

Effect of a Newly Developed Pelleted Papermill Biosolids on Crop and Soil

Morteza Mozaffari*, Hannah C. Hays

Northeast Research and Extension Center Keiser, Department of Crop Soil and Environmental Science,
Division of Agriculture University of Arkansas System, Arkansas, USA

Email: *mmozaff@uark.edu

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Abstract

The US is one of the leading global producers of paper industry with approximately 24 percent of the share of world paper supply. Despite diversity of the feedstock and production methods, C rich papermill biosolids (PB) is a major byproduct of paper production process. Landfilling is the predominant method of PB management. Increasing landfill cost and its potential environmental consequences have incentivized research and development efforts to find beneficial uses for PB. This sensible option reduces the overall paper production costs and increases environmental sustainability. Pelletization of PB increases its marketability by reducing transportation costs. This greenhouse study was conducted to gain a better understanding of the properties and effects of a recently developed pelletized papermill biosolids (PPB) on bell pepper (*Capsicum annuum* L.) and soil. Urea and PPB were each applied at four total N rates equivalent to 45, 90, 135, and 180 kg N ha⁻¹ and an additional control treatments of 0 N was included. The total C and N concentration in this PPB were 379 and 14 g·kg⁻¹ respectively and its C:N ratio was 27.2. Nitrogen treatment significantly ($P \leq 0.0839$) influenced pepper height, dry biomass, N concentration, and N uptake. Plant height ranged from 31.2 to 44.4 cm; 135 kg·ha⁻¹ urea-N and PPB-N produced the tallest and shortest plants respectively. Dry biomass of the pepper that did not receive any N, those treated with urea-N or PPB-N were 5.3, 5.7 - 7.5, and 5.9 - 6.5 g·plant⁻¹ respectively. Nitrogen concentration in control treatment (0 N) was 36.4 g·kg⁻¹ and that of pepper treated with any N ranged from 32.0 - 40.7 g·kg⁻¹. There was an inverse numerical, albeit not always statistically significant, relationship between PPB rate and plant N concentration. Generally, pepper treated with urea removed significantly more N from soil than control or PPB treated pepper. Nitrogen uptake by plants that did not receive any N and those amended with urea or PPB were 194, 229 - 270, and 155 - 164 mg·plant⁻¹ respectively. Pepper N uptake and concentration data indicate that

higher rates of PPB resulted in immobilization of native soil and PPB-N due to its wide C:N ratio (27.2). Nitrogen treatment significantly influenced soil pH, SOM, total C and N ($P > 0.1$). Soil organic matter and total C in post-harvest soil samples were 17.4 - 19.4 and 21.9 - 35.0 g·kg⁻¹ respectively. The observed increase in soil total C and SOM highlights the potential beneficial use of PPB as a means to improve soil health and sequester C in soil. Narrowing the C:N ratio of PPB, by coapplication or incorporation of the mineral N into the pellets will make it an attractive organic N fertilizer.

Keywords

Pelleted Papermill Biosolids, Pepper, Nitrogen, Urea, Beneficial Use

1. Introduction

The US paper industry produces more than 24% of the world paper using several chemical and mechanical methods for production of paper from pulp [1]. Regardless of the production method, biosolids is a major byproduct of paper production. Approximately 5.5 million Mg of dry papermill biosolids (PB) is produced by the US pulp and paper industry [2]. Traditionally landfilling has been the most widely used method of PB management. The increasing costs and environmental concerns about this practice have necessitated research and development efforts to find a more sustainable solution for PB management. Papermill biosolids is a mixture of organic compounds such as lignin, cellulose, hemicellulose and secondary treated PB contains N, P and additional nutrients. In recent decades beneficial utilization of PB as a soil amendment has emerged as a sensible alternative to landfilling. A median N and P values of 23.3 and 4.2 g kg⁻¹ respectively were reported for a secondary treated PB Camberato *et al.* (1997) [3], Camberato *et al.* (2006) [4]. The organic C, total N, and total P content of another secondary treated PB were 238, 26.7, and 15.3 g·kg⁻¹ respectively Price *et al.* (2007) [5]. These results demonstrate that plant nutrients (N, P) and organic matter content of PB is determined by the feedstock, paper production method and biosolids treatment process, thus they can be quite variable. This variability indicates that each specific type of PB should be evaluated and utilized based on its effect on soil plant system to ensure a successful beneficial use program.

The effect of various PB on crop and soil has been investigated in field and greenhouse studies with a variety of crops. Application of 100 Mg·ha⁻¹ PB with a C:N of 86 reduced barley (*Hordeum vulgare* L.) yield as compared to non-amended plants Aitken *et al.* (1998) [6]. Grain yield and N use efficiency of corn (*Zea Mays* L.) treated with 150 kg total N ha⁻¹ from two PBs with C:N ratio of 13 and 50 were 11.9 and 6.9 Mg·ha⁻¹ and 12% to 22% respectively ($P \leq 0.001$) Gagnon *et al.* (2012) [2]. According to Vagstad *et al.* (2001) [7] application of 10 Mg PB ha⁻¹ with C:N ratio of about 30 or more tended to decrease barley grain yield. Application of a PB with C:N ratio of 14 increased the yield and N uptake of

barley while application of another PB with C: N ratio of 31 did not influence the dry bean (*Phaseolus vulgare* L.) yield Ziadi *et al.* (2013) [8]. They also reported that anion exchange extractable soil $\text{NO}_3\text{-N}$ was decreased by application of a deinking PB with C:N ratio of 65 (N-immobilization) and increased with application of a combined (primary and secondary treated) PB with C:N ratio of 14 [8]. Net N mineralization in an Entisol amended with $10 \text{ Mg}\cdot\text{ha}^{-1}$ of a PB (C:N = 15.5) was not significantly more than that of the non-amended soil San Martin *et al.* (2016) [9]. In general application of PB with wide C:N ratio had increased N immobilization but application of PB with low C:N ratio had increased soil inorganic N Nunes *et al.* (2008) [10], Cabral *et al.* (1993) [11]. These outcomes indicate that the C:N ratio of the PB is a major determinant of the crop response to PB application. Most researchers have suggested that a $\text{C:N} > 30$ will negatively impact crop yield potential and some have even reported a critical C:N ratio of 20 - 30 Cordovil *et al.* (2007) [12], Sims (1990) [13].

Amending soil with PB increased soil C and thus organic matter and the magnitude of the increase was dependent on PB composition and application rate [4] [14] [15]. For example amending a soil with 40, 80, and $120 \text{ Mg}\cdot\text{ha}^{-1}$ of secondary treated PB increased the soil organic C by 0.4, 0.6, and $1.3 \text{ g}\cdot\text{kg}^{-1}$ respectively [10]. These studies clearly demonstrate that soil and plant response to PB depends on several factors including its chemical properties (a function of production methods), application rate, and crop grown. Thus a successful beneficial use program requires information on crop and soil response to PPB when it is applied at agronomically reasonable rates.

Only a small percentage of total amount of PB produced in the US is currently utilized as a beneficial soil amendment, despite environmental and economic benefits of this sustainable practice. Widespread beneficial use of PB has been hampered by its high moisture content and bulky nature which increases the cost of long-distance transport and the need for specialized field application equipment. Pelletization (by heat and pressure) of biosolids will help to overcome these obstacles. Cooperative efforts in the US have led to successful development of a new pelleted papermill biosolids (PPB) which is a mixture of PB and a byproduct of cow manure. This newly developed PPB is currently at testing stage of product development. The objectives of this research were to measure and compare the effects of several rates of PPB, urea, and a 0 N control on pepper: 1) plant height; 2) N concentration and uptake; 3) selected soil properties.

2. Experimental Procedures

A replicated greenhouse experiment was conducted at the University of Arkansas Northeast Research and Extension Center (NEREC) in Keiser Arkansas in 2017 (N: 35.674988° , W: -90.084732°). We evaluated pepper and soil response to urea and PPB each applied at four total N rates equivalent to 45, 90, 135, and $180 \text{ kg total N ha}^{-1}$. The PPB applications rates were approximately equivalent to 2.24, 4.48, 6.72, and 8.48 Mg ha^{-1} on as is basis. A control treatment of 0 N was

also included. Detailed experimental treatments are listed in **Table 1**. Experimental design was a randomized complete block and each treatment was replicated five times.

Greenhouse Cropping:

A bulk sample of the 0 - 15 cm depth of a soil mapped as Deerfield loamy fine sand (mixed, mesic Aquic Udipsamments) was collected, dried to constant moisture in a forced-air oven at 40°C, and ground to pass a 2-mm sieve. Dried soil sample was thoroughly mixed in a new cement mixer. Soil texture was measured by the hydrometer method [16]. Soil pH was measured by 1:1 soil: water [17] and soil organic matter (SOM) was measured gravimetrically by loss on ignition (LOI) [18]. Soil total carbon (C) and N were measured by combustion using an Elementar Variomax instrument [19]. Soil NO₃-N and NH₄-N were extracted by 2-M KCl and measured on a Skalar auto analyzer [20]. Those two are the inorganic sources of soil N which are taken up by plants. Mehlich-3 extractable nutrients were measured by the standard procedure [21]. The PPB was ground to fineness in a new coffee grinder and analyzed for pH, total C, N, NO₃-N and NH₄-N as mentioned before. Total P and K in the PPB were measured as outlined by Peters [22].

We amended the bulk soil sample with monocalcium phosphate ((CaH₂PO₄)₂), potassium chloride (KCl), sulfate of potash and magnesia (Sul-Po-Mag), zinc sulfate (ZnSO₄) and pelletized lime to supply the equivalent of 56, 112, 36, 48, 8.4, and 2800 kg·ha⁻¹ of: P₂O₅, K₂O, Mg, S, Zn, and lime respectively. This ensured that N was the only soil amendment that could limit pepper yield potential.

The experimental units consisted of 24-cm diameter-7.2-liter black plastic pots. The required amount of N-treatment for all five replications of each treatment was thoroughly mixed with the appropriate quantity of soil in a cement

Table 1. Nitrogen sources, total N application rates, and eight N-treatments for a pepper experiment conducted in a greenhouse at the University of Arkansas Northeast Research and Extension Center in Keiser, Arkansas in 2017.

N source	Amendment rate	Total N rate	N-treatment
	kg·ha ⁻¹	kg N ha ⁻¹	
None	0	0	none
Urea	98	45	Urea-45
Urea	195	90	Urea-90
Urea	292	135	Urea-135
Urea	382	180	Urea-180
PPB	2240	45	PPB-45
PPB	4480	90	PPB-90
PPB	6720	135	PPB-135
PPB	8480	180	PPB-180

mixer. Then 9 kg of N-treated soil was transferred from cement mixer into each plastic pot and the pot was tamped three times to create a uniform soil bulk density. Five seeds of a bell pepper Hybrid “Alliance F1” were planted in each pot on 5-July 2017 and thinned to one seedling per pot five days after germination. Pepper was grown for 89 days. Greenhouse temperature was set to maintain at 24°C. Supplemental light was provided 12 hrs·day⁻¹ from 7:00 am to 7:00 pm and pepper was watered as needed. Pepper was grown for 77 days and harvested on 3-October 2017.

Post-harvest Pepper and Soil Sample Collection and Analysis

At the end of the study, we measured and recorded the height of each pepper plant from the lowest node above the soil level to the top of the apical meristem, then cut the total above-ground portion of each plant at 1 cm above the soil level. Plant samples were dried to constant weight in a forced-air oven and their dry biomass was recorded. Plant samples, from all five replications, were ground in a Willey Mini-Mill to pass a 20-mesh sieve and analyzed for total N with combustion method [19]. Total N uptake per plant was calculated by multiplying the whole plant dry biomass by its respective N concentration.

After pepper harvest we transferred the soil from each pot (selected N-treatments, 0, 90 and 180 kg total N ha⁻¹) to a clean plastic tub, removed the roots manually, mixed the soil thoroughly, and collected representative samples by the quarter method. Postharvest soil samples were dried as described, ground to pass a 2-mm-sieve and analyzed for pH, total N, NH₄-N, NO₃-N, and SOM as described above.

Statistical Analysis

The effect of N-treatment on pepper growth parameters and selected soil properties were evaluated by analysis of variance using the SAS software package. When appropriate, means were separated by the least significant difference (LSD) method and interpreted as significant when $P \leq 0.10$.

3. Results and Discussion

Characterization of Soil and PPB

Analysis of the pre-amendment soil indicated that the soil texture was sandy loam where sand and clay were the most and least predominant soil particles respectively. Soil pH was 5.5, SOM, total C and N were 28, 12.4 and 0.64 g·kg⁻¹ respectively (**Table 2**). Soil inorganic N was low (13.2 mg·kg⁻¹) and was predominated by NH₄-N. Mehlich-3 extractable K was 28 mg·kg⁻¹ indicating the need for supplemental K fertilization.

The pH of PPB was 7.7 and was within the range of values reported by other PB researchers [5] [8] [23] [24] [25]. Total C and N concentrations were 379 and 14 g·kg⁻¹ respectively and the C:N ratio was 27.2 (**Table 3**). Concentration of total C and N in a mixed pelleted biosolids was 260 and 16.8 g·kg⁻¹ respectively [9] [5]. Total C and total N concentrations of 256 and 3.0 g·kg⁻¹ respectively were reported for a PB sample from a Papermill in Canada [5]. Total C and N

Table 2. Selected mean (n = 2) chemical properties of the thoroughly mixed untreated bulk soil sample of the surface horizon of a Deerfield loamy fine sand that was used in the greenhouse study with pepper at the University of Arkansas Northeast Research and Extension Center, in Keiser, Arkansas in 2017.

Soil pH	Sand	Silt	Clay	Soil Organic Matter	Total C	Total N	NO ₃ -N	NH ₄ -N	Mehlich-3-extractable nutrients			
									P	K	Ca	Mg
----- g·kg ⁻¹ -----					----- mg·kg ⁻¹ -----							
5.5	630	300	70	28	12.42	0.64	1.52	11.70	75	28	176	25

Table 3. Selected mean properties of the finely ground sample of the pelletized Papermill biosolids (PPB) that was used in a greenhouse study with pepper at the University of Arkansas Northeast Research and Extension Center in Keiser, Arkansas in 2017.

Test Material	pH	Total C	Total N	C/N ratio	NH ₄ -N	P ₂ O ₅	K ₂ O
Pelletized Papermill biosolids (PPB)	7.7	378.7	13.9	272	0.18	5.0	6.8

concentrations of 276 and 3 mg·kg⁻¹ and C:N of 92 respectively have been reported for another PB [10]. Total C and N concentration range of 329 - 438 and 10.5 - 31.5 g·kg⁻¹ respectively were reported for two combined PB [8].

The C and N concentration of this newly developed PPB suggest its potential as an organic source of N. Nitrate-N was below the detection limit and NH₄-N was very low (0.18 g·kg⁻¹) therefore organic N was the predominant source of N. This is in agreement with others including Gagnon *et al.* (2012) [2] who reported that only 1% of total N in a combined PB was in NH₄-N. The C:N ratio and low inorganic N content of this PPB suggested that N mineralization/immobilization was the key decisive factor in determining its N supplying capacity. The PPB contained several other plant essential nutrients including P and K, thus it is a potential high organic matter low grade source of those two nutrients.

Pepper Response to N-treatment

Pepper dry biomass, N concentration, and N uptake were significantly ($P \leq 0.0839$) influenced by N-treatment (Table 4). Plant height ranged from 31.2 to 44.4 cm where 135 kg ha⁻¹ of urea-N and PPB-N produced the tallest and shortest plants respectively. Dry biomass of the peppers that did not receive any N, those treated with urea-N or PPB-N were 5.3, 5.7 - 7.5, and 5.9 - 6.5 g·plant⁻¹ respectively. Pepper that did not receive any N and that treated with 45 kg urea-N ha⁻¹ produced the numerically smallest and largest plant dry biomass (5.3 vs 7.5 g·plant⁻¹). This numerical trend is consistent with Gagnon *et al.*, (2012) [2] who reported corn dry biomass of 7, 9.7, and 14 Mg·ha⁻¹ for plants that received 0 and 150 kg N ha⁻¹ from PB and mineral N fertilizer respectively. Pepper fertilized with 180 kg urea-N ha⁻¹ appeared dark green and produced significantly smaller dry biomass than plants treated with 45 kg urea-N ha⁻¹ reflecting excessive N supplied by that higher N-rate. Pepper amended with high PPB rates exhibited visual symptoms consistent with N deficiency (yellowing of the lower leaves).

Table 4. Pepper plant height, whole plant dry biomass, N concentration and total N uptake as affected by N-treatment from urea and pelletized Papermill biosolids (PPB) in a greenhouse study conducted at the University of Arkansas Northeast Research and Extension Center in Keiser, Arkansas in 2017.

N source	Total N-rate kg·ha ⁻¹	N-treatment	Plant height cm	Plant dry biomass g·plant ⁻¹	N concentration ---- g·kg ⁻¹ ----	N uptake mg N plant ⁻¹
None	0	none	35.3bc ^z	5.3c	36.4ab	194cde
Urea	45	Urea-45	37.2b	7.5a	36.2bc	270a
Urea	90	Urea-90	35.3bc	6.1bc	40.7a	249ab
Urea	135	Urea-135	44.4a	7.4a	36.3abc	269a
Urea	180	Urea-180	37.5b	5.7bc	39.8ab	229c
PPB	45	PPB-45	33.8bc	6.5ab	32.0cd	208cd
PPB	90	PPB-90	33.8c	6.0bc	28.9de	164ef
PPB	135	PPB-135	31.2c	5.9bc	27.8de	155f
PPB	180	PPB-180	32.5c	6.4abc	25.7e	163f
<i>P</i> -value			0.0021	0.0839	<0.0001	<0.0001

^zmeans followed by the same letter are not significantly different at *P*-value = 0.10.

Dry biomass of PPB treated pepper was 5.9 to 6.5 g·plant⁻¹, was not significantly affected by PPB rate and was not generally different than plants that did not receive any N. Similar to our work, Norris *et al.* (2012) [26] reported that increasing the PB application rate decreased the dry biomass of perennial ryegrass (*Lolium Perrenne* L.).

Nitrogen treatment significantly influenced pepper N concentration (*P* < 0.0001). The N concentration of pepper that did not receive any N was 36.4 g·kg⁻¹ and that of pepper receiving urea-N or PPB-N were 36.2 - 40.7 and 25.7 - 32.0 g·kg⁻¹ respectively. Increasing PPB rate consistently albeit not always significantly lowered the plant N concentration. This is in agreement with Simmard *et al.* (1998) [27] who reported that increasing PB application rate decreased N concentration in barley grain and straw. Similar results were reported by others. Nitrogen concentration of pepper treated with any PPB-N was 13% - 30% less than pepper that did not receive any N and 12% - 27% less than plants fertilized with any urea-N (*P* < 0.0001). The lower pepper N concentrations are consistent with the general yellowish green color of the PPB amended pepper. Reduction in crop N concentration in soil amended with PB has also been reported by others [26] [28].

Pepper N uptake was significantly (*P* < 0.0001) influenced by N-treatment and reflected the combined effect of N-treatment on plant dry biomass and N concentration. Nitrogen uptake by pepper that was not fertilized with any N, urea-N, and PPB-N was 194, 229 - 270, and 155 - 164 mg·plant⁻¹ respectively. In general urea-N treated pepper removed significantly more N from soil; pepper fertilized with 180 kg urea-N ha⁻¹ was an exception. A similar trend was observed by Gagnon *et al.* (2012) [2] who noted that N accumulation by silage corn

fertilized with 150 kg inorganic N ha⁻¹ was significantly more than corn treated with the same amount of total N from a PB with C:N ratio of 50. Smaller pepper N uptake at the highest rate of urea-N is a reflection of smaller plant dry biomass caused by excessive N supply as evidenced by dark green plant color. In general, pepper treated with urea-N removed more N (from the soil) than plants treated with PPB-N. Nitrogen uptake by plants amended with ≥ 90 kg PPB-N ha⁻¹ was significantly less than pepper that did not receive any N. The N concentration and uptake data indicate that higher rates of PPB resulted in microbial sequestration of the native soil and PPB-N due to its high organic C content. This is supported by the relatively wide C:N ratio (27.2) and very low inorganic N content of our PPB. Nitrogen immobilization has been reported when PB with C:N ratio > 30 was applied to soil by others [26] [27] [28]. Other researchers, have noted N immobilization at C:N ratio of 12 - 30 [12] [13] [29]. Our data indicates that for this particular PPB, N immobilization occurred at the C:N of 27.2. From a beneficial use perspective, N immobilization, caused by wide C:N ratio of an organic byproduct, can be mitigated by co-application of inorganic N [30] [31] or incorporation of inorganic N into the pellets Zerbath *et al.* (2005) [32], Pawlett *et al.* (2015) [33], Smith *et al.* (2015) [34], and Antille *et al.* (2014) [35].

Post-Harvest Soil Samples

Soil pH, SOM, total C, N, NH₄-N, and NO₃-N were significantly influenced by N treatment ($P > 0.1$, **Table 5**). Soil organic matter and total C in post-harvest soil samples ranged 17.4 - 19.4 and 21.9 - 35.0 g·kg⁻¹ respectively. Application of 180 kg PPB-N ha⁻¹ produced significantly more SOM than the other treatments and total C in soil treated with any 180 kg N ha⁻¹ was significantly more than all other treatments. This confirms that PPB is a good source of C and organic matter similar to the other types of bulk PB investigated by other researchers [2] [5] [15], Manirakiza *et al.* (2019) [36], Foley *et al.* (2002) [37], and N'Dayegamiye *et al.* (2003) [38]. The observed increase in SOM and total C brought about by PPB, points to the potential soil health benefits of PPB, provided that its C:N ratio can be reduced to control microbial sequestration of N. The concentration of NH₄-N was low (0.11 to 1.63 mg·kg⁻¹) and in general decreased with increasing

Table 5. Effect of urea and pelleted papermill biosolids (PPB), each applied at two total N rates on selected chemical properties of the soil samples taken from experimental pots after pepper harvest for a greenhouse study conducted at the University of Arkansas Northeast Research and Extension Center in Keiser, Arkansas in 2017.

N source	Total N-rate	Soil pH	Soil organic matter	Total C	Total N	NH ₄ -N	NO ₃ -N
	kg ha ⁻¹		----- g kg ⁻¹ -----			----- mg kg ⁻¹ -----	
None	0	6.2b ^z	17.8b	22.4c	1.8b	1.63a	7.2c
Urea	90	5.8c	17.4b	23.8bc	1.8b	1.29ab	46.4a
Urea	180	5.7d	18.0b	32.0a	1.1c	0.40cd	28.0b
Pelleted papermill biosolids (PPB)	90	6.1b	17.5b	21.9c	1.9c	0.62bcd	4.4c
PPB	180	6.4a	19.4a	35.0a	1.0c	0.11d	3.8c
<i>P</i> -value		<0.0001	0.0278	<0.0001	<0.0001	0.020	<0.0001

^zmeans followed by the same letter are not significantly different at P -value = 0.10.

N application rate. Total N in post-harvest soil samples ranged 1.0 - 1.9 g·kg⁻¹, the highest and lowest total N were observed in soil that did not receive any N and soil treated with 180 kg PPB-N ha⁻¹ respectively.

The pH of the soil that did not receive any N (control) was 6.2, and that of the soils treated with urea or PPB were 5.7 - 5.8 and 6.1 - 6.4 respectively. The lower pH of the urea-N treated soil, as compared to control, is attributed to hydrolysis of urea-N to NH₄ and its subsequent conversion to NO₃-N (nitrification). This is supported by the significantly lower concentration of NO₃-N (7.2 mg·kg⁻¹) in the control soil as compared to urea-N treated soils (28 - 46 mg·kg⁻¹, **Table 5**). It is well established that conversion of one mole NH₄ to NO₃ releases five H ions. Hydrogen ion secretion by plant roots during nutrient uptake may have also contributed to reducing the soil pH. Acidifying effect of mineral fertilizers has also been reported in similar experiments Vagstad *et al.* (2001) [7]. In contrast to that, application of high rate of PPB-N significantly increased the soil pH by 0.2 units. This is consistent with Nunes *et al.* (2008) [10], who noted that application of 40 Mg ha⁻¹ of PB increased the pH of two soils by 1 and 1.6 units. Similar results had been reported Cabral *et al.* (1993) [11]. The numerical decrease in post-harvest soil NO₃-N had been observed by Douglas *et al.* (2003) [28].

4. Concluding Remarks

Our greenhouse study established that this newly developed PPB is an excellent source of organic matter that will improve the soil health and enhance C sequestration. It also contains some P. The C:N ratio of this PPB should be reduced to make it an attractive source of organic N for a pepper crop. Future research to determine the optimal rate of supplemental N that should be co-applied or incorporated into this PPB will enhance its widespread use as a beneficial soil amendment.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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List of Abbreviations

g: gram.

mg: milligram.

kg: kilogram.

PPB: pelletized papermill biosolids.

SOM: soil organic matter.