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Corn and Soil Response to a Recently Developed Pelletized Papermill Biosolids

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Abstract

Beneficial utilization of industrial byproducts such as papermill biosolids (PB) provides a unique opportunity to reduce the overall production cost and increase environmental sustainability. Pelletization of a byproduct enhances its marketability by improving the transportation and application. This greenhouse study was conducted to gain a better understanding of the properties and effects of, a recently developed pelletized papermill biosolids (PPB), on corn (*Zea mays* L.) and soil. Urea and PPB were each applied at four total N rates equivalent to 45, 90, 135, and 180 kg·ha^{-1} and an additional control treatments of 0 was also included. The PPB contained 379 and 14 g·kg^{-1} total N and C and its C:N ratio was 27. Nitrogen treatment significantly ($P <$ 0.0001) influenced corn dry biomass, N concentration, and N uptake. Corn dry biomass ranged 26.9 - 41.1 g·plant^{-1} where application of 180 kg·ha^{-1} of PPB-N produced the smallest plant biomass. Numerically the dry biomass of corn that did not receive any N, corn fertilized with any PPB, and corn fertilized with any urea was 38.3, 26.9 - 41.1 and 38.1 - 40.92 g·plant^{-1} respectively. Nitrogen concentration in corn plants ranged 6.2 - 11.5 g·kg^{-1}. Nitrogen concentration in corn that did not receive any urea or corn that received urea was 8.7 - 11.5 g·kg^{-1} and was significantly more than corn treated with any PPB. Total N uptake (removed from soil) by the corn plant was 166 - 455 mg·plant^{-1}. Total N uptake by corn that did not receive any N, corn fertilized with any PPB, and corn fertilized with any urea were 327, 166 - 278, and 379 - 455 mg·plant^{-1} respectively. The data suggest that the high C/N ratio (27.2) of PPB resulted in immobilization of PPB-N. Thus the next step will be to research the optimal rates of inorganic N that should be incorporated into this PPB to reduce its C:N to make it an effective high organic matter content N fertilizer. Nitrogen treatment significantly ($P <$ 0.0001) influenced soil organic matter (SOM) which ranged 18.5 - 19.7 g·kg^{-1}. The SOM of the treatments fertilized with 90 and 180 kg·ha^{-1} of PPB-N was 19.4 - 19.7 g·kg^{-1} and was significantly higher than soil that did not receive any N. The application of PPB significantly increased the soil total C which was 36.0 and 23.6 g·kg^{-1} in the soil amended with 180 kg·ha^{-1} of PPB-N and the control respectively. The observed increase in SOM and total C in PPB treated soil points to the potential soil health and C sequestration benefits of PPB provided that its C/N ratio can be increased by incorporating inorganic N into it.

Keywords

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

1. Introduction

Paper production is a major industry, makes significant contribution to the US economy and generates approximately 5.3 million metric tons of wastewater solids [1]. Beneficial utilization of industrial and agricultural byproducts provides a unique opportunity to reduce the overall production cost and increase environmental sustainability. Developing beneficial uses for papermill biosolids (PB) is an example of this environmentally and economically sound approach. The PB is a mixture of organic compounds such as lignin, cellulose, hemicellulose and secondary treated PB contains N, P and additional nutrients. Camberato *et al.* [2] [3] reported median N and P values of 23.3 and 4.2 g·kg^{-1} respectively for a secondary PB. The organic C, total N, and total P content of a secondary treated PB was reported to be 238, 26.7, and 15.3 g·kg^{-1} respectively [4]. The total C, N, and P content of 276, 3, 11.9 g·kg^{-1} respectively have been reported [5]. Ziadi *et al.*, [6] reported that the total C content of two combined (primary and secondary) PB from Canada were 672 and 438 g·kg^{-1} and total N content the same PBs was 11 and 32 g·kg^{-1}. However, PB from a facility that used a deinking process contained 294 and 4.5 kg^{-1} of total C and N respectively. This data clearly indicate that plant nutrients (N, P) and organic matter content of PB is determined by the feedstock and paper production process and thus are quite variable [2] [3]. This variability highlights the need for evaluation of each specific type of PB for development of a successful beneficial application 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 a PB with a C:N ratio of 14 increased the yield and N uptake of barley (*Hordem vulgare* L.), but application of another PB with the C:N ratio of 31 did not significantly influence the dry bean (*Phaseolus vulgare* L.) yield [6]. Anion exchange extractable soil NO_3-N was immobilized by application of deinking PB with C:N of 65 and increased with application of combined (primary and secondary treated) PB with C:N ratio of 14 [6]. Application of 100 Mg·ha^{-1} primary treated PB with the C:N of 86 reduced barley yield as compared to treatment that did not received any PB [7].

Application of 120 Mg·ha^{-1} of a PB with a C:N ratio 10.6 significantly increased the soil mineral N as compared to the untreated soil. In general application of PB with high C:N ratio had resulted in immobilization of N and application of low C:N ratio PB increased soil inorganic N [4] [8]. The C:N ratio of the PB plays an important role in crop response to PB application. Most researchers suggested that C:N > 30 will negatively impact crop yield potential and some have reported acritical C:N ratio of 20 - 30 [9] [10].

Several Researchers have reported that application of PB had increased soil organic C content with the amount of increase in soil C being dependent on PB composition, processing, and application rate [3] [11] [12]. As an example, application of 40, 80, and 120 Mg·ha^{-1} of secondary treated pulp mill sludge increased the organic C in a soils by 0.4, 0.6, and 1.3 g·kg^{-1} respectively [4].

These studies clearly demonstrate that soil and plant response to PB is dependent on several factors including the nature of the PB, application rate, and crop grown. Thus the development of an economically and environmentally successful beneficial use of PB requires information on crop and soil response to PB when it is applied at agronomically reasonable rates.

Despite the environmental and economic benefits of beneficial use of PB, a small percentage of total amount of PB produced in US is currently utilized as a beneficial soil amendment. The difficulties in long distance transport and field application (requiring specialized equipment) are two major hindrances. Pelletization of PB is an effective means to overcome those obstacles. Cooperative efforts in the US have resulted in successful development of a pelleted papermill biosolids (PPB) which is a mixture of PB and 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, a 0 N control on corn: 1) plant height 2) N concentration and uptake, 3) selected soil properties.

2. Experimental Procedures

In 2017 a replicated greenhouse experiment was conducted at the University of Arkansas Northeast Research and Extension Center (NEREC) in Keiser Arkansas (N: 35.674988˚, W: −90.084732˚). The study evaluated corn and soil response to urea and PPB each applied at four total N rates equivalent to 45, 90, 135, and 180 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} of PPB on as is basis. A control treatments of 0 N was also included. Detailed experimental treatments are listed in **Table 1**. Experimental design was a randomized complete block, each treatment was replicated five times.

2.1. 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

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

N source	Amendment rate ($kg \cdot ha^{-1}$)	Total N rate (kg N ha^{-1})	N-treatment
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

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 pH was measured by 1:1 soil:water [13] and soil organic matter (SOM) was measured gravimetrically by loss on ignition (LOI) [14]. Soil total C and N were measured by combustion using an Elementar Variomax instrument [15]. Soil NO_3-N and NH_4-N were extracted by 2-M KCl and measured on a Skalar auto analyzer [16]. 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 [17]. Relative amount of sand, silt, and clay were measured by the hydrometer method [18]. The PPBwas ground to fineness in a new coffee grinder and analyzed for pH, total C, total N, NO_3-N, and NH_4-N as described. Total P and total K were measured by standard method and converted to P_2O_5 and K_2O equivalents [19].

We amended the bulk soil sample with monocalcium phosphate ($(CaH_2PO_4)_2$), potassium chloride (KCl), sulfate of potash and magnesia (Sul-Po-Mag), zinc sulfate ($ZnSO_4$) and pelletized lime to supply the equivalent of 56, 112, 36, 48, 8.4, and 2800 $kg \cdot ha^{-1}$ of: P_2O_5, K_2O, Mg, S, Zn, and lime respectively. This ensured that N was the only soil amendment that could limit the corn yield potential.

The experimental units consisted of 24-cm diameter-7.2-liter black plastic pots. The required amount of N source for all five replications of each treatment was thoroughly mixed with the appropriate quantity of soil in a cement mixer. Then 9 kg of N-treated soil was transferred into each plastic pot and the pot was tamped three times to create a uniform soil bulk density. Five seeds of corn hybrid DEKALB "DK C67-14", a high biomass producing hybrid, were planted in each pot on 5-July 2017 and thinned to one seedling per pot five days after germination. Greenhouse temperature was set to maintain at 24˚C. Supplemental light was provided 12 $hrs \cdot day^{-1}$ from 7:00 AM to 7:00 pm and corn was watered as needed. Corn was grown for 77 days and the experiment was terminated on 19-September 2017 when most plants had reached the silk growth stage (R-1).

2.2. Corn and Soil Sample Collection and Analysis

At the end of the study we measured and recorded the height of each corn plant from the lowest node above the soil level to the top of the tassel and 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 plant 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. Total N uptake per plant was calculated by multiplying the whole plant biomass by its respective N concentration.

After the corn 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 samples were ground to pass a 2-mm-sieve and analyzed for pH, total N, NH_4-N, NO_3-N, and SOM as described before.

2.3. Statistical Analysis

We performed analysis of variance (ANOVA) to evaluate and compare the effect of N-treatment on corn growth parameters and post-harvest soil properties 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

3.1. Characterization of Soil and N-Sources

Prior to addition of any soil amendment, the soil pH was 5.5 and SOM was 28 $g \cdot kg^{-1}$ (**Table 2**). Soil texture was sandy loam; sand and clay were the most and least predominant soil particles respectively. Soil inorganic N was 13.2 $mg \cdot kg^{-1}$ and NH_4-N was the predominant form. Mehlich-3 extractable K was 28 mg/kg indicating the need for supplemental K fertilization.

The PPB had a pH 7.7 which is within the range of values reported by Price *et al.* (2009) [5] and Ziadi *et al.* (2013) [6]. Carbon and N concentrations were 379 and 14 $g \cdot kg^{-1}$ respectively and C:N ratio was 27.2 (**Table 3**). The C and N concentrations of 276 and 3 $mg \cdot kg^{-1}$ and C:N ratio of 92 respectively have been reported by others [5]. The C and N concentrations of 329 - 438, 10.5 - 315 $mg \cdot kg^{-1}$ and C:N of 14 - 31 for two secondary treated PPB have been reported [6].

Nitrate-N was below the detection limit and NH_4-N was very low (0.18 $g \cdot kg^{-1}$) indicating that organic N was the predominant source of N. Therefore, N mineralization/immobilization is expected to be the key determinant of the N fertilizer value of PPB. The PPB contained several plant essential nutrients including

Table 2. Selected mean (n = 2) properties of the thoroughly mixed untreated bulk soil sample of the surface horizon of a Deerfield loamy fine sand that was used for the greenhouse study 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 papermillbiosolids (PPB) that was used in a greenhouse study with corn 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_2O_5	K_2O
				g·kg^{-1}			
Pelletized papermill biosolids (PPB)	7.7	378.7	13.9	272	0.18	5.0	6.8

(P) and potassium (K), thus it is a potential low grade and high organic matter source of those two nutrients.

3.2. Corn Response to N-Treatment

Nitrogen treatment did not significantly influence ($P \geq 0.1$) plant height which numerically ranged 175 - 183 cm (**Table 4**). However, it significantly ($P <$ 0.0001) affected corn dry biomass, N concentration, and N uptake. Corn dry biomass ranged 26.9 - 41.1 g·plant^{-1}. Dry biomass of corn that did not receive any N was 38.3 g·plant^{-1}and was not significantly different than urea-N treated corn (38.1 - 40.9 g·plant^{-1}) or corn treated with 90 kg PPB-N ha^{-1}. Application of 180 kg·ha^{-1} of PPB-N produced the significantly smallest plant biomass (26.9 g·plant^{-1}). Application of ≥135 kg PPB-N ha^{-1} significantly reduced the corn dry biomass as compared to lower rates of PPB, 0-N, or urea-N treatments. There was a trend of decreasing plant biomass with increasing PPB rate. This is consistent with Gagnon *et al.* (2012) [20] who reported corn dry biomass of 9.7, and 14 Mg·ha^{-1} for plants that received 150 kg N ha^{-1} from PB and mineral fertilizer respectively. It is also similar to the effect of a deinking PB, where increasing the PB application rate decreased the dry biomass of perennial ryegrass (*Lolium Perrenne*) (Simard *et al.*, 1998) [21]. However, N'Dayegamiye *et al.* (2003) [22] reported that application of 3.2 Mg·ha^{-1} (dry weight) PB, with C:N ratio of 14, significantly increased corn dry biomass as compared to 0 N control. In our study corn amended with high PPB rates exhibited visual symptoms consistent with N deficiency (yellowing of the lower leaves). The difference between our results and theirs can be attributed to the wider C:N ratio of the PPB we used (14 vs. 27.2). We believe that the wide C:N ratio (27.2) of our PPB

Table 4. Effect of urea, and pelletized Papermill biosolids (PPB) on plant height, whole plant dry biomass, N concentration, and total N uptake by corn plants in the greenhouse study conducted at the University of Arkansas Northeast Research and Extension Center in Keiser, Arkansas in 2017.

N source	Total N-rate ($kg{\cdot}ha^{-1}$)	N-treatment	Plant height (cm)	Plant dry biomass ($g{\cdot}plant^{-1}$)	N concentration ($g{\cdot}kg^{-1}$)	N uptake (mg N $plant^{-1}$)
None	0	none	179a[z]	38.3ab	8.7b	327bc
Urea	45	Urea-45	175a	40.9a	9.5b	379ab
Urea	90	Urea-90	178a	38.1ab	10.2 ab	388ab
Urea	135	Urea-135	175a	39.1ab	11.5a	455a
Urea	180	Urea-180	183a	39.5ab	10.1ab	421a
PPB	45	PPB-45	178a	41.1a	6.7c	278cd
PPB	90	PPB-90	180a	36.9ab	6.7c	245d
PPB	135	PPB-135	180a	32.7c	6.3c	210de
PPB	180	PPB-180	179a	26.9d	6.2c	166e
P value			0.9858	<0.0001	<0.0001	<0.0001

[z]means followed by the same letter are not significantly different at *P* value = 0.10.

promoted immobilization of soil and PPB-N. Several researchers have noted that application of PB with C:N ratio > 30 can result in microbial sequestration of N in soil [7] [23] [24]. Some other researchers, have noted a critical C:N ratio of 12 - 30 [12]. Our data indicates that N immobilization occurred at the C:N ratio of 27.2.

From a beneficial use perspective, the C/N ratio of the PPB can be reduced by incorporating inorganic N (from a nitrogen fertilizer) into the pellets. This approach have been tested by several researchers with generally successful outcomes [25] [26] [27].

Nitrogen concentration in corn ranged 6.2 - 11.5 $g{\cdot}kg^{-1}$ and there was no significant difference in N concentration between plants that did not received any N (8.7 $g{\cdot}kg^{-1}$) and plants fertilized with 45 or 90 kg urea-N ha^{-1} (9.5 - 10.2 $g{\cdot}kg^{-1}$). However, PPB application significantly reduced corn N concentration as compared with the corn that did not receive any N (6.2 - 6.7 vs 8.7 $g{\cdot}kg^{-1}$). On average the application of PPB reduced the corn N concentration by 26% as compared to the corn that did not receive any N. At any given N application rate, the N concentration of urea-N treated corn was significantly more than PPB-N treated corn. Increasing PPB application rate often numerically albeit not significantly reduced corn N concentration. This is in agreement with Simmard *et al.* [21] who reported that increasing PB application rate decreased N concentration in barley grain and straw. Similar results were reported by Feagly *et al.* (1994) [28] and Douglass *et al.* (2003) [29]. The trend in our corn N concentration indicates N immobilization by PPB application.

Total N uptake (removed from soil) by the corn plant reflected corn dry biomass and N concentration. Total N uptake by corn that did not receive any N and corn fertilized with urea-N or PPB-N were 327, 166 - 278, and 379 - 455 mg·plant^{-1} respectively. This is in agreement with Gagnon *et al.* (2012) [20] who noted that N uptake by silage corn fertilized with 150 kg inorganic N ha^{-1} was significantly more than corn that received the same amount of total N from a PB with C:N ratio of 50. Nitrogen uptake by any urea treated corn was significantly more than corn treated with any PPB. There was no significant difference in N uptake among the plants that did not receive any N and those fertilized with 45 or 90 kg urea-N ha^{-1}. Plants treated with 180 kg·ha^{-1} urea-N were very dark green, a symptom consistent with excessive N uptake.

3.3. Post-Harvest Soil Samples

Nitrogen treatment did not significantly influence the soil pH ($P > 0.1$, **Table 5**), but significantly ($P < 0.0001$) influenced SOM which ranged 18.5 - 19.7 g·kg^{-1}. The SOM of the treatments that received 90 and 180 kg PPB-N ha^{-1} were 19.7 and 19.4 g·kg^{-1} respectively and were significantly higher than soil that did not receive any N (SOM = 18.5 g·kg^{-1}). This is a small increase in SOM, however the amount of PPB applied at those two rates (**Table 1**) was ≤8480 kg·ha^{-1} which is considered a low rate. The observed increase in SOM, brought about by PPB, points to the potential soil health benefits of beneficial use of higher rates of PPB, provided that its C:N ratio can be reduced to control N-immobilization. Nitrogen treatment significantly ($P < 0.0001$) influenced soil total C and N. Total C in the soil that did not receive any N was 23.6 g·kg^{-1} and that of the soil treated with 180 kg N ha^{-1} by urea or PPB were 33.4 and 36.0 g·kg^{-1} respectively. The increase in soil C is consistent with Rotenberg *et al.* (2005) [30] where application of secondary PB increased soil organic C. Similar to the increase in SOM, amending soil with PPB is a potential means of C sequestration and thus it is environmentally appealing.

Table 5. Effect of urea and pelletized Papermill biosolids (PPB), each applied at two total N rates on selected chemical properties of the soil samples taken from experimental pots after corn harvest in the 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	Soilorganic matter	Total C	Total N	NH_4-N	NO_3-N
	lb N/acre		g·kg^{-1}			mg·kg^{-1}	
None	0	6.1b^z	18.5b	23.6b	2.1a	0.31b	6.7c
Urea	90	6.0b	18.0b	21.6b	1.8b	0.49b	14.6b
Urea	180	6.0b	19.6a	33.4a	1.3c	2.29a	20.2a
PPB	90	6.5b	19.7a	23.3b	1.8ab	0.18b	1.6d
PPB	180	6.2b	19.4a	36.0a	1.1c	0.27b	2.1d
P value		0.1112	<0.0001	<0.0001	0.0001	<0.0001	<0.0001

^{z}means followed by the same letter are not significantly different at *P* value = 0.10.

Total N in post-harvest soil samples ranged 1.1 - 2.1 g·kg^{-1} where the highest and lowest total N were measured in soil that did not receive any N and soil treated with 180 kg·ha^{-1} of PPB-N respectively. The inorganic forms of N (NH_4-N and NO_3-N) were significantly ($P < 0.0001$) affected by N-treatment. Nitrate-N was the predominant form of soil inorganic N (1.6 - 20.2 mg NO_3-N kg^{-1}) as compared to NH_4-N (0.18 - 2.29 mg NH_4-N kg^{-1}). Soil NH_4-N was highest in soil that was treated with 180 kg·ha^{-1} urea-N perhaps as a result of hydrolysis of urea in soil. Nitrate-N concentration was highest in the urea treated soil (14.6 - 20.2 mg NO_3-N kg^{-1}), lowest in soil treated with PPB (1.6 - 2 mg NO_3-N kg^{-1}), and that of soil that did not receive any N was 6.7 mg·kg^{-1}. The low concentration of NO_3-N in PPB treated soil is another indicator of microbial sequestration of PPB-N.

4. Concluding Remarks

The outcome of this research suggests that PPB is a good source of organic matter, will improve soil health and enhance C sequestration. However, its C:N ratio needs to be reduced such that it can supply N to a growing crop of corn. Therefore, future research with this PPB should focus on evaluating the optimal rate of supplemental N that should be added to make it an effective source of N.

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