further affects surface water quality of the river systems and surrounding lakes [87, 88]. Dissolved inorganic N and bioavailable P are generally the excessive nutrients that promote algal growth in water bodies [89–91] which can be hazardous to aquatic organisms and human health [92]. Maximum acceptable limit for nitrate in drinking water is 10 mg L⁻¹ according to World Health Organization. Hence, efficiency in application of nutrients should be the main objective in designing fertiliser plans for coarse-textured agricultural soils [86–88].

3. Potential Sandy Soil Management Using Amendments

3.1. Clay. Clay spreading is a sustainable and economically viable method for long-term remediation of water repellence in sandy soils [93] and claying is a common term used for top dressing transported clay materials on surface of the sandy soil, which is a practice used in south-western Australia. Increases in clay content of even 1–2% can play a crucial role in prevention of water repellence in a very sandy soil [70, 71]. Water repellence can be minimised by applying a higher amount of clay minerals (up to 100 t ha⁻¹) in sandy soils [70, 93, 94] and, in particular, use of kaolinite clay on a very severely water repellent soil has been shown as the best clay in overcoming water repellence [95]. Clay is attributed for its potential in increasing surface area that causes improved soil wettability [93, 95]. Conventionally, clay has been added at rates of 40–250 t ha⁻¹ on sandy soils in southern and south-western Australia to overcome the water repellence [70, 93, 94]. However, the technique of claying is continually being modified to attain maximum economic returns from degraded agricultural lands in various regions of Australia [6, 93].

3.2. Lime. Lime addition has been shown to decrease water repellence in sandy soils and liming is generally used as a common term for soil lime application [6, 69]. It has been demonstrated that lime added at rates of 3–15 t ha⁻¹ decreased water repellence under irrigated sandy soils in WA [6]. While liming is generally practiced to increase soil pH in acid soils, it has also been shown to increase microbial biomass and soil respiration that is associated with acid-intolerant microorganisms [62, 63, 96]. Increased soil microbial biomass and activities due to liming, in turn, increase mineralisation and nutrient availability for better plant productivity [63, 97]. However, increases in microbial activity, including mineralisation rates, are not consistent under different soil management systems [98, 99]. In a comparison of the practices of claying and liming, it was shown that lime applied up to 5 t ha⁻¹ could be used as an alternative for clay applied at 100 t ha⁻¹ in a sandy soil [6]. However, a combination of lime with clay was not included

in this investigation. Furthermore, higher soil pH has been shown to increase the abundance of wax-degrading bacteria corresponding with a reduction in the hydrophobic layer that causes water repellence in sandy soil [6]. Therefore, management practices that decrease water repellence under acid soil conditions could be strategically planned by choosing a combination of soil ameliorants.

3.3. Biochar. Biochar is a byproduct resulting from pyrolysis (process of thermal degradation in the absence of air) of organic materials [100, 101]. Besides being popularly known for its carbon sequestration values [102–106], its soil ameliorating and agronomic values have equally attracted research attention worldwide [107–111]. Biochar characteristics differ (e.g., pH 6.2–9.9) with the various feedstock sources used under different production temperatures (260–700°C) [101]. There is no supply of direct plant nutrients available in biochar to help enriching soil fertility status [101, 112]. However, the varying micro- and macropore structures of biochar (from nano-, <0.9 nm, micro-, <2 nm, to macropores, >50 nm) [113] help increase soil surface area and retention of nutrients supplied through other fertiliser sources. These factors increase soil agronomic values and plant productivity in addition to providing a physical niche for beneficial soil microorganisms [101, 108, 114–116]. Therefore, biochar has potential for improving soil fertility by manipulating aspects of soil physical, chemical, and biological properties when amended with a range between 0.5 and 135 t ha⁻¹ [101, 110, 117]. The nature of the manipulation depends on the origin of the biochar, as not all forms of biochar have the same characteristics [101].

The physical structure of biochar manipulates soil macro- and microporosity and provides microhabitat for soil microbial communities including fungi (arbuscular mycorrhizal (AM) fungi) and bacteria [108, 110, 118–120]. Management of microbial communities in agricultural soils depends on provision of soil conditions that suit their growth and activity [121]. Several studies have shown increased colonisation of AM fungi corresponding with biochar application [119, 122, 123]. The biochar particles buried in soil increased availability of micropore space which has been claimed to provide protective microhabitat for growth and extension of the hyphae of AM fungi into the biochar [124, 125]. The extended extraradical hyphae can thereby increase plant P uptake from soil [126–128]. Similarly, rhizobacterial activity and symbiotic nitrogen fixation with legume plants can be increased in association with biochar application [129]. Biochar applied to soil can protect rhizobia in pores <50 mm from predation in soils with low clay content and caused improved nitrogen fixation [130].

In addition to creating favourable microhabitat as a direct benefit to microbial communities, biochar application can help create favourable soil chemistry for their survival and plant uptake indirectly [124, 131–134]. For example, increased atmospheric N₂ fixation was observed in biochar-applied legume root nodules through improved symbiotic association with soilborne rhizobia [124, 129]. These effects are associated with a suppressive effect of soluble forms of N in soil solution on the N₂ fixation, while available soil P

can provide supportive effect in the bacterial growth when soils are amended with biochar [118, 124, 135]. It has also been suggested that biochar may have a role in presence of fertilisers if they stimulate the available native strains of beneficial microbial communities in the soil [112, 132].

From an environmental perspective, soil amendment with some forms of biochar has potential to minimise nearby surface and ground water pollution [112, 136–141] through the mechanism of adsorption of dissolved organic carbon [119], NH₄⁺ [112, 142, 143], and trace metals in leachates [144]. Soil amendment with biochar may also reduce heavy metal accumulation due to frequent applications of other organic amendments (e.g., biosolids) that contain potential metal contaminants [100]. Some studies have demonstrated increased bioavailability of plant nutrients and uptake of P, K, Ca, Zn, and Cu after charcoal application, while decreased N leaching has also been observed [112, 145–149]. A range of specific soil physical, chemical, or microbiological properties, therefore, may be manipulated by developing a careful soil amendment strategy with biochar, although not all biochars are expected to function in the same manner [110]. For instance, short-term negative impacts of biochars have also been attributed to volatile toxic organic compounds and phytotoxic salts that could have caused reduced microbial biomass C [143] and plant root growth activities, respectively [110].

3.4. Interactions between Clay, Biochar, and Biosolids. Characteristics of soil amendments are inherently different. They determine key roles in altering soil N mineralisation processes when applied independently or when coapplied with other amendments. While biosolids are usually considered as a source of nutrients (especially N) that accelerate N release into soil [150, 151], other amendments such as clay and biochar act as potential sinks and lead to retention of soil N and decrease N leaching into the immediate environment through various mechanisms [101, 110].

Dempster et al. [141] compared the efficiency of clay and biochar in achieving decreased N leaching in coarse-textured soils in south-western Australia. A lysimeter column study was investigated for 21 days after amending soil independently with 25 t ha⁻¹ of clay and biochar with different application methods. Both amendments significantly decreased cumulative NH₄⁺ leaching by about 20% and NO₃⁻ leaching by about 25%. However, biochar significantly decreased NO₃⁻ leaching more than clay did and this was mainly associated with a larger difference in anion retention capacity. While clay lacked the ability to retain NO₃⁻, the biochar used in this study had a dual role in NO₃⁻ sorption processes and increased water holding capacity [141]. Moreover, the capacity of biochar to control N leaching in this case was associated with decreased nitrification values in amended soil as shown in a previous study [143].

Previous studies with biochar identified the same two important mechanisms as described above, which were attributed as potential reasons for decreased N leaching capacity. First the high NO₃⁻ retention was due to positive charge of biochar [152]. Second, decreased NH₄⁺ leaching was due to improved gravimetric water holding capacity of

soil rather than its sorption effect onto the biochar [112, 141, 142].

Coapplication of clay with other organic composts has been shown to be a beneficial soil management practice in coarse textured soils of south-western Australia and for similar environments [153]. As discussed above for the experiments of Dempster et al. [141], a preliminary 21-day study showed that clay also had a significant effect on decreasing N leaching in sandy soils and its effect was comparable with that of biochar. However, that study did not investigate whether the nutrients retained by clay or biochar were plant available or whether they helped increase the efficiency of use of fertiliser in the amended agricultural soil. Further, these studies did not investigate the combined effect of clay and biochar on plant growth nor did they investigate the combined influence on slow release fertiliser such as biosolids when they are coapplied in multiple combinations.

The inclusion of biochar helped decrease nitrate leaching from biosolids amended soils over five months [42]. A lysimeter column leaching study was established with ryegrass and amended with combinations of biochar (102 t ha^{-1} equivalent) and biosolids (600 and $1200 \text{ kg N ha}^{-1}$ equivalent) in two types of silty loam soils. The inclusion of biochar and biosolids together resulted in significantly less nitrate leaching than in the biosolids alone treatment and suggested for higher rate application of the mixtures to rebuild degraded soils. However, developing an optimum mixture ratio and identification of the mechanisms responsible for alteration in the nitrogen cycle were not addressed in this study. There is potential that biosolids applied at a level equivalent to $1200 \text{ kg N ha}^{-1}$ could become an additional risk associated with excessive leaching in conditions similar to those used by Knowles et al. [42].

The use of clay minerals such as Na-bentonite and Ca-bentonite could be a potential tool for immobilising up to 70% of bioavailable form of heavy metals such as Zn, Cd, Cu, and Ni in sewage sludge contaminated soil [154]. In an incubation study of more than 110 days, soil biological parameters such as microbial biomass C, respiration, and organic C mineralisation rate were higher in clay-amended soil, and this was associated with reduced heavy metal toxicity on the microbial parameters.

In comparison to the studies considered above, the combination of lime, clay, and biosolids makes LaBC a unique biosolids product with potential for benefits to be multiplied when coapplied with biochar. This raises scope of investigating the biosolids amendment in combination with other N-absorbing ameliorants [54].

4. Soil Microbial Properties

A shift in soil microbial communities can be associated with soil textural changes. For example, mineralization is influenced by soil texture and structure as they both affect the aeration status, the physical distribution of organic materials, and other physical, chemical, and biological environmental characteristics of soil [155–157]. Bach et al. [157] demonstrated that soil microbial community (using phospholipid

fatty acid, PLFA method) responses to grassland restoration were moderated by soil texture during the monitoring of grassland ecosystem recovery for 19 years. Nevertheless, other studies have claimed that soil texture had no significant effect on the decomposition rate and/or microbial community structures when soils with contrasting textures were amended with carbon substrates such as low molecular weight carbon sources (e.g., glucose) [158–160]. However, addition of clay to soil could create new microhabitats, particularly if it has low clay content [161]. The added clay can contribute to an increase in soil microbial biomass [156, 162–164]. Despite conflicting observations, the respiration rate of preincubated soils could, at least to some extent, depend on the replacement of the labile substrate from soil organic matter, and the amount applied could decrease when soil texture is altered, for example, when texture is modified with increasing clay content [165].

Soil texture, in particular clay content, is an important factor which influences organic matter decomposition including the labile pool of carbon, that is, microbial biomass [166, 167]. Clay can physically protect living (microbial biomass C) and nonliving soil organic matter and causes reduced decomposition and CO_2 evolution in soils [165, 168, 169]. The direct physical protection of organic matter is possibly achieved by surface adsorption and entrapment of organic matter between clay layers and thereby prevents or reduces the rate of decomposition by soil microorganisms [170–173]. On the other hand, clay can protect the active component of living organic matter (known as soil microflora) by physically confining them in small pores, making them less active and protecting them against predation by soil protozoa [174]. Thus, clay is assumed to play a major role in minimising soil C mineralisation.

Umar [167] investigated the protective effect of clay minerals (5, 10, 20, and 40% w/w) on wheat residue (2% w/w) decomposition in commercial sand associated with decreased C mineralisation through 32-day incubation study. Clay applied at <20% decreased cumulative respiration until 28 days and this decreased C mineralisation was associated with a clay binding effect on organic matter. However, 20 and 40% clay increased the C mineralisation after 18 days which was associated with higher water retention capacity and corresponding increase in higher activities of microbial communities which were unable to survive in lighter-textured soils.

5. Conclusions

Biosolids disposal on soils with water repellent, acid, and coarse-textured characteristics will need to ensure that any nutrients from the modified product do not become a potential threat to the environment. While the potential agro-nomical influences of modified biosolids products use have been identified in terms of altering soil physical and chemical properties, their use in improving soil fertility with relevance to microbiological parameters has not been studied critically.

The potential benefits of combining biosolids with other amendments can include reduction in microbial processes such as mineralisation of organic matter. This could be

beneficial if it led to slow release of nutrients from biosolids. There is a need for further investigation of the C and N mineralisation patterns and related changes in soil microbiological properties when combinations of clay, lime, and biosolids and/or biochar are applied to sandy soil. This information would enable greater understanding of how modified biosolids products influenced the short- and long-term dynamics of soil nutrient cycling following application to soil. It would clarify effects of plant rotation, in terms of the build-up of soil N, microbial biomass N, and bioavailability of N. Investigation of impacts on soil microbiological processes across the scales of laboratory incubation through a series of glasshouse and field experiments would contribute to modification of biosolids products to suit particular soils, plants, agricultural management systems, and environmental conditions.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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Research Article

Agronomic Efficiency of Biosolid as Source of Nitrogen to Banana Plants

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Sewage sludge (SS) or biosolid has been studied as source of nutrient for several different plant species. It also contributes to soil fertility recycling organic matter and plant nutrients. This followup work examines a three-year (2001–2004) field experiment designed to evaluate the response of banana plants (Cavendish subgroup) to the application of biosolid as source of nitrogen. The treatments consisted of control (mineral PK, no N), three rates of sludge, and two rates of mineral NPK fertilizer. Plant and soil N concentration, fruit yield, plant height, stem diameter, and foliar endurance index were measured. Fruit yield with mineral fertilization or sludge applications did not differ statistically ($P > 0.05$). Application of biosolid resulted in statistically significant higher agronomic efficiency ($P < 0.05$) in comparison to mineral fertilizers. The concentration of soil mineral nitrogen increased using mineral fertilizer or sludge until 0.80 m after three years of application. The effect of the source of N was smaller than the effect of the rate. Biosolid can be used as source of N for banana growers.

1. Introduction

Among the alternatives to dispose biosolid, recycling on agricultural land offers economic and environmental benefits. The key concept of land application of biosolid [1–3] is the return of organic matter and plant nutrients removed by crops. Some researchers have reported that biosolid application also improves soil physical properties [4–6]. Biosolids were effective to substituted mineral fertilizers as source of nitrogen (N) for ryegrass [7], sugarcane [1, 2, 8], sunflower [9], and maize [10, 11]. In all of these, there were no indications of environmental hazards due to biosolid addition in soils. In Brazil, as in many other places, there is a specific regulation about the application of biosolids in agricultural soils [12] and for agronomic purposes, the sludge rate must be calculated by using the sludge's N mineralization rate (N_{MR}) and the plant requirement.

Andrade et al. [11] pointed out that the successive application of biosolid can cause an increase of the potential

mineralizable N, underestimating the soil capacity of providing N. Situations like that could represent environmental risks as the gradual increase of soil capacity to supply N should be coupled to a decrease in the rates of sludge applied as fertilizer preventing nitrate leaching [13, 14] and soil N_2O emissions [15, 16].

Besides the environmental concern, the use of biosolid as source of N for banana could be beneficial as the plant grows fast and requires high levels of nutrients to produce biomass and fruits. Literature review indicates that plant of banana producing around $50\text{ t fruits ha}^{-1}\text{ year}^{-1}$ demands from 198 to 388 kg ha^{-1} of N and 29 to 52 kg ha^{-1} of P [17]. Although the biosolid, as source of plant nutrients, has been evaluated for other plant species, there is a lack of knowledge about crops highly nutrient demanding.

In this study, we hypothesized that the biosolid was similar to mineral N fertilizer to supply the required nutrients to banana plants. The objectives of this work were to evaluate the banana response to the application of biosolid as source

of N in terms of fruit yield, N uptake, and agronomic effectiveness of N usage.

2. Materials and Methods

2.1. Site Description. The experiment described in this paper is complementary to that reported by Teixeira et al. [18] which discussed only soil parameter modifications by sludge application. Here, fruit yield from three cropping cycles, the agronomic efficiency use of N from sludge, and the concentration of soil nitrogen (N-NO_3^- and N-NH_4^+) were presented. The experiment was carried out between September 2001 and December 2004 at the city of Parqueira-Açu, state of São Paulo, Brazil ($24^\circ 39' \text{S}$; $47^\circ 55' \text{W}$). This region cropped around 38,500 hectares of land with banana, representing 70% of the total area planted with this species in the state of São Paulo. Historical climatic records from 01/10/1992 to 31/12/2004 showed that the monthly averaged minimum/maximum air temperature ranged from 23.2°C (July) to 31.1°C (February); monthly rainfall averaged from 54.7 mm (August) to 298.6 mm (January). Climate data of experimental period and most relevant management practices performed during cropping period were presented in Figure 1.

The soil was classified by Sakai and Lepsch [19] as a clayey texture (34.0% of clay, 0.9% of silt and 57.0% of sand) A horizon alic Yellow Oxisol (Parqueira Unit-I). Before trial installation, soil chemical characteristics were evaluated according Raij et al. [20] in composite samples ($n = 6$) collected from depths of 0–0.20 and 0.20–0.40 m resulting in values of $\text{pH CaCl}_2 = 4$ and 4; soil organic carbon = 23 g dm^{-3} and 18 g dm^{-3} ; $\text{K}^+ = 1.6 \text{ mmol}_{(+)} \text{ dm}^{-3}$ and $1.2 \text{ mmol}_{(+)} \text{ dm}^{-3}$; $\text{Ca}^{2+} = 8 \text{ mmol}_{(+)} \text{ dm}^{-3}$ and $7 \text{ mmol}_{(+)} \text{ dm}^{-3}$; $\text{Mg}^{2+} = 2 \text{ mmol}_{(+)} \text{ dm}^{-3}$ and $2 \text{ mmol}_{(+)} \text{ dm}^{-3}$; $\text{H}^+ + \text{Al}^+ = 90 \text{ mmol}_{(+)} \text{ dm}^{-3}$ and $92 \text{ mmol}_{(+)} \text{ dm}^{-3}$; and base saturation index as $(\text{K}^+ + \text{Ca}^{2+} + \text{Mg}^{2+})/\text{CEC} = 12.3\%$ and 10%, respectively. Based in this data, soil received dolomitic limestone (effective calcium carbonate equivalent = 67%) in order to increase the base saturation index (V) to 60%. Soil preparation consisted of disc plowing, harrowing and furrowing until the depth of 0.35 m.

2.2. Sludge and Application Rates. Biosolid was produced by the sewage treatment plant of the city of Mongagua, located 170 km far north of the experimental site. This plant processes municipal sewage by using the method of Sequencing Batch Reactor (SBR) or “Fill and Draw System” with hydraulic retention time of around 6-7 hours. Selected characteristics of the sludge, on different years, are in Table 1. Sludge available N was quantified according CETESB [21]. Organic carbon (OC) and nitrogen forms (Kjeldahl-N, NH_4^+ -N, $(\text{NO}_3^- + \text{NO}_2^-)$ -N) were determined according Raij et al. [20]. Nutrients and heavy metals were extracted according to the 3051 US-EPA method [22] and quantified by inductively coupled plasma atomic emission spectroscopy (ICP-AES). Sludge pH was determined at 1:10 water extract (m/v); humidity and volatile solids (VS) by mass loss at 60°C and 500°C , respectively.

Sludge rate was calculated to match the theoretical banana requirement for nitrogen. In the state of São Paulo, N

fertilizer recommendation to produce $50\text{--}60 \text{ t ha}^{-1}$ of fruits is 430 kg N ha^{-1} [23]. Therefore, rates equivalent to 0.5, 1.0 and 2 times of the mentioned N rate were applied. For first growing season, sludge rates were equal to $T_{L1} = 219 \text{ kg ha}^{-1}$ N; $T_{L2} = 400 \text{ kg ha}^{-1}$ N and $T_{L3} = 870 \text{ kg ha}^{-1}$ N (Table 2). For the subsequent two growing seasons the nitrogen rate were reduced by half (i.e., $T_{L1} = 100 \text{ kg ha}^{-1}$ N; $T_{L2} = 200 \text{ kg ha}^{-1}$ N and $T_{L3} = 400 \text{ kg ha}^{-1}$ N) in all treatments (Table 2). This modification on sludge rate was necessary because, in an unexpected way, fruit production reached $50\text{--}60 \text{ t ha}^{-1}$ in the first growing season. As the nitrogen mineralization rate of the biosolid was determined as 30% we supposed the full response of fruit yield would occur only in the second or third season, when most of the N was mineralized. Therefore, facing to the high productivity in the first cropping season and to prevent plant toxicity or environmental concerns we adopted a reduced rate of the sludge. This occurred because for normal agricultural practices in São Paulo State, the recommendation for N application is based on expected productivity.

Sewage sludge rate (SS_R) was calculated considering the amount of N required by the plants (N_{RP}), the content N available on the residue (SS_N) (Table 1) by using (1).

$$\text{SS}_R, \text{kg ha}^{-1} = \text{N}_{RP} \times \text{SS}_N. \quad (1)$$

The nitrogen mineralization rate (N_{MR}) was determined in a laboratorial trial (data not shown) and was equal to 30% of total N. For planting (2) and growing seasons 2 and 3 (3) amendments two different equations to estimate SS_N were used:

$$\text{SS}_N = \left(\frac{\text{N}_{MR}}{100} \right) \times (\text{Kjeldahl-N} - \text{NH}_3\text{-N}) \quad (2)$$

$$\begin{aligned} \text{SS}_N = & \left(\frac{\text{N}_{MR}}{100} \right) \times (\text{Kjeldahl-N} - \text{NH}_3\text{-N}) \\ & + 0.5 \times [\text{NH}_3\text{-N} + \text{NO}_3^- \text{-N} + \text{NO}_2^- \text{-N}], \end{aligned} \quad (3)$$

where Kjeldahl-N = total nitrogen (mg kg^{-1}); NH_4^+ -N = nitrogen as ammonia (mg kg^{-1}); NO_3^- -N = nitrogen as nitrate (mg kg^{-1}); NO_2^- -N = nitrogen as nitrite (mg kg^{-1}).

This was necessary because in 2002 and in 2003, biosolid was manually side-dressed without incorporation into the soil, over a one meter-wide strip alongside the plant rows.

2.3. Orchard Planting, Management and Sampling. Experiment consisted of plots with 100 m^2 ($10 \times 10 \text{ m}$) distributed in a complete randomized design, with five replicates ($n = 5$). Twenty banana plantlets per plot were planted ($2.5 \times 2.0 \text{ m}$ spacing) by using *in vitro* micropropagated Grand Naine cultivar (AAA group, Cavendish subgroup) material. By the time of the orchard establishment the plantlets presented, in average ($n = 50$), 4.4 leaves and 0.221 m height.

In 2001, biosolid was applied into the furrows, between the planting holes, and carefully incorporated to the soil. Fertilizer phosphorus was applied in all plots, twice during

TABLE 1: Chemical composition of the municipal biosolid applied to a banana plantation in the city of Pariquera-Açu as source of nitrogen over three crop seasons.

Parameter	Unit	Growing season			Parameter	Unit	Growing season		
		2001	2002	2003			2001	2002	2003
P	g kg ⁻¹ *	15.4	11.6	8.7	OC	g kg ⁻¹ *	222.7	302.8	172.4
K	g kg ⁻¹ *	0.9	0.8	1.0	pH	% (m/m)	9.9	12.7	12.6
Na	g kg ⁻¹ *	0.6	0.8	0.4	Humidity	% (m/m)*	71.2	64.3	62.9
As	mg kg ⁻¹ *	<0.1	<0.1	<0.1	Volatile solids	% (m/m)*	52.5	43.9	35.1
Cd	mg kg ⁻¹ *	10.3	1.2	0.5	Kjeldahl-N	g kg ⁻¹ *	32.0	31.1	21.5
Pb	mg kg ⁻¹ *	83.6	115.6	21.0	NH ₄ -N	mg kg ⁻¹ **	1941.5	806.7	591
Cu	mg kg ⁻¹ *	139.6	115.6	67.2	(NO ₃ + NO ₂)-N	mg kg ⁻¹ **	47.1	42.0	33.5
Cr	mg kg ⁻¹ *	43.8	19.3	14.5	S	g kg ⁻¹ *	7.7	7.0	6.74
Hg	mg kg ⁻¹ *	ND	ND	ND	Mn	mg kg ⁻¹ *	778.5	539.4	342.8
Mo	mg kg ⁻¹ *	ND	ND	ND	Fe	g kg ⁻¹ *	106.4	50.9	54.7
Ni	mg kg ⁻¹ *	22.2	12.7	9.9	Mg	g kg ⁻¹ *	5.9	4.8	3.8
Se	mg kg ⁻¹ *	ND	ND	ND	Al	g kg ⁻¹ *	7.0	6.7	5.5
Zn	mg kg ⁻¹ *	573.4	449.8	281.6	Ca	g kg ⁻¹ *	173.6	359.6	193.7
B	mg kg ⁻¹ *	22.9	13.9	7.4	Available-N	g kg ⁻¹ **	2.96	3.41	2.32

* On dry matter basis; ** determined on biosolid with natural moisture content.

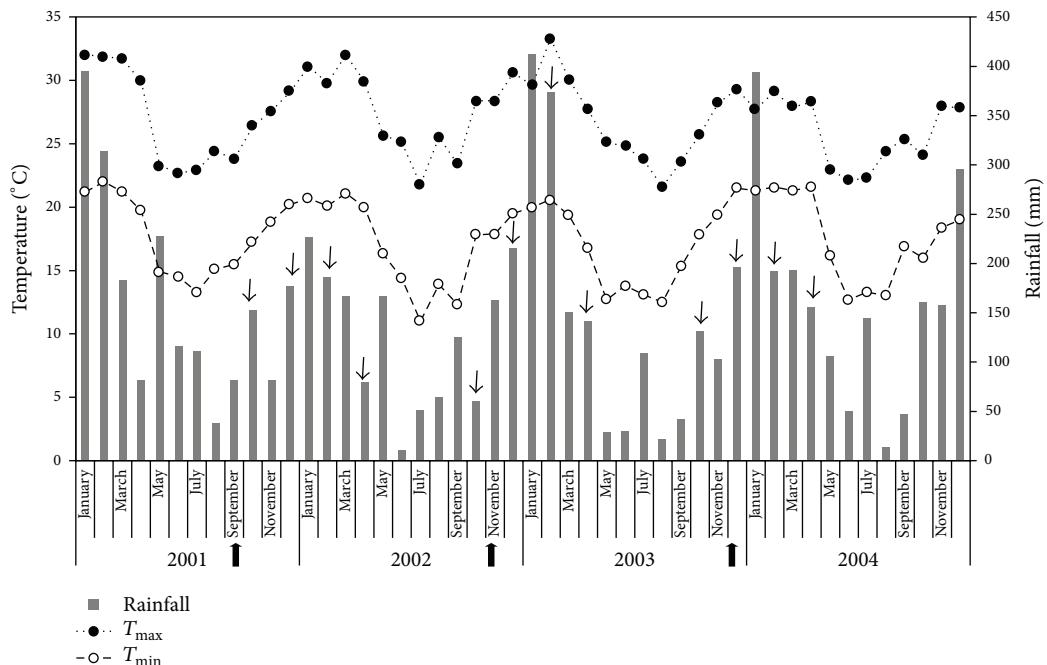


FIGURE 1: Climatic parameters over the period of 2001–2004 in the city of Pariquera-Açu at São Paulo State, Brazil. Upward fulfilled black arrows indicate the sludge application dates. Mineral fertilizer application was split in four events (25% each) within the same crop season and was represented by the downward arrows.

the trial: 150 kg ha⁻¹ of P₂O₅, into the furrows by the planting (in 2001) and 200 kg ha⁻¹ of P₂O₅ was broadcasted on soil surface in 2003. The source of P was phosphate (41% P₂O₅). Potassium was yearly applied at a rate of 342 kg ha⁻¹ year⁻¹ of K as KCl, and nitrogen as NH₄NO₃ (32% N) according to each treatment (Table 2). Fertilization with potassium

and nitrogen was split in four events during the crop cycle (Figure 1).

Due to its own biological characteristics, as banana plant grows new suckers from its rhizome comes out. The management of the plants consisted of pruning and keeping a clump formed by three plants (parent plant, first and second

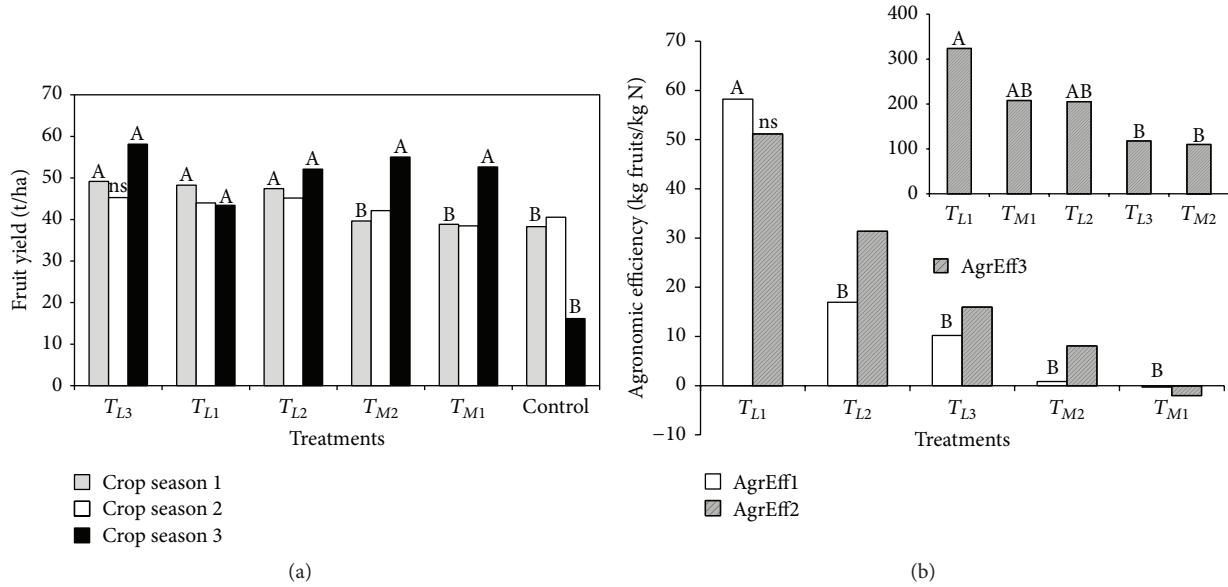


FIGURE 2: Biosolid and mineral fertilizer application on fruit yield (a) and agronomic efficiency index (b). Columns, within the same crop season, labelled with different letters on top are significantly different ($P < 0.05$). ns = not significant ($P > 0.05$). In (b) observe the difference of scale on y -axis. T_{L1} , T_{L2} , and T_{L3} correspond to the equivalent amount of approximately 100, 200, and 400 kg ha^{-1} year $^{-1}$ of N applied as SS. T_{M1} and T_{M2} were equivalent to the application of 200 and 400 kg ha^{-1} year $^{-1}$ of N as NPK mineral fertilizer.

suckers). Only the parental plant of each plot was used to record fruit yield, plant height, pseudo-stem diameter and to collect leaf samples. Soil sampling was done yearly, after the fruit harvesting, in 2002, 2003, and 2004. In 2002 and 2003 soil samples were taken from depths of 0–0.2, 0.2–0.4, 0.4–0.6 m. In 2004 the layer of 0.60–0.80 m was also collected. Fresh soil samples were analyzed for $\text{N}-\text{NO}_3^-$ and $\text{N}-\text{NH}_4^+$ after extraction with 2 mol L^{-1} KCl solution according to Raij et al. [20]. Fertility parameters were evaluated in 2 mm sieved air-dried soil samples and processed according Raij et al. [20].

To evaluate the effect of sludge and fertilizer applications on soil organic matter variations from the initial condition, before the application of sludge or fertilizer, were calculated every year as $\Delta \text{SOM} = \text{SOM}_{\text{year}} - \text{SOM}_{\text{initial}}$. Thus, a positive ΔSOM indicates accumulation of carbon for a specific treatment, depth and time.

Plant nutrition was evaluated by using the chemical leaf tissue analysis [24] prepared according to procedures described by Bataglia et al. [25].

The agronomic efficiency index (AgrEff) was calculated according to (4):

$$\text{AgrEff} = \frac{(Y_{\text{fert}} - Y_{\text{unfert}})}{Q_{\text{NA}}}, \quad (4)$$

where AgrEff = agronomic efficiency index in kg fruits kg^{-1} of N applied; Y_{fert} = Production of harvested biomass (Fruits + peduncle) with N, kg ha^{-1} ; Y_{unfert} = Yield without N, kg ha^{-1} (Control); Q_{NA} = Amount of N applied, kg ha^{-1} .

Pseudo-stem diameter was measured at the height of 0.30 m from the soil surface and the plant height was measured from soil surface to the insertion point of youngest leaf. In addition, the leaf endurance index (LEI), which is

the number of active leaves at the harvesting divided by the number of active leaves at bunch emission, was calculated. “LEI” represents the maintenance of active leaves by plants and the practical experience of the researches involved in this paper has showed that there was a direct and positive relationship between LEI and yield.

2.4. Statistical Calculations and Data Analysis. Data were analyzed using analysis of variance (ANOVA) for randomized complete block designs to test the effect of treatments. Means were compared by using Tukey’s HSD test. All calculations considered the level of significance of $P = 0.05$ and were performed in Minitab v.16.

3. Results and Discussion

As said at the beginning, this paper continues to explore the experiment previously reported by Teixeira et al. [18]. Also, data of first fruit yield became available after the publication of the soil parameters [18], allowing to consider that these were partially resulted from the sludge application in 2001. In this way, some information about the soil from the first growing season already presented by Teixeira et al. [18] is been showed again to facilitate discussions.

3.1. Fruit Productivity and Agronomic Efficiency. In the first growing season, treatments that received sludge produced higher amounts of fruits (Figure 2(a)). Unlikely, plants cultivated with the similar N rate, but applied as mineral fertilizer (T_{M2}), did not respond in the same way (Figure 2(a)). Besides the addition of sludge increases the availability of nitrogen and phosphorus other factors should be improving the biomass production.

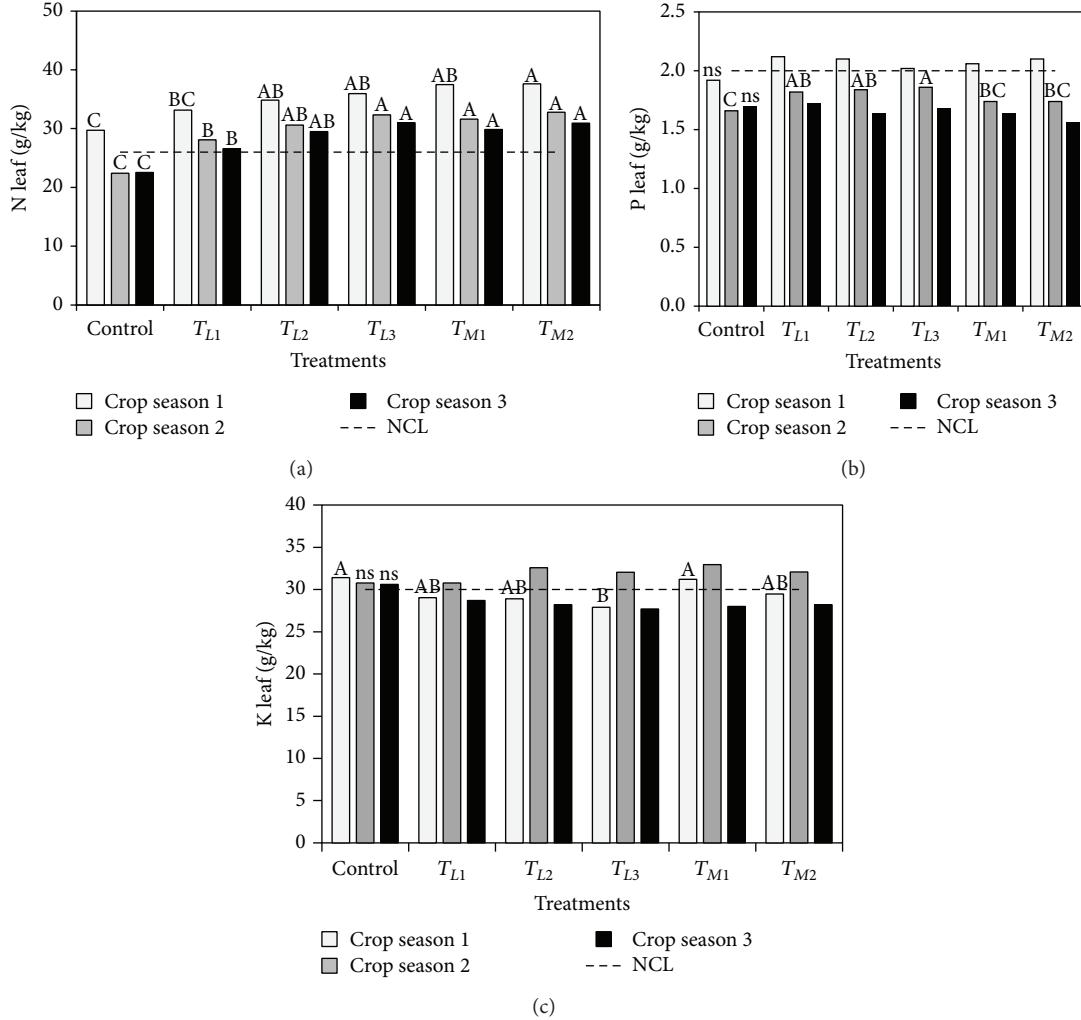


FIGURE 3: Nitrogen (a), phosphorus (b), and potassium (c) concentrations in banana leaves with sewage-sludge (T_L) and mineral fertilizer (T_M). Columns with the same letter on top, within crop season, indicate that the mean does not differ by Tukey's HSD test ($P > 0.05$). ns = ANOVA F test is not significant for treatments effects ($P > 0.05$). Dashed line represents the nutrient critical level (NCL) according to Raij et al. [37].

The precise contribution of sludge is difficult to measure comparing the yield from Control to amended treatments. Control also received P and K; thus part of the variation observed was caused by the supply of N. Besides, yield in first two growing seasons did not differentiated among treatments with biosolid rates.

The highest yield obtained with sludge amendment can be due to double effect of the organic material supplying nutrients and improving soil physical parameters as well [4, 5]. Unfortunately, soil physical properties were not measured in this work and more considerations cannot be made.

Absence of plant response in mineral fertilized treatments in comparison to the Control can be partially input to natural soil fertility. Although N supply is used as indicator to sludge rate application, there was no agreement about soil N evaluation. More about soil N is shown in Section 3.2 of this paper.

Fruit production in the Control treatment without N at similar levels to plants amended with mineral fertilizer (T_{M1} and T_{M2}) reinforces this statement.

The data from the third growing season (AgrEff3) were considered without influence of the natural soil fertility and analyzed with more detail (Figure 2(b)). It reflected the data of fruit yield but showed that the application of the smallest rate of sludge (T_{L1}) was the most effective then the mineral amendment. This reinforces the observation that biosolid is not a pure source of nutrients but also affects other soil parameters resulting in a more efficient use of them.

Furthermore, in general, biosolid increased the AgrEff within growing seasons, but the ratio of AgrEff between biosolid to fertilizer ($\text{AgEff}_{T_L}/\text{AgEff}_{T_M}$) diminished from 103.5 (growing season 1) to 10.77 (growing season 2) and 1.36 (growing season 3). Probably the biosolid re-application is increasing the amount of N potentially mineralizable and therefore, reducing the proportion of fruits per kilogram of applied N as postulated by others [11, 26].

3.2. Plant Nutrition. Foliar N and P contents agreed with the yield data (Figure 3). For the three crop cycles the N concentrations for Control treatment were lower than the observed

TABLE 2: Description of treatment and additional information.

Treatment	Fertilization	Biosolid rate t ha ⁻¹ (dry base)	Nitrogen kg ha ⁻¹
2001			
Control	No N + mineral fertilization (P + K)	—	—
T_{L1}	Biosolid rate 1 + K	21.3	219
T_{L2}	Biosolid rate 2 + K	42.6	435
T_{L3}	Biosolid rate 3 + K	85.2	870
T_{M1}	Mineral N (rate 1) + P + K	—	400
T_{M2}	Mineral N (rate 2) + P + K	—	800
2002			
Control	No N + mineral fertilization (P + K)	—	—
T_{L1}	Biosolid rate 1 + K	10.7	100
T_{L2}	Biosolid rate 2 + K	21.4	200
T_{L3}	Biosolid rate 3 + K	42.8	400
T_{M1}	Mineral N (rate 1) + P + K	—	200
T_{M2}	Mineral N (rate 2) + P + K	—	400
2003			
Control	No N + mineral fertilization (P + K)	—	—
T_{L1}	Biosolid rate 1 + K	13.9	100
T_{L2}	Biosolid rate 2 + K	27.8	200
T_{L3}	Biosolid rate 3 + K	55.5	400
T_{M1}	Mineral N (rate 1) + P + K	—	200
T_{M2}	Mineral N (rate 2) + P + K	—	400

in T_L and T_M treatments. In terms of foliar phosphorus concentration, biosolid presented similar effectiveness than mineral fertilizer amendments (Figure 3(b)) although no difference between T_{L1} and T_{L3} rates were observed.

These results agree with the observed for other species in which a significant amount of P was released by the biosolid [2, 27–32]. Also it is important consider that the establishment of biosolid application rate based on N availability, in this case, resulted in high amounts of P being added to soil ranging from 1278 kg ha⁻¹ (T_{L3} —in 2001) to 121 kg ha⁻¹ (T_{L1} —in 2003).

Statistical significant effect of treatments on potassium foliar concentrations was found only in the data of the first growing season (Figure 3(c)). Some can highlight the difference of K content between T_{L3} and T_{M1} but is important to observe that the treatment T_{M2} presented similar values. Because potassium was applied to all treatments and as there was no consistent alterations of foliar concentration with biosolid rates increase, further considerations about this nutrient are difficult to be made.

These results support the conclusion of Teixeira et al. [18] about the capacity of the biosolid to supply N to banana and also P. These results are important because practically there were no others references about the usage of biosolid as source of plant nutrients in banana orchards. More studies considering fruit quality parameters as well are encouraged.

TABLE 3: Leaf endurance index, N/K relationship in the shoots, plant height, and banana stem diameter due to biosolid or mineral fertilizer application.

Treatment	LEI, %	Foliar N/K	Plant height, m	Stem diameter, m
2002				
Control	39 ab	0.95 b	2.21 abc	0.19 ab
T_{L1}	33 bc	1.14 a	2.29 a	0.21 a
T_{L2}	32 cd	1.20 a	2.26 ab	0.20 a
T_{L3}	26 d	1.29 a	2.26 ab	0.21 ab
T_{M1}	39 ab	1.20 a	2.16 bc	0.19 b
T_{M2}	40 a	1.28 a	2.10 c	0.19 b
$P_{F5,4}$	<0.0001	<0.0001	<0.0001	<0.0001
2003				
Control	49 a	0.74 b	2.42 b	0.23 ab
T_{L1}	37 b	0.91 a	2.60 ab	0.23 ab
T_{L2}	34 bc	0.95 a	2.63 ab	0.23 ab
T_{L3}	28 c	1.02 a	2.71 a	0.23 a
T_{M1}	39 b	0.96 a	2.48 ab	0.21 b
T_{M2}	38 b	1.03 a	2.59 ab	0.21 ab
$P_{F5,4}$	<0.0001	<0.0001	0.023	0.012
2004				
Control	63	0.74 c	2.74	0.25
T_{L1}	61	0.92 b	2.96	0.26
T_{L2}	59	1.05 ab	2.95	0.26
T_{L3}	65	1.12 a	2.92	0.26
T_{M1}	67	1.07 ab	2.83	0.24
T_{M2}	62	1.10 a	2.89	0.25
$P_{F5,4}$	0.105	<0.0001	0.44	0.092

LEI: leaf endurance index; $P_{F5,4}$: significance of F test for treatment effects at 5 degrees of freedom (df) for treatments and 4 df for error; averages followed by same letters within crop cycles on columns do not differ to Tukey HSD test at $P = 0.05$.

Besides being effective, the application of 20 t ha⁻¹ sludge (dry basis) as source of N resulted in foliar N concentrations not statistically different ($P > 0.05$) to that with 400 kg ha⁻¹ N from fertilizer (Figure 3(a)).

Under Brazilian soil and climate conditions different plant species already have shown that the biosolid was efficient to sustain the crop yield at similar levels than the observed with purely mineral fertilizer treated plots [1, 7, 27, 31, 33–36]. The application of biosolid to sugarcane cropped soils as source of N and P supplied 100% of N and 35% of the P requirements on these nutrients [1]. Ryegrass cultivated on soils treated with different types of SS also recovered approximately 36–75% of the N and 4–7% of the P supplied [7]. This suggests that biosolid is an effective source of N to *Musa* spp. plants.

3.3. Leaf Endurance Index. There was no statistical significant difference on leaf endurance index, plant height, and stem diameter within treatments for the third growing season (Table 3).

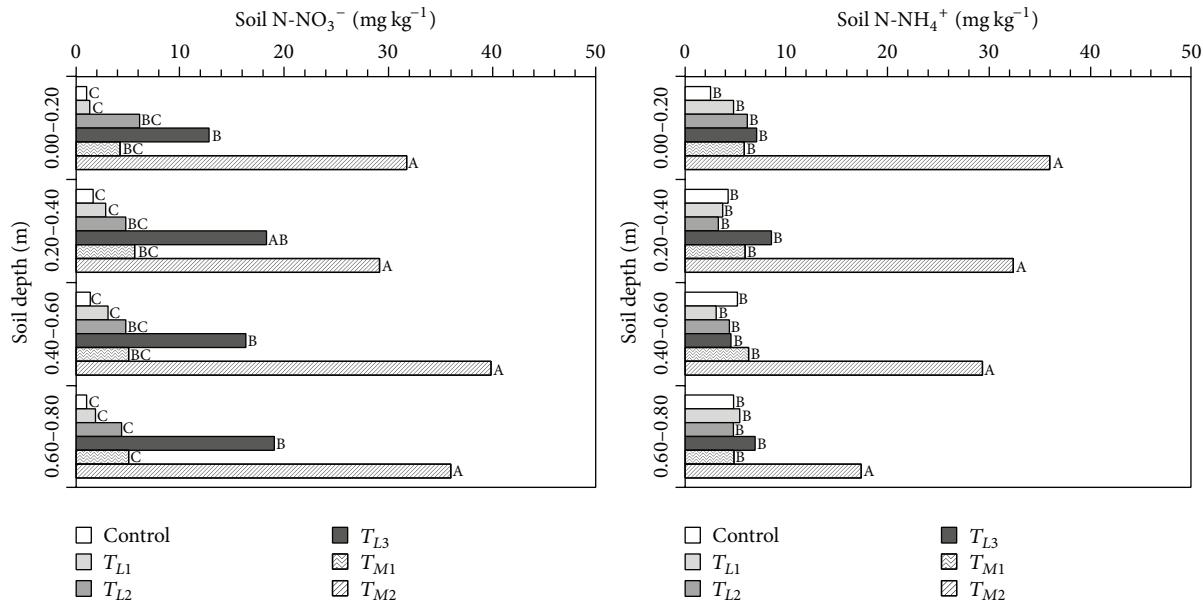


FIGURE 4: Soil nitrate (N-NO_3^-) and ammonium (N-NH_4^+) as effect of biosolid (T_{L1} , 100; T_{L2} , 200; and T_{L3} , 400 kg ha^{-1} N) and mineral fertilizer (T_{M1} , 200 and T_{M2} , 400 kg ha^{-1} N). Means followed by same letters, within depths, do not differ statistically by Tukey's test ($P > 0.05$).

Leaf endurance index (LEI) showed to be more sensitive to detect differences between biosolid and mineral fertilizer than height and diameter. For the same N rate addition, biosolid resulted in a LEI decrease of 6% (T_{L2} versus T_{M1}) and 4% (T_{L3} versus T_{M2}) in the first season. The same comparison, made with second season data, showed biosolid depressive effects on LEI of 5% (T_{L2} versus T_{M1}) and 10% (T_{L3} versus T_{M2}) (Table 3). Although blamed to be an important index to banana cropping management, LEI did not present relationship to fruit yield as the differences between T_{L2} versus T_{M1} and T_{L3} versus T_{M2} were favorable to biosolid addition in the first growing season (Figure 2). Biological relevance of differences of these magnitudes should be better investigated in future.

In general, the adequate supply of N tends to maximize banana growth [37]. There are some indications that the maintenance of the foliar N/K relationship around 1.5 is needed to keep banana plants in an adequate number of photosynthetically active leaves. In 2004 foliar N/K relationship for T_{L3} and T_{M2} were the highest (Table 3). Since K was supplied in a constant rate to all treatments, biosolid should supply N in order to keep the N/K relationship favorable to plants. This is supported in part, by the higher AgrEff 3 (Figure 2(b)).

3.4. Soil Nitrogen, Organic Carbon, and Available Phosphorus. Soil inorganic N was evaluated only in 2004, three years after the initial application of biosolid and mineral fertilizer. Effects on soil N content were observed in all soil layers (Figure 4). Only the highest rate of biosolid (T_{L3}) and all mineral fertilizer levels increased N-nitrate concentration in comparison to the control (Figure 4).

For the same amount of N, mineral fertilizer (T_{M2}) always resulted in higher concentrations of nitrate and ammonium than the biosolid (T_{L3}) at the same depth. This is a very

common observation as a high percentage of the N applied in the sludge is bounded in organic matter and has to be mineralized while mineral fertilizers rapidly became available forms.

There was no statistically significant difference between T_{L2} and T_{M1} for nitrate and ammonium at all depths. These were the treatments representing the reference values of N application for banana in Brazil.

These results were in agreement to the related to fruit productivity. Part of the yield variations can be coupled to soil N because it is one of the most important elements of banana plant nutrition [2, 28, 29, 31, 33, 38, 39]. Thus, the more available the N is, the more the plant grows and produces biomass and fruits. In a different perspective, the application of 400 kg ha^{-1} N as fertilizer (T_{M2}) is more potentially dangerous than the same amount as biosolid (T_{L3}) in a single growing season due to the increase of soil N-NO₃ and N-NH₄ concentration. The most interesting fact is that the application of half of the N rate as biosolid (T_{L1}) had practically no effect on soil inorganic N accumulation but resulted in the more agronomic effective practice (Figure 2(b)).

The soil organic carbon showed a distinct pattern (Figure 5). In the first year, there was an increment on soil C content with biosolid application at 0–20 cm layer (Figure 5).

For the same treatments, in the second year the content of SOC diminished only to rise again in the third growing season. In 2003, even the treatments with mineral fertilizer or the control showed an increase in SOC until the depth of 60 cm (Figure 5). This was the result of plant growth, mainly root system, and the recycle of biomass from the stems cut after the fruit production.

It was hypothesized that the addition of N by biosolid stimulated the microorganism to degrade the organic matter

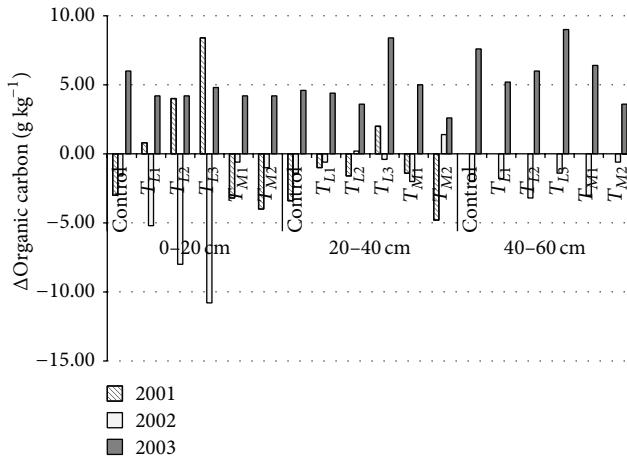


FIGURE 5: Soil organic carbon as effect of biosolid (T_L treatments) and mineral fertilizer (T_M treatments) rates at different depths and years.

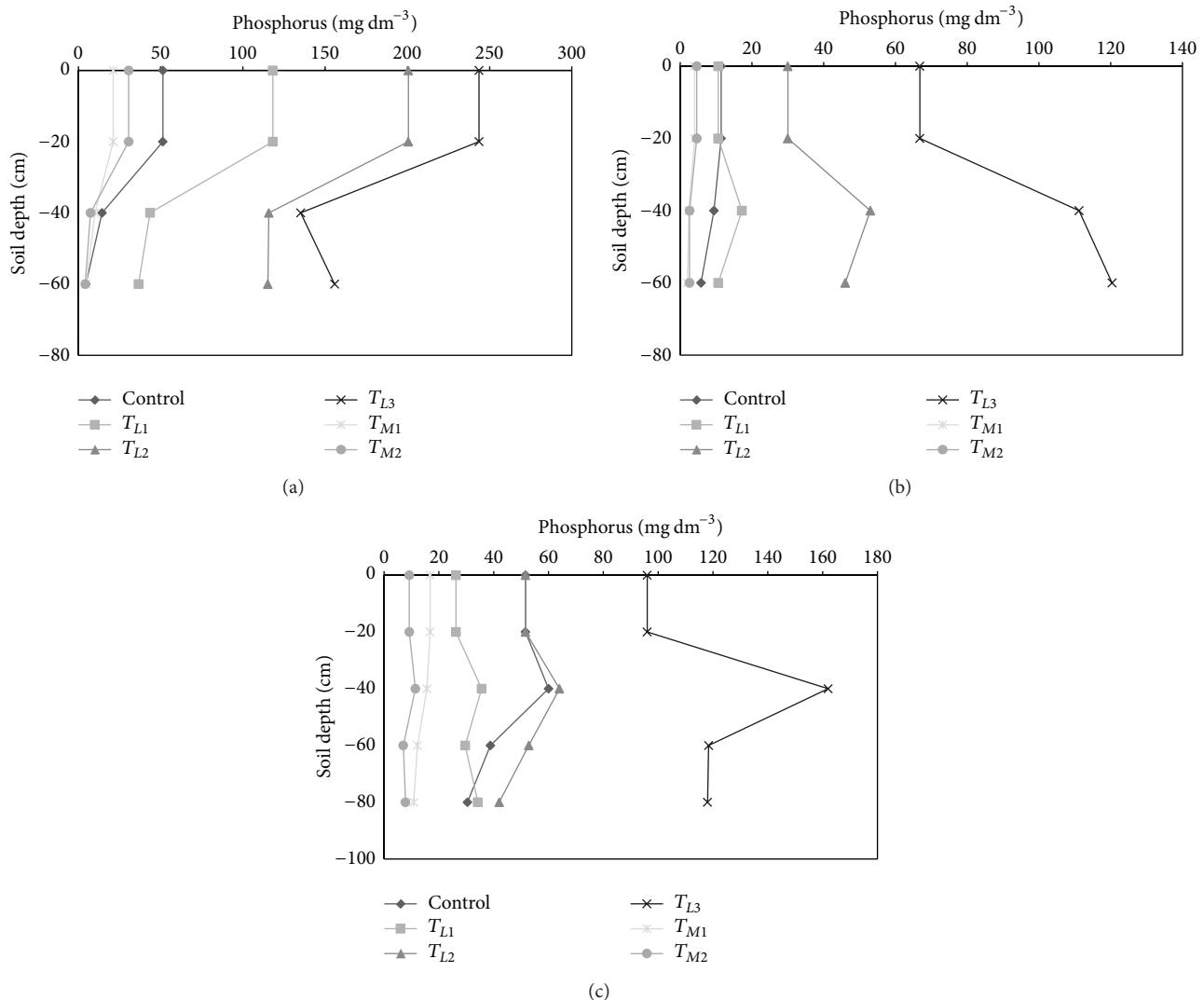


FIGURE 6: Available phosphorus in different growing seasons and depths as due to the application of biosolid and mineral fertilizer rates in soil cropped with banana. (a) Growing season 2001/2002; (b) growing season 2002/2003; (c) growing season 2003/2004.

preexisting into the soil in the second growing season. After this a new equilibrium was established resulting in the accumulation of new forms of carbon.

The two higher rates of biosolid (T_{L2} and T_{L3}) resulted in the higher concentrations of available phosphorus (Figure 6). For different species, others have reported that the biosolid can supply P [7, 40]. Chiba et al. [40] reported that for sugarcane, biosolid addition based on N release can supply approximately 35% of the P plant requirement in one growing season.

The soil profile distribution of available P needs to be more investigated. As can be seen, at T_{L2} and T_{L3} more P were available at deeper layers (Figure 5). Brazilian soils are normally rich in iron and aluminum oxides, which result in a high P retention capacity. In growing seasons 2 and 3, biosolid was spread in the soil surface and the mechanism that leads to the increase of P content at 60 to 80 cm is still unclear.

4. Conclusions

Biosolid is an affordable source of N and P for banana plants; biosolid as source of N to banana plants does not cause depressive effects on biomass production compared to mineral fertilization; the agronomic efficiency index is a reliable tool to describe the response of banana to biosolid application.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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Research Article

Influence of NaCl-Induced Salinity and Cd Toxicity on Respiration Activity and Cd Availability to Barley Plants in Farmyard Manure-Amended Soil

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The objective of this study was to evaluate the Cd availability and toxicity as affected by NaCl-induced salinity and farmyard manure addition. The Cd availability and toxicity were investigated in greenhouse pot and incubation experiments were conducted on a calcareous loamy sand soil contaminated with Cd (0.5, 1.5, 3, 6, 12, and 24 mg kg⁻¹ of soil) and amended with two rates of 0.0 and 30 g farmyard manure (FYM) kg⁻¹. Barley seeds (*Hordeum vulgare* L.) were sown in pots and irrigated with water containing different levels of salinity (0, 30, 60, and 120 mM NaCl). The results revealed that the DTPA-extractable Cd and its content in barley plant shoots tended to increase in line as Cd was applied and salt levels increased. Elevated decreases in the soil basal respiration with increased Cd applied and NaCl-induced salinity were found. However, applying FYM significantly reduced Cd availability and increased plant growth and soil respiration activity. The results clearly showed that adding farmyard manure as soil organic amendment decreased the availability of Cd to barley plants and mitigated the toxicity of both Cd and salinity to soil microbial activity.

1. Introduction

Cadmium (Cd) is one of the most dangerous heavy metals due to its high mobility and phytoavailability [1, 2]. Due to its high accumulation in the tissues of plants and animals, the Cd toxicity affects adversely human health. Moreover, Cd may have a negative effect on soil microbial activities altering soil nutrient cycle and subsequently resulting in changes in soil fertility [1, 3]. The bioavailability of heavy metals in soils depends on numerous factors such as pH, CaCO₃, clay content, organic matter nature and content, redox potential, and soil solution composition [2, 4, 5].

Soils and irrigation water of arid regions are characterized by high salinity levels, which may aggravate heavy metal pollution problems. In arid regions, irrigation water containing high levels of chloride ions might increase soil heavy metal mobilization through the formation of metal-chloride complexes [6, 7]. It has been reported that the consequences of soil salinity can increase or decrease heavy metal solubility

and availability, mainly depending on the composition of soil solution and its dominant inorganic ligands [6–8].

Application of organic amendments to soil has been shown to improve soil fertility and alleviate environmental stresses in soils, resulting in enhancing plant growth. Specifically, the addition of organic amendments to soils can result in situ immobilization of heavy metals and subsequently reduce their bioavailability [8–10]. Ok et al. [9] reported that the addition of organic residues to heavy metal-contaminated soils increased soil organic matter content, improved the chemical and biological properties, and decreased the heavy metal phytoavailability. The effect of organic amendments on metal availability to plants corresponds to several factors including their influences on biochemical soil properties [9–11]. Recently, it has been reported that the application of organic manure to heavy metal-contaminated soil decreased the metal toxicity and subsequently increased soil microbial activities [12]. To our knowledge, however, there is no available information on the role of organic amendments in terms

of their contribution to decrease metal phytotoxicity under saline conditions. Therefore, the objective of the present work was to study the influence of salinity and Cd toxicity on Cd availability to barley plants and soil respiration in farmyard manure-amended calcareous loamy sand soil.

2. Materials and Methods

2.1. Soil Samples and Characterization. The surface layer (0–30 cm) of agricultural loamy sand calcareous soils was collected from Assiut University Experimental Farm, Assiut, Egypt. All samples were air-dried and sieved by using a 2 mm mesh prior to soil characterization and experiments setup. The particle size distribution of soil samples was determined by the pipet method [13]. Soil chemical properties were measured according to standard methods [14]. Soil pH was measured using a digital pH meter in a saturation paste. The EC values were measured in paste extracts using a digital EC meter. Calcium carbonate content was determined using the calcimeter method. Soil organic matter (OM) was determined by a modified Walkley and Black method [15]. The particle size distribution was measured by the hydrometer method. The total Cd in the soil was determined using a GBC model 906 atomic absorption spectrophotometer (AAS) after conventional aqua regia digestion method [16]. The particle size distribution was 800 g sand kg⁻¹, 147 g silt kg⁻¹, and 53 g clay kg⁻¹, indicating a texture of loamy sand. The pH of the soil was 7.82 and the electrical conductivity was 3.64 dS m⁻¹. The organic C and CaCO₃ contents were 0.21% and 9.30%, respectively. The total soil content of Cd was 0.13 mg kg⁻¹. The pH and organic matter content of applied farmyard manure were 7.15 and 32.60%, respectively.

2.2. Pot Experiment. A greenhouse pot experiment was carried out to investigate the interactive effects of NaCl-induced salinity and farmyard manure (FYM) on soil Cd availability and its uptake by barley plants (*Hordeum vulgare* L.) grown on sandy calcareous soils. A greenhouse experiment with six levels of Cd (0.5, 1.5, 3, 6, 12, and 24 mg kg⁻¹ of soil, in the form of CdCl₂), two rates of farmyard manure (0.0 and 30 g FYM kg⁻¹), and four salinity levels was conducted. The four salinity levels were prepared by using deionized water with salinity of 0 (S0), 30 (S1), 60 (S2), and 120 (S3) mM NaCl. Plastic pots were filled with 3 kg of the artificially Cd-contaminated sandy calcareous soil samples treated and untreated with FYM. Ten seeds of barley plants were planted in each pot. The plants were thinned to five barley plants per pot after germination. The pots were irrigated with the water containing different salinity levels. Overall, in experiment period, all the pots were maintained at field capacity moisture content by weight. NPK basal fertilization was used with the first irrigation. The experiment was designed using a randomized complete block design with three replications.

2.3. Soil and Plant Analysis. At the end of the experiment, soil pH was measured using a digital pH meter in a saturation paste. Electrical conductivity (EC_e) and concentrations of

Ca⁺⁺, Mg⁺⁺, Na⁺, K⁺, SO₄⁻², HCO₃⁻, and Cl⁻ were measured in the paste extracts according to standard methods of Sparks [14]. The concentrations of Na and K were analyzed using a flame photometer. The concentrations of Ca⁺⁺, Mg⁺⁺, Cl⁻, and HCO₃⁻ were determined by titration. Sulfate (SO₄⁻²) was determined using the turbidimetric method. In order to determine the available fraction of soil Cd, the soil samples were extracted by DTPA (diethylene triamine penta acetic acid) according to Norvell et al. [17]. The suspension was then shaken and centrifuged. The supernatant was filtered through Whatman No. 42 filter paper. Cd concentrations in the filtered solutions were analyzed by atomic absorption spectrophotometer (AAS).

Barley plants were harvested after 50 days of planting. To determine dry shoots weight, shoot samples of barley plants were oven-dried at 70°C. Dried shoots were ground and digested using a 2:1 HNO₃ : HClO₄ acid mixture. The shoot Cd concentrations were analyzed by AAS.

2.4. Incubation Experiment. At the end of the greenhouse pot experiment, the soil samples of each pot were collected and used to investigate the interactive effects of NaCl-induced salinity and Cd toxicity on soil basal respiration in farmyard manure-amended sandy calcareous soil. This experiment was conducted with the soil samples which were treated with two levels of Cd (0.5 and 24 mg kg⁻¹ of soil) and amended with two rates of 0.0 and 30 g FYM kg⁻¹ and different salinity levels of 0 (S0), 30 (S1), 60 (S2), and 120 (S3) mM NaCl.

The soil mixtures (100 g) were put in glass vessels (250 mL). Deionized water was added to each soil mixture to bring it to 70% of field capacity. Each treatment was replicated three times. Small vials with 10 mL of 1 M NaOH solution were placed in vessels to trap CO₂. After the addition of NaOH, the vessels were closed air tight and incubated for 28 days at 30°C. The NaOH solutions in the vials were changed after 1, 3, 7, 14, 21, and 28 days. CO₂ evolved during the incubation periods was trapped in 1 M NaOH and the excess NaOH was titrated with 0.1 M HCl following the BaCl₂ addition [18]. The soil respiration was calculated as CO₂-C rate (mg C g⁻¹ soil day⁻¹).

2.5. Statistical Analysis. The mean values and standard deviation (\pm SD) of three replicates are calculated. The analysis of variance was performed using the statistical computer program of StatSoft [19].

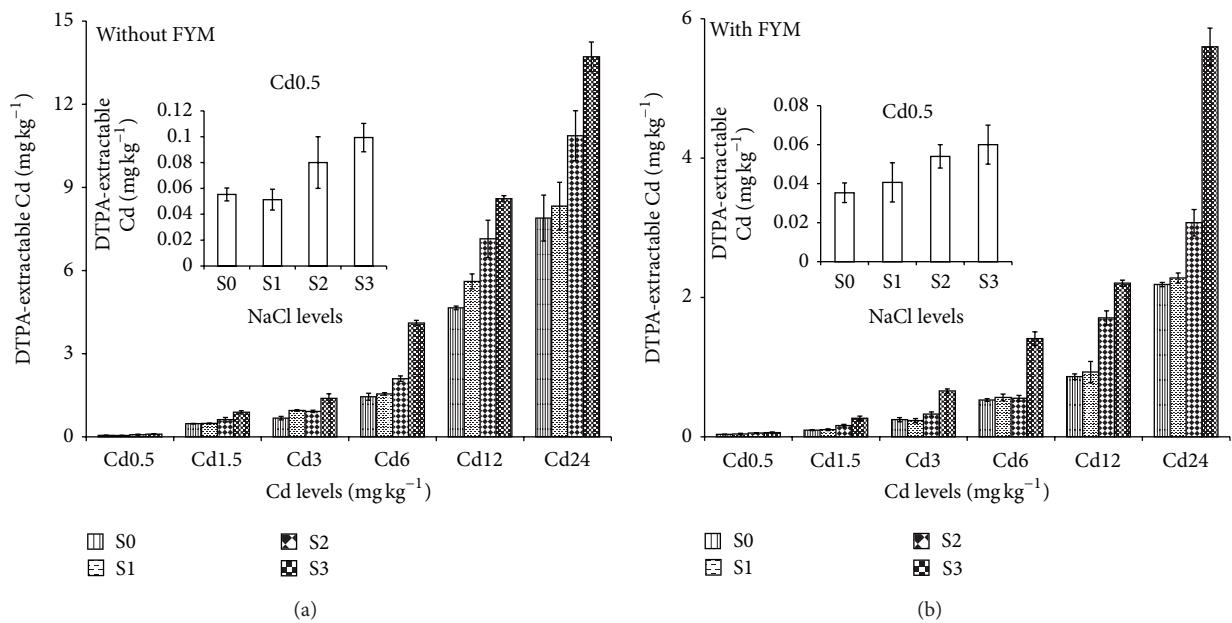
3. Results

3.1. Treatment Effects on Soil pH, EC, and Soluble Cations and Anions. Soil pH decreased slightly in the soils irrigated with saline water, especially with increasing NaCl salt levels (Table 1). As expected, the values of EC_e were significantly increased with increasing NaCl levels as well as in the presence of FYM. Na⁺ and Cl⁻ concentrations increased in line as salt levels increased in NaCl-treated soils. It was generally observed that the irrigation with saline water caused significant increases in soluble K⁺. The results clearly showed also that the addition of

TABLE 1: Treatment effects on pH, EC_e, and soluble cations and anions (\pm SD).

NaCl levels	pH	EC _e (dS m ⁻¹)	Ca ⁺⁺	Cations (mmol (+) L ⁻¹)			Anions (mmol (-) L ⁻¹)		
				Mg ⁺⁺	Na ⁺	K ⁺	Cl ⁻	SO ₄ ⁻²	HCO ₃ ⁻
Without FYM									
S0	7.86 \pm 0.05	3.69 \pm 0.05	9.01 \pm 0.72	4.02 \pm 0.40	9.11 \pm 1.18	0.39 \pm 0.04	17.4 \pm 0.83	8.66 \pm 0.62	1.62 \pm 0.10
S1	7.83 \pm 0.04	4.19 \pm 0.05	9.08 \pm 0.64	4.01 \pm 0.51	13.2 \pm 1.10	0.36 \pm 0.03	21.5 \pm 1.08	8.59 \pm 0.89	1.61 \pm 0.08
S2	7.80 \pm 0.03	4.95 \pm 0.10	9.06 \pm 0.84	4.19 \pm 0.49	21.3 \pm 1.42	0.44 \pm 0.01	29.5 \pm 1.94	8.92 \pm 0.86	1.66 \pm 0.06
S3	7.77 \pm 0.05	6.29 \pm 0.05	9.14 \pm 0.70	4.36 \pm 0.52	34.4 \pm 1.55	0.52 \pm 0.02	43.7 \pm 2.35	9.10 \pm 0.80	1.64 \pm 0.07
LSD _S	0.04	0.06	NS	NS	1.07	0.03	1.34	NS	NS
With FYM									
S0	7.67 \pm 0.03	3.84 \pm 0.06	10.2 \pm 0.71	4.74 \pm 0.31	9.41 \pm 1.00	0.62 \pm 0.11	17.5 \pm 1.52	8.90 \pm 0.97	1.66 \pm 0.07
S1	7.65 \pm 0.04	4.33 \pm 0.06	10.2 \pm 0.66	4.90 \pm 0.47	13.5 \pm 1.51	0.63 \pm 0.05	21.3 \pm 1.76	8.93 \pm 0.51	1.64 \pm 0.07
S2	7.56 \pm 0.05	5.16 \pm 0.04	10.3 \pm 0.76	5.21 \pm 0.54	22.5 \pm 1.13	0.68 \pm 0.03	30.7 \pm 1.60	9.01 \pm 0.54	1.65 \pm 0.06
S3	7.51 \pm 0.04	6.42 \pm 0.10	10.5 \pm 0.65	5.01 \pm 0.36	35.0 \pm 1.88	0.78 \pm 0.05	45.6 \pm 1.73	9.23 \pm 0.77	1.67 \pm 0.06
LSD _S	0.03	0.06	NS	0.35	1.15	0.05	1.33	NS	NS
LSD _{SF}	0.02	NS	0.23	0.15	NS	0.03	NS	NS	0.02

LSD_S ($P < 0.05$) = salt effect; LSD_{SF} ($P < 0.05$) = salt * FYM effect, S0 (without NaCl), S1 (30 mM NaCl), S2 (60 mM NaCl), and S3 (120 mM NaCl); NS = not significant.

FIGURE 1: Treatment effects on DTPA-extractable Cd (\pm SD).

organic manure caused a significant increase in soluble Ca⁺⁺, Mg⁺⁺, and K⁺.

3.2. Treatment Effects on DTPA-Extractable Cd. The impact of treatments on the DTPA-extractable Cd is shown in Figure 1. The DTPA-extractable Cd was increased with increasing Cd and NaCl levels. In the soils nontreated with NaCl and FYM, the DTPA-extractable Cd concentrations were 0.06, 0.48, 0.68, 1.45, 4.65, and 7.89 mg kg⁻¹ for Cd0.5, Cd1.5, Cd3, Cd6, Cd12, and Cd24, respectively. Moreover, the DTPA-extractable Cd increased with increasing the levels of NaCl

salinity and accounted for 0.05–8.33 mg kg⁻¹ for S1 and to 0.08–10.87 mg kg⁻¹ for S2 and 0.10–13.71 mg kg⁻¹ for S3.

The addition of FYM significantly decreased the extractability of soil Cd compared to untreated soils. In soil samples treated with FYM, the concentrations of DTPA-extractable Cd were 0.04–2.19 mg kg⁻¹ for S0, 0.04–2.28 mg kg⁻¹ for S1, 0.05–3.07 mg kg⁻¹ for S2, and 0.06–5.60 mg kg⁻¹ for S3.

3.3. Treatment Effects on Dry Matter and Shoot Cd Concentrations of Barley Plants. The impact of treatments on the shoot dry matter of barley plants is shown in Table 2. Increasing

TABLE 2: Treatment effects on shoot dry matter of barley plants (g pot^{-1}).

FYM treatments	NaCl levels	Cd levels (mg kg^{-1})						LSD_{Cd}
		0.5	1.5	3	6	12	24	
Without FYM	S0	0.83	0.80	0.85	0.83	0.82	0.71	NS
	S1	0.82	0.82	0.80	0.81	0.80	0.70	NS
	S2	0.77	0.78	0.77	0.74	0.68	0.60	NS
	S3	0.69	0.66	0.68	0.64	0.53	0.39	0.17
LSD _S		NS	NS	NS	0.11	0.13	0.13	
With FYM	S0	1.10	1.12	1.08	1.15	1.10	1.11	NS
	S1	1.15	1.11	1.09	1.13	1.12	1.10	NS
	S2	1.10	1.07	1.08	1.07	1.05	0.98	NS
	S3	1.07	1.05	1.06	0.97	0.92	0.78	0.20
LSD _S		NS	NS	NS	NS	NS	0.21	
LSD _{SF}		0.08	0.07	0.09	0.08	0.10	0.12	

$\text{LSD}_{\text{Cd}} (P < 0.05) = \text{Cd effect}$; $\text{LSD}_S (P < 0.05) = \text{salt effect}$; $\text{LSD}_{SF} (P < 0.05) = \text{salt * FYM effect}$, S0 (without NaCl), S1 (30 mM NaCl), S2 (60 mM NaCl), and S3 (120 mM NaCl); NS = not significant.

TABLE 3: Treatment effects on shoot Cd content (mg kg^{-1}) of barley plants.

FYM treatments	NaCl levels	Cd levels (mg kg^{-1})						LSD_{Cd}
		0.5	1.5	3	6	12	24	
Without FYM	S0	0.49	0.63	0.90	1.67	2.03	2.97	0.44
	S1	0.42	0.67	0.93	1.83	2.10	2.93	0.47
	S2	0.74	1.06	1.29	2.25	2.70	4.17	0.77
	S3	0.99	1.73	2.30	2.92	3.25	5.74	0.95
LSD _S		0.31	0.52	0.66	0.75	0.93	0.99	
With FYM	S0	0.33	0.34	0.70	1.13	1.57	1.90	0.42
	S1	0.35	0.26	0.67	1.47	1.53	2.05	0.45
	S2	0.52	0.64	0.91	1.48	2.04	2.38	0.54
	S3	0.47	1.03	1.17	1.89	2.17	3.08	0.66
LSD _S		NS	0.10	0.20	0.55	NS	0.95	
LSD _{SF}		0.18	0.36	0.41	0.43	0.50	0.85	

$\text{LSD}_{\text{Cd}} (P < 0.05) = \text{Cd effect}$; $\text{LSD}_S (P < 0.05) = \text{salt effect}$; $\text{LSD}_{SF} (P < 0.05) = \text{salt * FYM effect}$, S0 (without NaCl), S1 (30 mM NaCl), S2 (60 mM NaCl), and S3 (120 mM NaCl); NS = not significant.

irrigation water salinity combined with increasing levels of Cd decreased significantly the shoot dry matter of barley plants (Table 2). Through the applied salinity levels without FYM, the shoot dry matter accounted for $0.71\text{--}0.85 \text{ g pot}^{-1}$ in S0, for $0.70\text{--}0.82 \text{ g pot}^{-1}$ in S1, for $0.60\text{--}0.78 \text{ g pot}^{-1}$ in S2, and for $0.39\text{--}0.69 \text{ g pot}^{-1}$ in S3. On the contrary, applying FYM significantly stimulated the biomass production of barley plants compared to the unamended soil. Through the applied salinity levels with FYM, the shoot dry matter accounted for $1.08\text{--}1.15 \text{ g pot}^{-1}$ in S0, for $1.09\text{--}1.15 \text{ g pot}^{-1}$ in S1, for $0.98\text{--}1.10 \text{ g pot}^{-1}$ in S2, and for $0.78\text{--}1.07 \text{ g pot}^{-1}$ in S3. These results clearly showed that NaCl-induced salinity significantly reduced the growth and shoot dry matter yield of barley plants, especially when the highest salt level (120 mM) was in conjunction with the highest Cd content (24 mg kg^{-1}).

The shoot Cd concentrations were increased with increasing applied Cd and NaCl levels (Table 3). In soil treated and nontreated with FYM, the addition of NaCl resulted in a significant increase in Cd concentrations in the shoot of barley

plants. Through the applied Cd levels without FYM, the shoot Cd concentrations accounted for $0.49\text{--}2.97 \text{ mg kg}^{-1}$ in S0, for $0.42\text{--}2.93 \text{ mg kg}^{-1}$ in S1, for $0.74\text{--}4.17 \text{ mg kg}^{-1}$ in S2, and for $0.99\text{--}5.74 \text{ mg kg}^{-1}$ in S3. In soil treated with FYM, meanwhile, the shoot Cd concentrations were $0.33\text{--}1.90 \text{ mg kg}^{-1}$ for S0, $0.26\text{--}2.05 \text{ mg kg}^{-1}$ for S1, $0.52\text{--}2.38 \text{ mg kg}^{-1}$ for S2, and $0.47\text{--}3.08 \text{ mg kg}^{-1}$ for S3. These results indicated that applying FYM significantly reduced the Cd availability to barley plants.

The results also indicated that the increases in the shoot Cd concentration were correlated positively ($P < 0.05$) with the DTPA-extractable Cd ($r^2 = 0.82\text{--}0.87$), as shown in Figure 2.

3.4. Treatment Effects on Soil Respiration in Incubation Experiment. The $\text{CO}_2\text{-C}$ evolution rates are presented in Table 4. The Cd and NaCl treatments in this study significantly reduced soil respiration. On the contrary, the FYM significantly enhanced soil respiration. In soil without FYM, the $\text{CO}_2\text{-C}$ evolution rates were $0.0027\text{--}0.0276 \text{ mg C g}^{-1} \text{ soil}$

TABLE 4: Treatment effects on CO_2 -C evolution rate (mg C g^{-1} soil day $^{-1}$) during incubation time.

Cd levels	NaCl levels	Day 1	Day 3	Incubation time			LSD _T
				Day 7	Day 14	Day 21	
0.5 ppm Cd	S0	0.0276	0.0140	0.0065	0.0035	0.0033	0.0018
	S1	0.0263	0.0134	0.0062	0.0035	0.0033	0.0036
	S2	0.0222	0.0109	0.0056	0.0029	0.0031	0.0023
	S3	0.0164	0.0090	0.0049	0.0026	0.0025	0.0024
	LSD _S	0.0057	0.0025	0.0013	0.0016	0.0013	—
	S0	0.0197	0.0105	0.0051	0.0029	0.0025	0.0016
	S1	0.0181	0.0097	0.0046	0.0028	0.0023	0.0020
24 ppm Cd	S2	0.0094	0.0055	0.0028	0.0017	0.0015	0.0040
	S3	0.0069	0.0034	0.0017	0.0010	0.0009	0.0013
	LSD _S	0.0026	0.0044	0.0025	0.0011	0.0016	0.0008
	LSD _{CdS}	0.0040	0.0033	0.0018	0.0013	0.0013	0.0012
	Cd levels	With FYM					
	S0	0.1145	0.0653	0.0377	0.0226	0.0196	0.0177
	S1	0.1125	0.0643	0.0373	0.0229	0.0197	0.0180
0.5 ppm Cd	S2	0.1108	0.0631	0.0361	0.0225	0.0195	0.0178
	S3	0.1013	0.0569	0.0324	0.0196	0.0175	0.0157
	LSD _S	0.0055	0.0046	0.0018	0.0012	0.0006	0.0009
	S0	0.1154	0.0658	0.0373	0.0221	0.0194	0.0175
	S1	0.1137	0.0647	0.0367	0.0218	0.0193	0.0177
	S2	0.0942	0.0544	0.0318	0.0191	0.0171	0.0153
	S3	0.0769	0.0457	0.0275	0.0167	0.0144	0.0130
24 ppm Cd	LSD _S	0.0071	0.0040	0.0024	0.0010	0.0009	0.0011
	LSD _{CdS}	0.0058	0.0039	0.0019	0.0010	0.0007	0.0009
	LSD _{CdSF}	0.0047	0.0035	0.0018	0.0011	0.0010	0.0010

LSD_T ($P < 0.05$): time effect; LSD_S ($P < 0.05$): salt effect; LSD_{CdS} ($P < 0.05$): Cd * salt effect; LSD_{CdSF} ($P < 0.05$) = Cd * salt * FYM effect, S₀ (without NaCl), S₁ (30 mM NaCl), S₂ (60 mM NaCl), and S₃ (120 mM NaCl); NS = not significant.

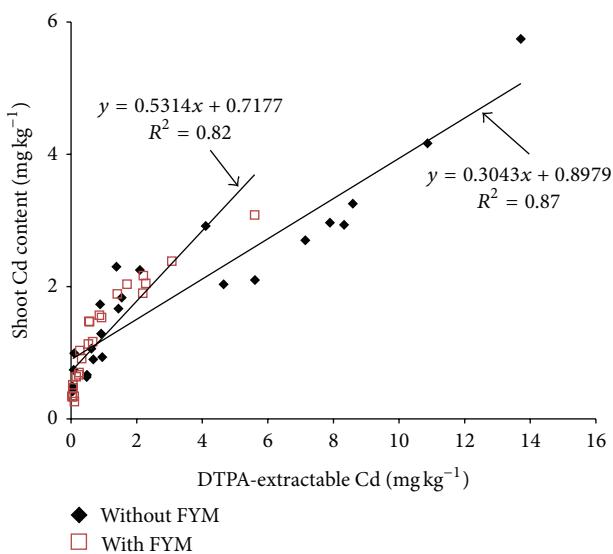


FIGURE 2: Relationship between shoot Cd concentrations and soil DTPA-extractable Cd.

day $^{-1}$ at Cd0.5 and 0.0009–0.0197 mg C g $^{-1}$ soil day $^{-1}$ at Cd24. Meanwhile, in soil treated with FYM, the CO₂-evolution rate

evolution rates were 0.0157–0.1145 mg C g $^{-1}$ soil day $^{-1}$ at Cd0.5 and 0.0130–0.1154 mg C g $^{-1}$ soil day $^{-1}$ at Cd24. Overall, the highest values of CO₂-C evolution rates were recorded at day 1 but tended to decrease sharply when incubation time increased. The results of this study also indicated that the salt stress and Cd affect adversely the microbial activity, as indicated by lower values of CO₂-C evolution rate. The lowest values of CO₂-C evolution rates were found in soil treated with the highest levels of NaCl (120 mM) and Cd (24 mg Cd kg $^{-1}$). It was observed that these decreases in CO₂-C evolution rate were higher in soil without FYM than those in FYM-treated soil. In the absence of FYM, compared to the soil nontreated with NaCl (S₀), S₁, S₂, and S₃ with Cd0.5 reduced the CO₂-evolution rate by 1.76–4.50%, 7.06–22.2%, and 19.4–40.5%, respectively. Additionally, S₁, S₂, and S₃ with Cd24 reduced the CO₂-evolution rate by 4.69–8.40%, 38.4–52.5, and 62.4–67.1%, respectively. In the presence of FYM, compared to the soil nontreated with NaCl (S₀), S₁, S₂, and S₃ with Cd0.5 reduced the CO₂-evolution rate by 1.10–1.80%, 0.6–4.39%, and 10.8–14.3%, respectively. However, S₁, S₂, and S₃ with Cd24 reduced the CO₂-evolution rate by 0.61–1.66%, 12.51–18.30, and 25.7–33.4%, respectively.

4. Discussion

The results of the present study showed that soil pH tended to be decreased slightly with increasing salt levels, mainly due to proton displacement by Na^+ of saline irrigation water. As expected, the values of EC_e , Na^+ , and Cl^- were significantly increased with increasing NaCl levels. Additionally, the concentration of soluble K^+ tended to increase significantly with increasing NaCl levels. This may be linked with the exchange of this ion by Na^+ on soil surface sites. Overall, the addition of FYM lowered the soil pH as compared to unamended soil (Table 1), mainly due to releasing H^+ ions that were associated with mineralized organic anions or by nitrification [9]. The results showed also that the addition of organic manure caused a significant increase in soluble Ca^{++} , Mg^{++} , and K^+ , perhaps due to the release of nutrients through the decomposition of organic amendments [20].

The concentration of DTPA-extractable Cd was increased with increasing Cd levels (Figure 1). Additionally, increasing levels of NaCl caused a significant increase in DTPA-extractable Cd. The highest increase in soil available Cd was pronounced when the soils treated with the highest Cd level (24 mg g^{-1}) were irrigated with the highest NaCl level (120 mM) (Figure 1). This observation confirms the earlier findings that chloride ligands can form particularly strong complexes with heavy metals such that they affect their adsorption reactions with the soil surface [6, 7, 17, 21, 22]. In this context, Ghallab and Usman [6] suggested that the increases in soil chloride ions by NaCl -induced salinity can form strong complexes with Cd, resulting in decreasing the positive charge of Cd and conveying it from the solid to the solution phase. Thus, this leads to enhancing solubility and availability of Cd. Another possible explanation is that Na^+ ions may also contribute to enhancing Cd solubility through the ion exchange of Na^+ for Cd on soil exchange sites. In a study conducted by Smolders et al. [22], to investigate the effect of NaCl and NaNO_3 on soil Cd availability, increasing the levels of NaCl addition to soil increased Cd availability and treatments with NaNO_3 did not significantly affect soil Cd availability. Therefore, the increases in soil Cd availability could be attributed to the formation of CdCl_{n-2-n} complexes but not to Na^+ exchange with Cd onto soil surfaces. These results are in accordance with those of several other researchers [6, 23, 24].

Results showed also that increasing levels of Cd and NaCl caused increases in the phytoavailability of Cd, as indicated by a significant increase in the shoot Cd concentration (Table 3). However, the addition of FYM significantly decreased DTPA-extractable Cd compared to unamended soils (Figure 1). Furthermore, addition of FYM caused a significant decrease in shoot Cd content of barley plants. These results are in accordance with those of Ahmad et al. [25], who observed that farm manure amendment significantly decreased AB-DTPA-extractable Cd and Pb concentrations and their content in wheat grains. Decreased bioavailability of Cd with organic amendments has also been reported by several other researchers [9, 26–30]. The lower Cd availability in the FYM amended soils could be mainly related to the Cd immobilization through sorption, chelation, and sequestration by

the solid and soluble organic matter. Indeed, soil metal availability can be reduced following the addition of organic materials through metal transformation from more readily available forms to less available forms such as fractions associated with organic materials, carbonates, or metal oxides, causing a reduction in metal uptake by plants [9, 30]. Organic matter contains reactive functional groups of hydroxyl, phenoxyl, and carboxyl, which may be responsible for governing the adsorption and complex of heavy metals with soil and subsequently their effect on metal bioavailability [26, 28, 31]. Narwal and Singh [26] found that the DTPA-extractable Cd, Cu, Ni, and Zn in soils were decreased with increasing application rates of cow and pig manure and peat except one case of pig manure where the DTPA-extractable Zn increased. They also found that the concentration of metals was lower in plants grown in the cow manure-amended soil as compared to those grown in the soil amended with either pig manure or peat soil. Our results also clearly showed that the increases in the shoot Cd concentration were correlated positively with the DTPA-extractable Cd (Figure 2). This result suggests that the availability of Cd to plants is dependent not only on its total content but also on its soil available fraction. Therefore, chemical extractants such as DTPA should be taken into account for evaluating the availability of Cd to plants.

Soil respiration can be considered as a main process as a result of organic matter turnover. The individual effects of salinity and metal toxicity on microbial activity are well known but less is known about their interactive effects. Our results indicated that CO_2 -C evolution rates were rapid initially and then tended to decrease sharply with incubation time (Table 4). It is possible that this is mainly due to the high readily available organic C to soil microorganisms at the beginning, which can easily be exhausted with time [12, 32, 33]. Adding FYM led to a significant increase in soil respiration compared to unmanured soil, as indicated by the higher CO_2 -C evolution rate. This might be caused by the unstable organic fraction in FYM [9, 12].

Salt stress affects adversely the soil respiration and organic C mineralization, as indicated by lower values of CO_2 -C evolution rate with increasing NaCl levels. Overall, the lowest values of CO_2 -evolution rates were found in soil samples treated with the highest levels of NaCl treatments. This result suggests that a portion of soil microorganisms might be not able to tolerate high osmotic stress. A lower osmotic potential by a higher salinity in the soil can result in lowering microbial activity and organic matter transformation [34]. The toxicities of specific ions may also be a reason for inhibiting microbial activity under saline conditions [35]. The obtained findings can explain the lowered CO_2 -C evolution rates in saline soils by the reduced size and activity of the microbial community [36]. In terms of heavy metal toxicity, it is also clear that the higher content of Cd caused significant decreases in the values of CO_2 -evolution rate. The highest depressed impact on soil respiration was found when the soil was treated with the highest Cd level (24 mg kg^{-1}) in conjunction with the highest NaCl level (120 mM). The increases in soil Cd availability in soils with increasing NaCl levels could enhance metal toxicity to soil microorganisms, resulting in the lowest microbial activity. It has been reported

that Cd has a toxic effect on soil microbial activity [1, 37].

On the other hand, the CO₂-eflux rate was affected by FYM application. Applying FYM to Cd-contaminated soil irrigated with NaCl showed higher CO₂-evolution rates compared to NaCl treatment in the absence of FYM. This suggests that organic amendment can significantly stimulate the microbial activity, even under metal contamination and saline conditions. Generally, organic amendment can increase microbial activity and diversity and microbial tolerance to stress conditions can be detected over time [1, 38]. On the other hand, soil organic matter may contribute to adsorption and immobilization of Cd and thus restrict its solubility and bioavailability. Therefore, organic amendments such as FYM may have an important role to improve quality and minimize metal toxicity of contaminated soils under heavy metals and salinity stress. More recently, it has been speculated that the application of organic manure to metal-contaminated soil may create good environmental condition for mitigating the toxicity induced by high metal concentrations [12].

5. Conclusion

This study suggests that Cd-contaminated soil irrigated with NaCl increased the inhibitory toxic effect on plant growth and soil respiration activity. However, Cd bioavailability in calcareous loamy sand soil reduced following the application of farmyard manure. Further researches are needed to fully understand the effects of FYM on the availability and transformation of heavy metal in different metal-contaminated soils of arid conditions.

Conflict of Interests

The author declares that there is no conflict of interests regarding the publication of this paper.

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Research Article

Effectiveness of Extractants for Bioavailable Phosphorus in Tropical Soils Amended with Sewage Sludge

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Urban wastes such as sewage sludge can be an economically viable alternative source for providing macro- and micronutrients to plants in tropical conditions. Sewage sludge is normally rich in phosphorus (P), which is present in soils mainly in organic forms, so that it is very important to establish methods for estimating its availability to plants. This study aimed to test three extractants that simulate P-uptake by maize (*Zea mays*) cropped in plots after 13 consecutive years of fertilization with sewage sludge, in a cycle of fertilized sugarcane (*Saccharum L.*) amended with sewage sludge and organic compost. Soil samples were collected at depths of 0–10, 10–20, and 20–40 cm in March 2010 from the two experimental areas. Soil P was extracted via ion exchange resin, Mehlich-I, and 0.025 M H₂SO₄ and determined via colorimetry. Maize and sugarcane diagnostic leaves were collected in the experiments, subjected to nitric-perchloric digestion, and the leaf-P content was determined via colorimetry. No significant correlations were found between phosphorus extracted from soils and phosphorus concentrations in diagnostic leaves. Resin extracted larger amounts of P in the short-term experiment, while acidic extractants yielded larger amounts in the long-term experiment.

1. Introduction

Applying urban wastes such as sewage sludge to agricultural soils is an economically viable alternative for their disposal, and amending soils with sewage sludge benefits crops and poses few environmental risks [1, 2]. Concerns remain, however, regarding the possible presence of trace elements, toxic organic compounds, or pathogenic microorganisms in sewage sludge [3].

Sewage sludge can increase soil concentrations of certain nutrients, such as phosphorus (P) [4]. Current Brazilian legislation [5] mandates that rates of sewage sludge be calculated based on crop nitrogen (N) requirements and does not take into consideration P needs or the fate of the P applied with wastes [6]. Because the P:N ratio of waste is typically higher than that required by plants [7], soils amended with

sludge usually accumulate P depending on climate and soil properties that command soil P adsorption and desorption. Ippolito et al. [8] showed that fertilization with sewage sludge based on crop nitrogen requirement can add to the soil more P that maize plants can remove.

Studies of long-term soil amendment with sewage sludge of agricultural soils are crucial because in addition to P accumulation soil amendment with sludge can potentially cause incremental and unpredictable effects such as increased levels of organic matter and toxic element accumulation [1]. Ideally, a P soil extractant should operate regardless of varying mineralogical, biological, and chemical attributes of soils. Many authors have used parameters of plant growth such as the P content and plant production to test the extent to which planned responses are correlated with P extracted from soils [9]. In many cases, however, P extractants commonly

used in laboratories are incapable of extracting from the soil quantities of the element correlated with those accumulated in plants. One possible reason for this variation is the fact that many extractants remove inorganic P while underestimating the influence of organic P in plant nutrition. In tropical soils, the organic material has high affinity for Fe and Al oxide surfaces [10], causing P fixation.

Available P contents may be underestimated if the extractant is not able to oxidize the organic fraction resulting from the quick decomposition of the fresh organic material or if organic matter are slightly modified, that are associated with the oxides surface [11]. In soils with low P content in which organic P is an important source of nutrient to plant nutrition, the selection of extractants that are able to estimate the available P may not be an easy task [12]. As of now, there is no known efficient extractant for organic P present in residues which is capable of estimating the labile organic P fraction that can be absorbed by the plants [13].

The aim of this study was to compare the effectiveness of three extractants widely used for the extraction of phosphorus in tropical soils considering the quantification of (i) available P in soils amended with sewage sludge for 13 consecutive years and the maize P-uptake and (ii) available P in soils amended with a single application of sewage sludge and compost sewage sludge and sugarcane P-uptake.

2. Materials and Methods

The maize experiment was part of an ongoing study established in the 1997-1998 growing season at Jaboticabal, in the state of São Paulo (SP), Brazil, on a Typic Eutrorthox, with the aim of evaluating the effects of 13 consecutive years of amendments with sewage sludge. The original treatments were a control (no sludge and no fertilizer added), 2.5 t ha^{-1} , 5 t ha^{-1} , and 10 t ha^{-1} of sewage sludge (dry basis). The 5 t ha^{-1} rate was established to supply the amount of nitrogen (N) required by maize crops, with the assumption that 1/3 of the N in the waste would become available to plants during the first year. Starting in the second year, mineral fertilizer was added each year to supply plant needs in NPK according to soil chemical analysis and the indications of van Raij et al. [14]. Starting in the fourth growing season, the 2.5 t ha^{-1} rate was changed to 20 t ha^{-1} to induce trace element phytotoxicity in plants. During the 13 years before the soil sampling, the cumulative amounts of sewage sludge added to the soils totaled 65, 130, and 207.5 t ha^{-1} for the 5, 10, and 20 t ha^{-1} treatments, respectively. The experiment consists of 60-m² plots in randomized complete blocks with four treatments (varying rates of sewage sludge) and five replicates. In 2009, the annual application of sewage sludge took place in December. The sludge had a pH_{H2O} of 5.8 and contained 81.3% water and the following contents (dry matter): 246.7 g kg^{-1} C, 20.3 g kg^{-1} P, 24.8 g kg^{-1} N, 2.4 g kg^{-1} K, 1.0 g kg^{-1} Na, 15.9 g kg^{-1} Ca, and 4.2 g kg^{-1} Mg. Heavy metal concentrations in the sludge (dry matter) were 5.1 mg kg^{-1} Cd, 19.6 mg kg^{-1} Co, 531.5 mg kg^{-1} Cr, 669.0 mg kg^{-1} Cu, $34,526.6 \text{ mg kg}^{-1}$ Fe, 320.2 mg kg^{-1} Mn, 290.7 mg kg^{-1} Ni, 106.6 mg kg^{-1} Pb,

and $1,398.5 \text{ mg kg}^{-1}$ Zn. The plots with sewage sludge were fertilized with mineral fertilizers in order to receive the same amounts of NPK as the control. Sewage sludge was spread over the entire area and incorporated to 0–10 cm, while the mineral fertilizers were applied in the furrow.

The second field experiment was part of a study underway at Piracicaba (SP), Brazil, on a Typic Hapludalfs in a sugarcane plantation that had been recently harvested (third harvest) at the time of the sampling. Soil acidity was corrected with 2.5 t ha^{-1} of dolomitic lime (15% water content) and sewage sludge and organic compost were applied on a single occasion on top of the sugarcane straw left behind after harvest. The sewage sludge contained (dry matter) 340 g kg^{-1} C, 22 g kg^{-1} N, 11 g kg^{-1} P, 5 g kg^{-1} K, 14 g kg^{-1} Ca, 3 g kg^{-1} Mg, and 9 g kg^{-1} S. Metal concentrations were 95 mg kg^{-1} Cu, 625 mg kg^{-1} Mn, and 715 mg kg^{-1} Zn. The composted sewage sludge had (dry matter) 210 g kg^{-1} C, 18 g kg^{-1} N, 11 g kg^{-1} P, 3 g kg^{-1} K, 20 g kg^{-1} Ca, 3 g kg^{-1} Mg, and 4.5 g kg^{-1} S, as well as 70 mg kg^{-1} Cu, 475 mg kg^{-1} Mn, and 527 mg kg^{-1} Zn. The treatments were complemented with 120 kg ha^{-1} of K₂O, since the sanitary sludge had low levels of potassium. The experiment consisted of subdivided plots with three replicates. The treatments (sludge or compost) were (i) no waste applied; (ii) 50% of the rate recommended by Brazil's National Environmental Council (CONAMA) for amendment with sewage sludge applied; (iii) 100% of the recommended rate applied; and (iv) 200% of the recommended rate applied. The CONAMA regulations are based on the assumption that 20% and 10% of organic N in anaerobically digested sewage sludge and organic compost, respectively, will be mineralized. To supply 100 kg ha^{-1} N for the sugarcane crop, 67 t ha^{-1} of sludge (wet basis) and 300 t ha^{-1} of compost (wet basis) were used; the water content of these organic sources was 73% and 60%, respectively. The experiment consisted of 24 plots. Each plot contained five rows of sugarcane measuring 7 m long and spaced 1.4 m between rows. The three central rows and 5 m to either side of them represent the primary sampling area of the plots.

Diagnostic leaves of maize (the leaf opposite to and below the ear) and sugarcane (plus three leaf) cultivated in the 2009–2010 growing season were collected from the two experiments to quantify P concentrations. Plant material was harvested, cleaned with deionized water, placed in paper bags, and dried at 60°C in a forced-circulation oven until reaching constant mass. Samples were ground in Wiley mills and subsequently subjected to nitric-perchloric digestion (0.5 g of plant material + 5 mL HNO₃ + 1 mL HClO₄) adapted from Johnson and Ulrich [15]. P concentrations of the extracts were determined by colorimetry (ammonium vanadate-molybdate method).

Soil samples were collected in March 2010 from the two experimental sites: (i) the soil amended with sewage sludge (cultivated with maize) and (ii) the soil amended with sewage sludge composted with straw pruning tree and sludge (cultivated with sugarcane). Twenty subsamples were collected at each of the three soil layers (0–10, 10–20, and 20–40 cm) and mixed to make a composite sample for each depth. In this study, we examined the surface layer (0–10 cm) to observe how higher levels of organic matter influenced

the contents of available P to plants. Samples taken for soil fertility analyses are typically collected at a depth of 0–20 cm because fertilization is commonly performed based on tables that consider soil sampling at this depth. Soil samples were air-dried and sifted through a 2 mm (10 mesh) screen. Phosphorus was extracted with ion exchange resin [16], Mehlich-I ($0.05\text{ M HCl} + 0.0125\text{ M H}_2\text{SO}_4$) [17], and $0.025\text{ M H}_2\text{SO}_4$ [18]. The latter is the extractant most commonly used to predict levels of P available to sugarcane crops in Brazilian soils. Phosphorus content of the extracts was determined by colorimetry.

The results were compared via analysis of variance and correlation tests. For all analyses SAS software was used [19] and significance levels of 1% and 5%.

3. Results and Discussion

3.1. Phosphorus Concentrations in Maize and Sugarcane Diagnostic Leaves. The P concentration in maize and sugarcane diagnostic leaves did not differ between different rates of sewage sludge and/or composted sewage sludge (Table 1). The P concentrations in diagnostic leaves were in the adequate range for maize ($2\text{--}4\text{ g kg}^{-1}$) and sugarcane nutrition ($1.5\text{--}3.0\text{ g kg}^{-1}$) [14]. Sewage sludge application for a long time did not add amounts of the element in the plant above the values recommended for the culture. The element may have been either strongly adsorbed by soil colloids or may not have accumulated at the part of the plant analyzed. Phosphorus bioavailability in soil is directly dependent on its lability in the residues [20, 21], and this can explain the absence of increment in the element concentration in the diagnostic leaves.

No significant correlations were found between the P extracted from soils by the extractants and that in the diagnostic leaves of maize and sugarcane suggesting that this sampled part of the plant is not adjusted for the soils we have studied, which have been amended continuously with a waste that is rich in organic matter and phosphorus. Some authors suggest other leaves for the nutritional diagnostic of maize and sugarcane plants [22]. The excess of extracted P, where found to occur, may have accumulated in another part of the plant. In Brazil, one special leaf has been used to predict the nutritional state for various crops, to facilitate the recommendation of additional fertilization during the plant growing cycle. However, sometimes there is no correlation between the nutrient absorbed by the plant and its content in the diagnosis leaf, depending on the crop considered. For maize, this is relatively well defined, whereas for sugarcane there is some controversy. The use of diagnostic leaves for nutritional diagnosis of cultures is often used in agricultural soils not receiving organic compounds over a long period, which may explain the absence of correlation of nutrients in soil and plant.

In highly weathered soils that are rich in Fe and Al oxides, P is strongly retained by colloids and the extractants we used may not be capable of removing the available quantity. This complicates fertilization management and may explain the weak correlations observed between P concentrations

TABLE 1: Phosphorus concentrations in diagnostic leaves of maize plants grown in a Typic Eutrorthox amended annually with sewage sludge for 13 consecutive years and in diagnostic leaves of sugarcane plants grown in a Typic Hapludalf amended once with sewage sludge and sludge compost ($n = 4$).

Rates	P (diagnostic leaves) g kg^{-1}
Maize	
0	2.78 ^a
5	3.02 ^a
10	3.11 ^a
20	3.07 ^a
RL	ns
RQ	ns
Sugarcane	
0	2.30 ^a
50	2.40 ^a
100	2.35 ^a
200	2.57 ^a
RL	ns
RQ	ns

*RL: linear regression; RQ: quadratic regression; ns: not significant at 5%.

**Values with the same letter within a column are not different (Tukey, $P < 0.05$).

extracted by common extractants and those absorbed by plants [23].

Sewage sludge possesses a vast gamut of physical and chemical properties. Schroder et al. [24] comment that the average level of total P in the samples of sewage sludge between 1993 and 2005 was 38.6 g kg^{-1} , ranging between 29.8 and 59.5 g kg^{-1} . Maguire et al. [25] concluded that forecasting P availability in sewage sludge can be a useful tool for predicting the extractable P in soils treated with this type of residue. They investigated the relationship between processes applied in the treatment of sewage sludge and the bioavailability of P and verified that the extraction pattern is different for soils depending on whether they are amended or not with residue (and amended or not with Fe or Al) and that the need for such extraction tends to reduce in line with the incubation period of soil with the residue.

Kamprath and Watson [26] compiled various correlations between the amount of P extracted by Bray-I solution and the amounts absorbed by plants and found a range of values from -0.10 to 0.94 . Their study is a good example of the difficulty of finding a P extractant that is effective across a broad range of plants and/or soils. Similarly, Sharpley et al. [27] compiled data on correlation coefficients and found values ranging from 0.41 to 0.93 between P absorption by plants and P extracted by anionic exchange membrane, Olsen, Bray-I, strips impregnated with Fe oxide, acetic acid, and dilute sulfuric acid. This lack of correlation between the P extracted from the soil and the P absorbed by the plant occurs in the majority of cases, where the extractants had been developed for a system designed for traditional crops, without significant presence of organic material, and this

TABLE 2: Mean extracted phosphorus by Mehlich-I, H_2SO_4 , and resin in the two experiments described in the text: one with amendment of sewage sludge and the other with the amendment of sewage sludge and composted sewage sludge ($n = 4$).

Extractant	Depth (cm)		
	0–10	10–20	20–40
Sewage sludge			
Mehlich-I	104.9 ^a	42.9 ^a	8.4 ^b
H_2SO_4	90.7 ^b	36.7 ^b	6.6 ^c
Resin	81.2 ^c	33.8 ^b	9.8 ^a
Sewage sludge and composted sewage sludge			
Mehlich-I	10.2 ^b	6.6 ^a	4.8 ^{ba}
H_2SO_4	8.4 ^c	5.4 ^b	4.0 ^b
Resin	13.6 ^a	7.5 ^a	6.0 ^a

* For each experiment, values with the same letter within a column are not different (Tukey, $P < 0.05$).

could impose difficulties for the extraction of organic P such that at the stage where the element is fractionated this P content is included in the fraction extracted by HCl. Huang et al. [28] managed, at least in part, to correct and eliminate the organic P in the HCl fraction, but this value was included in the residual fraction. This demonstrates the complexity of cultivation systems with a significant presence of organic material as far as it relates to a prediction of bioavailable P for the plants, once great variation becomes apparent in the attributes of the soils and residuals used as alternative sources of nutrients.

No extractant provided a reliable representation of the quantity of available P in a wide-range of soils. In addition, the fact that P forms in soils change quickly following the addition of organic waste material containing the element (e.g., when P is added as manure or sludge) raises many questions about the validity of extraction methods [29, 30].

3.2. Soil Phosphorus Content in the Long-Term Experiment. At depths of 0–10 and 10–20 cm levels of P-Mehlich-I were higher than those extracted by H_2SO_4 , which in turn were higher than those extracted by resin. At a depth of 20–40 cm the highest P contents were obtained with P-resin, followed by P-Mehlich-I and P- H_2SO_4 ($P < 0.01$) (Table 2). Soils formed in tropical conditions are corrected with limestone to the 0–20 cm layer, having the highest probability of P being bound to calcium forms. The Mehlich-I extracts the Ca-P fraction and to a lesser extent the fractions of P-Al and Fe-P. These findings could explain the high content extracted by Mehlich-I in the surface soil and the low extraction capacity in the subsurface layer, in which the concentrations of Fe and Al are higher. The same behavior was observed in tropical soils by Viégas et al. [31] observing high correlation among the P levels extracted by Mehlich I, with the action of two acids (HCl and H_2SO_4) and the increase in the soil pH, which decreased when the correlation with resin extractant was considered possibly due to the consumption of acidity (Mehlich I) and exchange anions SO_4^{2-} .

TABLE 3: Mean extracted phosphorus by Mehlich-I, H_2SO_4 , and resin from samples collected at three depths from soils treated with varying rates of sewage sludge, in the long-term experiment ($n = 4$).

Rates	Mehlich-I	H_2SO_4 mg kg ⁻¹	Resin
	0–10 cm		
0	56.7 ^c	51.4 ^c	43.3 ^b
5	68.8 ^c	60.6 ^c	53.0 ^b
10	125.2 ^b	102.4 ^b	104.5 ^a
20	169.0 ^a	148.3 ^a	124.1 ^a
RL	**	**	**
RQ	ns	ns	ns
10–20 cm			
0	21.7 ^b	19.4 ^b	19.2 ^b
5	24.4 ^b	21.1 ^b	22.2 ^b
10	47.5 ^{ba}	40.5 ^{ba}	40.8 ^a
20	77.9 ^a	65.8 ^a	53.2 ^a
RL	**	**	**
RQ	ns	ns	ns
20–40 cm			
0	5.3 ^b	3.9 ^b	6.4 ^b
5	6.0 ^b	4.5 ^b	7.6 ^b
10	8.3 ^{ba}	6.1 ^{ba}	10.9 ^{ba}
20	14.0 ^a	11.9 ^a	14.5 ^a
RL	*	*	**
RQ	ns	ns	ns

(i) RL: linear regression; RQ: quadratic regression; ns: not significant at 5%; * significant at 5%; ** significant at 1%.

(ii) Values with the same letter within a column and same depth are not different (Tukey, $P < 0.05$).

Mehlich-I solution is a mixture of strong acids with a pH of approximately 1.2. As an extractant based on strong acids, it is expected to mostly extract P bound to Ca, and, at lower levels, P bound to Fe and Al. Due to the nature of the acid-Mehlich I, it is expected that in addition to the resin extracted fraction, extracted from the fraction bound to calcium, amounts of nonlabile P can also be extracted, which can cause an overestimating of the levels of P available in soils for agricultural crops. In clayey soils where kaolinite predominates, extractant exhaustion has been observed, leading to an underestimating of P levels and the readsoption of phosphate to the soil colloids after the adsorption of chloride and sulfate ions by sites that were not initially occupied by phosphate [32].

The effect of the rates of sewage sludge was detected by all three extractants studied (Table 3). Even after 13 years of amendment with sludge, these soils showed increasing levels of P with increasing amounts of waste (accumulative effect). Sludge may diminish adsorption of P to soils because organic ions may compete with phosphate for adsorption sites [33].

Paz-Ferreiro et al. [34] carried out an experiment to assess the effect of cattle manure (30, 60, and 90 t ha⁻¹, dry basis) on nutrient accumulation in the surface layer of an acidic soil with high levels of OM and P and found that levels of

P extracted by anion exchange resin were lower than those extracted by Mehlich-III. The P levels extracted by the resin and Mehlich-III methods (determined by colorimetry or ICP) increased as the rates of cattle manure increased. Kidd et al. [35] carried out a greenhouse experiment to assess P availability to maize and two species of wild plants in soils treated with sewage sludge for more than ten years and found that amendment with sludge was associated with increased levels of Olsen P. The P absorption was higher in plants grown on amended soils (possibly luxury consumption by these plants), but the difference was lower for maize plants, suggesting that the excess P added to the soils cultivated with maize posed a higher risk for losses through leaching.

Between 1988 and 2000 Mantovi et al. [36] studied the application of liquid sewage sludge, dehydrated sludge, and composted sewage sludge (5 and 10 t ha⁻¹ yr⁻¹) to a silty loam soil in Italy cultivated in rotating crops of wheat, beets, and maize and observed a sharp increase in P contents extracted by Olsen. This finding could be explained not only by the large rates of sludge applied, but also by the increasing availability of the nutrient with time in the treated soils [37]. Mantovi et al. [36] reported that wheat grain from amended soils showed higher P concentrations than those of plants treated with mineral fertilizers. Citak and Sonmez [38] also noted a greater concentration of nutrients in the edible part of cabbage (*Brassica oleracea*) when grown in soil amended with organic residues (bovine and chicken manure and blood meal) when compared to vegetables composted with mineral fertilizers.

Despite the beneficial effects for plants, excess of mobile P represents an environmental risk [39] that can lead to the eutrophication of lakes and streams [40] in sandy soils. McDowell et al. [13] quantified available P to *Lolium* and *Pinus* using 12 extractants (five saline and seven resins) for seven soil types and reported that the mineralization of organic P was best characterized by increases in the correlation coefficients between extractable P and P absorbed by plants when organic and inorganic P were considered together. McDowell et al. [13] argued that the easily extractable forms of organic P in soils contribute, in the short term, to the absorption of P by plants and that those forms of P should be considered in common tests to predict the availability of P in soils.

We observed positive linear correlations between the methods we tested, with the highest correlation coefficients between Mehlich-I and H₂SO₄, but we did not detect any significant correlation between P levels extracted from soils and P concentration in diagnostic leaves by plants, probably because the extractants we used did not take into account the organic P added to the soils (Table 4).

3.3. Phosphorus Content in the Short-Term Experiment. In the experiment in which soils received a single application of sewage sludge and composted sewage sludge, resin extracted the greatest amounts of P from samples collected at depths of 0–10 and 20–40 cm, followed by Mehlich-I and H₂SO₄, which occurred due to less contact time of the residue with the soil, and the predominance of more soluble P, which is

TABLE 4: Correlation matrix for phosphorus extracted from soils at depths of 0–10, 10–20, and 20–40 cm and phosphorus levels in diagnostic leaves of maize plants grown in the long-term experiment (*n* = 4).

	Mehlich-I	H ₂ SO ₄	Resin	Plant
0–10 cm				
Mehlich-I	—	0.99**	0.92**	ns
H ₂ SO ₄		—	0.90**	ns
Resin			—	ns
Plant				—
10–20 cm				
Mehlich-I	—	0.99**	0.94**	ns
H ₂ SO ₄		—	0.94**	ns
Resin			—	ns
Plant				—
20–40 cm				
Mehlich-I	—	0.98**	0.95**	ns
H ₂ SO ₄		—	0.93**	ns
Resin			—	ns
Plant				—

ns: not significant at 5%; **significant at 1%.

poorly retained by the soil and easily extracted by the resin. At a depth of 10–20 cm, P extracted by resin and Mehlich-I were similar and greater than those extracted by H₂SO₄ (Table 2).

Variation in the rates of sludge and composted sewage sludge had no effect on the extracted phosphorus contents. This was mostly due not only to the short duration of the experiment (i.e., only three months between waste application and soil sampling), but also to the fact that waste was not incorporated into the soil (Table 5). After the initial increases, the pool of P extracted by laboratory tests declined gradually with time (due to the slow precipitation of mineral P). Lucero et al. [41], Reddy et al. [42], and Griffin et al. [43] reported differences in the amounts of P extracted from soils when the element was applied as inorganic fertilizers (KH₂PO₄, NH₄HPO₄) and manure. The effects of these differences on the availability of P have been attributed to variation in the speciation of the element, pH, the solubility of the applied sources, the quality and quantity of organic matter, and the presence of Al, Fe, and Ca [44, 45].

In two years Chiba et al. [46] applied rates of sewage sludge (with or without incorporation and at amounts based on N contents) to an Ultisol planted with sugarcane and showed that P contents were not altered by amendment. This result probably reflects that sewage sludge in that experiment remained on the surface of the soil, largely out of contact with the soil biota capable of breaking it down. Both thermal drying and exposure to sun tend to slow the degradation rate of sewage sludge, due to a reduction in water content and a consequent increase in the stability of organic compounds. This may also explain why different rates of amendment had no effect on levels of biologically available P in this short-term experiment, since the waste was placed on the soil surface in a single application. Any change in the phase or decomposition of organic material attributable to heat or dryness can

TABLE 5: Mean of phosphorus concentrations extracted by Mehlich-I, H_2SO_4 , and resin from samples collected at three depths from soils treated with varying rates of sewage sludge and composted sewage sludge, in the short-term experiment ($n = 4$).

Rates	Mehlich-I	H_2SO_4 mg kg ⁻¹	Resin
0–10 cm			
0	8.4 ^a	7.1 ^a	12.3 ^a
50	13.5 ^a	11.0 ^a	18.0 ^a
100	8.8 ^a	7.2 ^a	11.9 ^a
200	10.2 ^a	8.4 ^a	12.1 ^a
RL	ns	ns	ns
RQ	ns	ns	ns
10–20 cm			
0	5.5 ^a	4.4 ^a	4.9 ^a
50	7.2 ^a	5.8 ^a	8.5 ^a
100	6.6 ^a	5.4 ^a	7.1 ^a
200	7.1 ^a	5.9 ^a	9.5 ^a
RL	ns	ns	ns
RQ	ns	ns	ns
20–40 cm			
0	7.0 ^a	6.1 ^a	7.9 ^a
50	3.8 ^a	3.3 ^a	4.2 ^a
100	5.1 ^a	4.5 ^a	5.4 ^a
200	3.2 ^a	2.1 ^a	6.5 ^a
RL	ns	ns	ns
RQ	ns	ns	ns

* RL: linear regression; RQ: quadratic regression; ns: not significant at 5%.

** Values with the same letter within a column in the same depth are not different (Tukey, $P < 0.05$).

lead to the formation of P compounds with metals and/or minerals, although more thorough studies may be necessary to elucidate the mechanisms behind these alterations [47]. In a study of the sequential extraction of P in sewage sludge and poultry litter, He et al. [47] concluded that the solubility of P contained in the sewage sludge was controlled mainly by the metals Al, Mn, and Zn, with Al exerting the greatest influence due to its greater concentration, which should be a relevant consideration when working with soils that are highly weathered in moist tropical zones.

We observed positive linear correlations with a high correlation coefficient between extractants, but not between extractant and P levels in diagnostic leaves (Table 6). The strongest correlations were observed between acidic extractants, which provided the same effect on the release of the organic P on the soil surface. The effects of changes caused by the application of sewage sludge vary not only with the duration of application, but also with variation in speciation of the element, in pH, in the solubility of the applied source, in the quantity and quality of organic matter, in the presence of Al, Fe, and Ca, and other factors.

TABLE 6: Correlation matrix for phosphorus extracted from soils at depths of 0–10, 10–20, and 20–40 cm and phosphorus levels in diagnostic leaves of sugarcane plants grown in those soils, for the short-term experiment ($n = 4$).

	Mehlich-I	H_2SO_4	Resin	Plant
0–10 cm				
Mehlich-I	—	0.98**	0.94**	ns
H_2SO_4	—	—	0.97**	ns
Resin	—	—	—	ns
Plant	—	—	—	—
10–20 cm				
Mehlich-I	—	0.98**	0.75**	ns
H_2SO_4	—	—	0.77**	ns
Resin	—	—	—	ns
Plant	—	—	—	—
20–40 cm				
Mehlich-I	—	0.99**	0.80**	ns
H_2SO_4	—	—	0.77**	ns
Resin	—	—	—	ns
Plant	—	—	—	—

ns: not significant at 5%; ** significant at 1%.

4. Conclusion

- (i) No significant correlations were observed between P extracted from soils by the three extractants and P concentration in the diagnostic leaves of maize and sugarcane plants grown in those soils, suggesting that (1) the plant parts sampled were not appropriate for evaluating the availability of P in tropical soils that are amended with sewage sludge or composted sewage sludge or (2) the extractants used were developed for conventional crops, and they do not appear to be effective in systems where there is an elevated level of organic material. This underlines the importance to us of the supply of nutrient to plants, especially in soils where the level of the element is low.
- (ii) Resin extracted greater amounts of P from soils in the short-term experiment (P in more labile forms), while the acidic extractants removed greater amounts of the nutrient in the long-term experiment (P in less bioavailable fractions).
- (iii) An alternative for improving the extractants with the aim of rendering them useable in tillage systems where there is a presence of organic material is to develop potentially available mechanisms for extracting organic P or quantify the P in the residuals as well.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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Research Article

Seasonal Dynamics of N, P, and K in an Organic and Inorganic Fertilized Willow Biomass System

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The seasonal variations in soil nutrient supply and bioavailability were assessed in a willow biomass crop (*Salix miyabeana*, SX64) treated with 150 and 200 kg available N ha^{-1} of commercial fertilizer (CF), biosolid compost (BC), dairy manure (DM), and control (CT0) at Delhi, NY. Plant root simulator probes were used to measure nutrient supply (inside) and bioavailability (outside) of root exclusion cylinders. Measurements were made in September 2008 and May, August, and October of 2009. Soil moisture content (θ_d) and foliar nutrient concentrations were also determined. The BC treatments increased soil P supply more than CF and CT0. The supply of NH_4^+ and K in the soil increased in August but their bioavailability increased in May and October. Soil NO_3^- and P supply and bioavailability were both high in May. Foliar N and K concentrations were significantly high in May and low in August which could be due to dilution effect caused by increased soil moisture foliar dry weight. Foliar P concentrations increased in September and October. The observed higher soil NO_3^- mineralization and plant uptake in May suggest that in high soil NO_3^- conditions willow biomass crops can leach out of the root zone into groundwater.

1. Introduction

An adequate supply of nutrients is essential to support the high yields that are necessary to make short rotation woody crops (SRWC) like shrub willow viable both economically and biologically. Soil nutrient supply and bioavailability in short rotation woody crops (SRWC) systems may vary depending on the type and form of nutrient input added, conditions at the site, and how the system is managed. Systems that receive high organic inputs would be expected to have greater soil nutrient supplying ability than those that receive only mineral fertilizers [1]. The use of mineral N fertilizers (100 kg N per rotation cycle) in willow biomass production systems has been estimated to represent 10% of the production cost [2] and account for as much as 37% of the nonrenewable fossil energy inputs [3]. The use of commercial fertilizers for willow biomass production therefore reduces

the environmental and economic benefits of the feedstock production system [4].

Land application of organic residues represents an economically and environmentally safe way to recover value from these materials. The organic matter and nutrient contents of the waste materials can supply crops' needs and maintain soil fertility. However, due to increased public concerns about environmental issues, farmers are reluctant to use sewage sludge on lands used for food crop production. Since SRWC are nonfood crops, they are an attractive system for utilizing organic waste streams. The ability of willow to retain high amounts of nutrients in combination with its high biomass production makes it a preferred candidate for utilizing organic wastes [5].

The traditional method of measuring soil nutrient supply and availability represents only a "snapshot" of available nutrients and may not adequately reflect nutrient supply

throughout the growing season [6]. *In situ* burial of ion-exchange membranes (IEM), such as the plant root simulator (PRS) probe, on the other hand, allows the measurement of nutrient supplies according to the actual field conditions with minimal soil disturbance. The use of PRS probe provides insight into the dynamics of ion flux in the soil. When buried in the soil, it can serve as a measure of nutrient supply rate by continuously adsorbing charged ionic species over the burial period. This measured flux of soil nutrients over time can provide a reliable index of nutrient bioavailability [7].

In contrast to soil tests, foliar nutrient analysis can be used to provide insight into a plant's response to fertilization. It is a measure of plant's nutritional status and therefore reflects nutrient pools that are available for plant growth [8]. Leaves constitute the dominant sink for N in plants during the active growth period. At the end of the growing season, a significant part of the leaf N is withdrawn and stored in perennial tissues. The stored N is remobilized in the following spring and used for growth of new shoots [9, 10]. Phosphorus behaves in a manner similar to that of N, as a significant part of the leaf P is withdrawn before leaf abscission [11], as is potassium [12].

The seasonal dynamics of N and other macronutrients has been studied in willow biomass crops [9, 13, 14]. However, most of these studies were carried out in a controlled environment, either in the laboratory or as pot experiments in a green house. There is only limited information on the seasonal dynamics of foliar nutrient concentrations in short rotation willow biomass production systems under field conditions [15, 16]. No studies integrate the seasonal variations in soil nutrient supply and availability with foliar nutrient dynamics of SRWC systems under field conditions in organic and inorganic amended soils.

A better understanding of nutrient supply, bioavailability, and uptake over the entire growing season in SRWC systems fertilized with organic waste and commercial fertilizer is important since the timings of nutrient release and crop uptake are not always synchronized. The aim of this study is to investigate the seasonal dynamics of soil supply and bioavailability, as well as foliar concentrations of N, P, and K in a willow biomass production system that has received varying rates of organic amendments and commercial fertilizer. The specific objectives of this study are to

- (1) compare the effects of varying rates of organic and inorganic fertilizers on soil N, P, and K supply and bioavailability,
- (2) determine the effects of varying rates of organic and inorganic fertilizers on foliar N, P, and K concentrations as an indication of foliar nutrient response to fertilization,
- (3) assess the seasonal dynamics in the supply and bioavailability of soil N, P, and K and foliar concentrations in a shrub willow *Salix miyabeana* (SX64).

2. Materials and Methods

2.1. Site Description and Preparation. This study was conducted in Delhi, NY ($42^{\circ} 15' 8.65''$ N, $74^{\circ} 56' 22.03''$ W), on

a field that had previously been used for agricultural purposes but had been left fallow for about five years prior to the establishment of the research plots. The soil at the site is a Basher silt loam (mesic Fluvaquentic Dystrudepts) with a loamy alluvium parent material derived from acid, reddish sandstone, siltstone, and shale. The soil is moderately well drained with a slope of 0–3%.

Site preparation was done using a combination of mechanical and chemical treatments in the autumn of 2006. A contact herbicide, glyphosate, was sprayed at the rate of $2.3 \text{ kg a.i. ha}^{-1}$ followed by plowing and disking. Unrooted cuttings, about 20 cm in length, of the shrub willow cultivar *Salix miyabeana* (SX64) were planted flush with the ground in the spring of 2007 using a step planter. About 15,000 cuttings ha^{-1} were planted in double rows (1.5 m and 0.75 m distance between and within rows, respectively, and 0.6 m between cuttings in a row) to accommodate future weeding, fertilization, and harvesting operations. Preemergent herbicide, simazine, was applied at the rate of $4.5 \text{ kg a.i. ha}^{-1}$ immediately after planting.

2.2. Characterization of Organic Materials Used and Soil at the Study Site. Prior to treatment application, soil samples were collected to characterize the initial soil properties and to determine if there were differences in the blocks that might bias posttreatment results. Samples were collected from the 0–15 and 15–30 cm layer at three locations across the diagonal of each block and composited. Soil samples were air dried and crushed to pass through a 2 mm sieve after stones, and visible roots and plant parts were removed. Soil chemical analyses were done at the Ag Analytical Lab of Penn State University. Total N was determined by combustion method [17]. Available P, K, Ca, and Mg were determined via inductively coupled plasma (ICP) following Mehlich 3 extraction. Cation exchange capacity (CEC) was determined by summation and organic matter content by loss on ignition [18] and soil pH was determined in water (1:2).

Samples of biosolid compost (BC) and digested dairy manure (DM) were stored at 4°C until analysis could be completed. Subsamples of BC and DM were oven dried at 105°C until mass loss ceased. The dried samples were ground in a Wiley mill to pass through a 1 mm mesh sieve and used for chemical analysis at the Ag Analytical Lab services of Penn State University using the same methods as above.

2.3. Treatments and Experimental Design. Experimental plots were set up to accommodate six fertilization treatments and a control in a randomized complete block design with three replications. Each treatment plot was comprised of three double rows and measured $7.92 \text{ m} \times 6.86 \text{ m}$. The middle double row within each treatment plot, measuring $4.27 \text{ m} \times 2.29 \text{ m}$, was used as measurement plot for data collection. The fertilization treatments comprising two rates (150 kg N ha^{-1} and 200 kg N ha^{-1}) of each of the three fertilizing materials were applied in June 2008. Urea (46-0-0) was used as commercial fertilizer (CF). The biosolid compost (BC) was sourced from the Delaware County Waste Treatment Plant (DCWTP) at Delhi, NY, and the digested dairy manure (DM)

TABLE 1: Precipitation temperature and gravimetric soil moisture content in the sampling periods when PRS probes were installed in shrub willow plots treated with organic wastes and commercial fertilizers.

Code	Burial period	Season	Total rainfall/month (mm)	Average temp./month (°C)	Gravimetric soil moisture content (θ_d)
Sept.	Sept. 24–Oct. 7, 2008	Fall	189.74	14.61	0.32
May	May 20–June 2, 2009	Spring	89.92	11.97	0.18
Aug.	August 10–23, 2009	Summer	198.4	19.6	0.39
Oct.	October 11–24, 2009	Fall	112.78	7.54	0.29

Temperature and precipitation data for the burial periods was downloaded from a nearby weather station: <http://www4.ncdc.noaa.gov/cgi-win/wwcgi.dll?wwDI~StnSrch~StnID~20019716>.

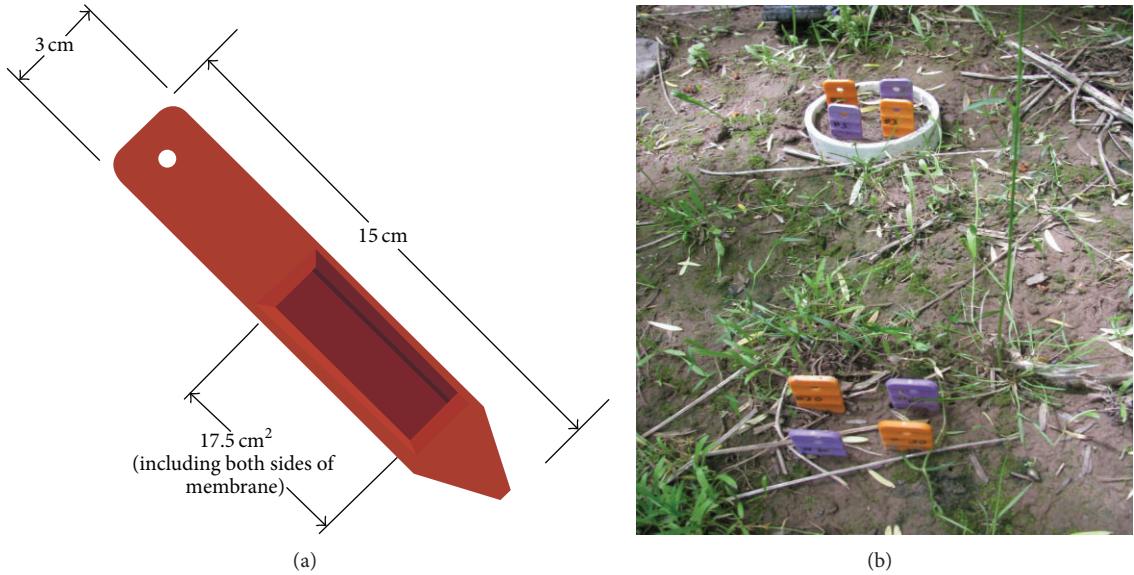


FIGURE 1: Dimensions of the PRS probe (a) and installation in the field (b). Two sets each of cation and anion probes were buried inside and outside RECs to measure soil nutrient supply and bioavailability, respectively, and in the double row and between the double rows in shrub willow biomass crops that had been amended with organic wastes and commercial fertilizer.

was sourced from AA Dairy Farms located in Candor, NY. The fertilizing materials were manually spread evenly over the treatment plots at the respective rates of application, using wheelbarrows and shovels.

2.4. Nutrient Supply and Bioavailability Measurement. Plant root simulator (PRS) probes manufactured by Western Ag Innovations Inc., Saskatoon, SK, Canada, were used to measure soil nutrient supply and bioavailability in the measurement plots. The PRS probes consist of a cation- or an anion-exchange resin membrane encased in a plastic holding device ($15 \times 3 \times 0.5$ cm, $L \times W \times H$). The probes were inserted vertically into the top 15 cm of the soil to expose the ion-exchange membrane surface (which measures 17.5 cm^2 , including both sides of the membrane) to the soil zone with the largest concentration of roots, microbial activity, and active biogeochemical processes. To measure the bioavailability of nutrients, four pairs each of cation and anion probes were installed outside of root exclusion cylinders (RECs). The same sets of probes were installed inside RECs to measure nutrient supply rate (Figure 1).

The probes were buried *in situ* for a period of two weeks in each of four sampling periods/seasons (Table 1). At the end of each burial period, the probes were removed from the soil, cleaned, and sent to Western Ag Innovations for extraction and analysis. The set of probes within each treatment plot at each burial spot were combined, much like a composite soil sample. This helped to account for any microscale variability. Nutrient accumulation rates are expressed as microgram (μg) of nutrient absorbed per 10 cm^2 per 2 weeks (i.e., $\mu\text{g } 10 \text{ cm}^2 2 \text{ wk}^{-1}$) and are used as a measure of nutrient supply and bioavailability. At probe retrieval, soil samples were collected in triplicate at spots near the points where the probes were installed and then composited and used to determine gravimetric soil water content by drying subsamples at 105°C . Temperature and precipitation data for the burial periods (Table 1) were downloaded from a nearby NOAA weather station [19].

2.5. Foliage Sampling and Analysis. Foliar analysis was used to determine if nutrient supply rate and uptake reflect the foliage nutrient concentrations and also to assess the nutrient

TABLE 2: Initial soil characteristics (mean and standard error) before organic wastes and commercial fertilizer were applied to shrub willow biomass crops.

Depth	N	P	K	Mg	Ca
	g kg ⁻¹		mg kg ⁻¹		
0–15	1.8 (0.1)	17.9 (3.8)	62.3 (21.9)	92.3 (2.9)	728.9 (29.2)
15–30	1.3 (0.2)	13.9 (3.3)	24.0 (7.8)	90.7 (15.0)	852.0 (138.3)
Depth	S	OM	pH	CEC	EC
	mg kg ⁻¹	%		meq/100 g	mS/cm
0–15	22.2 (1.1)	9.1 (3.0)	5.4 (0.1)	14.1 (1.1)	3.9 (1.6)
15–30	26.0 (7.8)	4.4 (1.3)	5.7 (0.1)	12.4 (1.7)	3.6 (1.8)

status of the plants. At the time of probe retrieval exactly 100 fully expanded leaves in the third quarter from the bottom of the plant canopy were sampled per plot. The foliage samples were oven dried at 60°C to constant mass and then ground in a Wiley mill to pass through a 1 mm mesh sieve and used for chemical analysis using the same methods as indicated above.

2.6. Statistical Analysis. Effects of the fertilization and sampling time (seasons) on soil nutrient supply and bioavailability as well as foliar nutrient concentrations measured in the various burial periods (seasons) were analyzed using analysis of variance with repeated measures (ANOVAR). The PROC MIX procedure in SAS was used. Pearson correlation coefficients were used to examine the relationship between soil moisture and the supply and bioavailability of soil N, P, and K and the correlation between soil availability and foliar concentrations of N, P, and K and amongst the nutrient elements in soil and foliage. Statistically significant differences are reported at $\alpha = 0.05$. All statistical analyses were done using SAS [20].

3. Results

3.1. Characteristics of Soil and Organic Materials. Analysis of the soil properties between blocks prior to treatment application did not show any significant differences that could bias the posttreatment results. This pretreatment analysis indicates that the nutrient levels were typical for the type of soil in the region (Table 2). Between the organic materials, biosolid compost showed significantly higher concentrations of most nutrient elements than the manure with the exception of K. Manure K concentration was significantly higher than that of biosolid compost (Table 3).

3.2. Temperature, Rainfall, and Soil Moisture Content. The August sampling period had the highest soil moisture level ($0.39 \theta_d$) as a result of the high total amount of rainfall received in that month (198.40 mm). Average air temperature was also higher in August (19.6°C) than in other sampling periods. Rainfall was lowest in May (89.92 mm) and thus the lowest soil moisture content ($0.18 \theta_d$). The similarity in the patterns of monthly rainfall and soil moisture content indicates that the total amount of rainfall received per month influenced soil moisture content (Table 1).

3.3. Seasonal Patterns in Soil Supply and Bioavailability of N, P, and K. Nitrogen (NO_3^- and NH_4^+) ions released into the soil solution exhibited significant seasonal patterns. Supply and bioavailability of NO_3^- were both higher in May than in August and October (Figure 2(a)). The index of NO_3^- uptake (supply rate-bioavailability) followed the same seasonal patterns as its supply rate; it was highest in May and declined in August. Supply and bioavailability of NH_4^+ followed a different seasonal pattern. While supply rate was higher in August than in October, bioavailability was higher in October than in May (Figure 2(b)). The uptake index of NH_4^+ was greatest in May, followed by August. In October, NH_4^+ ion accumulation on the probes outside the REC (bioavailability) exceeded accumulation on the probes inside the REC (supply rate). Consequently, the NH_4^+ uptake index was negative (Figure 2(b)). In general, NO_3^- was the dominant form of N in the soil, with ratio of $\text{NO}_3^- : \text{NH}_4^+$ ranging from 7:1 to 22:1 measured inside the RECs and 3:1 to 15:1 measured outside the RECs.

The supply rate, bioavailability, and uptake index of P and K also exhibited significant seasonal variability. Both supply and bioavailability of P increased significantly in May and August and then decreased in September and October (Figure 2(c)). The P uptake index was negative in all seasons because bioavailability exceeded supply rate.

The uptake index for K was positive only in May because its supply was significantly higher than bioavailability but the supply decreased below bioavailability levels in August and October. Though K bioavailability was also high in the spring and decreased in the summer and autumn, the decrease in supply rate in the summer and autumn was greater than the decrease in bioavailability. As a result, K uptake index was negative in the summer and autumn (Figure 2(d)).

3.4. Effect of Fertilization on Soil Supply and Bioavailability of N, P, and K. The fertilization treatments had no statistical significant effect on soil N and K supply and bioavailability (Figures 3 and 4). There was also no significant treatment by season interaction effect on soil N and K supply and bioavailability. The patterns for P were different from N and K. The bioavailability of P was also not affected by fertilization but the BC1 treatment raised P supply rate significantly compared to CF1 across all seasons (Figure 4(b)). There was significant interaction effect on soil P supply rate and bioavailability. Supply and bioavailability of P in DM2 treatment were significantly higher in May than in October (Figure 4(a)). The application of DM2 treatment also increased P supply rate compared to CF1 in May (Figure 4(b)). With the exception of CF2 in August and October, the P uptake index was negative for all treatments in all seasons.

3.5. Seasonal Patterns in Foliar N, P, and K Concentrations. Foliar dry weight and concentrations of N and P exhibited significant seasonal patterns but foliar K concentration did not show any significant seasonal variability. Foliar dry weight was low at the beginning of the growing season. It increased as the season progressed and reached a maximum

TABLE 3: Nutrient content (mean and standard errors) of the biosolid compost and manure used as organic amendments for shrub willow biomass crops.

Organic material	Total N	Org N	NH_4^+	P	K	Ca	Mg	S
	g kg^{-1}							
Biosolid	18.1 [†] (0.24)	17.8 [†] (0.24)	0.3 (0.01)	14.0 [‡] (0.48)	6.2 (0.07)	36.3 [‡] (1.12)	3.5 (0.15)	6.3 [‡] (0.13)
Manure	15.4 (1.39)	15.2 (1.45)	0.2 (0.12)	6.0 (0.39)	8.2 [‡] (0.69)	10.1 (0.59)	3.5 (0.18)	3.3 (0.17)

[†]Significant difference between biosolids and manure at alpha level ($P < 0.05$); [‡]significant at alpha level ($P < 0.01$).

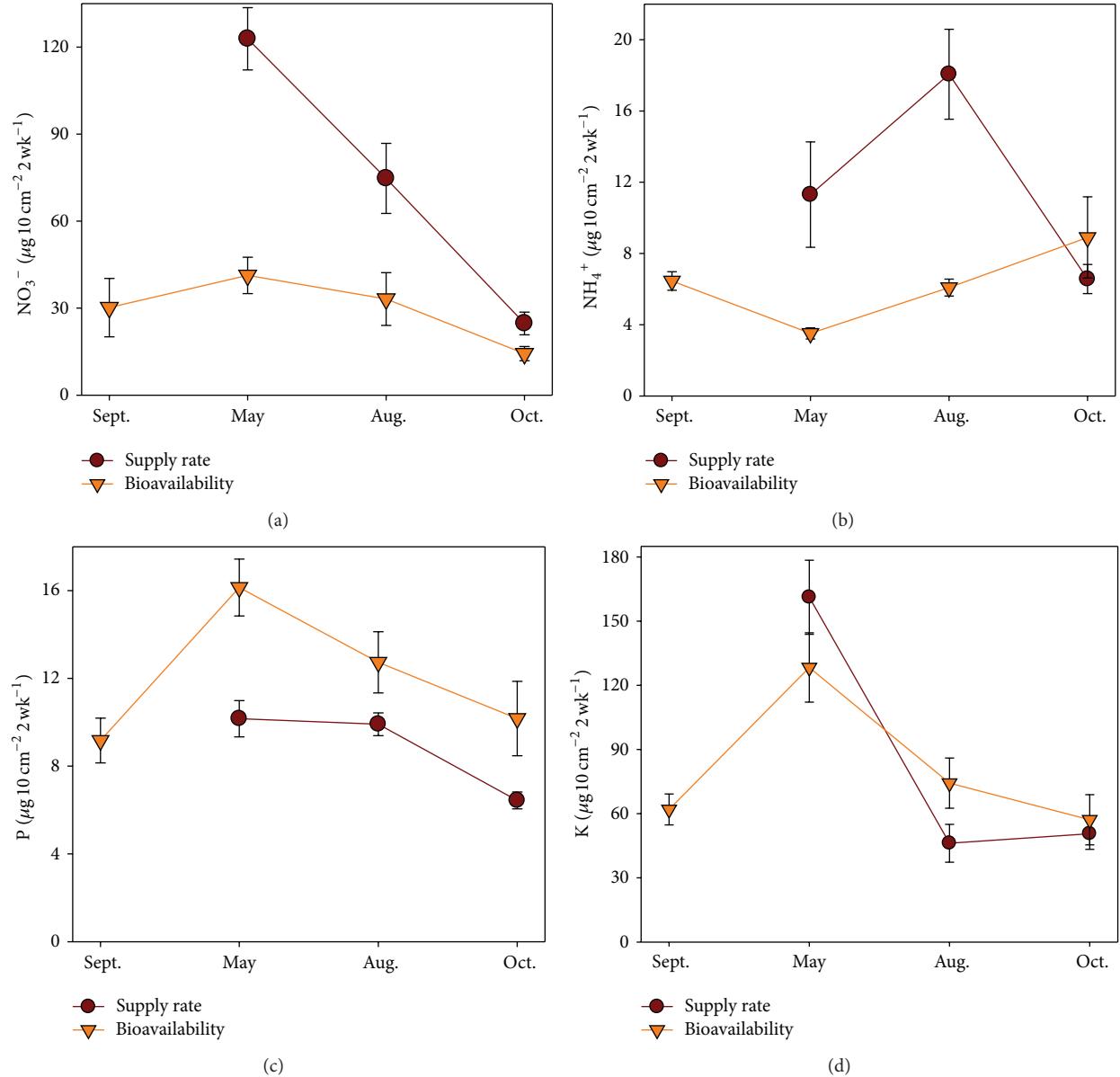


FIGURE 2: Seasonal dynamics in soil nutrient supply rate and bioavailability (mean and standard error) of four different compounds ((a) NO_3^- , (b) NH_4^+ , (c) P, and (d) K) in a shrub willow (*S. miyabeana* SX64) biomass crops that had been amended with organic wastes and commercial fertilizer.

in August before decreasing again towards the end of the growing season (Figure 5). Foliar N concentration was also significantly higher in May but decreased as leaf development progressed into the summer and rose again in the fall (Figure 5). Foliar P concentration was high in May, when

soil moisture content was low and leaf development had just begun. It then decreased to a minimum in August as leaf development progressed. However, towards the end of the growing season in October, foliar P concentration increased again (Figure 5).

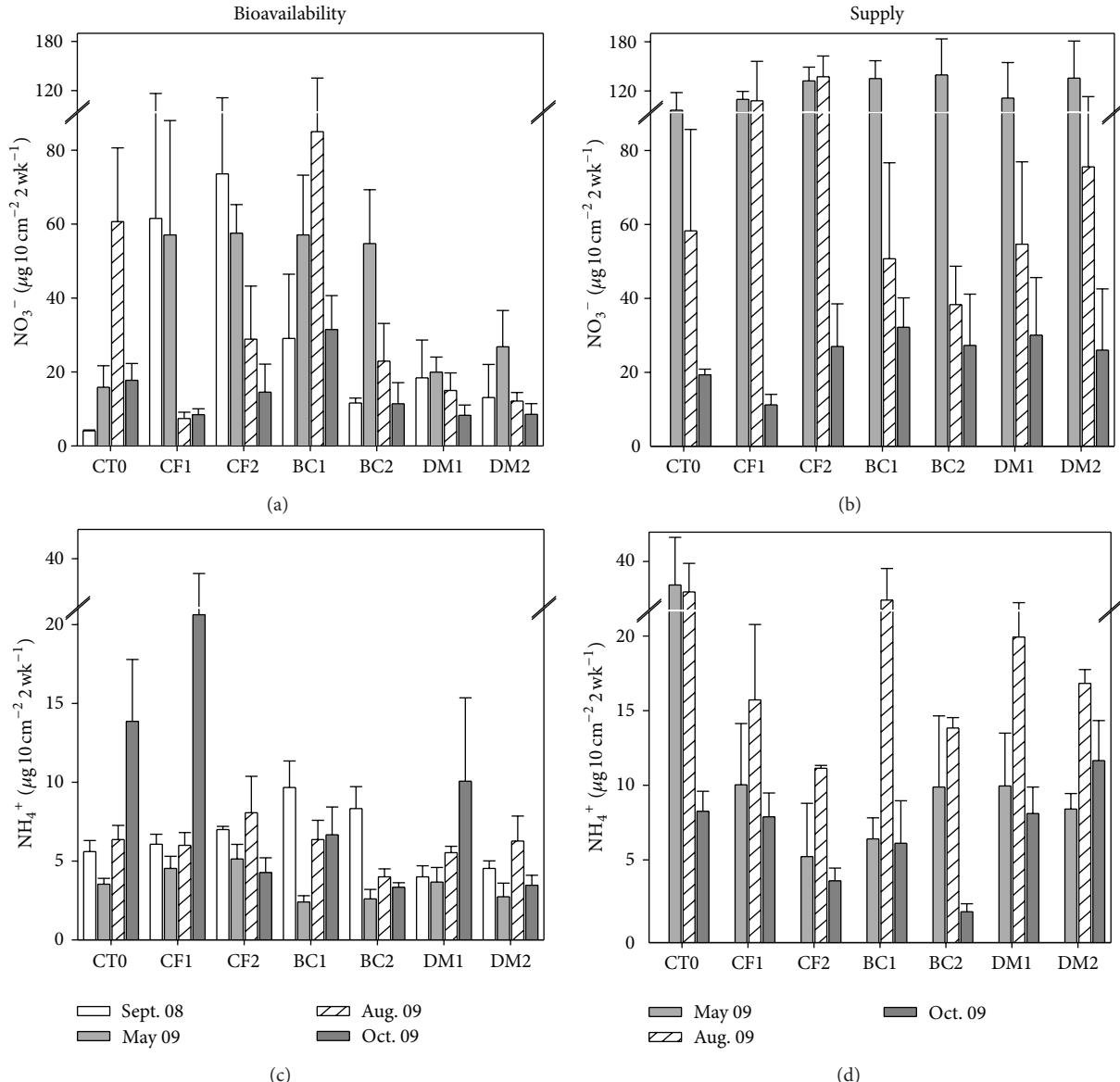


FIGURE 3: Soil N (NO₃⁻ and NH₄⁺) bioavailability and supply rate (mean and standard error) in four sampling periods (seasons) of a shrub willow (*S. miyabeana* SX64) field that has been treated with two rates each of commercial fertilizer (CF), biosolid compost (BC), and dairy manure (DM). CT0 is unfertilized control.

3.6. Effect of Fertilization on Foliar N, P, and K Concentrations. There was no significant fertilization effect on foliar dry weight and N, P, and K concentrations ($P > 0.70$). There was only an interaction (treatment \times season) effect on foliar N concentration ($P = 0.05$). Generally, this effect resembled more of a seasonal effect than a combination of treatment and seasonal effect. For example, foliar N concentration of the unfertilized plants was significantly higher in May than in September (Figure 6). It was also higher in all the fertilized plots in October than in August with the exception of DM1. Generally, foliar N concentration was significantly higher in May than in August for all treatments including CT0 (Figure 6).

4. Discussion

The seasonal variability in soil moisture appeared to be an important determinant in the seasonal dynamics of soil nutrient supply and bioavailability as well as foliar nutrient concentrations. The effect of soil moisture on plant nutrient supply and availability has been reported elsewhere [21]. Nitrate is soluble in the soil solution and is therefore drawn to the root through mass flow of water. Conversely, NH₄⁺, P, and K are primarily moved in the soil by diffusion [22, 23].

The characteristics of organic materials, particularly C : N ratio, determine its behavior in the soil, as well as the amount of N that becomes available through mineralization [1].

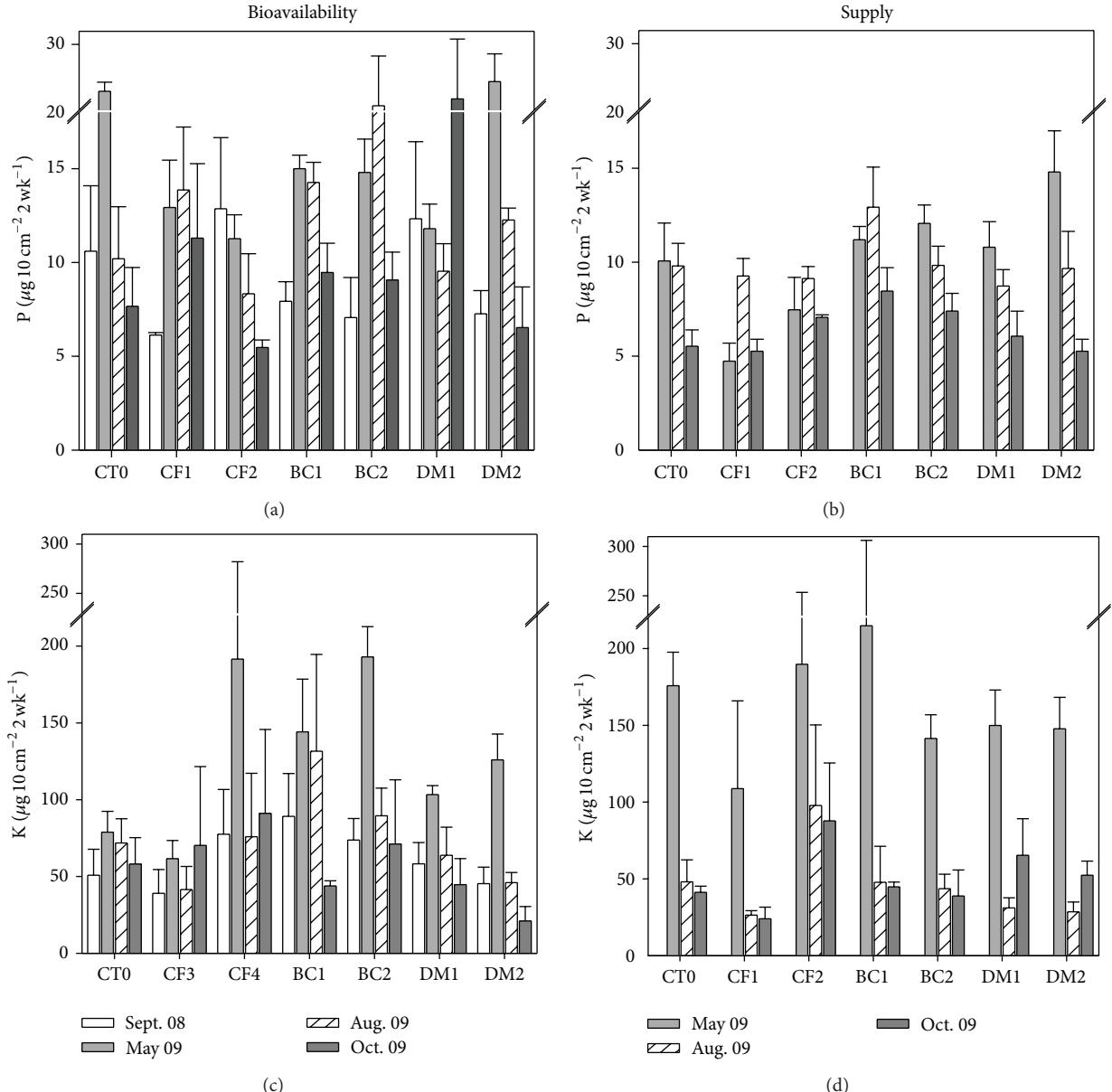


FIGURE 4: Soil P and K bioavailability and supply (mean and standard error) in four sampling periods (seasons) of a shrub willow (*S. miyabeana* SX64) field that has been treated with two rates each of commercial fertilizer (CF), biosolid compost (BC), and dairy manure (DM). CT0 is unfertilized control.

The organic materials used in this study contained an appreciable amount of macronutrients. The biosolid compost had significantly higher levels of N and P compared to the manure (Table 3) and was therefore expected to enrich the soil with these nutrients. The soil pH and CEC values indicate that the soil is moderately acidic and has good availability of basic cations. The characteristics of the fertilizing materials and the inherent soil properties are important factors to be considered when interpreting the results observed in this study. With a bulk density of 1.25 g cm^{-3} and soil N content of 1.8 g kg^{-1} , the total N content of the top 15 cm soil is about $3,375 \text{ kg N ha}^{-1}$. The addition of 200 kg N ha^{-1} is only

an increase of 6%. When this is added in organic amendments with most of the N being tied up initially, it can mask the response to fertilization. Even with the commercial fertilizer, where more of the N is available over a shorter time frame, the amount of N available in the soil may overwhelm the input.

4.1. Seasonal Dynamics in Soil Supply and Bioavailability of N, P, and K. In this study, maximum NO_3^- supply rate and bioavailability occurred in May while minimum NO_3^- supply rate and bioavailability occurred in October. The NO_3^- uptake index was also high in May, indicating that even the one-year-old, above-ground willow biomass crops

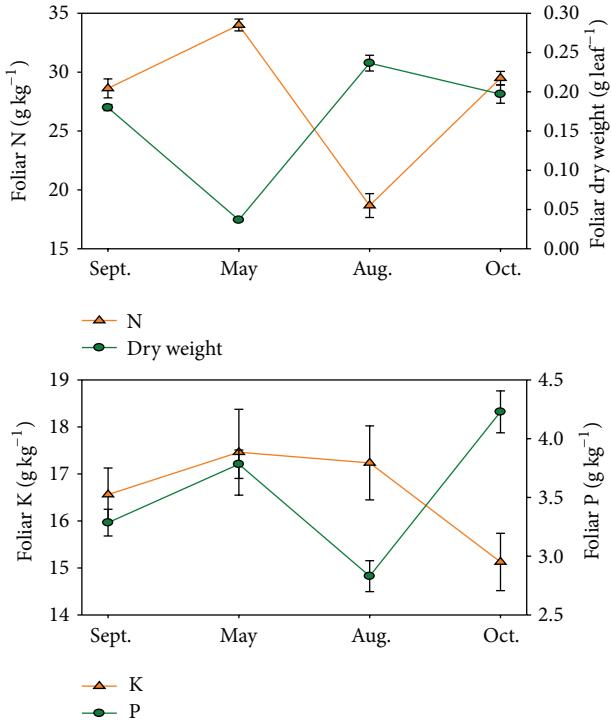


FIGURE 5: Seasonal dynamics in foliar dry weight and N, P, and K concentrations (mean and standard error) of a shrub willow (*S. miyabeana* SX64) crop that has been fertilized with organic and inorganic N sources.

are able to modulate the high supply of NO_3^- in the field in spring. This observation agrees with the findings of Mitchell et al. [21] who observed high NO_3^- concentration and flux in spring and low NO_3^- flux in summer and autumn. On the contrary, Huang and Schoenau [24] reported minimal soil NO_3^- supply in May and maximum supply later in the growing season in an aspen forest in Saskatchewan, Canada. Unlike the findings of Mitchell et al. [21], the decline in NO_3^- supply rate in August in this study cannot be attributed to vegetation uptake alone because the index of NO_3^- uptake was also higher in May than in August and October. Soil NH_4^+ supply rate was highest in August and lowest in October, while bioavailability was highest in October and lowest in May. The relatively higher NH_4^+ observed in August than in May could perhaps be due to inhibition of nitrification and high ammonification rates in the presence of favorable soil moisture and temperature in the summer. The negative correlations between NO_3^- and NH_4^+ supply and bioavailability support the existence of this condition in the soil. Similar observation was made by Casals et al. [17] who reported that mineral N uptake followed a seasonal pattern that matched N supply rate.

The supply rate and bioavailability of P in this study were high in May. On the contrary, Zhao et al. [25] found greater mineral P in the summer and attributed it to the combined effect of the high temperature and moisture which promote the biological transformations of soil P and lead to increased availability of inorganic P in the soil. However, other researchers have observed greater P accumulation

during autumn than in spring and summer [24, 26] and ascribed it to the greater plant uptake and increased biological activity in the soil.

In this study, phosphate ion supply rate was lower than bioavailability. Consequently, the P uptake index was negative across all seasons and treatments. Though P uptake could be low at this site, biologically, it cannot be negative. The low P supply rate could therefore be attributed to microbial immobilization due to disturbance caused by the installation of the cylinders. Inserting the PVC into the soil could have caused some root damage and lead to root exudation and elevated microbial activities [26]. A second possible factor that could contribute to this observation is soil moisture. The soil inside the REC could be drier relative to that outside and could thus affect P mineralization resulting in a lower P supply rate inside the REC than outside. P mineralization is also known to be higher in the presence of plant roots, because organic P from root exudates may be mineralized to an inorganic form [27]. As an anion, phosphate participates in outer sphere reactions and its release can be a function of root chemistry [23]. In fact, the two ways by which inorganic P is released into soil solution—ligand exchange and congruent dissolution—both require the chemical action of plant roots [23]. Soil P availability is controlled by the biogeochemical transformation of the different forms of organic and inorganic P in the soil [26]. Particularly, the mineralization-immobilization of organic P is strongly influenced by seasonal variations in temperature, moisture, plant growth, and root activity and by organic matter [10, 25]. Soil P availability is particularly sensitive to soil moisture regimes, not only because of the indirect impacts through microbial activity, but also because of the movement of phosphate ions in soils by diffusion through pores filled with water.

Soil K supply rate was higher in May than in other sampling periods and showed no particular pattern with soil moisture content. The negative uptake index for K in August and October could be attributed to microbial uptake, low soil water in the RECs, and the effects of roots exudates which could result in lower K supply than bioavailability [27].

4.2. Fertilization Effects on Soil Supply and Bioavailability of N, P, and K. Neither the fertilization treatments nor their interactions with the seasons had any significant effects on soil NO_3^- and NH_4^+ supply rate compared to unfertilized plots. This lack of treatment effect may be related to internal nutrient cycling, especially when high nutrient containing leaves remain on site [28]. Internal cycling of nutrients from litter and fine roots decomposition can reduce the demand for the addition of external nutrients [29]. For example, Adegbidi [30] reported that three-year-old unfertilized *S. dasyclados* (SV1) added $80.3 \text{ kg N ha}^{-1} \text{ yr}^{-1}$ to the soil via litter fall. Rytter [31] found that $34\text{--}69 \text{ kg N ha}^{-1} \text{ yr}^{-1}$ was recycled back into the soil from three-year-old willows due to rapid turnover rates of fine roots. In addition, this site is in a flood plain and it flooded during this trial which brought additional nutrients to this site, which may have masked some of the nutrient addition in the organic amendments.

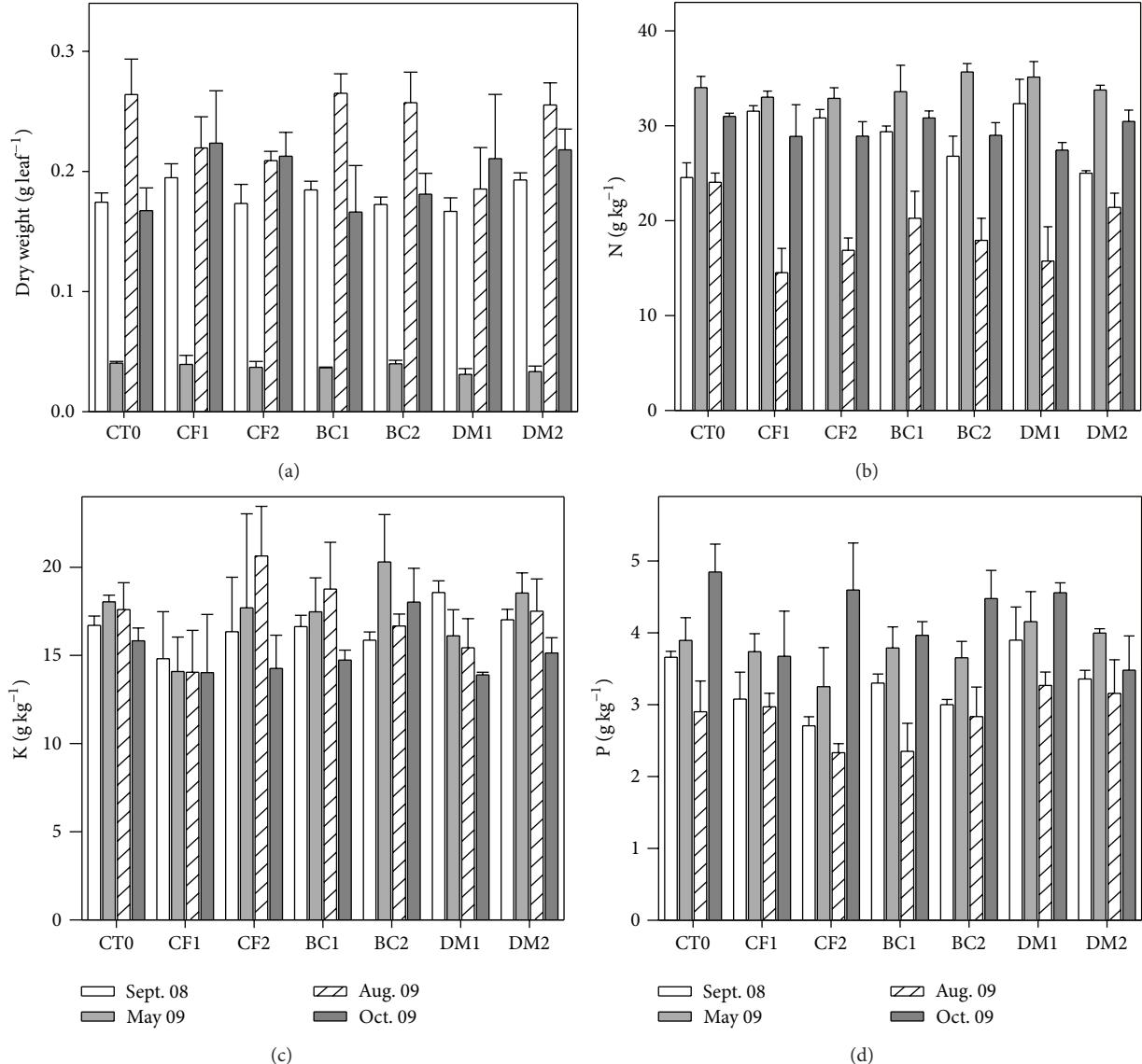


FIGURE 6: Foliar dry weight and N, P, and K concentrations (mean and standard error) in four sampling periods (seasons) of a shrub willow (*S. miyabeana* SX64) field that has been treated with two rates each of commercial fertilizer (CF), biosolid compost (BC), and dairy manure (DM). CT0 is unfertilized control.

The biosolid compost used in this study had twice the P concentration present in the manure. Urea contains only N and no other nutrient element. The significantly increased soil P supply rate in the organic amended plots compared to the urea treated plots was therefore expected. The contribution to mineralizable organic P content by the organic materials may have led to an increase in the P supply power in the organic amended plots. Organic matter and litter decomposition play an important role in soil P availability [22, 26]. Zhao et al. [25] also reported that P release from litter decomposition is generally higher than from the mineral soil. The increased P supply by the application of the organic wastes in this study could therefore be attributed to the decomposition of the organic matter added to the soil through the application of these materials.

Unlike N, P release showed a more variable pattern. The low values observed in September and May in the fertilized plots could be due to an initial P immobilization. Greater P mineralization occurred at later sampling periods after treatment application (August and October) as exhibited by high P levels in the organic amended plots relative to the urea treated plots. This initial immobilization followed by mineralization could be attributed to the influence of other nutrient elements such as N on P mineralization. The CF2 treatment increased soil K supply. Similarly, Hangs et al. [32] also reported an increase in soil K supply after fertilizer N addition. This may be explained by ammonium ions derived from the urea fertilizer which could displace potassium ions from exchange sites on the surface and interlayer of clay minerals into soil solution.

4.3. Seasonal Dynamics of Foliar Nutrients. The decrease in foliar N concentration between May and August was associated with increased foliar dry weight as the season progressed. This rapid increase in foliar dry weight caused dilution of N in the leaves. High foliar N concentration has been observed in deciduous trees in the spring followed by a decrease in foliar N as leaf development progressed [33]. The increase in foliar N concentration from August to October in this study is contrary to the observation made by von Fircks et al. [14]. They reported a rapid decrease in foliar N concentration of willow leaves from August to October. The higher foliar N concentration in May corresponded with the increased soil NO_3^- ion supply rate during the same period. This is consistent with NO_3^- being the predominant form of inorganic N in the soil and readily available N form for plant uptake. Secondly, it reveals that the release of plant available N from the soil N pool via mineralization in this study can be considered sufficient for normal plant growth. Thirdly, this common seasonality in soil NO_3^- supply and foliar N concentration indicates that the biogeochemical processes that influence soil N mineralization and supply rate are also determining factors in willow N uptake. Last and most importantly, it reveals the willows' ability to utilize the high soil NO_3^- in the spring, which could otherwise be leached and lost into groundwater.

Like N, foliar P concentration was also lowest in August when soil moisture and foliar dry weight were both high. Both N and P are mobile nutrient elements in plants [33] and are generally regarded as being in limited supply in most ecosystems. In other studies, P and N concentrations in leaves have been found to exhibit somewhat similar trends during the growing season [14, 33]. During the period of rapid leaf expansion in spring, both N and P exhibit increased concentrations. Though foliar P concentration in this study was relatively high in May and agrees with the above literature [14, 33], maximum foliar P concentration was observed in October, which is contrary to observation made in the literature. Foliar P concentrations in willow plants have been reported to decrease in autumn as a result of P withdrawal from the leaf prior to leaf abscission [11, 14]. The high foliar P concentration observed in this study towards the end of the growing season could be attributed to the early immobilization and late mineralization of P in the soil. The minimum foliar P concentration observed in August could also be attributed to leaching of foliar P due to the high rainfall amount received in that month. Foliar P leaching has been found to range between 5 and 15% and thus can be regarded as a major factor influencing foliage P concentration [33].

Foliar K concentrations in this study were fairly stable from September through August but decreased in October. This observation was expected because K is a mobile element in plants and thus it withdraws from the leaf before abscission [12, 14]. Like foliar N, maximum K concentration occurred in May (spring) when there was rapid accumulation in the newly formed leaves. Contrary to the results of this study, low foliar K concentrations of deciduous trees in the spring were reported by Grizzard et al. [33]. The readily available K in

the soil and its high degree of mobility within the plants probably influenced its seasonal trend more than the biogeochemical processes.

4.4. Response of Foliar Nutrient Concentrations to Fertilization. Foliar nutrient concentrations have been used as an indicator of tree response to fertilization, because it is a function of available nutrients as well as the factors that influence nutrient uptake by plants [34]. In this study, foliar nutrient concentrations did not show any significant response to the fertilization. In September, plants in the fertilization treatments had high foliar N concentration relative to the unfertilized plants. However, in the May–October sampling periods, the fertilization treatments depressed foliar N relative to CT0 with the exception of BC2 and DM1 in August. This pattern of foliar N concentration with respect to fertilization is the same as observed for soil NO_3^- and therefore portrays the synchrony between soil N supply and plant N uptake.

With the exception of the DM treatments in September, the fertilization regime depressed foliar P and K concentrations relative to CT0. Similarly, Jug et al. [35] observed reduction in foliar P concentration relative to control plots after applying N fertilizer to *S. viminalis* and attributed their observation to dilution effect due to larger leaves. This does not appear to be true in this study. It is however important to note that the reduction in foliar P by the fertilization treatments did not cause any increase in the foliar N : P ratio.

This lack of response of foliar nutrient concentrations to fertilization has been observed in an earlier study where *S. dasyclados* (SV1) was fertilized with paper sludge, manure, and urea [36]. Similar results have also been reported in other willow varieties that were fertilized with waste water in Quebec [37]. The nonresponsiveness of willow foliar nutrients to the applied fertilization materials in these different studies has been attributed to the relatively high internal nutrient cycling at the sites [36–38], which is also true for the site used for this study. Significant increases in willow foliar nutrient concentrations due to the application of fertilizer and organic amendments have been reported elsewhere [15, 37, 38], but it is evident from the literature that where plants can readily access internally supplied nutrients, they do not show growth response to applied fertilizer [39].

The foliar nutrient levels measured in this study reached or even exceeded values reported for optimal growth of field grown *Salix* species [13, 35, 39, 40]. Foliar N concentrations in this study fall within the 23–30 g kg⁻¹ reported by van den Burg [40] as required for normal willow growth. In August, however, the fertilization treatments showed mean foliar N concentrations of less than 23 g kg⁻¹ which could be considered suboptimal for normal growth. However, since the decline in foliar N in August may be attributable to the dilution effect, the actual N content of the plant was not affected. Previous studies have shown that this particular shrub willow cultivar (SX64) has high biomass production but falls in a group with significantly lower foliar N concentration than other varieties of willow with high biomass production [41] suggesting that this cultivar is more

effective at N use than other cultivars. Foliar P concentrations are above the range recommended for normal growth [40] and can be considered sufficient for optimal growth. The P levels are also higher than values reported by Labrecque and Teodorescu [39] and are comparable to values reported by Jug et al. [35] and Rytter and Ericsson [13] for various field grown willow clones. Values of foliar K concentrations are sufficient for normal growth and compared well with values reported by van den Burg [40], Labrecque and Teodorescu [39], and Rytter and Ericsson [13].

5. Conclusions

Both the supply rate and uptake index of NO_3^- were high in the spring indicating a synchrony of NO_3^- supply and plant uptake. This reveals the ability of the willow biomass crop to retain the high soil NO_3^- in the spring to minimize leaching out of the root zone into groundwater. The lack of a fertilization effect on soil N supply is related to the inherently high internal nutrient cycling of soil at the site. The P-rich biosolid compost increased soil P supply rate, but the digested manure which had a lower P loading rate did not have any impact on P supply rate. Although foliar nutrient concentrations in this study did not show any significant response to the fertilization treatments, they reached or even exceeded values reported for optimal growth of field grown *Salix* species. This nonresponsiveness of willow foliar nutrient to fertilization could be attributed to high internal nutrient cycling at the site. The significant effect of the organic amendments on soil P supply rate and their comparable effects on foliar nutrient concentrations make them a better fertilization option for SRWC systems considering the cost of commercial fertilizers and the added environmental benefits of land application of organic wastes. The common seasonal patterns and the significant correlations observed between soil moisture content and soil and foliar nutrient elements show the importance of the biogeochemical processes on soil supply and plant uptake of nutrients.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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Research Article

N Mineralisation from Bioresources Incubated at 12.5°C

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Soils treated with lime-amended biosolids (LAB), poppy seed waste (PSW), anaerobically digested biosolids (ADB) and poppy mulch (PM) and incubated at 12.5°C for 56 days released 45%, 36%, 25%, and -8%, respectively, of total applied N as plant available nitrogen (PAN) by the end of the incubation. The mineralisation rates were contrary to expectations based on the C:N ratios of the four products: LAB (5:1), PSW (7:1), ADB (3:1), and PM (16:1). PM showed a significant negative priming effect over the incubation period. These results have implications for production agriculture in temperate regions where application and incorporation of bio-resources traditionally occurs in autumn and spring when soil and air temperatures are relatively low. Current application times may not be suitable for nitrogen release to satisfy crop demand.

1. Introduction

The mineralisation and nitrification of nitrogen (N) in soil and in bioresources applied to soil depend on temperature and moisture [1–3]. In Australian cool-temperate climates, soil preparation for cropland, including application and incorporation of bioresources (e.g., composts, sewage sludge, and processing waste material), traditionally occurs in autumn and spring when average air temperatures range between 8 and 15°C and average soil temperatures range between 9 and 20°C (<http://www.bom.gov.au>). However, because crops in such climates are often not sown until spring, nitrogen release from the bioresources in these time periods may not be aligned with crop demand which is mostly in late spring or early summer, thus providing potential for either N loss (from the bioresources) or nutrient deficiency (of the crop).

Bergström and Brink [4] emphasised the importance of application rate and timing of inorganic fertilisers being calculated to meet crop demand, with new techniques such as organic resin coatings used to slow down the release of elemental N [5, 6]. Furthermore, stewardship programmes have been found to have a positive impact on water quality

by preventing soluble nutrient losses through leaching or overland flow from agriculture [7].

Incubation experiments to investigate N mineralisation of various soil-applied bioresources have been conducted by Flavel and Murphy [8], Burgos et al. [9], and Hsieh and Huang [10]. The incubation temperatures (and times) used for the amended soils were different for each study (15°C (142 days), 28°C (280 days), and 30°C (336 days), resp.). Although these studies were conducted for periods between 20 and 48 weeks, most changes occurred within the first 4 weeks following incorporation. N mineralisation studies conducted specifically on biosolids-amended soil have been incubated at 25°C [11, 12] and 20°C [13] with Smith et al. [12] concluding that biosolids type, soil temperature, and time from incorporation are dominant factors in determining N-release rate and nitrate formation.

Few studies have been conducted at soil temperatures typical of spring and autumn in temperate climates [14]. Furthermore, the Q10 principle, as described by Silvia and Machado [3], may not be adequate to predict mineralisation rates of bioresources at lower temperatures. Ågren and Bosatta [15] have suggested that soil organic matter (SOM) in cold climate soils mineralises faster when exposed to

warmer temperatures than it does in warm climate soils where the SOM is much more resistant to change. However, introduced organic matter from a bioresource may alter this temperature effect on SOM mineralisation because of the overall changes in chemical and physical soil characteristics from incorporation of external material. This suggests that a seasonal appropriate temperature is required for incubation studies to emulate field conditions.

Organic materials such as animal manures, crop residues, composts, and sewage sludge have been used in agriculture since cultivation of crops began, to supply plant nutrients and improve soil properties. Traditional agriculture in India and China has always considered these products as part of the farming system and a natural cycling of nutrients [16]. However, most developed nations have regarded agricultural residues and by-products of urbanisation and industrialisation as waste products for disposal. Therefore, amendment availability and logistical limitations have often determined application timing and rate for agricultural use rather than the demand for nutrients and organic matter [17]. If there is to be a change from conventional inorganic fertiliser inputs to organic material amendments, or a fusion of the two, to increase or maintain soil organic matter, the products and mechanisms of nutrient release from organic amendments within the soil matrix need to be understood.

In Tasmania, Australia, biosolids, poppy mulch, and poppy seed waste are three organic matter products produced in sufficient quantity for application to agricultural land. Biosolids are by-products from the treatment of urban sewage, poppy mulch is the by-product of alkaloid production, and poppy seed waste is the residue from poppy seed oil production. Although the annual state production of biosolids is by far the largest (about 40 000 wet tonnes), poppy mulch (10 000 wet tonnes) and poppy seed waste (5 000 wet tonnes) also contribute significantly to the overall organic matter resource available in the state. Ives et al. [18] conducted 2-year field trials with these materials, assessing soil characteristic and plant growth changes in response to their application in both incorporated and unincorporated crop production systems (to reflect minimum and no-tillage cropping situations). The results showed no significant difference in crop yields, grain total N, and postcrop soil NO_3^- over two growing seasons between incorporating and not incorporating lime-amended biosolids. However, it must be noted that while surface applied bioresources may be used in minimum and no-tillage cropping systems and not incorporated by cultivation, the planting operation provides some form of incorporation and/or mixing with the topsoil. Either way, the timing and availability of N from applying these bioresources under temperate soil/climatic conditions require further investigation.

The objectives of this study were as follows:

- (i) to quantify the rate of N release from poppy mulch (PM), poppy seed waste (PSW), lime-amended biosolids (LAB), and anaerobically digested biosolids (ADB) when mixed with a sandy loam soil at a temperature typical of autumn and spring in a temperate climate,

- (ii) to determine the peak mineralisation periods of the different products that may be used to influence application timing to match crop demand,
- (iii) to determine the effect of CaCO_3 in LAB on N release.

2. Methods and Materials

An incubation study was undertaken in a growth chamber over 56 days at 12.5°C. This temperature was selected based on a calculated average air temperature obtained from <http://www.bom.gov.au/climate/averages/> for five sites in the cropping regions of Tasmania, Australia (Cressy, Cambridge, Campbell Town, Ross, and Palmerston) for autumn and spring. A randomised complete block design with three replicates was used. Treatments included control (unamended), LAB, ADB, PM, and PSW. LAB was produced by Self's Point Wastewater Treatment Plant and ADB was produced by Macquarie Point Wastewater Treatment Plant, Hobart, both now managed under one authority, TasWater. PM was supplied by J. S. Aitken, Longford, and PSW was supplied by Rob and Kathy Henry, Woodrising Farms, Cressy. Two other treatments of NaNO_3 and NH_4Cl at 1% w/w soil were included for observing denitrification and N mineralisation, respectively [19]. A further control soil plus lime treatment (CaCO_3 at 4% of LAB wet rate) was used to determine the effect (if any) of additional calcium on the release of nitrogen in the absence of the biosolids treatment (i.e., LAB). Each replicate comprised seven samples for removal and analysis at days 0, 3, 7, 14, 28, 42, and 56.

Treatment preparation was derived from Smith et al. [12] with application rates based on treatments being incorporated in the soil to a depth of 10 cm at a wet weight equivalent rate of 7.5 dry solid (DS) t/ha, assuming a bulk density of 1 Mg m^{-3} . Although measured bulk density for this soil *in situ* was 1.4 Mg m^{-3} , the lesser value was used to reflect the state of soil immediately following cultivation. Soil to a depth of 10 cm was collected from an agricultural site near Cressy, Tasmania, sieved to < 4 mm and stored at 4°C. The soil had been previously classified as a Brown Sodosol [20]. The textual size (analysis undertaken by CSBP Soil and Plant Laboratory, Western Australia) for the trial soil was 51% fine sand, 20% coarse sand, 16% silt, and 13% clay, with an exchangeable Na percentage of 2.0. The gravimetric moisture content (GMC) of the soil at field capacity (FC) was determined using "Haines" apparatus [21] and calculated as 33%.

One and a half kilogram subsamples of field moist soil (20% GMC ≈ 61% FC) were spread loosely at an even thickness on 35 cm × 40 cm stainless steel trays. Each amendment was then evenly distributed over the soil samples at the required DS rate and mixed by hand using a broad spatula, turning the soil in a uniform motion. Both biosolids products were mixed into a slurry with 40 mL of distilled water before incorporating in the soil. A 40 mL aliquot of distilled water was added to all other treatments (including control) to ensure minimum soil water content of 70% field capacity at commencement of incubation. Subsamples (50 g each, seven for each replicate) were then placed in individual 125 mL

TABLE 1: Chemical characteristics of bioresources and soil.

	Units (DMB)	LAB	ADB	PM	PSW	Soil
Moisture	% (w/w)	70.1	80.3	55.1	10.8	20.0
pH (1:5 H ₂ O)		13	6.6	7.3	5.5	7.3
Organic C	% (w/w)	15.0	13.6	26.1	34.6	2.0
Soluble NH ₄ ⁺	mg/kg	1300	4300	8.6	46	<1.0
Soluble NO ₃ ⁻	mg/kg	1.7	1.2	<1.0	20	7.9
Soluble NO ₂ ⁻	mg/kg	1.2	<1.0	1.6	6	<1.0
Total N	% (w/w)	3.0	4.6	1.6	5.1	0.15
Total N _{AR} *	kg/ha	225	345	120	383	
Total P	mg/kg	18 000	11 000	9 300	15 000	340
Ca	mg/kg	248 000	20 700	89 400	23 600	7 790
C:N ratio†		5:1	3:1	16:1	7:1	13:1

Total N_{AR}* : total N in application rate (AR) 7.5 dry solid tonnes/ha of organic amendment; C:N ratio[†] assumes total C ≈ organic C.

plastic bottles with loose fitted lids (for gaseous exchange) and incubated in the dark at 12.5° ± 0.5°C. The treated and untreated soils were gently tamped down in the bottles (7 light taps on a bench) to achieve a similar bulk density (i.e., similar height in container). No additional water was added to the samples over the incubation period due to minimal moisture loss (72% FC at day 0 decreasing to 65% FC by day 56). The same dry weight application rate was used for all bioresources in the incubation in an effort to maintain similar soil to product contact, regardless of total N in the product. The C:N ratio was not kept constant because it has not been found a reliable indicator of mineralization rates [22].

On each sampling day (i.e., 3, 7, 14, 28, 42, and 56) a sample bottle from each treatment was removed, the soil placed in individual plastic bags and frozen at -19°C until analysis. Samples for day 0 were bagged and frozen immediately after mixing.

Frozen samples were thawed to room temperature before subsampling (10–15 g), drying at 105°C for 24 hours and reweighing to determine GMC. Five grams of each moist sample was also weighed into a 125 mL PPE screw top container and shaken with 2M KCl solution at a 1:10 ratio (w/v) for 1 hour. Extracts were then filtered through Whatman number 42 filter paper, analysed colorimetrically by CSBP Laboratories for NH₄⁺ and NO₃⁻, with results corrected for moisture using GMC.

The total inorganic N content was calculated as the sum of NH₄⁺ and NO₃⁻ extracted from each sample throughout the incubation and the net N mineralised from the applied products was calculated as the difference between inorganic N in each treatment and the control soil [9]. Reported values are actual concentrations on each respective sampling day. Extract concentrations in mg/L were converted to mg/kg using the following formula.

In the following formula, CA = concentration of analyte, CE = concentration in extract, EV = extract volume, and SDW = sample dry weight:

$$CA \text{ (mg/kg)} = \frac{CE \text{ (mg/L)} \times EV \text{ (L)}}{SDW \text{ (kg)}}. \quad (1)$$

The chemical compositions of LAB, ADB, PM, and PSW, together with the soil used in the trial, are shown in Table 1. Analysis was undertaken by Analytical Services Tasmania, with results shown as a Dry Solid basis.

3. Results and Discussion

3.1. N Mineralisation. The NO₃⁻ and NH₄⁺ concentrations of treated soils are shown in Tables 2 and 3, respectively. The moist control soil contained 8.5 mg/kg DS of NO₃⁻ at day 0, and after 56 days of incubation at 12.5°C in the dark it contained 48.3 mg/kg DS NO₃⁻. The ammonium chloride (1% NH₄Cl = 3372 mg/kg NH₄⁺) treatment still contained 2626 mg/kg (78%) of applied NH₄⁺ as NH₄⁺ by day 56 (Table 3) but its NO₃⁻ concentration (Table 2) was 41 mg/kg less than the control by day 56. This suggests that the NH₄⁺ added in the NH₄Cl treatment inhibited rather than stimulated nitrification. The soil with added sodium nitrate (1% NaNO₃ = 7295 mg/kg NO₃) still contained 1745 mg/kg (24%) of the added NO₃⁻ as NO₃ by day 56 (Table 2), concomitant with an increase in NH₄⁺ from 19.2 mg/kg to 46.1 mg/kg. In a similar study Rouch et al. [19] found after 70 days of soil incubation at 20°C in the dark that 84% of added NH₄⁺ was converted to NO₃⁻, whilst NO₃⁻ concentrations only increased by 8.7% in NaNO₃-amended soil. The differences between our results and those of Rouch et al. [19] are probably because of the different incubation temperatures (12.5°C and 20°C, resp.) and they demonstrate the potential effects on mineralisation from applying bioresources in cooler periods.

The concentration of NH₄⁺ in the lime treatment (CaCO₃) was not significantly different than that of the control or LAB (that contains lime as CaO). However, the concentration of NO₃⁻ in LAB at day 56 was significantly higher than the lime treatment. This difference may be due to the different adsorption rates of Ca²⁺ from the two different liming materials onto the colloidal complex, increasing base saturation and ultimately increasing soil pH. Lyngstad [23] found an increase in N mineralisation over a 3-year period as a result of adding CaCO₃ lime, whilst Mühlbachová and Tlustoš [24] found that although soil microbial activity initially decreased after application of CaO compared to

TABLE 2: NO_3^- concentration of treated soils (dry weight) after incubation at 12.5°C for 56 days.

		Day 0	Day 3	Day 7	Day 14	Day 28	Day 42	Day 56
ADB	(mg/kg)	9.75 ± 0.2^a	14.4 ± 2.1^{ab}	19.6 ± 11^a	73.8 ± 4.0^e	96.0 ± 59^c	135 ± 1.4^c	169 ± 15^c
Control	(mg/kg)	8.47 ± 1.4^a	12.0 ± 1.6^a	14.6 ± 3.4^a	19.2 ± 2.3^{abc}	31.5 ± 8.7^b	37.3 ± 7.6^b	48.3 ± 5.9^b
LAB	(mg/kg)	9.37 ± 1.4^a	11.6 ± 3.7^a	14.1 ± 9.9^a	52.0 ± 37^d	130 ± 29^d	167 ± 8.9^d	187 ± 16^c
Lime	(mg/kg)	9.49 ± 0.2^a	13.9 ± 0.5^a	17.7 ± 1.1^a	25.5 ± 4.1^{bc}	33.4 ± 9.0^b	41.5 ± 1.4^b	48.2 ± 4.5^b
NH_4Cl	(mg/kg)	9.28 ± 0.5^a	8.45 ± 0.6^a	8.96 ± 0.5^a	8.33 ± 0.4^{ab}	8.20 ± 1.2^a	7.90 ± 0.5^a	7.32 ± 0.8^a
PM	(mg/kg)	9.57 ± 1.2^a	5.68 ± 1.7^a	0.49 ± 0.2^a	3.84 ± 4.7^a	2.50 ± 0.01^a	14.0 ± 6.8^a	29.0 ± 9.7^b
PSW	(mg/kg)	9.79 ± 0.7^a	0.79 ± 0.6^a	1.10 ± 0.3^a	33.9 ± 5.7^{cd}	168 ± 7.3^e	231 ± 13^e	235 ± 15^d
NaNO_3	(mg/kg)	1919 ± 55	2052 ± 179	1892 ± 287	1781 ± 268	1710 ± 80	1882 ± 46	1745 ± 61

Note: different letters indicate significant differences between treatment means within the same row (LSD = 20.7, $P < 0.001$).

TABLE 3: NH_4^+ concentration of treated soils (dry weight) after incubation at 12.5°C for 56 days.

		Day 0	Day 3	Day 7	Day 14	Day 28	Day 42	Day 56
ADB	(mg/kg)	65.2 ± 2.7^b	70.0 ± 1.9^b	80.7 ± 15^c	23.2 ± 2.0^{ab}	8.32 ± 1.5^a	41.4 ± 4.1^b	8.47 ± 1.1^a
Control	(mg/kg)	20.0 ± 1.8^a	22.5 ± 0.8^a	22.6 ± 6.5^a	16.6 ± 0.6^{ab}	8.33 ± 1.4^a	7.01 ± 0.8^a	14.0 ± 0.9^a
LAB	(mg/kg)	34.9 ± 2.8^a	80.7 ± 17^b	97.9 ± 9.8^c	69.6 ± 1.9^c	25.7 ± 0.9^{ab}	11.2 ± 1.4^a	8.65 ± 1.6^a
Lime	(mg/kg)	23.2 ± 3.8^a	22.0 ± 0.3^a	25.1 ± 10^a	10.4 ± 2.1^a	8.80 ± 1.1^a	9.63 ± 0.2^a	8.72 ± 4.5^a
NaNO_3	(mg/kg)	19.2 ± 2.5^a	31.0 ± 3.2^a	41.0 ± 6.5^{ab}	31.3 ± 6.0^b	46.1 ± 14^c	51.4 ± 18^b	46.1 ± 1.7^b
PM	(mg/kg)	22.7 ± 0.5^a	23.0 ± 1.0^a	23.4 ± 4.4^a	14.2 ± 6.7^{ab}	19.5 ± 9.9^{ab}	17.5 ± 11^a	21.1 ± 1.4^a
PSW	(mg/kg)	22.5 ± 1.4^a	29.5 ± 3.1^a	50.9 ± 11^b	109 ± 5.8^d	34.5 ± 7.3^{bc}	11.5 ± 0.5^a	8.68 ± 0.4^a
NH_4Cl	(mg/kg)	2578 ± 293	2632 ± 203	2330 ± 41	2534 ± 114	2630 ± 67	2633 ± 25	2626 ± 78

Note: different letters indicate significant differences between treatment means within the same row (LSD = 19.3, $P < 0.001$).

CaCO_3 in the first days of incubation, CaO ultimately caused rapid mineralisation of the organic matter compared to CaCO_3 . Alternatively, the difference in the mineralisation of N between these two products may be due to water soluble Ca^{2+} from LAB stimulating microbial aggregation within the soil matrix soon after incorporation, subsequently accelerating decomposition and mineralisation of N. Mahoney et al. [25] found evidence of microbial aggregation when lime was added to an anaerobic sludge digester. Unfortunately, changes in soil pH in response to added treatments were not measured due to the limited volume of soil used in the incubation. Although a pH measure may have provided clarity, Corrêa et al. [26] reported no change in pH over 23 weeks from the addition of lime stabilised biosolids to an Oxisol. However, their application rate of 1 dry t/ha and subsequent concentration with the soil was much lower than that used in this study (7.5 dry t/ha).

The NO_3^- concentrations for both LAB and ADB increased over the 56-day period, with concentrations in the ADB treatment significantly higher than those in the LAB treatment at day 14 and the reverse at days 28 and 42. Conversely, there was a decrease in soil NO_3^- concentration for the PSW treatment after 3 days before they recovered to be significantly more than those in all other treatments by day 56. There was also a decrease in soil NO_3^- concentration in the PM treatment in the first 7 days before a similar recovery. The ADB, LAB, and PSW treatments all showed peaks in NH_4^+ concentration, at days 7, 7, and 14, respectively, before decreasing to similar concentrations at day 56. On the other

hand the PM treatment did not show an initial increase in NH_4^+ concentration and was not significantly different from the other treatments by the end of the incubation.

3.2. C:N Ratio. Using the assumption that the microbial activity and subsequent N mineralisation are inversely proportional to the C:N ratio of residues added to soil [27, 28], the N mineralisation rates of the treatments should follow the sequence ADB > LAB > PSW > PM, with C:N ratios of 3:1, 5:1, 7:1, and 16:1, respectively. However, the results in this experiment showed the extent and rate sequence of N mineralisation of the organic amendments to be in the order of LAB > PSW > ADB > PM. The initial loss of NO_3^- from PSW and PM (Figure 3) could have been due to denitrification or a negative priming effect (N drawdown) associated with the introduction of organic residues to soil [28]. The C:N ratio has been used to predict short-term N availability from solid manure amendments [29]; however Griffin and Hutchinson [22] found that the C:N ratio was poorly correlated with the rate and extent of mineralisation from soil-applied organic materials. Qian and Schoenau [29] found limited release of nitrogen over 67 days from cattle manure with a C:N ratio of between 13 and 15, which is close to the C:N ratio for PM (16:1). Furthermore, they suggested that if the C:N ratio exceeds 25:1, the microbes would source nitrogen from soil reserves (N drawdown, or negative priming). This does not explain why the PSW treatment, which had a preapplication C:N ratio of 7:1, exhibited a similar negative priming effect to the PM treatment (CN =

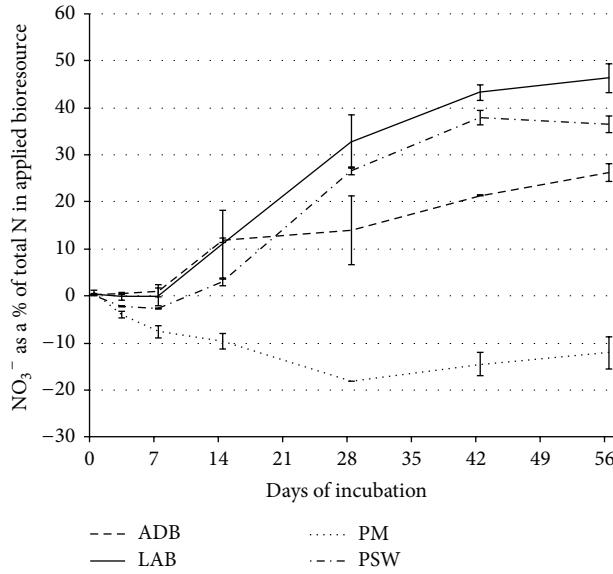


FIGURE 1: NO₃⁻ concentration of treated soils (dry weight) as a percentage of total N of added amendment (error bars are standard error of the means). Corrected for NO₃⁻ in control soil.

16 : 1). The disparity between treatments with regard to their C:N ratios and subsequent mineralization may be due to the C:N ratio of the soil, as mineralization of the treatments would not occur in isolation.

3.3. % Total N Released. In order to compare between mineralization rates of ADB, LAB, PM, and LAB, the results were corrected relative to the total N contained in each product after mixing with soil. Results are shown as a percentage of total N of the product for NO₃⁻, NH₄⁺ and plant-available N (PAN = NO₃⁻ + NH₄⁺) concentrations, respectively, and are corrected by subtraction for N from the control soil. Regardless of total N concentration, the percentage of total N present as NO₃⁻ (Figure 1) and NH₄⁺ (Figure 2) followed similar trends to those of the dry weight concentrations of NO₃⁻ and NH₄⁺ in the soil, when products were applied at the same dry weight rate. There was a 7-day lag time in %NO₃⁻ release for ADB and LAB with an estimated 10-day lag time in %NO₃⁻ release from PSW. There was a steady decline in %NO₃⁻ for the PM treatment until day 28, before a slight recovery to day 56. However, values for PM were still below 0, indicating that NO₃⁻ was either denitrified or taken up by microbial biomass. The %NH₄⁺ concentration for LAB (33.5%) was significantly higher than for ADB (16.8%) at their respective peaks after 7 days of incubation. The peak for NH₄⁺ as a percentage of total N for the PSW treatment did not occur until day 14, whilst for PM the peak, or plateau, began at day 28 but was not significantly different from any of the other treatments at that time.

The PAN results (Figure 3) show that 45%, 36%, 25%, and -8% of total N applied in LAB, PSW, ADB, and PM, respectively, were recovered as PAN at day 56, with the negative values in the PM treatment indicating a significant N drawdown for the whole period. The implications of

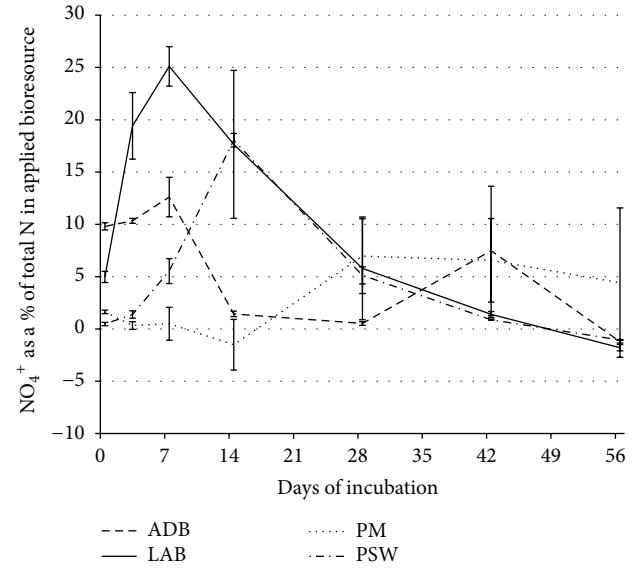


FIGURE 2: NH₄⁺ concentration of treated soils (dry weight) as percentage of total N of added amendment (error bars are standard error of the means). Corrected for NH₄⁺ in control soil.

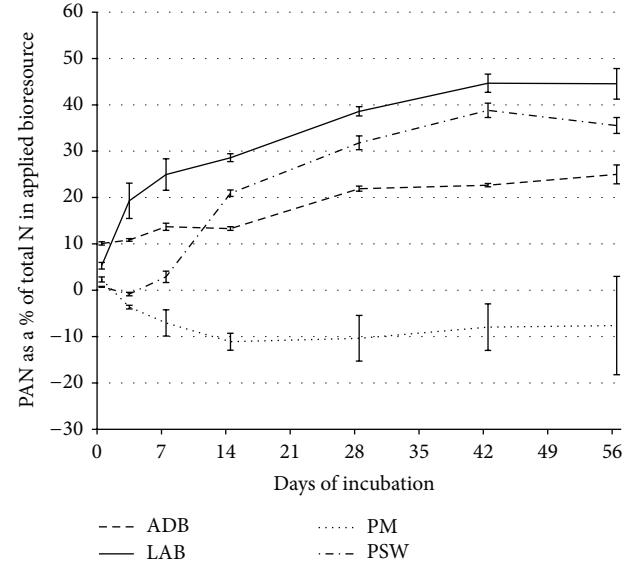


FIGURE 3: PAN (NO₃⁻ + NH₄⁺) of treated soils (dry weight) as percentage of total N of added amendment (error bars are standard error of the means). Corrected for PAN in control soil.

this drawdown from the PM treatment include determining application timing (either before crop is planted or when crop nutrient demand is low) and the application timing and amount of additional fertiliser N to satisfy plant requirements. Application timing may also need to be changed to satisfy plant demand when using PSW to take advantage of the early availability of N from the product. The practical limitations of shifting application to a more suitable time for plant uptake may increase risks associated with the season. For example, summer application may be suitable for autumn

nutrient release but may not be suitable for cultivation. Furthermore winter application may be suitable for spring nutrient release but paddocks may not be accessible at this time due to waterlogging or the risk of compaction of over wet soils and the increased risk of denitrification of mineralised N. The results for LAB in this study support the suggestion by Rigby et al. [30] that current biosolids guidelines do not reflect actual N release. This assertion was based on their study that found up to 65% of total N was released as PAN in the first season after application of lime-amended biosolids to sandy soils in Western Australia. Al-Dhumri et al. [31] also found that 39% of total N was mineralised 120 days after application of anaerobically digested biosolids to Sodosols in Victoria. However, the results of this incubation experiment contrast with the Tasmanian Biosolids Reuse guidelines that suggest only about 20% of total nitrogen in the product is released in the first twelve months following application [32]. Furthermore, the results of Rigby et al. [20] indicated that applying biosolids at guideline rates in autumn and spring may produce mineral nitrogen in excess of plant requirements at those times of year and increase the potential for leaching and denitrification. Similar to assertions by Al-Dhumri et al. [31] regarding the Victorian biosolids guidelines, Eldridge et al. [33] also questioned the adequacy of the current New South Wales biosolids guidelines [34] for calculating application rates.

4. Conclusion

The results of this study confirm that N mineralisation from different organic amendments is far from uniform and that predictions of mineralisation rates may not be reliably based on the C:N ratio of the applied product, at least for sandy loam soils as used here. Results also showed that, despite being incubated at lower than optimum mineralisation temperature, nitrogen mineralisation continued to occur, with 45%, 36%, and 25% of total N from LAB, PSW, and ADB, respectively, released as PAN by the end of the incubation period. The difference in N mineralisation between LAB and ADB may be due to the water soluble Ca²⁺ from LAB stimulating microbial activity and accelerating decomposition. The mineralisation rates at the temperature used suggest that application timing is critical to ensure that mineralisation of nitrogen from the applied products coincides with plant nutrient requirements and that mineralised N is not exposed to leaching loss and denitrification. These situations can potentially occur in the winter/early spring period in temperate climates such as Tasmania when rainfall is high and evapotranspiration is low, suggesting that autumn and early spring applications may not be appropriate. Although there are potential risks of nutrient build-up (i.e., phosphorus from PSW) associated with annual or periodical applications of PSW and PM (which are not regulated by EPA guidelines), it is suggested that regular soil tests be undertaken to detect any nutrient imbalances. The results also demonstrated that further work is required to understand the relationship between N mineralisation and composition of bioresources and whether the interaction of bioresources with sandy soils is similar with other soil types.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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