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Agronomic effectiveness of nitrogen and phosphorus recovered from swine manure

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Abstract

New technologies have been developed to extract and recover manure nitrogen (N) and phosphorus (P) that can be upcycled as substitutes for conventional fertilizers. Yet, there are uncertainties over the agronomic effectiveness of these recovered nutrients. In a greenhouse study, we evaluated annual ryegrass (*Lolium multiflorum* Lam.) response to recovered N (RN), conventional N (CN), recovered P (RP), and conventional P (CP) in four nutrient combinations: CN + CP, RN + CP, CN + RP, and RN + RP at five N rates: 0, 23, 46, 92, and 184 mg N kg^{-1} soil; and three P rates: 0, 39, and 78 mg P kg^{-1} soil. Results showed a synergistic response to N and P, where dry matter yield and nutrient uptake increased with both nutrients application. Under CP, ryegrass supplied with 92 and 184 mg N kg^{-1} of CN produced 16%–35% more dry mass yield than RN. In contrast, ryegrass dry mass yield under RP was 17%–114% greater with RN than CN. Ryegrass dry mass yield under CP was 17% greater with CP than RP at 78 mg P kg^{-1} soil, but RP produced 27%–66% more dry mass yield than CP under RN. There was no difference in soil inorganic N between the two N sources, but Mehlich-1 soil test P was less in RP than CP treated soil. The combination of RN and RP was an effective fertilizer blend for substituting CN and CP. This finding is critical for the advancement of the new circular nutrient economy.

Plain Language Summary

There is an increasing call to adopt a circular approach to nutrient management by recycling nutrients from agricultural wastes. This has resulted in the development of new technologies that can recover nitrogen and phosphorus from animal manure in a concentrated form, which can then be returned to the farm as substitutes for commercial fertilizers. This greenhouse study evaluated the agronomic effectiveness of

Abbreviations: CN, conventional nitrogen; CP, conventional phosphorus; EC, electrical conductivity; GPM, gas-permeable membrane; RN, recovered nitrogen; RP, recovered phosphorus.

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concentrated nitrogen and phosphorus recovered from swine manure versus commercial nitrogen (urea) and phosphorus (triple superphosphate) fertilizers using different nutrient combinations. Annual ryegrass was used as the test crop. The results demonstrated that the combination of recovered nitrogen and phosphorus was an effective fertilizer blend for substituting conventional nitrogen and phosphorus fertilizers. This finding is important for advancing the new circular nutrient economy.

1 | INTRODUCTION

Since their development, conventional nitrogen (CN) and phosphorus (CP) fertilizers such as urea (CH_4N_2O) and triple superphosphate ($Ca(H_2PO_4)_2$) have played a crucial role in maintaining food production to match its ever-increasing demand. However, there is growing concern over the agricultural use of conventional fertilizers due to their high environmental impacts (Brunelle et al., 2015; Gerardo et al., 2015). Conventional fertilizer production accounts for 1.2% of global energy consumption (International Fertilizer Industry Association [IFA], 2009) and up to 28% of the total energy consumed on farms in the United States (Garcia-Gonzalez & Vanotti, 2015). It is also estimated that production of each kilogram of synthetic N fertilizer emits 5.5–9.5 kg CO_2-eq into the atmosphere (Gerardo et al., 2015; Straaten, 2008), while production of P fertilizers accounts for 1.3–8.9 kg CO_2-eq. kg^{-1} of P_2O_5 (Walling & Vaneeckhaute, 2020). These reports urge the need to limit our reliance on conventional fertilizers and adopt a circular approach to nutrient management.

Despite the high cost of synthetic fertilizers, large quantities of animal manure loaded with plant nutrients, especially N and P, are disposed of annually without utilizing their nutrient-supplying potential. In the United States, confined animal production accounts for the largest share of organic waste, generating over 38×10^6 dry metric ton of animal manure each year (Milbrandt et al., 2018). This has resulted in an estimated 8.7×10^5 and 3.9×10^5 metric ton of excess farm-level manure N and P, respectively, in the United States (Gollehon et al., 2016; Vanotti, 2023). Excess nutrients from manure are also a problem in other regions of the world with agglomerated animal production. To reduce nutrient pollution associated with excess farm-level manure, the European Union has implemented a "Nitrates Directive," which not only identifies "Nitrate Vulnerable Zones," but it also enforces a strict manure application limit (Riaño et al., 2021; Vigerhoets et al., 2023). Regions with high density of livestock and manure production often face an increased risk of P runoff, which causes eutrophication of water bodies (Garske et al., 2020). A more sustainable approach to P management is to use nutrient recovery technologies to capture and reuse P from both solid and liquid waste streams and close the global P cycle in today's society (Desmidt et al., 2015; Keyzer, 2010).

Several studies have evaluated the fertilizer potential of raw animal manure (Paye et al., 2023) or manure digestate (Alburquerque et al., 2012; Leogrande et al., 2013; Nicoletto et al., 2014). Unlike the raw manure, concentrated nutrients recovered from manure using nutrient recovery technologies can be transported over longer distance to nutrient-deficient areas (Spiegal et al., 2020). However, uncertainties over the agronomic effectiveness of these recovered manure nutrients warrant further research.

The call for adopting a circular nutrient management approach is gaining momentum among agricultural scientists and policymakers. This is supported by the development of novel technologies that can extract and recover N (Vanotti & Szögi, 2015) and P (Vanotti et al., 2005) from manure in concentrated forms. The recovered N (RN) and recovered P (RP) can be used as substitutes for conventional fertilizers. The RN and RP are much cleaner and safer to handle, and contain higher nutrient density than raw manure (Vanotti & Szögi, 2015; Vanotti et al., 2020, 2005). This study evaluated the fertilizer potential of RN and RP as substitutes for synthetic CN and CP fertilizers. The objective was to assess the agronomic effectiveness of the combination of RN and RP as a fertilizer blend compared to CN (urea 46% N) and CP (triple superphosphate [TSP], 48% P_2O_5) fertilizer blends. We hypothesized that the recovered nutrients blend is as effective as conventional nutrients blend.

2 | MATERIALS AND METHODS

2.1 | Recovery of RN from liquid swine manure

The RN was extracted from liquid swine manure using the gas-permeable membrane (GPM) process (Vanotti & Szögi, 2015; Vanotti et al., 2020). The liquid swine manure was flushed manure from a finishing swine farm in Sampson County, North Carolina (Vanotti et al., 2017). The GPM process involved the passage of gaseous ammonia (NH_3) from liquid manure through a microporous hydrophobic membrane. The gaseous NH_3 was subsequently captured and

concentrated in an acidic stripping solution (0.5 M H_2SO_4) on the other side of the membrane. Tubular membrane manifolds were submerged in the liquid manure, and the NH_3 was removed from the liquid manure before it escaped into the air. Concentrated acid was added to a concentrator tank containing the acidic solutions to an endpoint pH of <1 whenever the pH of the acidic solution increased to 2. The acidic solution was recirculated into the GPM manifold in a closed loop. Ten batches were run using raw liquid swine manure containing approximately 1400 mg NH_4-N L^{-1}. The same stripping acidic solution was used in all consecutive batches, allowing the N concentration in the stripping solution to increase to high levels (Vanotti & Szögi, 2015). After 10 batches, the NH_3 in the liquid manure was recovered and concentrated in a clear solution containing approximately 53,000 mg NH_4^+-N L^{-1}. This concentrated NH_4^+-N solution was used in this research.

2.2 | Recovery of RP from liquid swine manure

The RP was extracted from liquid swine manure following Vanotti et al. (2005), which uses nitrifiers to lower the pH and eliminate bicarbonate and produce high-grade calcium phosphates. The method is based on the distinct chemical equilibrium between phosphorus and calcium ions when the carbonate and ammonium interferences are substantially eliminated before adding hydrated lime to precipitate the phosphate as calcium phosphate (Vanotti et al., 2020). The RP was recovered from liquid swine manure in a swine finishing operation in Duplin County, North Carolina (Vanotti et al., 2007). After biological N removal (97% NH_4^+- N removed) and alkalinity removal (88% alkalinity removed), soluble P was recovered in a P module (Vanotti et al., 2007). In this module, the liquid was first mixed with hydrated lime slurry in a reaction chamber using a process pH of 10.5. The liquid and the fine P precipitate were separated by gravity in a settling tank, and the resulting P-sludge was dewatered using Draimad filter bags (Vanotti et al., 2007). Anionic polymer was added to the P-sludge to enhance separation by filter bags, recovering 99% of total P with respect to unfiltered material (Szögi et al., 2006). Bags containing the wet calcium phosphate were left to dry on a drying concrete pad and removed from the farm monthly. The RP precipitate solid obtained was amorphous calcium phosphate with a concentration grade of 24.4 ± 4.5% P_2O_5 (Vanotti et al., 2007). The RP precipitate solid was ground and used in this research.

2.3 | Products characterization

The chemical composition of the RN and RP materials is analyzed and reported in Table 1. Since the RN (~5% N) was only in liquid form, granular urea (46% N) was also dissolved in deionized (DI) water to similar N concentration to ensure that both N sources were applied as liquid nitrogen solutions. The N content of the RN and CN solutions was determined by total Kjeldahl nitrogen (TKN) analysis to quantify the presence of all N forms in each solution. Similarly, the particle size of RP (16% P_2O_5) was <0.5 mm; thus, TSP (48% P_2O_5) was also crushed to similar particle size using a mortar and pestle. The pH and electrical conductivity (EC) of all materials were measured using an Orion Star A215 pH/Conductivity meter (Thermo Scientific). For the RN and CN solutions, pH and EC were determined without further dilution. However, CP and RP were first mixed in a 5:1 DI water-to-solid ratio, shaken on a reciprocal shaker for 1 h and allowed to settle for another 1 h before measuring pH and EC readings. The total elemental composition of both P materials was determined following Peters et al. (2003). The organic carbon content in both P materials was determined following APHA, AWWA and WEF (1998).

Core Ideas

- Phosphorus recovered from swine manure increased ryegrass yield more than conventional phosphrous.
- Recovered nitrogen produced less dry mass yield than conventional nitrogen when using conventional phosphorus.
- Recovered nitrogen produced 17%–114% greater dry mass yield than conventional nitrogen when using recovered phosphorus.
- Recovered nutrients could be effective substitutes for conventional nutrients.

2.4 | Trial location and soil preparation

The study was conducted under greenhouse conditions at the USDA-ARS Coastal Plain Soil, Water, and Plant Research Center in Florence, SC (34.245° N, −79.807° W). The experimental soil was obtained from a field located at the Clemson University Research and Extension Center in Florence, SC (34.311° N, −79.748° W). The soil is an Uchee sand (loamy, kaolinitic, thermic, Arenic Kanhapludults) based on USDA soil classification. It was collected from an area with low fertility and no fertilizer or agricultural lime application history. Bulk Uchee soil was obtained from the top 15-cm depth, air-dried for a week, and then sieved through a 2-mm sieve to remove pebbles and visible plant residues. Granulometric analysis showed the soil contained 91% sand, 6% silt, and 3% clay. The initial soil test nutrient analysis is presented in

TABLE 1 Chemical properties of conventional nitrogen and phosphorus (CN and CP) and recovered nitrogen and phosphorus (RN and RP).

Unit	Parameters[a]	RN solution	CN solution	RP	CP
–	pH[a]	1.03 ± 0.01	5.61 ± 0.02	7.71 ± 0.11	2.94 ± 0.01
dS m^{-1}	EC	3.00 ± 0.01	1.49 ± 0.01	0.01 ± 0.00	0.18 ± 0.00
	NH_4^+-N	49,585 ± 267	5.29 ± 1.11	–	–
mg L^{-1}	NO_3^--N	0.00 ± 00	0.00 ± 00	–	–
	NO_2-N	0.15 ± 0.03	0.08 ± 0.00	–	–
	TKN	50,063 ± 151	47,625 ± 546	–	–
mg g^{-1}	Organic C			76.79 ± 0.52	41.58 ± 0.41
	Al	–	–	1.17 ± 0.00	1.77 ± 0.08
	Ca	–	–	293 ± 1.22	177 ± 0.17
	Cu	–	–	0.22 ± 0.00	0.03 ± 0.00
	Fe	–	–	1.32 ± 0.03	0.97 ± 0.01
g kg^{-1}	K	–	–	6.58 ± 0.03	1.56 ± 0.14
	Mg	–	–	12.1 ± 0.15	3.40 ± 0.04
	Mn	–	–	0.15 ± 0.00	0.02 ± 0.00
	Na	–	–	1.76 ± 0.00	4.01 ± 0.06
	P	–	–	67.5 ± 1.03	212 ± 0.64
	S	–	–	2.12 ± 0.00	17.6 ± 0.09
	Zn	–	–	0.27 ± 0.05	0.52 ± 0.01

[a]pH and EC of CN and RN solutions were measured without further dilution, but in a 5:1 water extract for CP and RP. Organic matter was determined following APHA, AWWA and WEF (1998). Nitrogen was analyzed using the total Kjeldahl nitrogen (TKN) method. All other mineral content was determined using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES).

TABLE 2 Experimental soil chemical properties before and after liming.

Units	Parameter[a]	Before liming	After liming
–	pH	4.90 ± 0.13	6.80 ± 0.12
dS m^{-1}	EC	0.01 ± 0.00	0.02 ± 0.00
mg kg^{-1} [a]	Al	64.0 ± 13.6	87.0 ± 0.80
	Ca	13.0 ± 0.90	75.0 ± 6.12
	Cu	0.12 ± 0.03	0.13 ± 0.03
	Fe	8.00 ± 1.26	12.0 ± 0.79
	K	6.10 ± 0.98	7.00 ± 0.50
	Mg	4.10 ± 0.33	5.00 ± 0.33
	Mn	1.00 ± 0.19	1.30 ± 0.07
	Na	3.10 ± 0.14	3.98 ± 0.38
	P	3.42 ± 0.72	3.74 ± 0.47
	Zn	0.21 ± 0.01	0.31 ± 0.02

[a]Soil test nutrients were extracted using Mehlich-1 solution and analyzed using ICP-OES. Soil pH and EC were determined in a 2:1 DI water:soil ratio.

Table 2. The soil had an acidic pH (4.9, 2:1 water), which was adjusted by liming it to 6.8 by thoroughly mixing it with a reagent-grade calcium hydroxide $Ca(OH)_2$ at the rate of 0.14 g kg^{-1} soil, changing the moisture content to 60% water-holding capacity, and incubating for 1 week. Post-liming soil analysis showed increased pH and availability of all soil test nutrients (Table 2). After incubation, the soil was air-dried for another week. Afterward, each experimental pot of 12-cm diameter, 17 cm height, a volume of 1.1 L, and a total surface area of 113.0 cm^2, was filled with 1.5 kg of dry soil packed to a bulk density of 1.4 g cm^{-3} before treatment application and ryegrass planting.

2.5 | Experimental design and treatments

The experimental design was a randomized complete block with four replications. The 2 × 5 × 2 × 3 factorial treatment combinations included two N sources: CN or RN solution applied at five N rates: 0, 23, 46, 92, and 184 mg N kg^{-1} soil (equivalent to 0, 50, 100, 200, and 400 kg N ha^{-1} assuming 2,241,676 kg as the mass of 1 ha furrow slide to 15-cm soil depth), in combination with two P sources: CP or RP applied at three P rates: 0, 39, and 78 mg P kg^{-1} soil (equivalent to 0, 88, and 176 kg P ha^{-1} assuming 2,241,676 kg as the mass of 1 ha furrow slide to 15-cm soil depth). All nutrients were applied on total N and total P basis. Our nutrient application rates were based on the high N and P removal rates (384–486 kg N ha^{-1} $year^{-1}$ and 77–86 kg P ha^{-1} $year^{-1}$) of bermudagrass (*Cynodon dactylon*) hay fields in the Southeastern United States (Newton et al., 2003). Moreover, annual ryegrass overseeded on bermudagrass can remove 100–275 kg N ha^{-1} $year^{-1}$ (McLaughlin et al., 2005). Since each experimental pot contained 1.5 kg of soil, these

nutrient application rates resulted in 0, 34.5, 69, 138, and 276 mg N pot^{-1} and 0, 58.5, and 117 mg P pot^{-1} at the corresponding kg ha^{-1} N and P application rates. Thus, the following nutrient combinations were established for each N and P source and application rate: CN + CP, CN + RP, RN + CP, and RN + RP. All N sources and rates were applied as a single dose in combination with P rates at 0 and 39 mg kg^{-1}, but the 184 mg kg^{-1} N rate was supplied in two equally split applications when combined with 78 mg kg^{-1} P. Before planting, P fertilizers were applied, thoroughly mixed with the soil, and returned to each experimental pot. The N fertilizers (CN and RN) were applied as solutions. Therefore, the volume of solution required at each N rate was calculated and added to 150 mL of DI water before applying it to the soil in each pot. The 150-mL DI water was used to keep soil moisture content at 10% (w/w) to ensure optimum ryegrass growth. This moisture regime was maintained for the entire study by periodically weighing and adding water to each pot. Potassium (K) was supplied as potassium chloride (KCl) at the rate of 0.3 g pot^{-1} to ensure that K deficiency was not a plant growth limiting factor.

2.6 | Crop cultivation, harvesting, and analysis

Following all treatment applications, annual ryegrass was sown at a depth of ~0.5 cm and a seeding rate of 1 g pot^{-1} (~450 seeds pot^{-1}). The plants were grown in the greenhouse under controlled conditions for 45 days. Throughout the study, the ambient temperature was maintained at 26 ± 6°C and 60 ± 20% relative humidity. Supplemental light was provided to give a 12 h/12 h day/night photoperiod using an overhead LED lighting system. Ryegrass was harvested three times on 18, 30, and 45 days after planting by hand-clipping the biomass at 2.5 cm aboveground during the first two harvests and at the soil surface during the final harvest. The biomass samples were weighed to obtain fresh weights and then oven-dried to a constant weight at 60°C. After recording the dry weights, the samples were ground using a mortar and pestle, and total N content in plant tissue was determined using TKN analysis, while P content was determined by digesting the samples in concentrated sulfuric acid and P concentration was determined by an automated ascorbic method modified for digested extracts (Technicon Instruments, 1977). The N and P uptake was calculated as the aboveground dry matter yield (g) × nutrient content (%), while the recovery efficiency of fertilizer N (REfrtN) and recovery efficiency of fertilizer P (REfrtP) were calculated using Equations (1) and (2), respectively.

$$\text{REfrtN} = \frac{\text{N uptake in fertilized pots}\left(\frac{\text{mg}}{\text{pot}}\right) - \text{N uptake in unfertilized pots}\left(\frac{\text{mg}}{\text{pot}}\right)}{\text{applied N}\left(\frac{\text{mg}}{\text{pot}}\right)} \times 100 \quad (1)$$

$$\text{REfrtP} = \frac{\text{P uptake in fertilized pots}\left(\frac{\text{mg}}{\text{pot}}\right) - \text{P uptake in unfertilized pots}\left(\frac{\text{mg}}{\text{pot}}\right)}{\text{applied P}\left(\frac{\text{mg}}{\text{pot}}\right)} \times 100 \quad (2)$$

During the second harvest, the effect of each treatment on ryegrass total chlorophyll content was determined based on the method of Arnon (1949). For this, 0.1 g of fresh leaves was extracted overnight in 10 mL of 80% acetone at 4°C. The next day, 5 mL of the extract was filtered through a 1.5-µm glass fiber filter into cuvettes, and absorbance readings were taken at 663 and 645 nm using a HACH DR 2700 spectrophotometer (Hach Company). Total chlorophyll was calculated as follows:

$$\text{Total Chlorophyll} = \left[8.02 \times A_{663}\right] + 20.2 \times A_{645}] \times V/1000 \times \text{FW} \quad (3)$$

where A is the absorbance reading at each wavelength, V is the volume of the extract (mL), and FW is the fresh weight (g).

2.7 | Post-harvest soil sampling and analysis

After the last ryegrass harvest, the soil in each experimental pot was emptied individually into a 10-L plastic bucket, and after thoroughly mixing and removing plant roots, ~100 g subsamples were taken for chemical analysis. Soil gravimetric moisture content was determined by weighing 20 g of moist soil sample and oven-drying (at 105°C for 24 h). Soil residual inorganic N (sum of NO_3^- and NH_4^+) was determined by extracting 6 g of moist sample in 25 mL of 1 M KCl

solution for 1 h and the extract was analyzed colorimetrically using Sims et al. (1995), after which absorbance readings were taken at 650 nm using an EL-800 microplate reader (Bio-Tek Instruments, Inc.). The post-harvest soil test P was determined using a Mehlich-1 solution and analyzed for total elemental content by Inductively Coupled Plasma Optical Emission Spectroscopy.

2.8 | Statistical analysis

It is only possible to compare mean responses to conventional versus recovered nutrient sources when a nonzero amount of nutrient is applied. For this reason, zero and nonzero nutrient treatments were differentiated in the statistical analysis so that the main effect and interaction coefficients could be interpreted properly. A general linear mixed model was fitted to the dry mass yield, N uptake, P uptake, soil N, and soil P responses. All response variables other than dry mass yield had right-skewed distributions and therefore were log-transformed before model fitting. In the statistical model, N source, P source, N rate, P rate, and their interactions were considered fixed effects, while replicate was treated as a random effect. To compare the estimated marginal means within treatment groups, N rates were nested within N source because the nonzero N rates were only present within the CN and RN treatments, while the zero N rate was only present within the control (no added N treatments). The same was done for P source and rate. The estimated marginal means were calculated at each rate for both CN and RN, and for CP and RP using Kenward–Roger approximation for the denominator degrees of freedom in all cases (Kenward & Roger, 1997). We also calculated the contrasts on the estimated marginal means with adjusted p-values. Multiple comparison adjustment was done where appropriate, using the Holm correction. For example, when CN and RN were compared at four N rates, we corrected for $m = 4$ comparisons, and for CP and RP, we corrected for $m = 2$. All confidence intervals were adjusted using the Bonferroni method (Dunn, 1961). For the correlation analysis between total chlorophyll and N uptake, we fitted a linear mixed model to obtain the least-square mean values for both variables. We then calculated the Pearson correlation between the two variables across all nutrient combinations as well as within each nutrient combination. The statistical analysis was done using R software version 4.3.3 (R Core Team, 2024) with packages lme4 version 1.1-35.2 (Bates et al., 2015) and emmeans version 1.10.1 (Lenth, 2024). A linear regression analysis was performed on the treatment means obtained from each nutrient combination to predict ryegrass leaf chlorophyll content as a function of N uptake.

3 | RESULTS

3.1 | Ryegrass response to N source and rate within each P source

Ryegrass dry mass yield, and N and P uptake averaged over zero-P treatments showed no response to N application in the CN and RN treatments (Figure 1). These results were attributed to the low available P concentrations in the zero P treatments. Averaged over the nonzero CP treatments, dry mass yield, and N and P uptake increased with N application up to 92 mg kg^{-1} and declined thereafter. The dry mass yield was similar between CN and RN at 23 and 46 mg kg^{-1}, but it was 16% and 35% greater with CN than RN at 92 and 184 mg kg^{-1}, respectively. We observed no difference in N and P uptake between the two N sources in the nonzero CP treatments. However, dry mass yield averaged over the nonzero RP treatments was 17%, 29%, and 114% greater with RN at 46, 92 and 184 mg kg^{-1} than with respective application of CN. The N uptake did not differ between the two N sources at each rate, except at 184 mg kg^{-1} where it was greater ($p < 0.0001$) with RN than CN. Under both P sources, the P uptake of CN-treated grass decreased with N application in excess of 46 mg kg^{-1}, but it increased linearly with RN under the nonzero RP treatments, where it was greater ($p < 0.0001$) than CN at 92 and 184 mg kg^{-1} N. We also observed a synergistic ryegrass response to N and P application under all the nutrient combinations, where dry mass yield and N and P uptake increased with the supply of both nutrients (Figures S1–S3).

3.2 | Ryegrass response to P source and rate within each N source

The ryegrass responses to P source and application rate were similar to the N responses (Figure 2). The dry mass yield and N uptake averaged over the zero N treatments showed no response to P fertilization. The average P uptake of the zero N treatments only varied at 78 mg kg^{-1} P where it was 68% greater with CP than RP. When averaged over the nonzero CN treatments, ryegrass dry mass yield and P uptake only varied between the two P sources at 78 mg kg^{-1} P, where CP produced 17% more dry mass yield and resulted in 97% greater P uptake than RP. For both P sources, increasing the application to 78 mg P kg^{-1} soil did not increase yields. These results show that 39 mg kg^{-1} P was the optimum P rate for ryegrass in the tested soil. There was also no difference in average N uptake between the two P sources under the nonzero CN treatments. In contrast, dry mass yield averaged over the nonzero RN treatments showed difference (<0.0001) between the two P sources, with RP applied at 39 and 78 mg kg^{-1} producing

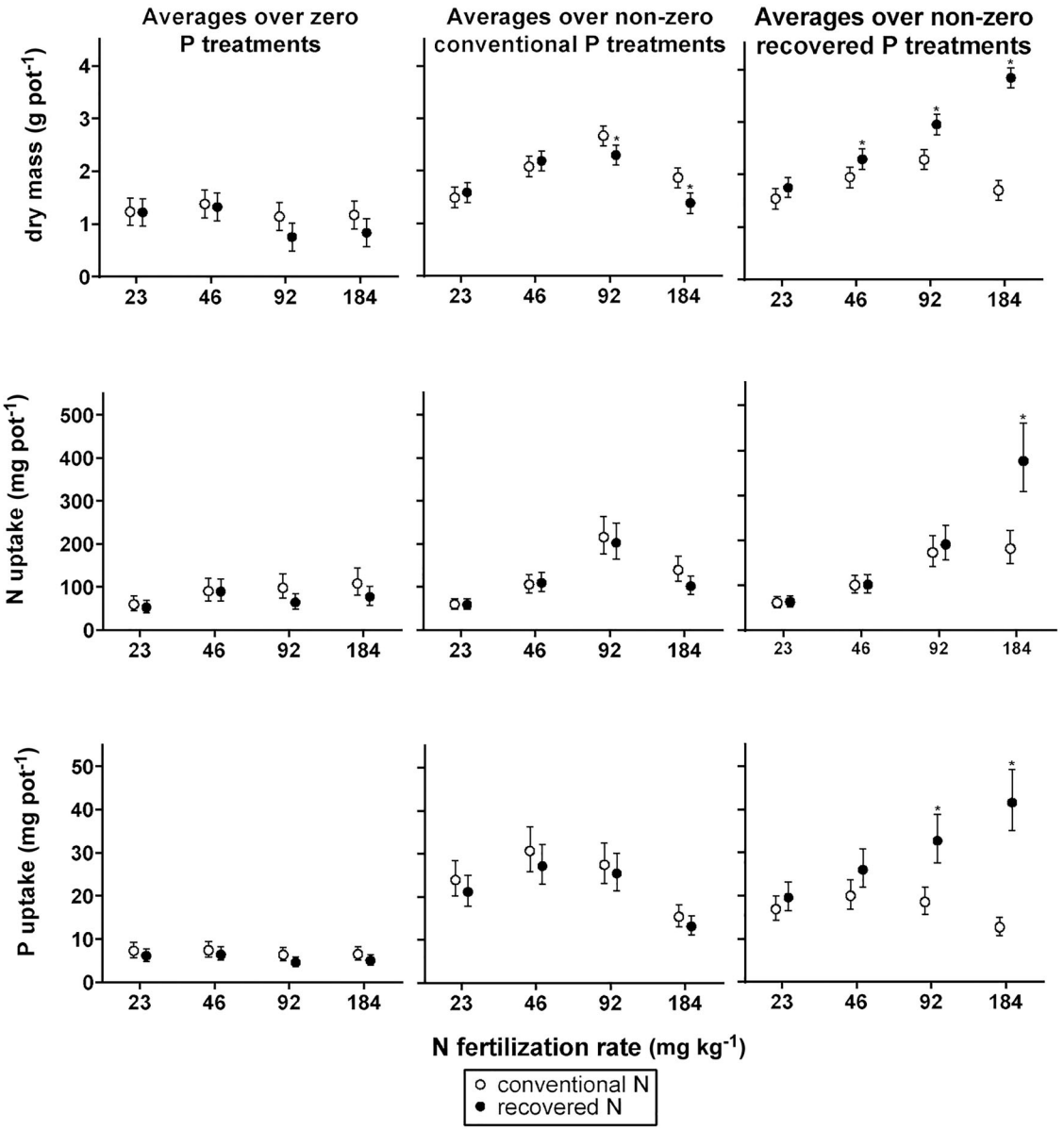


FIGURE 1 Ryegrass dry matter yields, and N and P uptake as influenced by N source and rates. Error bars represent 95% confidence intervals around the modeled means. Asterisks indicate a significant difference between the means at $p < 0.05$.

66% and 27% greater dry mass yield than CP at respective rates. RP also resulted in 73% and 111% greater N and P uptake, respectively, than CP at 39 mg kg^{-1} P, but there was no difference in these variables between the two P sources at 78 mg kg^{-1} P.

3.3 | Recovery efficiency of applied nutrients

Table 3 shows the N uptake and REfrtN for both N sources under each P source. Averaged over zero P treatments, CN had greater ($p < 0.0001$) REfrtN than RN at 34.5 and 138 mg N pot^{-1}. Averaged over the nonzero CP and nonzero RP treatments, both N sources had similar REfrtN at all N rates, except at 276 mg N pot^{-1} where it was greater ($p < 0.001$) with RN than CN under the RP treatments. Under CP, the REfrtN of both N sources declined drastically at the highest rate of 276 mg pot^{-1}, which is typical with excessive N application. However, under RP, REfrtN of RN increased with N rate. Table 4 shows the REfrtP for both P sources under each N source. Averaged over zero N and nonzero CN treatments, CP had greater ($p < 0.0001$) REfrtP than RP at 117 mg P pot^{-1}, but no difference at 58.5 mg pot^{-1}. In contrast, when averaged over nonzero RN treatments, RP had 130% greater REfrtP than CP at 58.5 mg pot^{-1}, but no difference at 117 mg pot^{-1}.

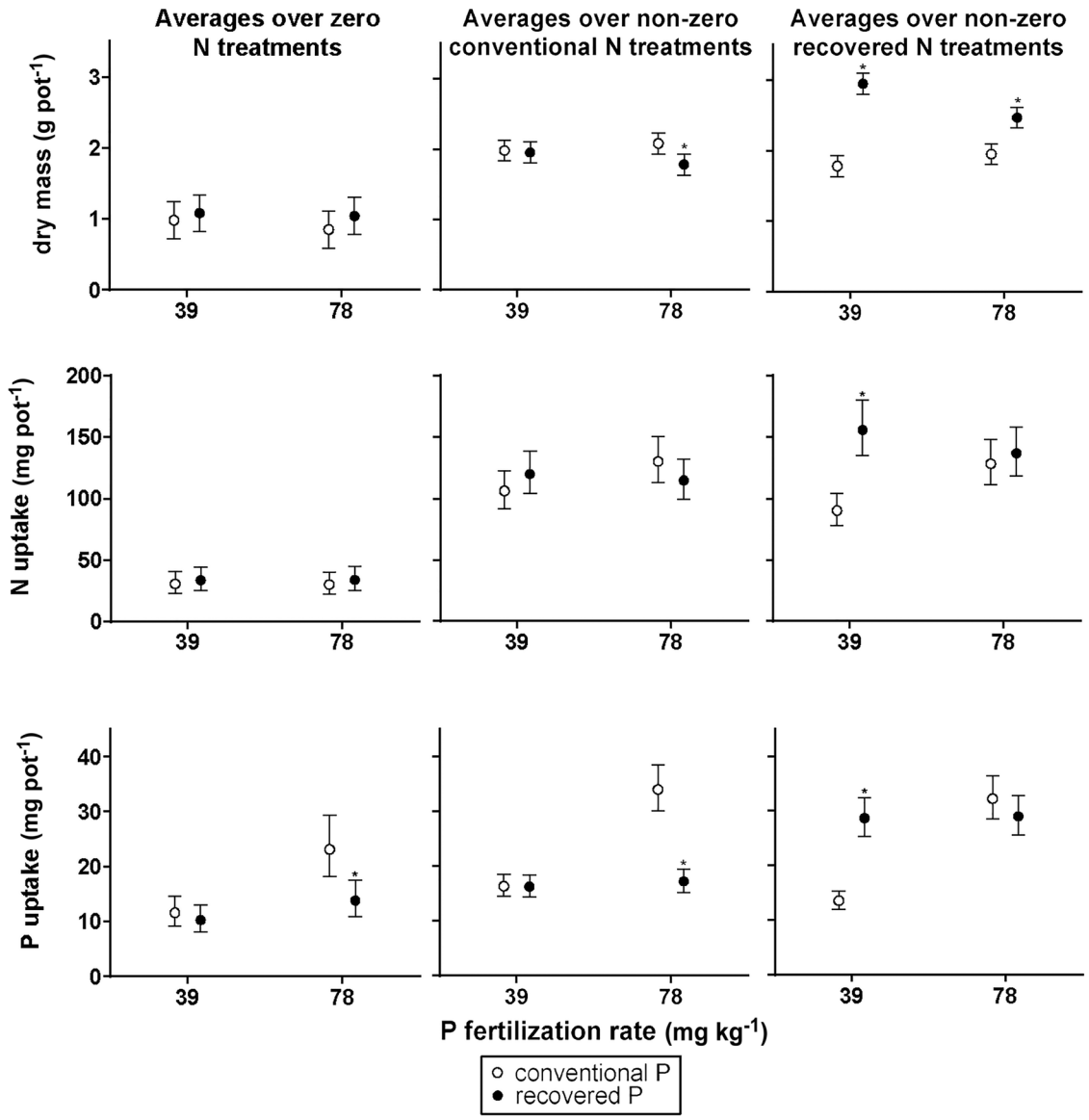


FIGURE 2 Ryegrass dry matter yields, and N and P uptake as influenced by P source and rates. Error bars represent 95% confidence intervals around the modeled means. Asterisks indicate a significant difference between the means at $p < 0.05$.

3.4 | Relationship of chlorophyll with N uptake

Correlation analysis between ryegrass total chlorophyll content and N uptake showed an overall significant ($p < 0.0001$) positive relationship ($r = 0.683$) when all nutrient combinations were considered (Table 5). However, when taken individually, the RN and CP (RN + CP) combination showed a weak negative correlation ($r = -0.244$, $p = 0.560$) (Table 5). Despite all other nutrient combinations showing positive correlations of leaf chlorophyll content with N uptake, RN + RP and CN + RP combinations showed the strongest positive correlations than the all-conventional (CN + CP) nutrient combination. Regression analysis performed on treatment means obtained from each nutrient combination showed an increase in ryegrass leaf chlorophyll content with N uptake (Figure 3).

3.5 | Residual soil N and P

The post-harvest residual soil N averaged zero P treatments were similar between both N sources at each N rate, except at 23 mg kg^{-1}, where it was significantly greater under CN (Figure 4). Without P, applying both N sources at 92 and 184 mg kg^{-1} resulted in significantly greater residual soil N. Averaged over the nonzero CP treatments, we observed no difference in soil residual N between CN and RN at each rate, but it was significantly greater at the highest N application rate (184 mg N kg^{-1}) compared to lower rates. When averaged

TABLE 3 Total N uptake and fertilizer N recovery efficiency of annual ryegrass treated with different N and P combinations.

Applied N (mg pot^{-1})	N uptake (mg pot^{-1})		REfrtN (%)	
	CN	RN	CN	RN
	Averaged over zero P			
0	22.5 ± 5.63c	22.5 ± 5.63d	–	–
34.5	59.6 ± 1.92b	52.6 ± 2.07c	108 ± 5.59aA	87.3 ± 6.00aB
69	90.8 ± 5.13a	89.8 ± 3.94a	99.0 ± 7.44a	97.5 ± 5.70a
138	99.7 ± 9.99aA	64.7 ± 4.80bcB	55.9 ± 7.24bA	31.3 ± 3.48bB
276	110 ± 11.3aA	79.2 ± 10.8abB	31.7 ± 4.10c	20.5 ± 3.92b
	Averaged over nonzero conventional P			
0	30.6 ± 1.99c	30.6 ± 1.99d	–	–
34.5	60.8 ± 4.26b	59.9 ± 4.16c	87.4 ± 12.3b	85.0 ± 12.1b
69	107 ± 5.79b	116 ± 14.4bc	111 ± 8.39ab	123 ± 20.9ab
138	224 ± 25.2a	200 ± 16.1a	140 ± 18.3a	128 ± 11.7a
276	173 ± 35.7a	33 ± 26.6b	51.6 ± 12.9c	36.9 ± 9.65c
	Averaged over nonzero recovered P			
0	38.3 ± 9.11c	38.3 ± 9.11d	–	–
34.5	61.3 ± 4.32b	64.2 ± 8.85c	66.72 ± 12.52bc	75.2 ± 15.22b
69	102 ± 7.17b	104 ± 17.2c	91.93 ± 10.39ab	95.2 ± 12.82ab
138	177 ± 16.1a	196 ± 35.0b	100.41 ± 11.67a	114 ± 12.49a
276	182 ± 5.93aB	386 ± 38.7aA	52.13 ± 2.15cB	126 ± 13.83aA

Note: Means with different lowercase letters within a column indicate significant difference between P rates, and means with different uppercase letters within a row indicate significant difference between P sources.

Abbreviations: CN, conventional N; RefrtN, recovery efficiency of fertilizer N; RN, recovered N.

TABLE 4 Total P uptake and fertilizer P recovery efficiency of annual ryegrass treated with different N and P combinations.

Applied P(mg pot^{-1})	P uptake(mg pot^{-1})		REfrtP (%)	
	CP	RP	CP	RP
	Averaged over zero N			
0	4.24 ± 0.26c	4.24 ± 0.26b	–	–
58.5	11.78 ± 1.42b	10.44 ± 1.17a	12.89 ± 2.42a	10.60 ± 2.00a
117	23.17 ± 0.99aA	13.85 ± 0.73aB	16.18 ± 0.85aA	8.21 ± 0.63aB
	Averaged over nonzero conventional N			
0	6.99 ± 0.33c	6.99 ± 0.33b	–	–
58.5	19.21 ± 2.06b	17.09 ± 1.20a	20.89 ± 3.52a	17.27 ± 2.06a
117	35.07 ± 2.44aA	17.37 ± 0.59aB	24.00 ± 2.08aA	8.87 ± 0.51bB
	Averaged over nonzero recovered N			
0	5.62 ± 0.34c	5.62 ± 0.34b	–	–
58.5	16.03 ± 1.62bB	29.65 ± 2.00aA	17.79 ± 2.78aB	41.07 ± 3.42aA
117	32.77 ± 1.57aA	30.97 ± 3.18a	23.20 ± 1.35a	21.66 ± 2.30b

Note: Means with different lowercase letters within a column indicate significant difference between P rates, and means with different uppercase letters within a row indicate significant difference between P sources.

Abbreviations: CN, conventional N; REfrtP, recovery efficiency of fertilizer P; RN, recovered N.

over the nonzero RP treatments, soil residual N was similar between both N sources, except at N application of 184 mg kg^{-1} where it was greater ($p < 0.0001$) under CN than RN.

Soil residual P averaged over zero N treatments increased with P application rate but did not differ between the two P sources (Figure 5). Also, soil P averaged over the nonzero CN and nonzero RN treatments showed no difference between both P sources at 39 mg kg^{-1}. However, at 78 mg kg^{-1} P, CP resulted in 22% and 54% more residual soil P, respectively, than RP, which is likely due

TABLE 5 Relationship between total chlorophyll content and N uptake by ryegrass treated with different combinations of conventional and recovered N and P.

Nutrient combo	Pearson correlation	95% Cl lower	95% Cl upper	*p*-value
Overall	0.683	0487	0.814	$<1 \times 10^{-5}$
CN + CP	0.697	−0016	0.940	0.055
CN + RP	0.835	0316	0.969	0.010
RN + CP	−0.244	−0810	0.556	0.560^{ns}
RN + RP	0.908	0566	0.984	0.002

Note: For overall relationship ($n = 45$ least-squares means across all treatment combinations), for each nutrient combination ($n = 8$ least-squares means for each combination of N and P source).

Abbreviations: CI, confidence interval; CN, conventional nitrogen; CP, conventional phosphorus; ns, not significant; RN, recovered nitrogen; RP, recovered phosphorus.

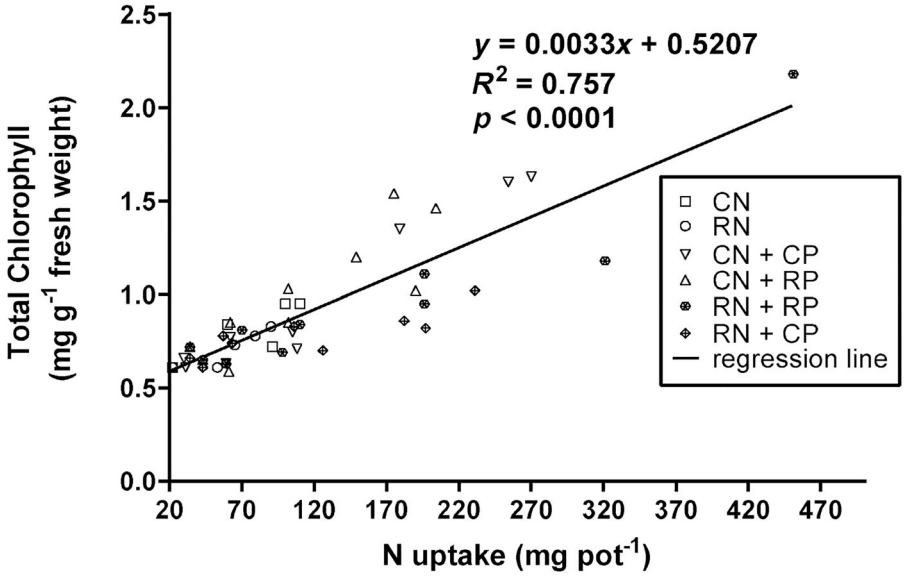


FIGURE 3 Linear regression between ryegrass total chlorophyll content and nitrogen uptake. Each data point represents the mean obtained from each nutrient combination; for conventional nitrogen (CN) and recovered nitrogen (RN) ($n = 5$ N rates × 1 N source = 5), for each nutrient combination ($n = 5$ N rates × 2 P rates = 10). CP, conventional phosphorus; RP, recovered phosphorus.

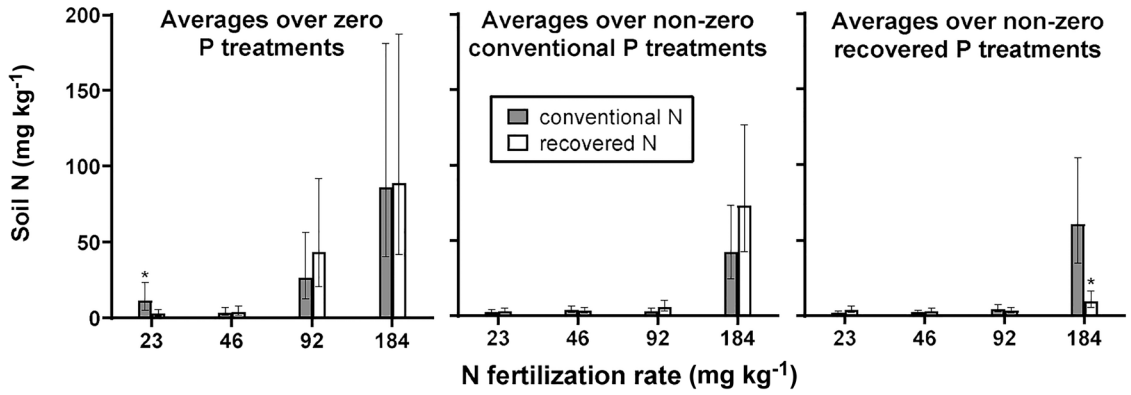


FIGURE 4 Soil residual inorganic N averaged for each N source at each N rate within each P source. Error bars represent 95% confidence intervals around the modeled means. Asterisks indicate a significant difference between the mean for conventional versus recovered N within a given N fertilization rate at $p < 0.05$.

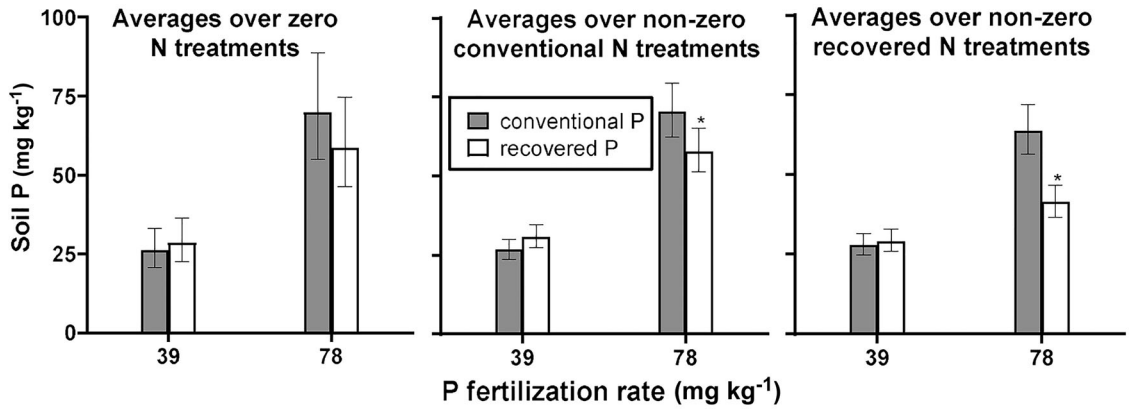


FIGURE 5 Soil residual P averaged for each P source at each P rate within each N source. Error bars represent 95% confidence intervals around the modeled means. Asterisks indicate a significant difference between the means for conventional versus recovered P within a given P fertilization rate at $p < 0.05$.

to the greater dry matter yield and P uptake of the RP treatments.

4 | DISCUSSION

4.1 | Crop yield and nutrient uptake

Our results show no difference in ryegrass biomass yield and N uptake between CN and RN when both were applied at increasing rates without P (Figure 1).This is because the two N sources contained 100% inorganic N (Table 1) and were therefore immediately plant available to an equal degree upon their application. No difference in crop yield between recovered ammonium nitrate and ammonium water versus synthetic calcium ammonium nitrate has also been reported by Saju et al. (2022) and Shrivastava et al. (2023). Crop responses to CP versus RP were also compared at two P rates without N application, and we found no difference in ryegrass dry matter yield between the two P sources (Figure 2). This is also because both CP and RP predominantly consisted of calcium (Ca) and P (Table 1). Further, the plant availability of RP (which is 99.88% total citrate-soluble) (Bauer et al., 2007) is similar to those of TSP or single superphosphate (SSP) (which are 97%–100% citrate-soluble) (Follet et al., 1981). Despite similar dry matter yield between the CP and RP at both rates, P uptake was only similar between the two materials when applied at a lower rate of 39 mg kg^{-1} without N application, but significantly greater with CP at 78 mg kg^{-1}. This difference in P uptake at the higher P rate was likely influenced by differences in the proportion of water-soluble P content in both materials. The TSP (CP in this study) is 43.5% water soluble (Ehmann et al., 2017), compared to RP, which is only 0.57% water soluble (Bauer et al., 2007). This explains the greater uptake of CP than RP at the highest rate when applied without N or in combination with CN. Additionally, RP is slightly alkaline (pH = 7.71), compared to TSP, which is acidic (pH = 2.94) (Table 1). Hence, without N application, the highest rate of RP applied to a soil already limed to pH 6.8 could have further increased the soil pH and reduced the solubility of RP at its highest application rate. This low solubility of RP resulted in lower REfrtP of the material when applied at high rate without N or in combination with CN (Table 4). In contrast, REfrtP of RP averaged over nonzero RN treatments was 53% greater than CP. Moreover, the 21%–41% REfrtP of RP under nonzero RN treatments is substantially greater than the 10%–15% P recovery typically found with mineral P fertilizer in crop production (Roberts & Johnston, 2015). This demonstrates that combining RN and RP generates a synergistic crop response to both nutrients, thus increasing their agronomic efficiency. Taken together, the lack of differences in ryegrass yield between the two N sources without P (Figure 1), and also between the two P sources without N (Figure 2), confirms our hypothesis that these new recovered nutrients are equally effective and can replace commercial synthetic fertilizers in a circular agricultural system.

An interesting pattern emerged when ryegrass responses to the different nutrient combinations were compared over nonzero conventional nutrients and nonzero recovered nutrient treatments (Figures 1 and 2). Under nonzero CP treatments, crop response pattern was similar between N sources, increasing with N rate up to 92 mg kg^{-1} and then declining. At 184 mg kg^{-1} N rate, there was less of a yield benefit of both N sources compared to 92 mg kg^{-1} N, but the yield declined more sharply with RN than CN. This is likely a result of the higher electrical conductivity and strong acidity of both RN and CP (Table 1), which may have acidified the soil or increased osmotic stress and reduced plant growth (Allakhverdiev et al., 2000; Gupta & Huang, 2014). In contrast, crop response averaged over nonzero RN and RP treatments (Figures 1 and 2) showed overall greater dry matter yield and N and P uptake for the all-recovered

nutrient combination than the all-conventional or the conventional plus recovered nutrient combinations. For all nutrient combinations, ryegrass supplied with 92 and 184 mg kg^{-1} N had a difficult start because of delayed germination that resulted in substantially less biomass production during the first 3 weeks of growth. These negative effects of high N rates remained visible in pots treated with the CN and CP, and those treated with RN and CP, up to the final biomass harvest. However, this growth-depressing effect of high N rates completely disappeared in pots receiving the RN and RP combination after the third week of growth, resulting in a dramatic increase in biomass production, which also led to significantly greater N and P uptake (Figures 1 and 2), and recovery efficiencies (Tables 3 and 4) for this nutrient combination.

High concentration of NH_4^+ in soil solution can curb root growth and result in overall poor crop performance (Fuchs et al., 2008; Möller & Müller, 2012). Thus, high concentrations of NH_4^+ in pots supplied with 92 and 184 mg N kg^{-1} soil may have suppressed ryegrass growth during the first 3 weeks after application. Currently, it is unclear why this phytotoxic effect of NH_4^+ only lasted briefly with the all-RN and RP nutrient combination and warrants further studies. However, the overall greater crop response to the RN and RP combination shows better efficiency and greater crop utilization of the all-recovered nutrients compared to all-conventional nutrients, or conventional plus recovered nutrient combinations. Our results are similar to those of other researchers (Herencia et al., 2011; Masarirambi et al., 2010; Vaneeckhaute et al., 2013) who reported better crop response to waste-derived nutrients than synthetic fertilizers.

Due to the strong correlation between chlorophyll and N content in crop biomass (Kalacska et al., 2015; Wang et al., 2014), precision nutrient management heavily relies on leaf chlorophyll content as a diagnostic tool for in-season crop nutritional health related to N fertilization. In our study, total chlorophyll content of ryegrass generally correlated well with N uptake when all nutrient combinations were considered (Table 5; Figure 3). Taken individually, the strongest correlation between total chlorophyll and N uptake was observed with the RN and RP ($r = 0.908$, $p = 0.0018$), and the CN and RP ($r = 0.835$, $p = 0.009$) combinations. This is likely because plants fertilized with RP had better N uptake than those fertilized with CP (Figures 1 and 2), resulting in more chlorophyll and dry matter production. It is also worth noting that the RN + CP combination resulted in a weak negative correlation between total chlorophyll and N uptake (Table 5). This nutrient combination also produced significantly less dry matter yields at the two highest N rates (Figure 1) and at both P application rates (Figure 2). This is likely a consequence of the acidity of both RN and CP (Table 2), which may have acidified the soil solution at higher N rates (Hort Americas, 2018; Shrivastava et al., 2023) and exacerbated the toxic effect of the high concentration of NH_4^+ in this treatment. We therefore caution against the use of this combination.

4.2 | Residual soil N and P

Assessing the soil residual nutrients provides insight into the potential impact of RN and RP as it relates to environmental quality when applied under field conditions (Buysse, 2015; Sigurnjak et al., 2019). Soil N under zero P treatments increased with N application (Figure 4), but it was not different between the two N sources. This shows that RN poses no greater risk to soil and groundwater quality than CN. Sigurnjak et al. (2019) also reported no difference in soil residual N between recovered ammonium nitrate and conventional calcium ammonium nitrate. Soil N averaged over nonzero CP treatments also showed no difference between CN and RN at each rate, but it was less with RN than CN when averaged over nonzero RP treatments. However, at the highest N rate, possibly due to the overall greater dry matter yield and N uptake (Figure 1), less inorganic N remained in the soil under RN at the completion of the study (Figure 4). These results agree with other researchers (Vaneeckhaute et al., 2013, 2014; Chen, 2014; Sigurnjak et al., 2019), who monitored residual soil N under recovered ammonium sulfate and ammonium nitrate versus synthetic N sources, and reported no difference with respect to their environmental impact related to groundwater quality. Despite increasing with P application, residual soil P averaged over zero N treatments did not differ between the CP and RP. The soil P only differed between the two P sources at 78 mg kg^{-1} where CP resulted in greater soil P than RP (Figure 5). This is possibly due to the greater biomass yield and P uptake of ryegrass treated with RP (Figure 1).

The finding of this study is particularly interesting because the nutrient combinations were compared on the basis of the same total N and P applied. As such, the reasons for the better agronomic performance of the RN and RP combination are not currently understood. Horta et al. (2022) also found that the N recovered using the same GPM method produced higher biomass and N uptake in triticale than the conventional fertilizer (Hoagland solution). A Raman spectra analysis of ammonium sulfate solution recovered from a mixture of swine manure and orange peel effluent using the GPM technology revealed the presence of several volatile organic compounds, which may have a biostimulant effect on crops (Horta et al., 2022).

It is important to recognize that the current study was a pot experiment that only lasted for 6 weeks of ryegrass growth. Consequently, extrapolating these results to a full-scale production under field conditions may be challenging. However, the controlled conditions of this study, including adequate lighting and stable temperature, provided an ideal setting for a proper evaluation of the nutrients tested. Hence, our results

provide sufficient evidence that recovered manure nutrients can be an effective replacement for urea or triple superphosphate. The greater overall biomass yield and nutrient uptake of the RN and RP combination demonstrate this as a novel nutrient combination that could be critical for improving crop yield and nutrient use efficiency in a circular agricultural system. Therefore, additional research is necessary to confirm the complete chemical composition of the RN and RP and verify their enhanced agronomic benefits under field conditions.

5 | CONCLUSION

This study shows that combining N and P recovered from liquid swine manure using advanced waste treatment technologies can be a novel fertilizer blend for improving crop yield. We observed no difference in crop response to CN versus RN when applied without P, nor between CP and RP when applied without N, which is evidence that both RN and RP are effective substitutes for the conventional nutrients used in this study. Ryegrass dry matter yield and nutrient uptake were greatly improved by the RN and RP nutrient combination, demonstrating that this nutrient blend is agronomically more efficient than CN and CP, or conventional and recovered fertilizers blend. Future research should focus on long-term utilization and validation of the responses of multiple crops to the RN and RP combination under field conditions while evaluating their impact on overall soil health, fertility, and environmental quality. The result of this study shows that N and P recovery technologies are not only critical for reducing greenhouse gas emission and soil P buildup in regions with high density of livestock production, but they also create an avenue for the valorization of excess manure nutrients, which is critical for closing the nutrient loop in circular agricultural systems.

AUTHOR CONTRIBUTIONS

Wooiklee S. Paye: Conceptualization; data curation; formal analysis; investigation; methodology; writing—original draft. **Raul Moral**: Conceptualization; investigation; methodology; writing—review and editing. **Matias B. Vanotti**: Conceptualization; data curation; formal analysis; project administration; supervision; writing—review and editing. **Ariel A. Szögi**: Conceptualization; supervision; writing—review and editing. **Quentin D. Read**: Data curation; formal analysis; writing—review and editing.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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