agronomy

Article

Annual Ryegrass (*Lolium multiflorum* Lam.) Growth Response to Nitrogen in a Sandy Soil Amended with Acidified Manure and Municipal Sludge after "Quick Wash" Treatment

Wooiklee S. Paye, Ariel A. Szogi *, Paul D. Shumaker and Eric D. Billman

Coastal Plain Soil, Water and Plant Research Center, Agricultural Research Service, United States Department of Agriculture, 2611 W. Lucas Street, Florence, SC 29501, USA; wooiklee.paye@usda.gov (W.S.P.); paul.shumaker@usda.gov (P.D.S.); eric.billman@usda.gov (E.D.B.)
* Correspondence: ariel.szogi@usda.gov

Abstract: The Quick Wash (QW) treatment extracts phosphorus (P) from manure and municipal sludge (MS), producing an organic acidified by-product with adequate nitrogen (N):P ratio to meet crop N requirements. Yet, data on crop response to N using QW by-products are lacking. We evaluated the response of annual ryegrass (*Lolium multiflorum* Lam.) and potential N leaching in sandy soil to N applications using raw wastes, their corresponding QW by-products, and ammonium sulfate (AMS) fertilizer. Treatments included a control (no amendment added), raw and acid-washed chicken litter, dairy and swine manure, MS, and AMS at 100, 200, and 400 kg N ha^{-1}. We found no significant differences in annual ryegrass yield and N uptake between the raw and acidified organic QW by-products. However, ryegrass produced 4–30% more biomass with AMS than organic amendments. The total residual soil inorganic N under AMS treatments ranged between 6.3 and 67.9 mg pot^{-1} and accounted for 5–17% of the total N applied, but it was <1% for all the organic amendments. We found no differences in soil N leaching between raw and acid-washed forms of each organic soil amendment. Our results indicated that acidified organic QW by-products can improve environmental quality by substantially reducing the amount of applied P with no penalties for crop yield losses compared to raw manure and MS.

Keywords: acidification; manure; municipal sludge; nitrogen; phosphorus recovery; soil amendments; quick wash

Citation: Paye, W.S.; Szogi, A.A.; Shumaker, P.D.; Billman, E.D. Annual Ryegrass (*Lolium multiflorum* Lam.) Growth Response to Nitrogen in a Sandy Soil Amended with Acidified Manure and Municipal Sludge after "Quick Wash" Treatment. *Agronomy* **2023**, *13*, 2655. https://doi.org/10.3390/agronomy13102655

Academic Editor: Andrea Baglieri

Received: 29 August 2023
Revised: 11 October 2023
Accepted: 19 October 2023
Published: 22 October 2023

1. Introduction

In the United States (U.S.), animal manure derived from confined animal feeding operations accounts for the largest and most significant organic waste production, generating 38 × 10^{12} dry g annually; in comparison, municipal sludge (MS) solids account for 12.5 × 10^{12} dry g per year [1]. Animal manure and MS are valuable agricultural inputs because they are excellent sources of essential nutrients such as nitrogen (N), phosphorus (P), and organic carbon, which are critical for sustainable crop production and soil health [2,3]. The application of manure and MS as soil amendments can substantially reduce commercial fertilizer use in crop production while promoting the recycling of nutrients in a circular economy. Typically, the amounts of manure and MS applied to agricultural lands are based on the crops' N requirement [4,5]. However, repeated land application of these organic wastes contributes to P buildup in soils because of the relatively low N:P ratio (<4.0) in manure or MS versus harvested crops, raising concerns of environmental pollution because of excess soil P accumulation and transportation by runoff or leaching to water resources [6,7]. On the other hand, the organic N and carbon (C) in these organic wastes stimulate soil microbial mineralization of organic N and C by producing ammonium (NH_4^+-N) and eventually nitrate (NO_3^--N) through microbial nitrification. However, losses of these inorganic N forms from soils receiving manure or MS applications through ammonia (NH_3)

gas volatilization or leaching can limit N availability to the crop and promote pollution of air and water resources [8,9]. Nevertheless, the livestock and municipal waste sectors can benefit from new technologies that manage excessive losses of inorganic N and P and reduce the environmental impact of land spreading of manure and municipal wastes.

A report by Vanotti et al. [10] covered an overview of alternative technologies for removing and recovering N and P from animal manure, including the agronomic utilization of by-products' nutrients. Here, we focus on the agronomic use of acidified manure and MS as sources of N to reduce the environmental impact of soil P and N losses. Several studies on the acidification of organic wastes show that this treatment effectively decreases NH_3 gas losses during their handling and disposal [11–13]. Acidification impacts multiple chemical and microbial processes in organic wastes, changing their composition and promoting the release of soluble P [14], thus increasing the risk of P pollution following land application [6,15,16]. As an alternative to reduce the environmental risks from land disposal of P-rich wastes, the U.S. Department of Agriculture (USDA) developed and patented a process called "Quick Wash" (QW) to selectively extract and recover P from organic wastes, such as poultry litter, livestock manure, and MS, before land application [17,18]. The process generates two by-products: (1) concentrated P and (2) an acid-washed, N-rich, organic material with a lower P content and a more balanced N:P ratio than untreated waste. In addition to reducing the P content of the treated manure and MS, the QW can also reduce NH_3 volatilization losses. A study of acid-washed poultry litter applied on surface soil reduced the NH_3 emission by 76% compared to untreated poultry litter [19]. Yet, the agronomic effectiveness of acidified organic QW by-products as soil amendments requires further research. In particular, low P acidified organic QW by-products could serve as a slow N release source and substitute commercial N fertilizer. The use of acid-treated QW products as soil amendments could create an avenue for the valorization of waste in a circular economy while maintaining the materials, services, and economic value of products and changing the linear life cycle pattern of take–make–consume–dispose of current closed-loop product life cycles [20].

Crop response to N fertilization using the acidified organic QW by-products is mainly unknown. Therefore, we conducted this study with the following objectives: (1) compare crop response to N application using the raw forms versus organic acidified QW by-products of three animal manures and MS, (2) document the agronomic effectiveness of raw and organic acidified QW by-products of animal manures and MS in comparison to commercial inorganic N fertilizer, and (3) determine the potential leaching of N in sandy soil of the Southeastern Coastal Plain, U.S., amended with raw and acidified organic QW by-products, and inorganic commercial N fertilizer. We hypothesized that crop response (i.e., biomass and N uptake) to N application is similar between the raw and organic acidified QW by-products. Furthermore, we hypothesized that crop response to N application is similar between organic acidified QW by-products and inorganic N fertilizer. However, the application of inorganic N fertilizer may result in more significant leaching losses of inorganic N in sandy Coastal Plain soils of the Southern U.S.

2. Materials and Methods

2.1. Quick Wash Treatment of Manure and Municipal Sludge

The QW process consists of three steps [17]. Animal manure or MS (raw waste material) is first mixed with water and inorganic or organic acids such as sulfuric or citric acid at pH < 5.0 (citric acid to pH 4.5 and a 6:1 water to solids ratio in this study); and allowed to settle for 20 min (Figure 1). This step allows the extraction of insoluble P attached to organic and inorganic phosphates. The second step involves the precipitation of P by increasing the solution pH to a range of 9–10 using a highly alkaline earth base (e.g., calcium hydroxide). In the third step, an organic flocculant is added to improve the grade of the precipitated P compound. Acidification in the first step of the QW process can remove as much as 80–90% of P from animal manure and MS solids without substantially altering the C and N content of its organic by-product [17,21].

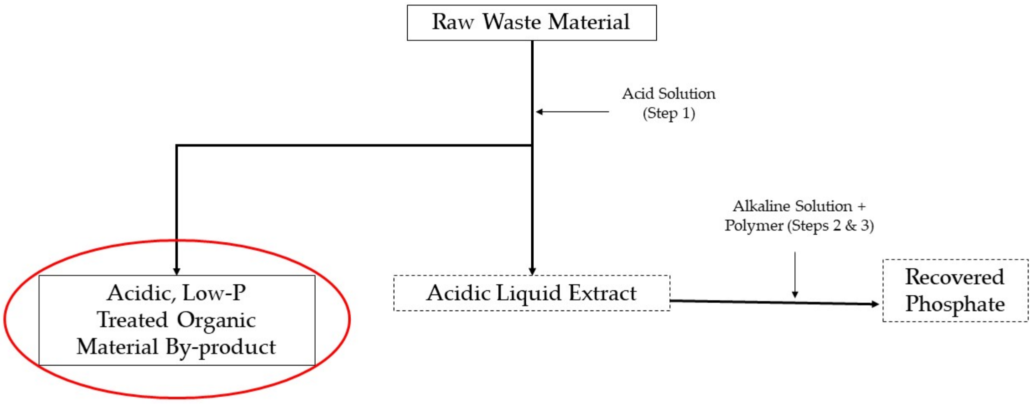

Figure 1. Schematic of the Quick Wash process. The acidified low P organic by-products (in red circle) of different animal (chicken, dairy, and swine) manures and municipal waste after the Quick Wash process were evaluated as sources of N in comparison to their respective raw forms.

The organic amendments used in this study consisted of chicken litter (CHL), fresh swine manure (SM), fresh dairy manure (DM), and MS. The MS originated from a municipal wastewater treatment plant in South Carolina. The CHL was collected from a 25,000-bird broiler house in a poultry farm (Lee County, South Carolina). The DM was collected from a concrete settling basin receiving scraped manure slurry from the milking center in a dairy operation (Laurens County, SC, USA). The SM was obtained from a barn with a shallow pit under a slatted floor in a finishing swine farm (Sampson County, North Carolina). Following the collection, each material was divided into two halves. One half was subjected to the acid wash step of the QW process, while the other half remained unwashed. This study focuses only on using the low P organic acidified QW by-products from the first step of the QW process as soil amendments. Before application to soil, all organic amendments were air-dried and homogenized to pass through a 2 mm sieve and analyzed for total C and N content by dry combustion with an Elementar VarioMax CN analyzer (Elementar Americas Inc., Ronkonkoma, NY, USA) and total P content determined in acid digested samples by inductively coupled plasma analysis (ICP-AES; Agilent Technologies Inc., Santa Clara, CA, USA) [22].

2.2. Experimental Soil, Treatments and Design

We conducted greenhouse trials to evaluate crop response to N application using the raw and QW acidified forms of three animal manures and MS as soil amendments and "Gulf" annual ryegrass (*Lolium multiflorum* Lam.) as the test crop. Gulf annual ryegrass was selected as the test crop due to its rapid growth rate, high cold tolerance, and adaptability to sandy soils. As such, it is widely grown as winter forage along the Southeastern Coastal Plain of the United States. The study was repeated twice at the USDA-ARS Coastal Plain Soil, Water and Plant Research Center in Florence, SC, USA. The soil used for both experiments was a Norfolk loamy sand (Fine, loamy, siliceous, thermic Typic Kandiudults) obtained from the Clemson University Pee Dee Research and Education Center near Florence, SC. Bulk Norfolk soil samples were collected from the top 15 cm (Ap horizon) and air-dried in the greenhouse for one week. The soil was then sieved through a 2 mm sieve to remove plant materials and large aggregates. The Norfolk soil has a loamy sand texture with a particle size distribution of 74% sand, 25% silt, and 1% clay (Soil Characterization Lab, The Ohio State University, Columbus, OH, USA). The soil test analysis showed a pH of 5.8, 22 mg P kg^{-1}, 44 mg K kg^{-1}, and a cation exchange capacity of 2.4 cmolc kg^{-1} (Soil Testing Lab, Clemson University, Clemson, SC, USA). The dry combustion method measured the soil total C and N contents which were 16.8 g total C kg^{-1} and 1.26 g total N kg^{-1}, respectively. After that, the soil pH was adjusted to 6.5 using calcium hydroxide. Each experimental pot was then filled with 1.5 kg of soil packed to a bulk density of 1.4 g cm^{-3} before sowing annual ryegrass seeds. Each experimental pot had a volume of 2.1 L, a 15 cm diameter, a 12 cm height, and a surface area of 176.6 cm^2. Treatments included CHL, acid-washed chicken litter (WCHL), DM, acid-washed dairy manure (WDM), SM, acid-washed swine manure (WSM), MS, acid-washed municipal sludge (WMS), AMS, and a control (no organic amendment or fertilizer added). We included the AMS (commercial synthetic inorganic fertilizer) treatment as a positive control. Each amendment was applied to pots on a dry weight basis at equivalent N application rates of 100, 200, and 400 kg ha^{-1}. After each treatment application, the soil and amendments were thoroughly mixed in a 10 L plastic bucket to mimic incorporation of field-applied organic soil amendment and then returned to each pot. The pots were then arranged in a randomized complete block design with three replications. Before planting, the pots were irrigated to 60% water-filled pore space using distilled water and incubated for two weeks to initiate N mineralization of the organic amendments. Moisture content was adjusted periodically by weighing the pots and adding distilled water when needed. This irrigation scheme was maintained throughout the experiment. Following incubation, each experimental pot was planted with 1.1 g (450 seeds per pot) of Gulf annual ryegrass seeds. This weight of seeds was equivalent to 100 kg ha^{-1} seeding rate.

2.3. Harvesting and Plant Analysis

The duration of each experiment was 70 days with a greenhouse temperature of 26 ± 6 °C, and relative humidity of 61 ± 20%. For both experiments, we harvested ryegrass biomass four times at 20, 35, 50, and 70 days after planting. Ryegrass harvest consisted of hand-clipping the biomass in each pot at a height of 2.5 cm during the first three harvests and at the soil surface during the last harvest. Plant biomass from each harvest was oven-dried at 60 °C to a constant weight. After that, the samples were weighed to determine dry matter yield. The dry biomass from each sampling was finely ground using mortar and pestle and analyzed for total C and N by dry combustion. Plant N uptake was calculated as the biomass yield (g) × N content (%), and the total biomass yield and total N uptake were calculated as the sum of biomass yield and N uptake from the four ryegrass harvests during both experiments.

2.4. Post-Harvest Leachate Collection, Soil Sampling and Analysis

After the last ryegrass harvest, each pot was leached with 1 L of de-ionized (DI) water (2.0 pore volumes of DI water) [23]. This volume of DI water simulated rainfall of 57 mm and was allowed to drain for 24 h. The volume of leachate collected from each pot was then measured and sampled for N analysis. After leachate collection, the soil in each experimental pot was emptied into 10 L plastic buckets and homogenized thoroughly by mixing, and 200 g of fresh composite soil samples were then taken from each pot for analysis. All plant root residues were removed from the soil samples by hand, and the samples were freeze-dried before laboratory analyses. The dry soil samples were extracted for inorganic N as NH_4^+-N, NO_3^--N, and nitrite (NO_2^--N) using a 2 M KCl solution. The pH of organic materials and soil samples was measured electrometrically using a combination pH electrode in a 1:2 solid: DI water suspension (Mettler Toledo, S-470, Columbus, OH, USA) [22]. Before analysis, soil leachates were filtered through a 0.45 μm nylon filter. The leachate and soil sample extracts were then analyzed for NH_4^+-N, NO_3^--N, and NO_2^--N by colorimetric method [24]; absorbance readings were taken using an EL-800 microplate reader (Bio-Tek Instruments, Inc., Winooski, VT, USA) set at 650 nm. The NO_2^--N concentration in the sample extracts was determined to be below the detection limit of this colorimetric analysis method. Thus, the total residual soil inorganic N was the sum of NO_3^--N and NH_4^+-N concentrations.

2.5. Statistical Analysis

All measured variables were tested for normality and homogeneity of variance using Levene's test, followed by two-way analysis of variance (ANOVA) for a randomized complete block experimental design using a MIXED model procedure in SAS version

9.4 (SAS Institute, Cary, NC, USA). Initial data analysis showed no difference ($p > 0.05$) in the measured variables for each treatment between the two experiments. Hence, the data for each treatment were averaged across the two experiments before final analysis. Thus, the ryegrass biomass yield and N uptake data are the sum of the four cuttings in each experiment, averaged across both experiments for each treatment. Similarly, the soil leachate NH_4^+-N and NO_3^--N concentrations and residual soil inorganic N were determined at each experiment's end and averaged across the two experiments for each treatment. The N sources, application rates, and their interactions were considered fixed effects during the analysis, while treatment replications were considered random effects. Because of the significant N source × application rate interaction effect on all measured variables, treatment means were separated using the Tukey post hoc test at $p \leq 0.05$.

3. Results

3.1. Soil Amendments pH, Nutrient Content and Application Rates

The initial pH and nutrient content of the organic soil amendments and AMS are presented in Table 1. Although the QW acidification process lowered the organic material's pH to 4.5, it became >5.0 after air drying them. The pH of the organic acidified QW by-products WCHL, WDM, WMS, and WSM were 17, 36, 6.8, and 7% lower than their respective raw forms. The QW process had a limited effect on the C content of the organic materials as indicated by the limited change in C contents between the raw and acidified forms of each organic amendment.

Table 1. Initial pH, carbon (C), nitrogen (N) and phosphorus (P) content of chicken litter (CHL), washed chicken litter (WCHL), dairy manure (DM), washed dairy manure (WDM), municipal sludge (MS), washed municipal sludge (WMS), swine manure (SM), and washed swine manure (WSM), and ammonium sulfate (AMS) evaluated as N sources.

Source	pH	C %	N %	P %	C:N Ratio	N:P Ratio
CHL	7.1	32.9	3.7	1.3	8.9	2.8
DM	8.5	44.9	3.5	0.5	12.8	7.0
MS	6.4	31.2	5.0	2.8	6.3	1.8
SM	7.2	43.4	3.1	0.9	14.0	3.4
WCHL	5.9	37.6	3.3	0.7	11.4	4.7
WDM	5.4	45.9	3.9	0.3	11.8	13.0
WMS	6.0	33.1	5.1	2.1	6.5	2.4
WSM	6.7	43.2	3.7	0.5	11.6	7.4
AMS	-	-	21.2	-	-	-

Except for WCHL, in which the N concentration slightly decreased by 11% compared to CHL following acid P extraction, the QW increased the N concentration in WMS, WDM, and WSM by 9, 10, and 20% compared to their respective raw forms (Table 1). As a result of acidification, the concentration of P in WCHL, WDM, WMS, and WSM were 46, 40, 25, and 45% less than their respective raw forms, and resulted in improved N:P ratios for all organic acidified QW by-products. Therefore, the change in N concentrations of the organic amendments after acid P extraction affected the application rates of each material. For instance, the 11% decrease in N content of the WCHL meant 11% more mass of WCHL was needed for the same N application rate than the raw CHL (Table 2). However, the increases in N concentration of WMS, WDM, and WSM after QW also resulted in a lesser application rate of these organic acidified QW by-products than their respective raw, untreated forms. As expected, applying all QW forms of the organic soil amendments resulted in a substantial (~50%) decrease in the amount of P applied with their respective raw forms (Table 2).

Table 2. The application rate of each amendment as a nitrogen (N) source to meet targeted N rate and the associated phosphorus (P) rate at the targeted N rate using chicken litter (CHL), washed chicken litter (WCHL), dairy manure (DM), washed dairy manure (WDM), municipal sludge (MS), washed municipal waste (WMS), swine manure (SM), washed swine manure (WSM), and ammonium sulfate (AMS).

Source	Targeted N	Material Applied	P Applied at Targeted N
		(kg ha^{-1})	
	100	2703	35
CHL	200	5405	71
	400	10,811	141
	100	3021	22
WCHL	200	6042	44
	400	12,085	88
	100	2849	15
DM	200	5698	31
	400	11,396	62
	100	2597	7.3
WDM	200	5195	15
	400	10,390	29
	100	2012	56
MS	200	4024	111
	400	8048	223
	100	1961	42
WMS	200	3922	84
	400	7843	167
	100	3226	28
SM	200	6452	55
	400	12,903	111
WSM	100	3155	14
	200	6309	28
	400	12,618	57
	100	472	-
AMS	200	944	-
	400	1887	-

3.2. Ryegrass Biomass Yield and N Uptake

Ryegrass biomass yield and N uptake showed significant differences between N sources, application rate, and the source × rate interaction (Table 3). The biomass yield was statistically similar between the organic acidified QW by-products and their respective raw forms at each N application rate (Table 4). At 100 kg N ha^{-1}, the ryegrass yield of AMS was similar to both CHL and WCHL and significantly greater than the other organic amendments, all of which produced similar biomass yield. However, AMS-treated ryegrass produced 11–34% more biomass on average than the organic amendments at 200 kg N ha^{-1}. Similarly, AMS application at 400 kg N ha^{-1} resulted in biomass production that was 14–45% greater than the organic amendments. When applied at 400 kg N ha^{-1}, ryegrass biomass production under CHL and WCHL was similar and 23–28% greater than the other organic amendments. Except for DM, ryegrass biomass production increased with the N application rate for all N sources.

Nitrogen uptake in aboveground biomass followed a similar trend as the biomass yield (Table 4). Ryegrass treated with AMS had N uptake of 64.9, 94.8, and 129 mg pot^{-1} at N application rates of 100, 200, and 400 kg ha^{-1}, significantly greater than all organic amendments. The N uptake of ryegrass was similar among the control and the organic amendments at an N application of 100 kg ha^{-1}. However, when N was applied at 200 kg ha^{-1}, CHL, WCHL, MS, and WMS, all had similar N uptake that was greater than the control, which had similar N uptake as the DM, WDM, SM, and WSM. Ryegrass supplied with 400 kg N ha^{-1} had N uptake of 60 and 66 mg pot^{-1} under CHL and MS, and 64 and 58 mg pot^{-1} under WCHL and WMS, respectively, significantly greater than the other amendments. Ryegrass N uptake increased with N application rate under each amendment, except for DM, which showed no statistical increase in N uptake with increasing N application rates.

Table 3. Analysis of variance on ryegrass biomass yield, nitrogen (N) uptake, ammonium (NH_4^+-N), and nitrate (NO_3^+-N) recovered in post-harvest soil leachate concentration and residual soil inorganic N content.

SV	DF	Biomass Yield		N-Uptake		NH_4^+-N in Leachate		NO_3^--N in Leachate		Soil Inorganic N	
		F-Value	***p*-Value**	**F-Value**	***p*-Value**	**F-Value**	***p*-Value**	**F-Value**	***p*-Value**	**F-Value**	***p*-Value**
N source (S)	9	48.2	<0.0001	220.5	<0.0001	2490.2	<0.0001	4.7	<0.0001	133.9	<0.0001
Rate (R)	2	55.9	<0.0001	71.7	<0.0001	488.9	<0.0001	1.6	0.2151	26.3	<0.0001
S × R	18	4.3	<0.0001	20.7	<0.0001	1185.9	<0.0001	1.76	0.0312	56.2	<0.0001

Note: initial analysis showed no significant variation between the two experiments, thus final ANOVA was performed on data averaged across both experiments. SV is source of variation; DF is degrees of freedom.

Table 4. Ryegrass biomass yield and nitrogen (N) uptake least square means under different organic soil amendments: chicken litter (CHL), washed chicken litter (WCHL), dairy manure (DM), washed dairy manure (WDM), municipal sludge (MS), washed municipal sludge (WMS), swine manure (SM), washed swine manure (WSM), and ammonium sulfate (AMS) as N sources.

N Source	N Rate (kg ha^{-1})					
	100	**200**	**400**	**100**	**200**	**400**
	Biomass Yield (g pot^{-1})			**N Uptake (mg pot^{-1})**		
Control	1.87 ± 0.03 b [†]	1.87 ± 0.03 c	1.87 ± 0.03 c	37.6 ± 1.43 b	37.6 ± 1.43 b	37.6 ± 1.43 c
CHL	2.02 ± 0.07 abB	2.23 ± 0.14 bB	2.69 ± 0.26 bA	43.3 ± 4.21 bB	45.5 ± 4.90 bB	60.2 ± 8.50 bA
WCHL	2.30 ± 0.11 aB	2.39 ± 0.10 abB	2.79 ± 0.13 bA	45.5 ± 4.10 bB	49.7 ± 5.25 bB	63.5 ± 7.93 bA
DM	1.93 ± 0.13 bA	1.99 ± 0.17 bA	2.23 ± 0.07 bA	39.5 ± 4.08 b	38.0 ± 2.81 b	46.8 ± 2.80 bc
WDM	1.93 ± 0.19 bB	2.01 ± 0.13 bB	2.35 ± 0.11 bA	39.3 ± 1.54 bB	43.5 ± 4.77 bB	52.7 ± 5.60 bA
MS	1.96 ± 0.11 bB	2.10 ± 0.10 bcB	2.45 ± 0.12 bA	44.1 ± 4.53 bB	51.7 ± 5.10 bB	66.1 ± 5.33 bA
WMS	1.99 ± 0.08 abB	2.17 ± 0.12 bAB	2.34 ± 0.10 bA	42.7 ± 4.02 bAB	47.3 ± 4.15 bcB	57.8 ± 5.97 bA
SM	1.98 ± 0.13 abB	2.24 ± 0.15 bAB	2.47 ± 0.20 bA	38.9 ± 2.05 bB	44.2 ± 3.74 bAB	49.8 ± 5.06 bA
WSM	1.69 ± 0.11 bB	1.98 ± 0.15 bcAB	2.18 ± 0.12 bcA	32.9 ± 3.27 b	37.7 ± 3.97 b	41.4 ± 0.86 bc
AMS	2.29 ± 0.04 aC	2.66 ± 0.07 aB	3.17 ± 0.03 aA	64.9 ± 0.17 aC	94.8 ± 1.49 aB	129 ± 1.02 aA

Data represent annual ryegrass biomass yield and N uptake averaged across two experiments. [†] Least square mean values ± standard error followed by different lowercase letters within a column indicate significant differences among N sources at each N application rate, while different uppercase letters within a row indicate differences among N rates within each N source based on Tukey's test at $p < 0.05$.

3.3. Soil Leachate NH_4^+-N and NO_3^--N and Residual Soil Inorganic N

A significant effect of N source, rate, and source × rate interaction was also observed on soil leachate NH_4^+-N and NO_3^--N concentrations and post-harvest residual soil inorganic N (Table 3). The amount of NH_4^+-N in soil leachate was statistically similar among the organic amendments at each N application rate (Table 5). However, applying AMS resulted in a significantly ($p < 0.0001$) greater mass of NH_4^+-N in the soil leachate at each N application rate. With the increasing application, the mass of NH_4^+-N recovered in leachates of AMS-treated soil were 3.34, 11.00, and 51.00 mg pot^{-1} at 100, 200, and 400 kg N ha^{-1}, respectively, but increases of such magnitude were not observed with the organic amendments. However, there was a general increasing trend in soil leachate NH_4^+-N concentration with N application regardless of the source. Averaged across N rates, application of WCHL, WMS, and WSM resulted in a slightly reduced amount of NH_4^+-N in soil leachate by 23, 19, and 13% compared to CHL, MS, and SM, respectively, but applying WDM increased the amount of NH_4^+-N in soil leachate by 15% compared to DM.

Table 5. Nitrogen (N) sources and application rate effect on mass of ammonium (NH_4^+-N) and nitrate (NO_3^--N) recovered in soil leachate under chicken litter (CHL), washed chicken litter (WCHL), dairy manure (DM), washed dairy manure (WDM), municipal sludge (MS), washed municipal sludge (WMS), swine manure (SM), washed swine manure (WSM), and ammonium sulfate (AMS).

N Source	**N Rate (kg ha^{-1})**					
	100	**200**	**400**	**100**	**200**	**400**
	NH_4^+-N in Soil Leachate (mg pot^{-1})			**NO_3^--N in Soil Leachate (mg pot^{-1})**		
Control	0.39 ± 0.03 b [†]	0.39 ± 0.03 b	0.39 ± 0.03 b	0.21 ± 0.04 b	0.21 ± 0.04 b	0.21 ± 0.04 b
CHL	0.41 ± 0.08 b	0.74 ± 0.13 b	1.18 ± 0.23 b	0.22 ± 0.06 b	0.35 ± 0.15 ab	0.31 ± 0.06 b
WCHL	0.30 ± 0.08 b	0.57 ± 0.20 b	0.78 ± 0.22 b	0.14 ± 0.02 b	0.21 ± 0.06 b	0.16 ± 0.03 b
DM	0.44 ± 0.11 b	0.47 ± 0.08 b	0.64 ± 0.14 b	0.21 ± 0.11 b	0.27 ± 0.09 ab	0.34 ± 0.11 b
WDM	0.42 ± 0.08 b	0.55 ± 0.13 b	1.04 ± 0.27 b	0.46 ± 0.16 ab	0.20 ± 0.05 b	0.22 ± 0.08 b
MS	0.45 ± 0.14 b	0.58 ± 0.18 b	1.24 ± 0.15 b	0.26 ± 0.11 abB	0.26 ± 0.11 abB	1.66 ± 1.33 aA
WMS	0.43 ± 0.09 b	0.43 ± 0.12 b	0.85 ± 0.22 b	0.24 ± 0.09 b	0.30 ± 0.19ab	0.52 ± 0.29 ab
SM	0.35 ± 0.04 b	0.37 ± 0.08 b	0.60 ± 0.13 b	0.19 ± 0.07 b	0.11 ± 0.02 b	0.13 ± 0.03 b
WSM	0.23 ± 0.06 b	0.30 ± 0.04 b	0.40 ± 0.06 b	0.12 ± 0.02 b	0.12 ± 0.00 b	0.11 ± 0.04 b
AMS	3.34 ± 0.16 aC	11.0 ± 0.42 aB	51.0 ± 0.57 aA	0.74 ± 0.08 a	0.70 ± 0.11 a	0.49 ± 0.05 b

Data represent ammonium (NH_4^+-N) and nitrate (NO_3^--N) in soil leachate annual averaged across two experiments. [†] Least square means followed by different lowercase letters within a column indicate significant differences among N sources at each application rate, while different uppercase letters within a row indicate significant differences among N application rates for each N source at the 0.05 probability level of the Tukey test.

The NO_3^--N in soil leachate also varied among N sources and source × rate interaction, but not among N application rates (Table 3). The interaction effect shows that soil leachate NO_3^--N under AMS (0.74 mg pot^{-1}) was similar to both MS (0.26 mg pot^{-1}) and WDM (0.46 mg pot^{-1}), and greater than all other organic amendments at 100 kg N ha^{-1} (Table 5). At 200 kg N ha^{-1}, soil leachate NO_3^--N measured under AMS was 0.70 mg pot^{-1}, similar to CHL, DM, MS, and WMS, but 71–83% greater than SM, WDM, and WSM. At 400 kg N ha^{-1}, the greatest amount of soil leachate NO_3^--N (1.66 mg pot^{-1}) was measured under MS, but all other N sources had similar NO_3^--N concentrations at this N rate. Soil NO_3^--N level also increased with N application under MS, but this was not observed with AMS and the other organic amendments. The total inorganic N (NH_4^+-N + NO_3^--N) recovered in the soil leachate increased with N application under AMS accounting for 5, 8, and 17% of total N applied at 100, 200, and 400 kg ha^{-1}, respectively, but N recovered in leachates of organic amendment treated soils accounted for <1% of the total N applied at all application rates.

The post-harvest soil inorganic N varied among N source, rate, and the source × rate interaction (Table 3). The interaction effect showed no difference in soil inorganic N among all the organic amendments at 100 kg N ha^{-1} (Table 6). At this N rate, AMS had significantly greater soil inorganic N (6.32 mg pot^{-1}) than organic amendments all of which had similar soil inorganic N content ranging between 0.88–2.46 mg pot^{-1}. At 200 and 400 kg N ha^{-1}, the soil inorganic N did not differ among the organic amendments, and there was no difference between the raw and acidified QW by-products of each organic amendment. However, AMS had 81–92% more inorganic on average than the organic amendments. Similarly, AMS applied at 400 kg N ha^{-1} had 93–98% more inorganic N than the organic amendments. While there were increasing trends of soil inorganic N with N rate under all N sources, these increases were only statistically significant under AMS, not for the organic amendments.

Table 6. Nitrogen (N) sources and application rate effect on soil residual inorganic N remaining after ryegrass harvest under chicken litter (CHL), washed chicken litter (WCHL), dairy manure (DM), washed dairy manure (WDM), municipal sludge (MS), washed municipal sludge (WMS), swine manure (SM), washed swine manure (WSM), and ammonium sulfate (AMS).

	N Rate (kg ha^{-1})		
	100	**200**	**400**
	Soil Inorganic N (mg pot^{-1})		
Control	1.54 ± 0.17 b [†]	1.54 ± 0.17 b	1.54 ± 0.17 b
CHL	2.16 ± 0.75 b	1.97 ± 0.59 b	2.37 ± 0.63 b
WCHL	1.25 ± 0.27 b	2.53 ± 0.83 b	1.55 ± 0.25 b
DM	1.28 ± 0.40 b	1.83 ± 0.89 b	2.81 ± 0.89 b
WDM	2.46 ± 1.02 b	3.46 ± 1.00 b	3.68 ± 1.29 b
MS	2.10 ± 0.84 b	2.32 ± 0.82 b	3.49 ± 1.18 b
WMS	1.20 ± 0.28 b	1.91 ± 0.79 b	4.58 ± 3.36 b
SM	1.38 ± 0.49 b	3.33 ± 1.23 b	2.06 ± 0.52 b
WSM	0.88 ± 0.38 b	1.49 ± 0.41 b	2.59 ± 0.67 b
AMS	6.32 ± 0.45 aC	18.3 ± 2.04 aB	67.88 ± 3.62 aA

Data represent residual soil inorganic N averaged across two experiments. [†] Least square means followed by different lowercase letters within a column indicate significant differences among N sources at each application rate, while different uppercase letters within a row indicate significant differences among N application rates for each N source at the 0.05 probability level of the Tukey test.

4. Discussion

4.1. N and P Content of Organic Acidified QW By-Products

Analysis of the organic amendments showed variation in N and P content between the organic acidified QW by-products versus raw forms of each organic N amendment (Table 1). Specifically, the QW process reduced the total N concentration in WCHL by 11% compared to CHL due to N losses via NH_4^+-N solubilization during the P extraction process. Poultry litter has a significant fraction of its total N in the form of NH_4^+-N [25], which explains the reduction in the total N of WCHL. In contrast, animal manures such as SM, DM, and MS mostly contain organic N [25,26], making them less susceptible to NH_4^+-N loss during the acidification step of P extraction of the QW process.

Animal manure and MS are typically applied to crops at rates calibrated to meet crop N requirements [27,28]. Because manures and MS typically contain N:P ratios unbalanced with crop nutrient requirements, application rates on crops' N requirement often increase the risk of over-application and buildup of soil P due to recurrent spreading of manure or MS. In the U.S., federal and state regulations require confined animal feeding operations to implement a utilization plan called a comprehensive nutrient management plan (CNMP) as part of a manure management system. A CNMP aims to reduce the environmental impacts of land application of manure by managing manure nutrients' rate, source, placement, and timing [29]. Our study aimed to control N losses as ammonia gas by incorporating the amendments into the soil and maintaining 60% soil moisture to prevent denitrification and leaching losses, focusing on evaluating the N source and rate effect on annual ryegrass biomass and N uptake. Based on soil test results, most nutrient management plans apply N according to agronomic N requirements at rates not to exceed crop P removal rate or the soil test recommended P rate. We based our highest manure N application rate of 400 kg N ha^{-1} on the high N removal rates (300 to 400 kg N ha^{-1}) of bermudagrass (*Cynodon dactylon*) hay fields in the Southern US [30]. Indeed, annual ryegrass overseeded on bermudagrass can remove 100 to 275 kg N ha^{-1} during winter [31]. However, runoff and leaching potentials can be high with these high application rates, and they must be considered in the nutrient utilization plan. After the QW acidification step, the total N content resulted in slightly different application rates of the acidified versus raw forms of each organic amendment (Table 2). Indeed, increases in N content of WDM, WMS, and WSM compared to their respective raw forms resulted in slightly lower application rates of these materials. Every organic amendment met the exact N requirement, but the amount of P applied when the targeted N rates were met using the different organic amendments was on average, 50% less with the organic acidified QW by-products than with the respective raw materials (Table 2). The reduction in P of the organic acidified QW by-products presents an opportunity to build soil organic matter (SOM) via optimal N application rates for plant growth while reducing P loading in soil and soil P legacy [32,33].

4.2. Effect of Organic Acidified QW By-Products on Crop Production

The agronomic response of annual ryegrass supported our first hypothesis of similar crop response to N application using the raw versus organic acidified QW by-products used in this study. The similar crop response (i.e., biomass yield and N uptake) (Table 4) between each organic amendment's raw and QW forms demonstrates that recovering P from these materials does not diminish their capacity to supply N to crops. Interestingly, we observed a similar yield between the raw versus acidified QW by-products of the organic amendments despite the substantial (>50%) reduction in P concentration of the acidified by-products compared to their respective raw forms (Table 2). The significant N source × rate interaction effect on ryegrass biomass yield and N uptake indicates the differences between the organic amendments and AMS. Ryegrass biomass yield increased with increasing N application for each form of the organic amendments and AMS, but AMS showed a significantly ($p < 0.0001$) greater biomass yield at each N rate than the organic amendments. This biomass difference can be attributed to the chemical dynamics of AMS and the organic amendments. Since the AMS fertilizer is an inorganic salt that dissolves rapidly upon its application to soil, inorganic N becomes immediately available for crop uptake, mainly in the NH_4^+-N form. In contrast, a fraction of the total N in the organic amendments becomes gradually bioavailable as inorganic N through microbial mineralization.

The organic N applied with the amendments must first mineralize to NH_4^+-N and convert to NO_3^--N through gradual microbial mineralization and nitrification [34,35]. Each material's C:N ratio affects the evolution of inorganic N content after adding the organic materials to the soil. Generally, adding organic materials with C:N ratios of less than 20:1 to soil leads to a release of NH_4^+-N early in the mineralization process [36]. In our study, all organic materials had C:N ratios below 20:1 (Table 1). These C:N ratios warranted the mineralization and release of NH_4^+-N and subsequent NO_3^--N accumulation due to biological nitrification (Table 5). The lesser yield and N uptake for all the organic soil amendments concerning AMS in this study indicates that N mineralization was insufficient to reach similar yields obtained with AMS within the duration of the experiment. This result contradicts our second hypothesis of equal crop response to N application between AMS and organic acidified QW by-products. This result concords with Park et al. [37], who reported greater perennial ryegrass yield under urea fertilizer than pig manure slurry application. However, when only the organic amendments were considered in our study, CHL and WCHL generally both had greater ryegrass yields at each N application rate than the other amendments (Table 4), possibly due to higher initial contents of NH_4^+-N in poultry litter than in the other organic materials [38].

Despite the more significant response of ryegrass to N application under AMS than the organic amendments, we must emphasize that the dry matter yield and N uptake measurements in this study lasted only 70 days after planting in each of the two experiments, which was probably not long enough to allow for sufficient N mineralization from the organic N sources used in this study. Several studies [39–41] have reported that N availability in soil treated with animal manure generally increases with time, and net N mineralization of field-applied organic amendments typically occurs several months after their application to soil [42]. Therefore, field studies of longer duration with recurring applications of raw versus acidified QW by-products may reveal how N mineralization of the acidified organic QW by-products would impact crop yield and environmental quality during the entire crop season.

4.3. NH_4^+-N and NO_3^--N in Soil Leachate and Residual Inorganic N

The application of AMS in this study significantly increased ryegrass yield but also resulted in a significant (5–17% of applied N) loss from the soil via leaching (Table 5). In contrast, the total inorganic N leached from soil treated with the organic amendments was <1%. These results reflect the common characteristic of mineral fertilizers, which quickly dissolve and release larger amounts of bioavailable N in soil than organic sources. The greater NH_4^+-N and NO_3^--N concentrations in soil leachate and residual inorganic N in AMS-treated soil confirm our third hypothesis. It demonstrates that its application poses a greater risk for groundwater quality than the organic soil amendments when applied to sandy soils at equal N rates. Overall, there was no difference in soil leachate NH_4^+-N and NO_3^--N and the residual inorganic N between the raw versus acidified QW by-products (Table 5). In a similar pot experiment, Macedo et al. [43] also reported no difference in NO_3^--N levels of soil treated with acidified and untreated cattle manure slurry. However, greater potential of NO_3^--N leaching in sandy soil treated with non-acidified manure slurry than with acidified manure slurry has been reported by other researchers [44]. For all the organic amendments, crop yield, N uptake, and soil inorganic N fractions were less than the mineral fertilizer AMS.

5. Conclusions

We evaluated annual ryegrass response to N application using the organic acidified QW by-products versus their raw forms of different animal manures and MS, as sources of N; and compared their agronomic effectiveness to AMS. The dry matter yield of ryegrass treated with increasing N application using AMS ranged between 2.3–3.2 g pot^{-1} and N uptake of 65–129 mg pot^{-1} was more significant than any acid-treated or untreated animal manure and MS evaluated. However, applying AMS also resulted in a significantly greater loss of NH_4^+-N and NO_3^--N from the soil and more residual soil inorganic N than the organic amendments. Despite the more excellent crop response to AMS application, results show that its application in sandy soils increases the risk for N leaching into groundwater resources compared to manure or MS application. The QW process did not affect the N-supplying potentials of the organic amendments, as indicated by the similar ryegrass yield and N uptake, as well as the soil residual inorganic N fractions between the acidified QW by-products versus raw forms of each organic amendment. Therefore, this study shows that when manure or MS is the primary source of N, applying organic acidified QW by-products can substantially reduce the amount of soil-applied P without reducing crop yield compared to raw manure or MS application scenarios.

Author Contributions: Conceptualization, A.A.S.; methodology, A.A.S. and P.D.S.; formal analysis, W.S.P. and E.D.B.; investigation, P.D.S.; data curation, A.A.S., W.S.P. and E.D.B.; writing—original draft preparation, W.S.P.; writing—review and editing, A.A.S., P.D.S. and E.D.B.; supervision, A.A.S.; project administration A.A.S. All authors have read and agreed to the published version of the manuscript.

Funding: The research did not receive external funding. This research was part of USDA-ARS National Program 212 Soil and Air, ARS Project 6082-12630-002-00D.

Data Availability Statement: The data presented in this study will be published and openly available to the public in a data repository within 30 months after the date of publication and cataloged in the National Agricultural Library (NAL) Ag Data Common.

Acknowledgments: Mention of trade names or commercial products in this article is solely for the purpose of providing specific information and does not imply recommendation or endorsement by the U.S. Department of Agriculture.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Milbrandt, A.; Seiple, T.; Heimiller, D.; Skaggs, R.; Coleman, A. Wet waste-to-energy resources in the United States. *Resour. Conserv. Recycl.* **2018**, *137*, 32–47. [CrossRef]
2. Pagliari, P.; He, Z.; Waldrip, H.M. *Animal Manure: Production, Characteristics, Environmental Concerns, and Management*; Wiley: Hoboken, NJ, USA, 2020; ISBN 9780891180135.
3. Arlo, L.; Beretta, A.; Szogi, A.; del Pino, A. Biomass production, metal and nutrient content in sorghum plants grown on soils amended with sewage sludge. *Heliyon* **2022**, *8*, e08658. [CrossRef]
4. U.S. EPA. *A Guide to the Biosolids Risk Assessments for the EPA Part 503 Rule*; Tech. Rep. EPA/832-B-93-005; Office of Wastewater Management: Washington, DC, USA, 1995.
5. Szogi, A.A.; Vanotti, M.B.; Ro, K.S. Methods for Treatment of Animal Manures to Reduce Nutrient Pollution Prior to Soil Application. *Curr. Pollut. Rep.* **2015**, *1*, 47–56. [CrossRef]
6. Shepherd, M.A.; Withers, P.J. Phosphorus leaching from liquid digested sewage sludge applied to sandy soils. *J. Agric. Sci.* **2001**, *136*, 433–441. [CrossRef]
7. Lory, J.A. *Managing Manure Phosphorus to Protect Water Quality*; M.U. Extension; University of Missouri: Columbia, MO, USA, 2018; Volume G, p. 9182.
8. Lu, Q.; He, Z.L.; Stoffella, P.J. Land Application of Biosolids in the USA: A Review. *Appl. Environ. Soil Sci.* **2012**, *2012*, 201462. [CrossRef]
9. Sajeev, E.P.M.; Winiwarter, W.; Amon, B. Greenhouse Gas and Ammonia Emissions from Different Stages of Liquid Manure Management Chains: Abatement Options and Emission Interactions. *J. Environ. Qual.* **2018**, *47*, 30–41. [CrossRef]
10. Vanotti, M.B.; García-González, M.C.; Szögi, A.A.; Harrison, J.H.; Smith, W.B.; Moral, R. Removing and recovering nitrogen and phosphorus from animal manure. In *Animal Manure*; Waldrip, H.M., Pagliari, P.H., He, Z., Eds.; American Society of Agronomy: Madison, WI, USA, 2020. [CrossRef]
11. Li, Y.; Li, W. Nitrogen transformations and losses during composting of sewage sludge with acidified sawdust in a laboratory reactor. *Waste Manag. Res. J. Sustain. Circ. Econ.* **2015**, *33*, 139–145. [CrossRef] [PubMed]
12. Fangueiro, D.; Hjorth, M.; Gioelli, F. Acidification of animal slurry—A review. *J. Environ. Manag.* **2015**, *149*, 46–56. [CrossRef] [PubMed]
13. Jatana, B.S.; Gami, S.K.; Ketterings, Q.M. Assessing the Impact of Mixing Acid Whey with Dairy Manure On pH and Nitrogen Dynamics During Manure Handling. *J. Soil Sci. Plant Nutr.* **2023**, *23*, 2878–2890. [CrossRef]
14. Sica, P.; Kopp, C.; Müller-Stöver, D.; Magid, J. Acidification and alkalinization pretreatments of biowastes and their effect on P solubility and dynamics when placed in soil. *J. Environ. Manag.* **2023**, *333*, 117447. [CrossRef]
15. Roboredo, M.; Fangueiro, D.; Lage, S.; Coutinho, J. Phosphorus dynamics in soils amended with acidified pig slurry and derived solid fraction. *Geoderma* **2012**, *189–190*, 328–333. [CrossRef]
16. Rosemarin, A.; Ekane, N.; Andersson, K. Phosphorus Flows, Surpluses, and N/P Agronomic Balancing When Using Manure from Pig and Poultry Farms. *Agronomy* **2021**, *11*, 2228. [CrossRef]
17. Szogi, A.A.; Vanotti, M.B.; Hunt, P.G. Process for Removing and Recovering Phosphorus from Animal Waste. U.S. Patent No. 8,673,046, 18 March 2014.
18. Szögi, A.A.; Vanotti, M.B. Phosphorus recovery prior to land application of biosolids using the "Quick Wash" process developed by USDA (Ta-P-27). In Proceedings of the RAMIRAN 16th International Conference 2015: Rural-Urban Symbiosis, Hamburg, Germany, 8–10 September 2015.
19. Szogi, A.A.; Shumaker, P.D.; Ro, K.S.; Sigua, G.C. Nitrogen Mineralization in a Sandy Soil Amended with Treated Low-Phosphorus Broiler Litter. *Environments* **2019**, *6*, 96. [CrossRef]
20. Hollas, C.E.; Rodrigues, H.C.; Oyadomari, V.M.A.; Bolsan, A.C.; Venturin, B.; Bonassa, G.; Tápparo, D.C.; Abilhôa, H.C.Z.; da Silva, J.F.F.; Michelon, W.; et al. The potential of animal manure management pathways toward a circular economy: A bibliometric analysis. *Environ. Sci. Pollut. Res.* **2022**, *29*, 73599–73621. [CrossRef] [PubMed]
21. Szögi, A.A.; Vanotti, M.B.; Hunt, P.G. Phosphorus Recovery from Poultry Litter. *Trans. ASABE* **2008**, *51*, 1727–1734. [CrossRef]
22. Peters, J.; Combs, S.M.; Hoskins, B.; Jarman, J.; Kovar, J.L.; Watson, M.E.; Wolf, A.M.; Wolf, N. *Recommended Methods of Manure Analysis*; University of Wisconsin Cooperative Extension Publishing: Madison, WI, USA, 2003; p. A3769.
23. Szogi, A.A.; Bauer, P.J.; Vanotti, M.B. Vertical distribution of phosphorus in a sandy soil fertilized with recovered manure phosphates. *J. Soils Sediments* **2012**, *12*, 334–340. [CrossRef]
24. Sims, G.K.; Ellsworth, T.R.; Mulvaney, R.L. Microscale determination of organic nitrogen in water and soil extracts. *Commun. Soil. Sci. Plant Anal.* **1995**, *26*, 303–316. [CrossRef]
25. Chastain, J.P.; Camberato, J.J.; Albrechat, J.E.; Adams, J., III. Chapter 3a. Swine manure production and nutrient contents. In *Confined Animal Manure Managers Certification Program Manual B Swine Version 3*; Clemson University Cooperative Extension Service: Beaufort, SC, USA, 2001.
26. Pettygrove, G.S.; Heinrich, A.L.; Eagle, A.J. *Dairy Manure Nutrient Content and Forms*; Cooperative Extension Manure Technical Bulletin Series; University of California: San Diego, CA, USA, 2009. Available online: http://manuremanagement.ucdavis.edu (accessed on 5 May 2023).
27. Sharpley, A.N.; Heron, S.; Daniel, T. Overcoming the challenges of phosphorus-based management challenges in poultry farming. *J. Soil Water Conserv.* **2007**, *62*, 375–389.

28. Silveira, M.L.; O'Connor, G.A.; Vendramini, J.M.B. *Utilization of Biosolids in Forage Production Systems in Florida*; University of Florida Extension Publication: Gainesville, FL, USA, 2017; p. SL444. [CrossRef]
29. USDA-NRCS. Conservation Practice Standard. Nutrient Management. Code 590. Available online: https://www.nrcs.usda.gov/sites/default/files/2022/Nutrient_Management_590_NHCP_CPS_2017.pdf (accessed on 5 October 2023).
30. Newton, G.; Bernard, J.; Hubbard, R.; Allison, J.; Lowrance, R.; Gascho, G.; Gates, R.N.; Vellidis, G. Managing Manure Nutrients Through Multi-crop Forage Production. *J. Dairy Sci.* **2003**, *86*, 2243–2252. [CrossRef] [PubMed]
31. McLaughlin, M.R.; Sistani, K.R.; Fairbrother, T.E.; Rowe, D.E. Overseeding Common Bermudagrass with Cool-Season Annuals to Increase Yield and Nitrogen and Phosphorus Uptake in a Hay Field Fertilized with Swine Effluent. *Agron. J.* **2005**, *97*, 487–493. [CrossRef]
32. Doydora, S.; Gatiboni, L.; Grieger, K.; Hesterberg, D.; Jones, J.L.; McLamore, E.S.; Peters, R.; Sozzani, R.; Broeck, L.V.D.; Duckworth, O.W. Accessing Legacy Phosphorus in Soils. *Soil Syst.* **2020**, *4*, 74. [CrossRef]
33. Lemming, C.; Oberson, A.; Magid, J.; Bruun, S.; Scheutz, C.; Frossard, E.; Jensen, L.S. Residual phosphorus availability after long-term soil application of organic waste. *Agric. Ecosyst. Environ.* **2019**, *270–271*, 65–75. [CrossRef]
34. Powell, J.M.; Jokela, W.E.; Misselbrook, T.H. Dairy Slurry Application Method Impacts Ammonia Emission and Nitrate Leaching in No-Till Corn Silage. *J. Environ. Qual.* **2011**, *40*, 383–392. [CrossRef] [PubMed]
35. Thangarajan, R.; Bolan, N.S.; Tian, G.; Naidu, R.; Kunhikrishnan, A. Role of organic amendment application on greenhouse gas emission from soil. *Sci. Total Environ.* **2013**, *465*, 72–96. [CrossRef]
36. Nguyen, T.T.; Cavagnaro, T.R.; Ngo, H.T.T.; Marschner, P. Soil respiration, microbial biomass and nutrient availability in soil amended with high and low C/N residue—Influence of interval between residue additions. *Soil Biol. Biochem.* **2016**, *95*, 189–197. [CrossRef]
37. Park, S.H.; Lee, B.R.; Cho, W.M.; Kim, T.H. Comparative nitrogen use efficiency of urea and pig slurry for regrowth yield and nutritive value in perennial ryegrass sward. *Asian-Australas. J. Anim. Sci.* **2016**, *30*, 514–522. [CrossRef] [PubMed]
38. Li, L.-L.; Li, S.-T. Nitrogen Mineralization from Animal Manures and Its Relation to Organic N Fractions. *J. Integr. Agric.* **2014**, *13*, 2024–2040. [CrossRef]
39. Cavalli, D.; Cabassi, G.; Borrelli, L.; Fuccella, R.; Degano, L.; Bechini, L.; Marino, P. Nitrogen fertiliser value of digested dairy cow slurry, its liquid and solid fractions, and of dairy cow slurry. *Ital. J. Agron.* **2014**, *9*, 567. [CrossRef]
40. Cavalli, D.; Cabassi, G.; Borrelli, L.; Geromel, G.; Bechini, L.; Degano, L.; Gallina, P.M. Nitrogen fertilizer replacement value of undigested liquid cattle manure and digestates. *Eur. J. Agron.* **2016**, *73*, 34–41. [CrossRef]
41. Nevens, F.; Reheul, D. Agronomical and environmental evaluation of a long-term experiment with cattle slurry and supplemental inorganic N applications in silage maize. *Eur. J. Agron.* **2005**, *22*, 349–361. [CrossRef]
42. Peters, K.; Jensen, L.S. Biochemical characteristics of solid fractions from animal slurry separation and their effects on C and N mineralisation in soil. *Biol. Fertil. Soils* **2011**, *47*, 447–455. [CrossRef]
43. Macedo, S.; Vasconcelos, E.; Semitela, S.; Coutinho, J.; Fangueiro, D. Effects of soil application techniques and cattle slurry pre-treatment on NH3 emissions and soil solution composition—A pot experiment. In Proceedings of the 2013, 15th RAMIRAN Conference, Versailles, France, 3–5 June 2013.
44. Semitela, S.; Martins, F.; Coutinho, J.; Cabral, F.; Fangueiro, D. Ammonia emissions and potential nitrate leaching in soil amended with cattle slurry: Effect of slurry pre-treatment by acidification and/or soil application method. In Proceedings of the 2013, 15th RAMIRAN Conference, Versailles, France, 3–5 June 2013.