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Effectiveness of Five Biosolids as Nitrogen Sources to Produce Single and Cumulative Ryegrass Harvests in Two Australian Soils

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ABSTRACT: Biosolids have been produced by various stabilization processes of sewage sludge, but little attention has been given to the effects of such treatments on their effectiveness to supply nitrogen for plant growth. Here, we investigated these effects by cultivating *Lolium perenne* (ryegrass) in two Australian soils, a sandy Spodosol and a clayey Oxisol. Biosolids stabilized by either aerobic digestion, composting, CaO-liming, 250 °C heat-drying or solar irradiation of domestic sewage sludge were applied to soils at 10 increasing rates (0.25-8.0 dry Mg ha^{-1}), and chemically fertilized soils were used as reference. Results showed that the stabilization processes affected biosolids-N agronomic rates and effectiveness to yield plant biomass, which was a function of organic-N contents mineralized in soils. In the short term, biosolids were from 1/5 (digested biosolids in Oxisol) to over twice (solar-irradiated biosolids in Spodosol) as effective as chemical fertilizer to produce a first single harvest. As long-term N-sources, they significantly increased the effectiveness to produce plant biomass, being from 2.0 to 4.1 times more effective than chemical fertilizer in Spodosol and 1.5-2.4 times in Oxisol. Biosolids could substitute for N fertilizer with similar or higher effectiveness to yield plant biomass, depending on the sewage sludge stabilization process, soil type and cultivation term considered. Therefore, the sound management of sewage products as N sources for crop production should consider the interaction among these factors rather than solely their N content.

Keywords: sewage sludge, organic-N, plant nutrients, Oxisol, Spodosol.

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INTRODUCTION

Sewage sludge contains significant concentrations of organic matter, nitrogen, phosphorus and other elements that improve soil structure and fertility, stimulate microbial activity, enhance root penetration and increase crop yield (Al-Dhumri et al., 2013; Jorge-Mardomingo et al., 2013; Jeke et al., 2015). The idea of recovering organic matter and plant nutrients by applying sewage sludge to agricultural lands has been widespread as a disposal option, a cost-effective practice, and a way to close the nutrient loop in agriculture and remediated soils (Torri et al., 2014; Yoshida et al., 2015). Prices of chemical fertilizers and favorable costs of sludge land application compared to other disposal options have been decisive for recycling sewage sludge components in soils (Winker et al., 2009). The term biosolids is currently used for treated sewage sludge deemed acceptable for land application, in order to emphasize the distinction between such material and untreated sludge (Silva et al., 2015).

The benefits of biosolids soil incorporation have to be assessed against potential hazards, including those associated with metals, human pathogens and nitrate leaching to groundwater (Jorge-Mardomingo et al., 2015; Yoshida et al., 2015). As a result of such health and environmental concerns, legislation worldwide requires the sound management of biosolids in soils (USEPA, 1995; EPA, 2004; Conama, 2006). Concentrations of metals, organic contaminants, N and P are often the controlling design parameters for long-term land application of biosolids, while pathogens, odors and vector attraction may prevent a site or a crop from receiving a single application of sewage products (Winker et al., 2009). Processes of sludge sanitation and stabilization have been employed to overcome the biological hazards and nuisance problems associated with the use of sewage materials. Sludge digestion, composting, lime stabilization, heat treatment and solar irradiation are commonly employed for turning putrescible sewage materials into stable biosolids (USEPA, 1995; EPA, 2004; Conama, 2006).

On sites where biosolids can be applied, environmental agencies demand soil application at N-agronomic rates, which means balancing crop N demand with N delivered from biosolids to soils in order to avoid nitrate leaching into groundwater (USEPA, 1995; EPA, 2004; Conama, 2006). From the economic point of view, N-agronomic rates provide enough N to achieve near maximum yield and well-nourished plants (Al-Dhumri et al., 2013; Antille et al., 2013). As N-agronomic rates maximize crop yields and minimize risks of groundwater contamination (Winker et al., 2009; Al-Dhumri et al., 2013), a safer and more effective management of different kinds of biosolids in soils demands an evaluation of their capacity to promote plant growth at N-agronomic rates (Al-Dhumri et al., 2013; Jorge-Mardomingo et al., 2015).

Currently, stabilization processes of sewage sludge and soil type have been reported to affect both the dynamics of biosolids-N degradation in soils and the contents of N leached through soil profile (Corrêa et al., 2006; 2012; Paula et al., 2011; Antille et al., 2013; Yoshida et al., 2015). Based on these results, this study tested the hypotheses that stabilization processes of sewage sludge and soil type also affect biosolids N-agronomic rates and their effectiveness to produce plant biomass. These hypotheses were tested by measuring the effects of the most common sewage sludge stabilization processes (digestion, composting, liming, heating and solar-irradiation) on the effectiveness of stabilized biosolids to yield single and cumulative ryegrass harvests in two soils of contrasting textures.

MATERIALS AND METHODS

Sludge stabilization

A 500 kg sample of freshly digested domestic sewage sludge was collected from Coliban Water Treatment Works in Victoria (Australia) and analyzed in triplicate for gravimetric water content (105 °C for 48 h), bulk density (BD), total N and total C (dry combustion method, Carbo-Erba NA 1500 analyzer, Thermo Scientific, Inc. Odessa, Texas, USA), mineral N (NH_4^+-N + NO_3^--N) (Kjeldahl steam distillation method), total P (HNO_3 + H_2SO_4 digestion followed by molybdate-vanadate color development), and available P (Bray 1-P). The freshly digested sewage sludge (878 g kg^{-1} moisture, BD = 1.2 Mg m^{-3}, C/N ratio = 6.2) was mixed with hardwood sawdust (96 g kg^{-1} moisture, BD = 0.3 Mg m^{-3}, and C/N ratio = 668) and woodchips (bulk agent) to achieve a C/N ratio = 25:1 in the feedstock blend. Then, three 450 L composting piles were pitched on a sheltered cement pavement, run at 35-65 °C for 34 days, allowed to mature for another 60 days and sieved at 2 mm. Another sample of the same freshly digested sewage sludge was treated with CaO at a 30 % ratio to sludge dry solids (w/w). Likewise, heat drying of fresh sewage sludge was performed in a furnace at 250 °C until a constant weight was achieved. The 250 °C heat-dried biosolids were ground and passed through a 2 mm sieve. For the solar irradiation process, three 10 kg fresh sludge samples were stored in freely drained plastic bowls under transparent plastic covers in sunny conditions for 14 days during Melbourne's summer (Australia), with daily temperatures ranging from 12.8 °C to 26.5 °C. The processing criteria established in USEPA (1995) were achieved in all the employed processes. All biosolids were analyzed in triplicate for total-N, mineral-N, total-P and Bray 1-P using the same analytical methods for the freshly digested sewage sludge. The results are shown in table 1. Analysis of variance and Tukey test were performed in GenStat® for Windows 5th edition.

Soils

Two soils were selected to be applied with the biosolids (Table 2): a sandy Spodosol and a clayey Oxisol (Soil Survey Staff, 1999). A 200-kg sample of each soil was collected from 0.10-0.20 m soil depth in Cranbourne (Spodosol) and Emerald counties (Oxisol) in Victoria, Australia. Soil top layers were removed prior to collecting to minimize the effects of native organic matter. Soil samples were allowed to air-dry for two weeks and were then passed through a 4-mm sieve.

Pot experiment

Soil samples (1.8 kg) treated with biosolids at ten increasing rates (Table 3) were placed in triplicate in 2 L pots. Although mineral-N concentrations of 10-20 mg kg^{-1} in soils are usually enough for adequate ryegrass growth (Al-Dhumri et al., 2013; Antille et al., 2013), an appropriate comparison among different sources of a given nutrient requires the cover of the whole plant demand range for this nutrient (Barrow and Bolland, 1990; Bolland, 1997). Thus, aiming to investigate complete plant responses to biosolids-N application, rates of N applied to soils as biosolids ranged from null to 261.4 mg kg^{-1}. Likewise, a chemical source of N was applied to soil samples at 0-512 mg kg^{-1} (Table 3).

Table 1. Nitrogen and phosphorus concentrations in biosolids

Property	Digested biosolids	Composted biosolids	30 %-CaO biosolids	250 °C-dried biosolids	Irradiated biosolids
			dry weight		
Total-N (g kg^{-1})	65.1 a	15.9 b	40.1 c	64.8 a	65.3 a
Total-P (g kg^{-1})	72.1 a	24.2 b	50.6 c	72.5 a	72.6 a
Mineral-N (mg kg^{-1})	624 a	277 b	93.8 c	356 d	803 e
Bray 1-P (mg kg^{-1})	268 a	377 b	11.9 c	678 d	199 e

Means (n = 3) followed by the same letter for each parameter are not statistically different by the Tukey test ($p<0.05$).

Table 2. Chemical and physical properties of the selected soils (mean ± standard error)

Property	Spodosol	Oxisol
Total-N (g kg^{-1})	0.38 ± 0.03	1.61 ± 0.11
Mineral-N (mg kg^{-1})	0.13 ± 0.01	5.02 ± 0.08
Total-P (g kg^{-1})	0.15 ± 0.01	0.40 ± 0.02
Bray 1-P (mg kg^{-1})	4.51 ± 0.1	14.6 ± 0.5
Total-C (g kg^{-1})	6.0 ± 3	13.0 ± 5
pH(H_2O) (1:5 w/v)	4.9 ± 0.1	5.0 ± 0.1
Bulk density (Mg m^{-3})	1.6 ± 0.1	0.9 ± 0.1
Clay (g kg^{-1})	48 ± 3	499 ± 38
Silt (g kg^{-1})	37 ± 1	145 ± 9
Sand (g kg^{-1})	915 ± 21	356 ± 19
Porosity (m^3 m^{-3})	0.41 ±0.01	0.67 ±0.08
Field capacity (m^3 m^{-3})	0.07 ± 0.02	0.31 ± 0.02

Mean (n =3).

Table 3. Total-N applied to air-dried soils based on the N content in biosolids

Equivalent	Digested biosolids	Composted biosolids	30 %-CaO biosolids	250 °C-dried biosolids	Irradiated biosolids	$NH_4H_2PO_4$
dry Mg ha^{-1}			N applied to soils (mg kg^{-1})			
0.25	8.1	2.0	5.0	8.1	8.2	1
0.50	16.3	4.0	10.0	16.2	16.3	2
1.0	32.6	8.0	20.1	32.4	32.7	4
2.0	65.1	15.9	40.1	64.8	65.3	8
3.0	97.7	23.9	60.2	97.2	98.0	16
4.0	130.2	31.8	80.2	129.6	130.6	32
5.0	162.8	39.8	100.3	162.0	163.3	64
6.0	195.3	47.7	120.3	194.4	195.9	128
7.0	227.9	55.7	140.4	226.8	228.6	256
8.0	260.4	63.6	160.4	259.2	261.2	512

Biosolids-amended soil samples were wetted with deionized water to their field capacity and allowed to rest on saucers for a week at 25 °C (±2 °C) in a glasshouse environment before sowing. A non-fertilized control soil and soil samples containing chemical fertilizer were placed among the biosolids-amended soil pots and treated alike (Table 3). Analytical reagent (AR) grade ammonium phosphate ($NH_4H_2PO_4$) was employed as the chemically combined source of N and P to be used as the reference for biosolids effectiveness to produce plant biomass. Fertilizer application rates aimed to cover the whole N range applied as biosolids (Table 3). Soil samples applied with $NH_4H_2PO_4$ also received a basal application of 78 mg kg^{-1} of K and 58 mg kg^{-1} of Mg. A commercial formulation containing 120 g kg^{-1} of $FeSO_4$, 25 g kg^{-1} of $MnSO_4$, 10 g kg^{-1} of $ZnSO_4$, 5 g kg^{-1} of $CuSO_4$, 1 g kg^{-1} of Na_2BO_3 and 1 g kg^{-1} of Na_2MoO_4 was also applied at a soil rate of 1.0 g kg^{-1}.

Plant test

Pots containing the biosolids-amended soils (five types of biosolids × 10 application rates × two soil types), unamended soils (two soil types) and chemically fertilized soils (10 application rates × two soil types) were sown with 10 seeds of *Lolium perenne* cv. concord at a depth of 0.5 cm. Two weeks after sowing, pots were thinned to five plants per pot, leaving in pots the most uniform plants. A volume of 10-20 mL of distilled water was daily applied to pots to meet plant requirements. The experiment was run in a glasshouse at 25 °C (±2 °C) and a 12 h day/night light cycle.

The first ryegrass harvest occurred eight weeks after thinning, and plants were allowed to re-grow in pots to be harvested at eight-week intervals until responses to the application of biosolids and fertilizers flattened out. The trial lasted for 11 months and pots were harvested six times after a single application of either biosolids or the $NH_4H_2PO_4$ + basal application. Plant materials were oven-dried (70 °C for 72 h) and yields were calculated on a dry matter basis. All pots were randomized weekly and the eventual leachate collected in saucers was reapplied onto soil surfaces to avoid nutrient losses.

Soil analysis

Soil samples were collected from pots on ryegrass sowing day and after the last harvest. Collected soil samples were placed in a 5 °C cold room (±1 °C) before the analyses of ammonium-N (NH_4^+-N) and nitrate-N (NO_3^--N), which were done by the Kjeldahl steam distillation method (Rayment and Higginson, 1992) within 48 h after each sampling. Concentrations of NH_4^+-N and NO_3^--N in each soil sample were summed as mineral-N. A Carbo-Erba NA 1500 analyzer (Thermo Scientific, Inc. Odessa, Texas, USA) was used to measure total-N in soils by the dry combustion method (Carbo-Erba NA 1500 analyzer, Thermo Scientific, Inc. Odessa, Texas, USA).

Analysis of data

The first and cumulative dry matter yields (six harvests) were plotted against N rates applied to soils and fitted by a modified Newton method of maximizing the likelihood to the following power function by using GenStat® for Windows 5th edition:

$\hat{y} = \alpha - \beta P^X$, P positive and <1 Eq. 1

where $\hat{y}$ is the yield (g dry matter per pot), x is the amount of N applied to soils (mg kg^{-1}), P is the model slope, α is the maximum yield and β is the yield response to the rates of N applied either as biosolids or $NH_4H_2PO_4$. A curvature factor (γ) was derived from -ln P for calculation of the effectiveness of each biosolids against $NH_4H_2PO_4$, based upon plant yield and N applied to soils. Equation 1 is equivalent to the Mitscherlich equation ($\hat{y} = \alpha - \beta^{-\gamma x}$). As the value of γ increases, less N from a source is required to produce the same plant yield or less N is required to reach the maximum yield (Bolland, 1997).

The effectiveness of each material to yield dry matter was calculated by the product of β and γ (βγ), since:

if $\hat{y} = \alpha - \beta P^X \rightarrow dy/dx = -\beta P^X \ln P = \beta P^X \gamma$

when $x = 0$, $P^X = 1 \rightarrow dy/dx = \beta\gamma$

βγ, as an effectiveness measure of each N source to yield plant biomass, has been used as a surrogate for N-agronomic rate (Al-Dhumri et al., 2013). The ratio of biosolids-βγ to βγ of $NH_4H_2PO_4$ ($\beta_1\gamma_1/\beta_2\gamma_2$) gives the relative effectiveness (RE) of biosolids against $NH_4H_2PO_4$, as described in Weatherley et al. (1988) and Bolan et al. (1990). When RE = 1, products are of equal effectiveness; when RE <1 or RE >1, a given biosolids is respectively less or more effective than the chemical reference. Scales on x axes were cut for a better representation of the curves.

Standard errors values for β and P (SE_β and SE_P respectively) were derived from parameters given by GenStat®. Standard errors for βγ ($SE_{\beta\gamma}$) were derived from the equation below, as described in Beers (1957):

$SE_{\beta\gamma} = (SE_\beta{}^2 + SE_P{}^2)^{0.5}$ Eq. 2

where the asymptotic model did not fit equation 1, linear regressions ($\hat{y} = \alpha + \beta_1 x$) of plant yield on N applied to soils were chosen for the calculation of RE, according to Barbarick and Ippolito (2000) and Al-Dhumri et al. (2013):

$$RE = \frac{slope_{(biosolids)}}{slope_{(NH4H2PO4)}} \qquad \text{Eq. 3}$$

N concentrations in soils on ryegrass sowing day (day 0) and after the last harvest (last day) were used to account for biosolids organic-N mineralized in soils throughout plant trial (Jeke et al., 2015), according to the equations below:

$$\text{Organic – N = total-N – mineral-N} \qquad \text{Eq. 4}$$

$$\text{Organic – N}_{\text{mineralized}} = \text{Organic – N}_{\text{day 0}} - \text{Organic – N}_{\text{last day}} \qquad \text{Eq. 5}$$

RESULTS AND DISCUSSION

Effectiveness of biosolids-N to produce a single harvest

Concentrations of mineral-N in soils significantly increased soon after biosolids application (Table 4), and plants positively responded to it in a short eight-week trial. N availability affects plant growth (Jeke et al., 2015), and ryegrass responses to the amendments varied among treatments. Different biomass yields were measured at N-agronomic rates, which varied according to biosolid and soil type (Figures 1 and 2). Spodosol at N-agronomic rate could receive N loads of 80 mg kg^{-1} as digested biosolids, 150 mg kg^{-1} as composted biosolids, 76 mg kg^{-1} as 30 %-CaO biosolids, 220 mg kg^{-1} as 250 °C-dried biosolids or 68 mg kg^{-1} as solar-irradiated biosolids (Figure 1). Plants in Oxisol reached biomass asymptotes at higher N-agronomic rates, which were 145 mg kg^{-1} for digested biosolids, 90 mg kg^{-1} for 30 %-CaO biosolids, 162 mg kg^{-1} for 250 °C-dried biosolids and 98 mg kg^{-1} for solar-irradiated biosolids (Figure 2). Nutrient use efficiency has been defined as the amount of dry matter produced per unit of an element supplied (Bolland, 1997; Al-Dhumri et al., 2013), and it is often more important than competition for light in determining the success of a plant to survive and thrive in the first stages of development (Vaz and Gonçalves, 2002). In this regard, N applied to soils as digested, 30 %-CaO and solar-irradiated biosolids were more efficiently used by plants to yield biomass than N stocked in 250 °C-dried and composted biosolids (Figures 1 and 2, Table 5).

Relative to the chemical-N source used as reference ($NH_4H_2PO_4$), biosolids presented different effectiveness to yield plant biomass (Table 5). Values greater than 1 for relative effectiveness

Table 4. Range of N concentrations in unamended soils and in soils at biosolids application rates of 0.25-8.0 dry Mg ha^{-1} on ryegrass sowing day and after the sixth-last harvest

Time	Unamended control soil	Digested biosolids	Composted biosolids	30 %-CaO biosolids	250 °C-dried biosolids	Irradiated biosolids
			Total-N in Spodosol (g kg^{-1})			
Sowing day	0.38 a	0.39 a – 0.59 b	0.36 a – 0.38 a	0.36 a – 0.42 a	0.39 a – 0.51 b	0.40 a – 0.56 b
After last harvest	0.37 a	0.32 a – 0.47 ab	0.32 a – 0.36 a	0.34 a – 0.36 a	0.35 a – 0.46 ab	0.34 a – 0.45 ab
			Mineral-N in Spodosol (mg kg^{-1})			
Sowing day	0.13 a	1.32 b – 3.73 c	0.77 d – 1.80 b	1.78 b – 2.16 bc	1.47 b – 3.49 c	3.22 c – 3.85 c
After last harvest	0.09 a	0.23 a – 1.11 b	0.13 a – 0.61 d	0.34 a – 0.97 d	0.14 a – 0.95 d	0.15 a – 1.32 b
			Total-N in Oxisol (g kg^{-1})			
Sowing day	1.61 a	1.59 a – 1.81 b	1.58 a – 1.60 a	1.55 a – 1.62 a	1.54 a – 1.76 b	1.62 a – 1.78 b
After last harvest	1.24 c	1.13 c – 1.51 a	1.18 c – 1.51 a	1.21 c – 1.45 a	1.34 c – 1.52 a	1.01 c – 1.48 a
			Mineral-N in Oxisol (mg kg^{-1})			
Sowing day	5.02 a	6.19 b – 17.99 c	5.83 ab – 9.38 d	5.97 b – 10.09 d	8.23 d – 12.11 e	6.56 b – 16.98 c
After last harvest	3.51 f	2.94 f – 5.15 a	2.83 f – 3.97 f	2.48 f – 3.95 f	2.50 f – 4.14 af	2.19 f – 6.17 b

(1) Means followed by the same letter for each soil and parameter are not statistically different by the Tukey test at 5 %.

(RE) indicate that digested, 30 %-CaO and irradiated biosolids produced more dry matter as sources of N than $NH_4H_2PO_4$ + basal application was able to do in the Spodosol (Table 5). These biosolids were 2.0-2.3 times more effective to yield dry biomass than $NH_4H_2PO_4$ in an eight-week trial. The better performance of biosolids to produce plant biomass compared to chemical fertilizers has also been reported for other soils and crops (Fresquez et al., 1990; Silva et al., 2000; Fernández et al., 2008; Mbarki et al., 2008; Rivero et al., 2009).

Plants cultivated in Spodosol amended with digested, 30 %-CaO and irradiated biosolids at N-agronomic rates yielded twice the ryegrass biomass than the chemical treatment (Figure 1, Table 5). Sewage sludge stabilization processes affect both the nutrient content and the mineralization rates of biosolids in soils (Corrêa et al., 2012). The less stable sewage products, such as digested, 30 %-CaO and solar-irradiated biosolids, showed higher RE values in Spodosol relative to the more stable products, like composted and 250 °C-dried biosolids (Table 5). Biosolids application modifies the main soil regulating parameters, particularly the nutrient stoichiometry, activity and diversity of soil microbial communities, which may have variable effects on organic matter decomposition (Gomes et al., 2010; Jorge-Mardomingo et al., 2013). Incorporation of putrescible organic materials into soils provides labile C, which results in considerable initial microbial growth and quick organic matter decomposition. The intensity of microbial growth and organic matter decomposition is generally related to N availability and the kind of incorporated organic C (Jeke et al., 2015).

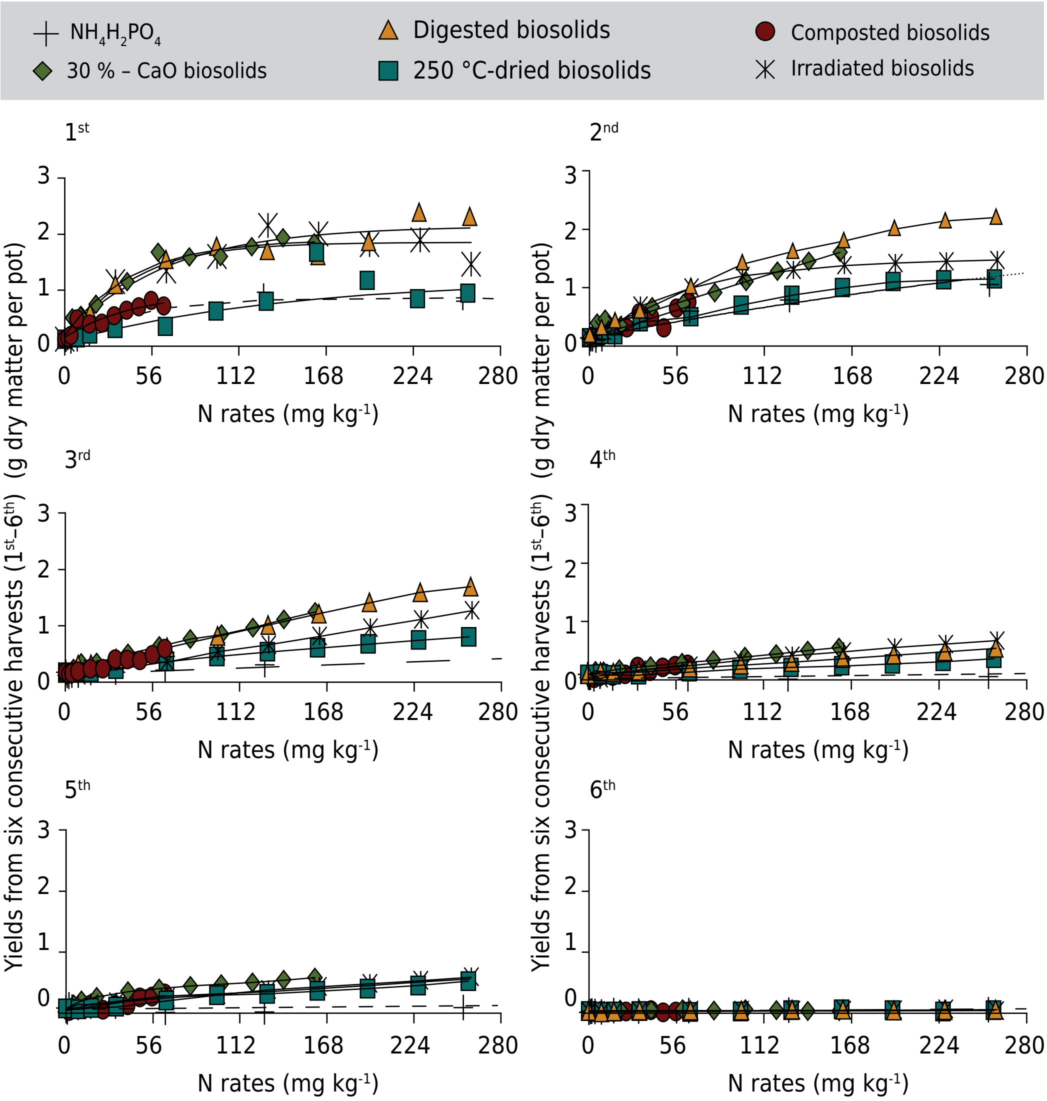

Figure 1. Yield responses to biosolids-N application and to $NH_4H_2PO_4$ + basal application in Spodosol.

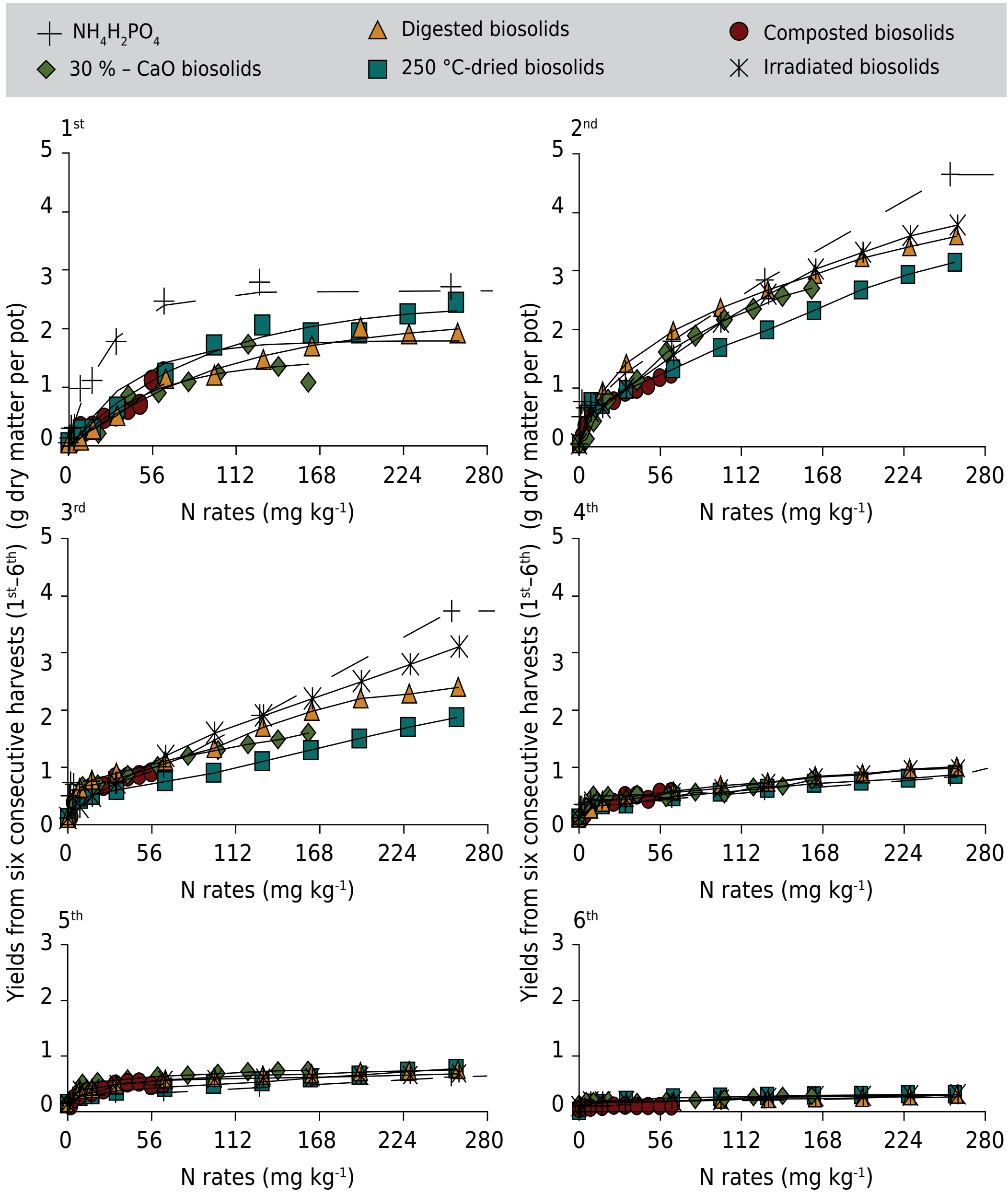

Figure 2. Yield responses to biosolids-N application and to $NH_4H_2PO_4$ + basal application in Oxisol.

The composting process stabilizes labile C from putrescible organic feedstocks, dilutes and volatilizes N, yielding a product relatively poor in N (Corrêa et al., 2006; Carvalho et al., 2015) (Table 1). Nitrogen mineralization is a microbial-mediated process (Jeke et al., 2015) and despite the low N content in composted materials (Table 1), they are sources of C and nutrients for microbial growth, which triggers organic-N mineralization in the soil environment (Gomes et al., 2010; Jorge-Mardomingo et al., 2013). In this work, composted biosolids were as effective as the chemical treatment ($NH_4H_2PO_4$ + basal application) to produce plant biomass in the Spodosol (Table 5). The 250 °C-dried biosolids, which contained four times more N than composted biosolids (Table 1), showed poorer performance in terms of yielding plant biomass than both the composted biosolids and chemical fertilization (Table 5).

Thermal treatment stabilizes labile components and sterilizes fresh residues, which reduces soil microbial activity and decomposition rates of organic matter (Gomes et al., 2010; Jorge-Mardomingo et al., 2013; Carvalho et al., 2015). Mineralization has to make nutrients from organic sources plant available (Al-Dhumri et al., 2013), and thermally dried biosolids may not support microbial metabolism upon soil application (Gomes et al., 2010; Jorge-Mardomingo

Table 5. Coefficients for yield responses to the treatments fitted to either asymptotic regression models ($\hat{y} = \alpha - \beta P^X$) or linear regression model ($\hat{y} = \alpha + \beta_1 x$) for the first and six cumulative harvests

Treatment	α	β	P	γ	βγ	RE
First yield responses to N applied to Spodosol						
$NH_4H_2PO_4$	0.856 ± 0.062	0.855 ± 0.075	0.978 ± 0.007	0.022	0.019 ± 0.006	1.00
Digested biosolids	2.147 ± 0.164	1.984 ± 0.191	0.981 ± 0.005	0.019	0.038 ± 0.001	1.98
Composted biosolids	0.959 ± 0.115	0.807 ± 0.293	0.975 ± 0.016	0.025	0.020 ± 0.005	1.06
30 %-CaO biosolids	1.901 ± 0.092	1.716 ± 0.101	0.977 ± 0.004	0.023	0.039 ± 0.007	2.08
250 °C-dried biosolids	1.233 ± 0.028	1.200 ± 0.393	0.991 ± 0.009	0.010	0.012 ± 0.001	0.63
Irradiated biosolids	1.854 ± 0.114	1.822 ± 0.192	0.976 ± 0.007	0.024	0.044 ± 0.004	2.30
First yield responses to N applied to Oxisol						
$NH_4H_2PO_4$	2.752 ± 0.086	2.678 ± 0.112	0.964 ± 0.003	0.037	0.096 ± 0.009	1.00
Digested biosolids	2.179 ± 0.150	2.199 ± 0.150	0.991 ± 0.004	0.009	0.020 ± 0.009	0.21
Composted biosolids	Linear, $\hat{y} = 0.042 + 0.014^{*} x$			$R^2 = 0.94^{**}$		0.38
30 %-CaO biosolids	1.461 ± 0.020	1.461 ± 0.216	0.981 ± 0.008	0.019	0.029 ± 0.012	0.29
250 °C-dried biosolids	2.446 ± 0.166	2.449 ± 0.158	0.989 ± 0.002	0.011	0.027 ± 0.005	0.28
Irradiated biosolids	1.790 ± 0.209	1.790 ± 0.369	0.975 ± 0.013	0.025	0.045 ± 0.025	0.47
Cumulative yield responses to N applied to Spodosol						
$NH_4H_2PO_4$	$\hat{y} = 0.731 + 0.007^{*} x$			$R^2 = 0.90^{*}$		1.0
Digested biosolids	$\hat{y} = 1.301 + 0.022^{*} x$			$R^2 = 0.91^{*}$		3.1
Composted biosolids	$\hat{y} = 0.599 + 0.024^{*} x$			$R^2 = 0.98^{**}$		3.4
30 %-CaO biosolids	$\hat{y} = 1.366 + 0.029^{*} x$			$R^2 = 0.95^{**}$		4.1
250 °C-dried biosolids	$\hat{y} = 0.613 + 0.014^{*} x$			$R^2 = 0.96^{**}$		2.0
Irradiated biosolids	$\hat{y} = 1.227 + 0.019^{*} x$			$R^2 = 0.92^{*}$		2.7
Cumulative yield responses to N applied to Oxisol						
	α	β	P	γ	βγ	RE
$NH_4H_2PO_4$	13.659 ± 0.858	11.750 ± 0.854	0.997 ± 0.001	0.003	0.035 ± 0.012	1.00
Digested biosolids	11.83 ± 2.44	10.32 ± 2.26	0.995 ± 0.002	0.005	0.052 ± 0.008	1.49
Composted biosolids	4.326 ± 0.354	3.744 ± 0.334	0.982 ± 0.007	0.018	0.067 ± 0.018	1.93
30 %-CaO biosolids	6.794 ± 0.502	5.953 ± 0.476	0.986 ± 0.003	0.014	0.084 ± 0.009	2.40
250 °C-dried biosolids	11.16 ± 1.44	9.95 ± 1.34	0.995 ± 0.001	0.005	0.050 ± 0.012	1.43
Irradiated biosolids	9.627 ± 0.629	8.512 ± 0.602	0.990 ± 0.002	0.010	0.085 ± 0.008	2.43

* and **: significant at 5 and 1 %, respectively, by Tukey test.

et al., 2013). Consequently, a shortage of available nutrients for plant growth may be expected in some soils amended with thermally treated biosolids (Corrêa et al., 2012).

Plants respond to various environmental factors and increasing rates of soil amendments usually increment plant yield, but such response also depends on soil type (Navas et al., 1999; Corrêa et al., 2012; Jeke et al., 2015). In this regard, the full potential of plant growth and the highest plant effectiveness for using N in Oxisol in an eight-week trial were achieved with chemical fertilization (Figure 2, Table 5). Organic sources of nutrients may mineralize at lower rates than plant demand for optimum growth in certain soils and a lower asymptote is reached (Barrow and Bolland, 1990). Lower efficiency of sewage materials relative to chemical fertilizers in loam and clayey soils has been reported and attributed to the mineralization rates of organic pools that do not match plant nutritional demands (Boeira and Maximiliano, 2009). In such a situation, chemical sources of nutrients applied on the top of biosolids as supplemental fertilization may be a sound management strategy for crop production (Antille et al., 2013).

Otherwise, nutrient availability can be increased by soil incorporation of organic sources some weeks prior to planting (Corrêa et al., 2012). Solar-irradiated biosolids, which rested for two weeks under environmental conditions before soil incorporation, were more effective at producing plant biomass in both soils than the original digested biosolids (Table 5). Differences in plant yields are chiefly determined by nutrient availability when water, light and heat do not limit growth (White, 1997). Where organic sources of nutrients are used for biomass production, their performance has been related to their mineralization rates in soils rather than to their nutrient contents (Barbarick and Ippolito, 2000; Corrêa et al., 2012).

Effectiveness of biosolids-N to produce cumulative harvests

Approximately 95 % of nutrients in biosolids are in organic form (Table 1) (Haynes et al., 2009) and for plant uptake, mineralization has to convert organic forms of nutrients into ionic elements and molecules after soil application, a biochemical process dependent on the degree of organic source stability, soil type, microorganisms, temperature, moisture and time (Corrêa et al., 2012; Al-Dhumri et al., 2013; Jeke et al., 2015). The biosolids significantly increased total-N concentrations in soils at the highest application rate of 8.0 dry Mg ha^{-1} (Table 4), except composted and 30 %-CaO biosolids, which respectively contained 25 and 62 % of the total-N present in the other three sewage products (Table 1). Organic-N mineralization brought total-N concentrations back to values similar to unamended soils after eleven months of ryegrass cultivation (Table 4).

Regardless the N source, ryegrass yields decreased with subsequent harvests (Figures 1 and 2). Chemical fertilizer applied to the Spodosol supported two harvests of similar yields, but plant production decreased considerably in subsequent harvests (Figure 1). Yields on Spodosol amended with digested, 30 %-CaO and irradiated biosolids respectively decreased by 25 % – 32 %, 55 % – 61 %, 72 % – 82 %, 85 % – 93 % and >95 % from the first to the sixth last harvest (Figure 1). The first two harvests yielded over twice the dry biomass as the last four harvests in this soil. From a practical point of view, these biosolids applied at N-agronomic rates could render two harvests in Spodosol, and further plant production would demand the application of supplemental N (Antille et al., 2013). Spodosol treated with composted and 250 °C-dried biosolids produced lower yields at each harvest compared to the other three biosolids (Figure 1), but yields were more evenly distributed along harvests. Such a figure confirms the slow mineralization pattern of composted and thermally dried biosolids in sandy soils (Mbarki et al., 2008; Corrêa et al., 2012).

In Oxisol, the second ryegrass yield doubled relative to the first one in all treatments (Figure 2). Then, yields from the third harvest were slightly higher than the first cut, and ryegrass biomass drastically dropped in subsequent cuts (Figure 2). A single soil application of biosolids at the N-agronomic rate could render three harvests in Oxisol without the need for supplemental N-fertilization. In another study, the second corn harvest from an Oxisol treated with digested biosolids also outperformed the first harvest, and biosolids-N was more efficiently used by plants than a chemical-N source when multiple corn harvests were assessed (Silva et al., 2000).

Efficiency is this work was measured as a function of plant yield in response to biosolids-N applied to soils (Barrow and Bolland, 1990). As mineralization makes N available in soils for plant uptake over the course of time (Jeke et al., 2015), the effectiveness of biosolids as N sources remarkably increased over the 11-month period relative to the first ryegrass harvest (Table 5). In this sense, cumulative ryegrass yields followed the amounts of organic-N mineralized in soils irrespective of soil and biosolids type (Figure 3). Mineralization is a major event when organic matter sources are incorporated into soils (Boeira and Maximiliano, 2009; Haynes et al., 2009), and the effectiveness of biosolids as N sources to yield plant biomass was mostly determined by the content of organic-N mineralized in soils while plants were growing on them (Figure 3).

In the Spodosol, biosolids relative effectiveness (RE) values ranged 0.6-2.3 for the first harvest and 2.7-4.1 for the six cumulative harvests having $NH_4H_2PO_4$ + basal application as reference (Table 5). The composted and 250 °C-dried biosolids, which mineralized slowly in Spodosol (Corrêa et al., 2012), showed the largest increases in efficiency to produce plant biomass as N sources when short-term cultivation was compared to long-term cultivation

(Table 5). Only solar-irradiated biosolids applied to the Spodosol did not significantly enhance effectiveness as a result of longer term ryegrass cultivation (Table 5). Such increases in effectiveness when multiple harvests are assessed suggest that biosolids are more appropriate for supplying N to perennial crops or for long-term cultivation.

Cumulative yields from six consecutive harvests in the Spodosol did not fit asymptotic models and the data graphically displayed linear responses (Figure 4a). Biosolids are recognized sources of N and P (Al-Dhumri et al., 2013; Antille et al., 2013), and the shift from asymptotic models to linear regression indicates a shortage of plant available N rather than P, because plants demand 7-15 times more N than P (Rivero et al., 2009). Cumulative yields in the Oxisol satisfactorily fitted asymptotic models (Figure 4b, Table 5) even for composted biosolids, for which the first harvest displayed a linear response (Table 5). In the Oxisol, no biosolid was as effective to produce ryegrass biomass as the chemical fertilizer ($NH_4H_2PO_4$ + basal application) in the first harvest, but all biosolids

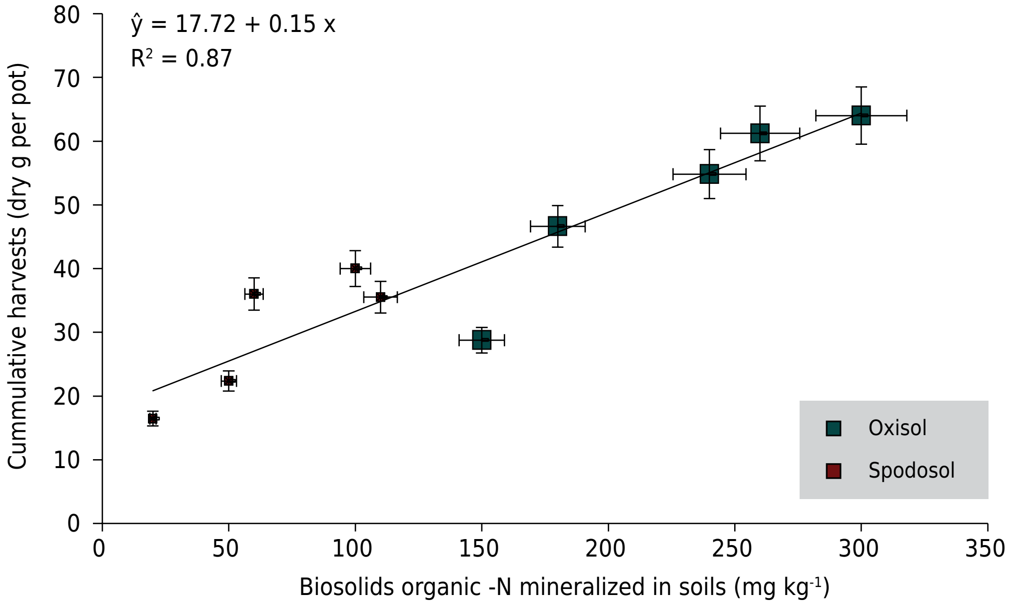

Figure 3. Ryegrass cumulative harvests as a function of biosolids-N mineralized in soils in 11 months of a plant trial.

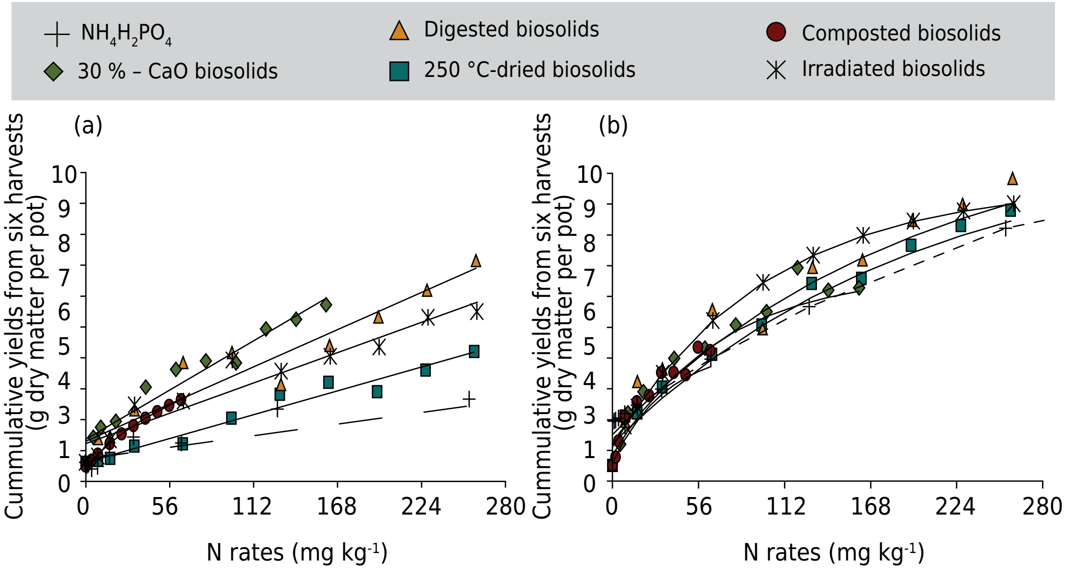

Figure 4. Cumulative yields in responses to biosolids-N application and to $NH_4H_2PO_4$ + basal application in Spodosol (a) and Oxisol (b).

outperformed chemical fertilization on a cumulative basis (Table 5). An advantage of biosolids over chemical fertilizers is the slow release of nutrients that keeps soil fertility elevated for a longer time (Mitchell et al., 2000).

CONCLUSIONS

The stabilization processes of sewage sludge affected both the biosolids-N agronomic rates and the effectiveness to produce plant biomass, which was a function of biosolids-N content mineralized in soils. In this regard, solar-irradiated biosolids showed the highest efficiency to produce plant biomass in a short eight-week trial. In an eleven-month period, solar-irradiated and 30 %-CaO biosolids outperformed digested, composted and 250 °C-dried biosolids.

Biosolids-N agronomic rates varied from 68 to 220 mg kg^{-1}, according to soil and biosolids type. Because biosolids-N mineralized more in Oxisol than in Spodosol, ryegrass yields were from 1.5 (digested biosolids) to 2.5 (250 °C-dried biosolids) times higher in the former soil than in the latter one.

Regardless of the soil type, biosolids were more effective as long-term N sources than when used for short-term crops. Overall, biosolids could substitute for N fertilizers with similar or higher effectiveness to produce plant biomass, depending on the sewage sludge stabilization process, soil type and cultivation term considered.

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