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Granulated fertilizers are more efficient in reducing potassium leaching losses than combining biosolids with inorganic sources

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ABSTRACT: Widespread use of soluble mineral fertilizers derived from non-renewable sources has raised concerns about environmental impacts, energy expenditures, and sustainability. Combining biosolid and mineral sources of phosphorus and potassium to produce organomineral fertilizers (OMF) can be used as an alternative nutrient source while reducing the environmental impact of sewage residues. As this approach simultaneously provides nutrients and incorporates organic matter, we hypothesized the presence of an organic source (biosolid) granulated with mineral sources of potassium (K) and phosphorus (P) would reduce leaching due to the benefits of the organic source and the slower release caused by granulation. Our goal was to evaluate the effect of different OMFs on the leaching losses of nutrients. Two forms of isolated N, P, and K sources (granulated and non-granulated), five OMFs with different NPK proportions (1-2-0, 1-4-0, 1-0-2, 1-2-2, 1-2-4), and a control (unfertilized) were evaluated over ten weeks in a leaching column experiment. Non-granulated potassium sulfate exhibited the highest K leaching and did not differ from OMFs with K in their formulation (granulated PS, 1-0-2, 1-2-2, and 1-4-2). Planned contrasts showed that granulation was particularly effective at reducing K leaching, resulting in a 70 % reduction compared to non-granulated PS. While formulation and granulation showed a trend of potential benefits in reducing N-(NO_3^- + NO_2^-) leaching, the effect was not statistically significant. Interaction between fertilizer treatments and leaching events was marginally significant for NH_4^+ leaching, indicating temporal variations of OMFs in nutrient dynamics may be influenced by mineralization. As granulation modulated how OMFs affected nutrient leaching dynamics, this highlights the importance of the physical characteristics of fertilizers for effective nutrient management.

Keywords: nitrogen leaching, nutrient management, NPK formulation, organomineral fertilizer.

INTRODUCTION

Most fertilizers used worldwide consist of soluble mineral forms originating from non-renewable sources. Synthetic fertilizers provide readily available nutrients to crops effectively, allowing to bring into production areas that were previously deficient in nutrients. However, producing these fertilizers involves high energy expenditures, and their use is associated with many environmental problems (Havlin et al., 2017). For example, the high solubility of nitrogen (N) and phosphorus (P) inorganic fertilizers could favor losses to nearby ecosystems, resulting in the eutrophication of water resources (Reza et al., 2019; Ullah et al., 2020). Moreover, countries with no or few resources for mineral fertilizer production rely on imports that increase agricultural production costs. For example, 80 % of the N, 71 % of the P_2O_5, and 85 % of the K_2O available for agriculture in Brazil were imported in 2021 (ANDA, 2021).

Increasing nutrient management efficiency is crucial to reducing fertilizer applications and minimizing farmers dependence on external inorganic fertilizers (Zonta et al., 2016). Strategies to enhance nutrient use efficiency try to balance production and environmental aspects of soil fertility to develop more sustainable systems. Reusing organic wastes or byproducts as organic fertilizers is a low-cost and sustainable approach that promotes nutrient recycling while utilizing wastes that would otherwise need to be treated or discarded, generating environmental liabilities (Cruz et al., 2017). Organic sources that have the potential to be used as fertilizers, such as animal wastes (e.g., manure), biochar, humic acids, filter cakes, and sewage sludge, are usually rich in organic matter and nutrients. Among those options, biosolids consist of treated sewage waste with a high nutrients and organic matter content that complies with pollutant and pathogen limits for proper surface disposal or soil application (Usepa, 1993; Cetesb, 1999; Conama, 2006; Cabreira et al., 2017). Biosolid suitability as an organic fertilizer depends upon its nutrient content and availability (Cassity-Duffey et al., 2020), which vary according to biosolid origin and processes used to stabilize it (Usepa, 1993; Abreu et al., 2017; Chavarin-Pineda et al., 2022). Nutrient availability of organic residues can be enhanced by combining organic and mineral sources into organomineral fertilizers (OMFs). The mineral fraction of OMFs is responsible for nutrient release, whereas the organic fraction provides both nutrients and carbon (C) that can fuel soil microbial activity, which increases nutrient availability and stimulates nutrient cycling (Kominko et al., 2017; Smith et al., 2020; Ullah et al., 2020). Moreover, adding organic sources to the soil can benefit cation exchange capacity (CEC), particle aggregation, aggregate stability, bulk density, and water dynamics (Li et al., 2021; Morais et al., 2023). The OMFs can increase CEC and aggregation, favoring nutrient retention and improving water percolation to mitigate nutrient losses, especially cation losses (e.g., NH_4^+, K^+, Ca^{2+}, and Mg^{2+}).

As OMFs are usually granulated or pelleted to combine the inorganic and organic sources, the physical composition and solubility of OMFs should also affect nutrient dynamics. Reza et al. (2019) reported that the low solubility of struvite (i.e., the mineral source) in an OMF reduced P losses from leaching. Furthermore, the granulation of biomass ashes can affect cations bioavailability in soil (Pesonen et al., 2017). For example, granulation or pelletization of K-enriched sewage sludge fertilizers reduced K release compared to powdered amendments and KCl (Fachini et al., 2022). As the physical and chemical composition of OMFs are directly related to fertilizer solubility, this affects nutrient release and retention and, subsequently, the potential of OMFs to limit nutrient losses (Kominko et al., 2017).

Although several studies assessed the effects of combining organic and inorganic fertilizers on nutrient availability and leaching (Magela et al., 2019; Reza et al., 2019; Liu et al., 2021; Llovet et al., 2022; Morais et al., 2023), few focused on the effects of formulation and granulation on nutrient leaching dynamics when biosolid is used as the organic matrix. This study evaluated the effect of different biosolid-based OMFs (N-P-K formulations) on N, P, and K leaching losses when compared to their isolated sources [biosolid, potassium sulfate (PS), and rock phosphate (RP)]. We hypothesized that the presence of an organic source (biosolid) granulated with mineral sources of K and P would reduce leaching, especially cations ($N-NH_4^+$ and K), due to the increase in CEC from the organic source and the slower release caused by granulation.

MATERIALS AND METHODS

Organomineral fertilizer formulation

A biosolid supplied by the State Water and Sewerage Company of Rio de Janeiro (CEDAE – Ilha do Gorvernador Station) was used for the formulation of the OMFs. Potassium sulfate (Paulifertil Fertilizantes LTDA.) and Rock Phosphate (Reactive Natural Phosphate) were used as sources of potassium and phosphorus for the OMFs, respectively. Components (biosolid, potassium sulfate, and rock phosphate) used in the granulation of the OMFs were analyzed for nitrogen and carbon by combustion (LECO TruSpec ® CHN). Phosphorus and K were determined according to the Manual of Official Analytical Methods for Fertilizers and Amendments (MAPA, 2014). Contents of calcium (Ca), magnesium (Mg), sodium (Na), micronutrients, and toxic elements [nickel (Ni), iron (Fe), cupper (Cu), manganese (Mn), chromium (Cr), zinc (Zn), lead (Pb), cadmium (Cd)] were determined using the Usepa 3050b methodology (Usepa, 1996). In the digestion extracts, total P concentration was determined by colorimetry, K and Na by flame photometry, and Ca, Mg, and micronutrients by atomic absorption spectrometry. Chemical composition of fertilizer sources is described in table 1.

The N-P-K granulation proportions used in this study were established according to rates previously assessed with organic sources of N in N-P formulations (Ferrari, 2017) and N-K formulations (Dias, 2017). The N-P-K formulation was included to determine the combined effect of macronutrients and their possible interaction on leaching dynamics. Hence, the formulations tested were preset to 1-0-0, 0-1-0, 0-0-1, 1-2-0, 1-4-0, 1-0-2, 1-2-2, 1-2-4 based on percentages of $N-P_2O_5-K_2O$. The suitability for agriculture application of biosolids and the sources of P and K were classified according to the Brazilian Normative Instruction (IN) No. 25, of July 23, 2009, by the Ministry of Agriculture, Livestock and Supply (MAPA, 2009). Subsequently, the components were ground in a mortar and then sieved in a 35-mesh sieve to homogenize the particle size of the three sources.

Table 1. Chemical characterization of organic and mineral sources used in fertilizer granulation

Property	Biosolid	Potassium sulfate	Rock phosphate
C (%)	12.00 ± 0.50	3.00 ± 0.64	4.00 ± 0.02
N (%)	2.00 ± 0.05	0.10 ± 0.03	0.20 ± 0.02
P (g kg^{-1})	5.50 ± 0.70	4.10 ± 0.30	224.00 ± 12.0
K (g kg^{-1})	0.70 ± 0.00	456.0 ± 14.50	0.90 ± 0.08
Ca (g kg^{-1})	0.03 ± 0.02	0.10 ± 0.09	0.06 ± 0.02
Mg (g kg^{-1})	0.03 ± 0.00	0.20 ± 0.00	0.06 ± 0.00
Ni (mg kg^{-1})	8.00 ± 1.80	6.86 ± 0.40	1.43 ± 0.05
Fe (mg kg^{-1})	236.00 ± 6.28	1.90 ± 0.11	71.00 ± 1.7
Cu (mg kg^{-1})	2.30 ± 0.16	0.06 ± 0.01	0.30 ± 0.06
Mn (mg kg^{-1})	2.30 ± 0.46	0.05 ± 0.00	1.60 ± 0.010
Cr (mg kg^{-1})	0.30 ± 0.03	0.04 ± 0.01	3.00 ± 0.10
Zn (mg kg^{-1})	10.40 ± 1.34	0.50 ± 0.4	1.70 ± 0.26
Pb (mg kg^{-1})	1.00 ± 0.09	0.00 ± 0.00	0.14 ± 0.14
Cd (mg kg^{-1})	0.03 ± 0.01	0.01 ± 0.00	0.85 ± 0.10

Numbers represent the mean of quadruplicates ± standard deviation.

Table 2. Chemical characterization of the organomineral fertilizers

Fertilizer	N	P_2O_5	K_2O	Organic C	C:N	pH
	kg kg^{-1}					
BGR	2.1	0.5	0.7	11.7	5.0	4.5
RPGR	0.5	5.2	0.4	1.1	2.0	6.3
PSGR	0.9	0.5	25.9	1.8	1.5	7.2
1-2-0	1.6	0.9	0.7	10.4	6.5	5.5
1-4-0	1.9	1.8	0.8	10.2	5.3	5.0
1-0-2	1.9	0.6	1.4	10.8	5.7	6.0
1-2-2	1.4	1.2	1.6	10.7	6.1	6.2
1-4-2	1.7	2.0	1.3	11.4	6.7	6.3

BGR: Granulated biosolid; RPGR: Granulated rock phosphate; and PSGR: granulated potassium sulfate.

Granulation of the OMFs was carried out in a pelletizer disk, and 3 % of polyvinylpyrrolidone (PVP) additive and washed sand were added to the dry mixture of each formulation to aid granulation and complete the formula. After granulation, OMF granules were transferred to a forced-air ventilation oven at a temperature of 45 °C for 12 h. At the end of this period, the fertilizers were sorted according to the particle size of granules, and the 2 to 4 mm diameter class was used in this experiment.

In each OMF formulation, the content of N, P, and K were determined according to the Manual of Official Analytical Methods for Mineral, Organic, Organomineral, and Corrective Fertilizers (MAPA, 2014), pH was measured in water (1:2.5) (Table 2), and total organic carbon was analyzed through wet oxidation (Yeomans and Bremner, 1988) due to technical issues with the elemental analyzer used in the preliminary characterization of the organic and mineral sources.

Leaching trials

To determine N, P, and K leaching from the different fertilizers, an experiment was conducted with leaching columns by adapting the methodology of Bamberg et al. (2012), as illustrated in figure 1. The experiment was conducted in a greenhouse located in the experimental area of the Department of Soils – Institute of Agronomy of the *Universidade Federal Rural do Rio de Janeiro* (UFRRJ), Seropédica, Rio de Janeiro, Brazil.

The experiment was conducted in a randomized complete block design. Evaluated treatments are described in table 2, with the addition of the natural ungranulated sources and a control with no fertilizer addition. Thus, there were 36 experimental units, i.e., 12 treatments × 3 replications.

Experimental units consisted of 0.10 m diameter × 0.60 m tall PVC leaching columns filled with sandy-textured soil (Figure 1). This soil was collected from the top layer (approximately 0.00-0.15 m) of a soil classified as *Planossolo Háplico* (Santos et al., 2018), equivalent to a Typic Fragiaquult in the US Soil Taxonomy classification (Soil Survey Staff, 1999), with its chemical properties described in table 3. This soil order was chosen as it is representative of the soils within the region under study (i.e., the state of Rio de Janeiro, Brazil).

Dolomitic limestone with a relative neutralizing value of 80 % was added to the soil at a rate of 0.9 g kg^{-1} (equivalent to a dose of 2 Mg ha^{-1}) and homogenized with a concrete mixer. Leaching columns were filled with 4.3 kg of the sieved and limed soil, compacting it to a height of 0.45 m to reach a density of 1.2 g cm^{-3}, equivalent to the density of the *Planossolos* class. The moisture was adjusted to 9 % (gravimetric moisture) for 20 days prior to the beginning of the leaching trial to allow time for the limestone to neutralize acidity and achieve an ideal pH of 5.5-6.5 that favors NPK availability in the soil solution.

Table 3. Chemical and physical properties of the soil used in the experiment

Property	Value/content
pH(H_2O) (1:2.5)	4.7
Organic carbon	2.2 g kg^{-1}
Base saturation	72 %
Organic matter	3.9 g kg^{-1}
P	3.5 mg dm^{-3}
K	15.5 mg dm^{-3}
H+Al	51 $cmol_c$ dm^{-3}
Al	35 mg dm^{-3}
Ca	80 mg dm^{-3}
Mg	72.5 mg dm^{-3}
Na	34.5 mg dm^{-3}
Sand	800 g kg^{-1}
Silt	100 g kg^{-1}
Clay	100 g kg^{-1}

The rate of OMF, 1-0-0, and biosolid added in each leaching column was 4.4 g per column, equivalent to the addition of 80 kg of total N ha^{-1} (computed based on the area of the tube). This input rate is comparable to the recommendation for corn in the Fertilization and Liming Manual for Rio de Janeiro (Freire et al., 2013). In treatments without added N, the rates applied were equivalent to the minimum amount of P and K found in the treatments with NPK due to OMF application. For P fertilizers, 0.95 g of RP or 0-1-0 were added, whereas for K, 0.23 g of PS or 0-0-1 applied per container. Isolated sources and OMFs were incorporated into the soil at a 0.05 m depth in the leaching column.

Leaching volume corresponded to the application of a volume of distilled water (V) equivalent to the balance between the annual averages of Precipitation (P) and Potential Evapotranspiration (Etp) of *Seropédica* (local climatic conditions). As P = 1354 mm and

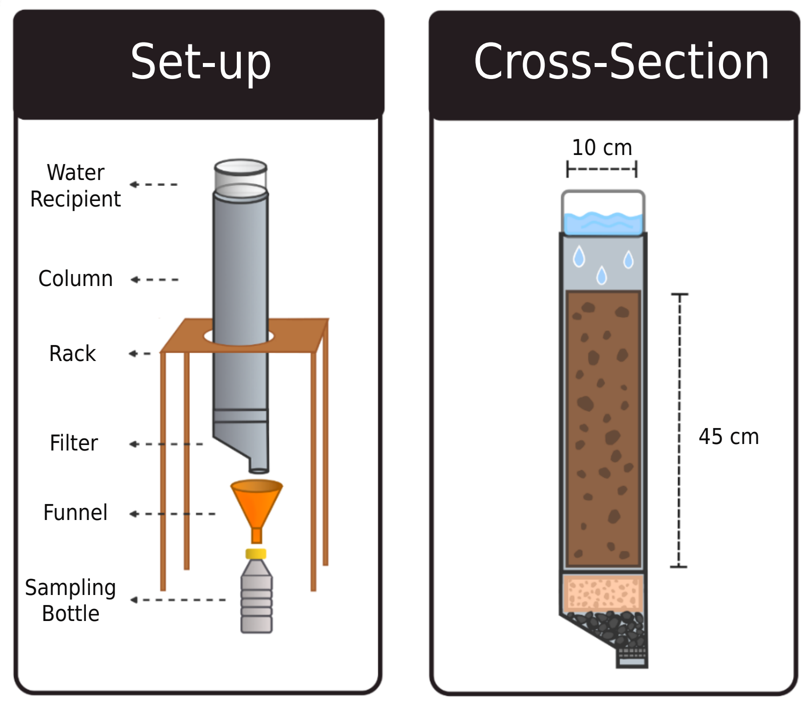

Figure 1. Diagram of the experimental set-up and composition of leaching columns.

Etp = 912 mm (Carvalho et al., 2015), the leaching volume would be V = 442 mm yr^{-1} or V = 1.2 mm day^{-1}, assuming a uniform distribution of leaching throughout the year.

As the leaching frequency that allows for nutrient quantification of the leachate is in the order of 7 to 14 days, the use of this leaching interval is recommended (Bamberg et al., 2012). To simulate a weekly leaching event, the amount of leaching applied was 8.5 mm per week (1.2 mm day^{-1} × 7 days). This value was multiplied by a process acceleration factor equal to 5 (it can vary from 5 to 10, depending on the method) to one leaching per week over ten weeks, to get a total of 10 leaching events corresponding to what would have been leached over 50 weeks in field conditions.

As the estimated pore volume of the column was 1932 mL, 1650 mL water was applied weekly per experimental unit, equivalent to 42.5 mm or roughly 85 % of the estimated pore volume. Leaching volumes were distributed using a pot with holes placed at the opening of the column, which allowed an approximate flow rate of 25 mL min^{-1} (0.8 mm min^{-1}), as recommended by Bamberg et al. (2012). This allowed water percolation without creating reducing conditions (Figure 1). Volume of water applied in the first leaching event did not exceed the field capacity and did not produce any leachate; hence, there were nine leachate extracts instead of 10.

Leachate samples were collected in plastic bottles, then preserved and analyzed for elemental concentrations following the methodology described in the Standard Methods for the Examination of Water and Wastewater (Rice et al., 2012). Briefly, samples were acidified and refrigerated, and concentrations were determined by colorimetry (P), flame spectrometry (K), and distillation + titration [N-NH_4^+ and N-(NO_3^- + NO_2^-)]. However, no P was detected in the leachate samples during this trial. This could have happened due to the P-immobilization potential of the soil used in the experiment or limitations in the methodology chosen, hence P is not discussed further.

Estimation of the net amount of K, N-NH_4^+, and N-(NO_3^- + NO_2^-) leached from the soil as a function of fertilizers added was obtained with equations 1, 2, and 3:

$$K\,net = Kr - Kc \qquad \text{Eq. 1}$$

$$NH_4^+net = NH_4^+r - NH_4^+c \qquad \text{Eq. 2}$$

$$(NO_3^- + NO_2^-)net = (NO_3^- + NO_2^-)r - (NO_3^- + NO_2^-)c \qquad \text{Eq. 3}$$

in which: “net” is the net amount leached of each nutrient; “r” is the amount leached by the fertilizer treatment; and “c” is the amount leached in the control treatment. All net leaching values are given in mg L^{-1}.

Data analysis

Data were analyzed using the R software (R Development Core Team, 2022) with a linear mixed model using the *nlme* package (Pinheiro and Bates, 2000; Pinheiro et al., 2022). A one-way analysis of variance (ANOVA) was performed to test the cumulative leaching response to fertilizer treatments. As some fertilizers did not contain P and K, the fertilizer treatments were incorporated as the within-group heteroscedasticity structure to improve the homogeneity of variances (Pinheiro and Bates, 2000). Means were compared using the Bonferroni adjustment at $\alpha = 0.05$.

A two-way analysis of variance was performed using a repeated measures model (Piepho et al., 2003) to test the significance of net leaching [K^+, N-NH_4^+, and N-(NO_3^-+NO_2^-)] as a function of treatments and leaching events. Simple effects and interactions were considered

Table 4. Analysis of variance of cumulative and net leaching as a response to fertilizer treatments and leaching events

Factor	Cumulative leaching							
			K		$N\text{-}NH_4^+$		$N\text{-}(NO_3^- + NO_2^-)$	
	DF	residual DF	F-value	p-value	F-value	p-value	F-value	p-value
Fertilizer	10	20	3.9	0.005	1.1	0.392	0.7	0.674
Factor	**Net leaching**							
			K		$N\text{-}NH_4^+$		$N\text{-}(NO_3^- + NO_2^-)$	
	DF	residual DF	F-value	p-value	F-value	p-value	F-value	p-value
Fertilizer	10	20	8.2	<0.0001	0.9	0.563	0.7	0.718
Leaching events	8	176	7.1	<0.0001	31.2	<0.0001	22.2	<0.0001
Fertilizer x Leaching	80	176	1.2	0.222	1.3	0.082	0.9	0.787

BGR: Granulated biosolid; RPGR: Granulated rock phosphate; and PSGR: granulated potassium sulfate.

significant at $\alpha = 0.05$ probability and marginally significant at $\alpha = 0.1$, according to Sulc et al. (2001). Treatment and leaching events were set as fixed effects, and block and the interaction between block and fertilizer as random effects. These variables had a repeated measure nature because evaluations were performed on the same experimental unit through all the leaching events. In addition to the heteroscedasticity structure, a covariance structure (First Order Autocorrelation) was added to account for possible correlation between variables sampled in the same experimental unit and leaching event. The addition of heteroscedasticity and covariance structures was evaluated through the improvement in the heterogeneity of variances and normality of the residuals. The goodness of fit between the simple and more complex models was assessed according to the AIC fit statistic.

Planned contrasts were performed as described in table 4 to isolate the influence of granulation and organic source (biosolid) in affecting K cumulative leaching and the influence of formulation (adding sources of P and K) and granulation in affecting total leaching of $N\text{-}NH_4^+$, and $N\text{-}(NO_3^-+NO_2^-)$ (Piepho et al., 2006). Planned contrasts were performed using the Bonferroni adjustment at $\alpha = 0.05$ probability. Mean comparisons and contrasts were performed using the *emmeans* package (Lenth et al., 2022).

RESULTS

Fertilizer had a significant effect only for K cumulative and net leaching, with no fertilizer effect on N leaching (Table 4). Leaching events significantly impacted the net leaching of all nutrients ($p<0.001$), indicating that leaching dynamics varied across time.

Soil K leaching increased through the first four leaching events, then decreased in the following two weeks, and increased slightly at the seventh event (Figure 2). Among the fertilizers with K, leaching was constantly higher for PS without granulation. Non-granulated PS had the highest net K leaching, with an average of 10 mg L^{-1} per leaching event, although this was not significantly different from other fertilizers containing K (data not shown). Leaching of PSGR increased at the sixth leaching event, following a slower but similar trend as non-granulated PS from that point.

Similarly to net leaching, PS had the highest cumulative K leaching (90 mg L^{-1}), which did not differ from the other fertilizers containing potassium (PSGR, 1-0-2, 1-2-2, and 1-4-2) (Figure 2). Excluding PS, fertilizers containing K had leaching values ranging from 30.8 to 19.8 mg L^{-1}, with the highest values recorded under the 1-0-2 treatment and the lowest with PSGR. Despite not being statistically different, the association of PS with granulation or organomineral fertilizers decreased cumulative K leaching 4.5- and 3.0-fold (averaged through OMFs), respectively. Furthermore, fertilizers that did not contain K (Biosolid, BGR, 1-2-0, 1-4-0, RP, and RPGR) increased K leaching relative to

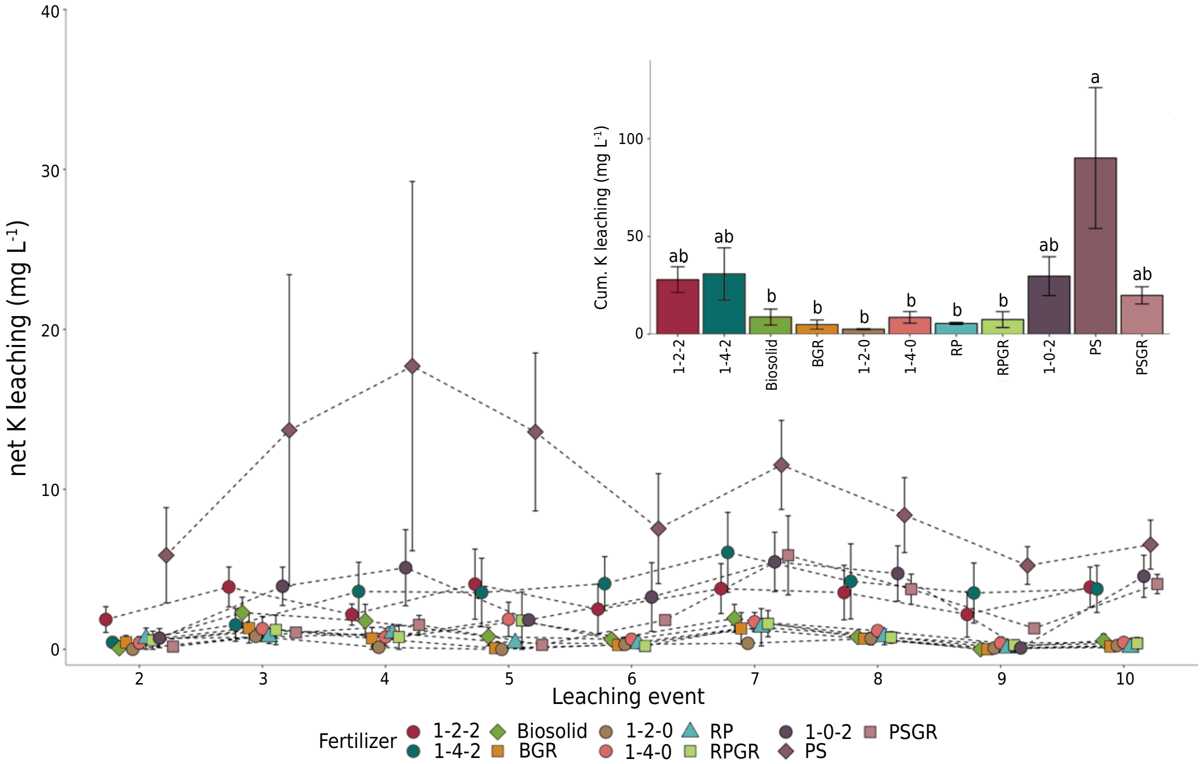

Figure 2. Mean (± standard error) of K leaching as a response to organomineral fertilizers. Treatments that share at least one letter do not differ when using a Bonferroni adjustment, at $p<0.05$. BGR: Biosolid granulated; RP: Rock phosphate; RPGR: Rock phosphate granulated; PS: Potassium sulfate; PSGR: Potassium sulfate granulated. Some net leaching points at a given leaching event were moved slightly to avoid overlapping points and error bars, but all measurements for a given leaching event were taken on the same day. Points with no error bars had small standard errors (e.g., <0.5 mg L^{-1}).

the control, even though no K was added, although they had significantly less cumulative K leaching than PS.

There were two main peaks in net N leaching, despite the variability observed in N-(NO_3^- + NO_2^-) net leaching (Figures 3 and 4). First peak occurred in the third leaching event for NH_4^+ and at the second and third leaching events for NO_3^- + NO_2^-. Second peak happened at the seventh leaching event for NH_4^+ and at the sixth and seventh leaching events for NO_3^- + NO_2^-. At the seventh leaching event, 1-4-2 and 1-2-2 had the highest net NH_4^+ leaching (15.1 and 11.2 mg L^{-1}, respectively), which was not statistically different from most of the fertilizers containing biosolid (1-2-0, 1-4-0, Biosolid and BGR). Net NO_3^- + NO_2^- leaching varied through time but did not differ among treatments (Figure 4).

Cumulative N leaching in the forms of NH_4^+ and NO_3^- + NO_2^- (Figures 3 and 4) was not different among fertilizers, with a mean cumulative leaching of 18.2 (11.1 to 24.7) mg L^{-1} for NH_4^+ and 98 (57.9 to 143) mg L^{-1} for NO_3^- + NO_2^-. Although not significant, the presence of biosolid in the composition enhanced NH_4^+ and NO_3^- + NO_2^- leaching by approximately 1.5 and 1.2 folds, respectively.

A contrast analysis compared the effects of formulation and granulation on leaching by simplifying the variable effects of the 11 fertilizers previously described (Table 5 and Figure 5). The "formulation" contrasts describe the mix of organic and mineral forms as single-source fertilizers, regardless of granulation. The "granulation" contrasts compare granulated fertilizers to single-source fertilizers without granulation, regardless of whether they are OMFs or single sources. Overall, the effect of formulation did not lead to significant differences in leaching (p = 0.236 or higher for both N forms and K). In contrast, the effect of granulation was significant for cumulative and net leaching of K (p = 0.008).

Granulation effectively reduced both net and cumulative K leaching (Figure 5). Cumulative leaching was 63 mg L^{-1} lower (i.e., 7 mg L^{-1} per leaching event on average) when

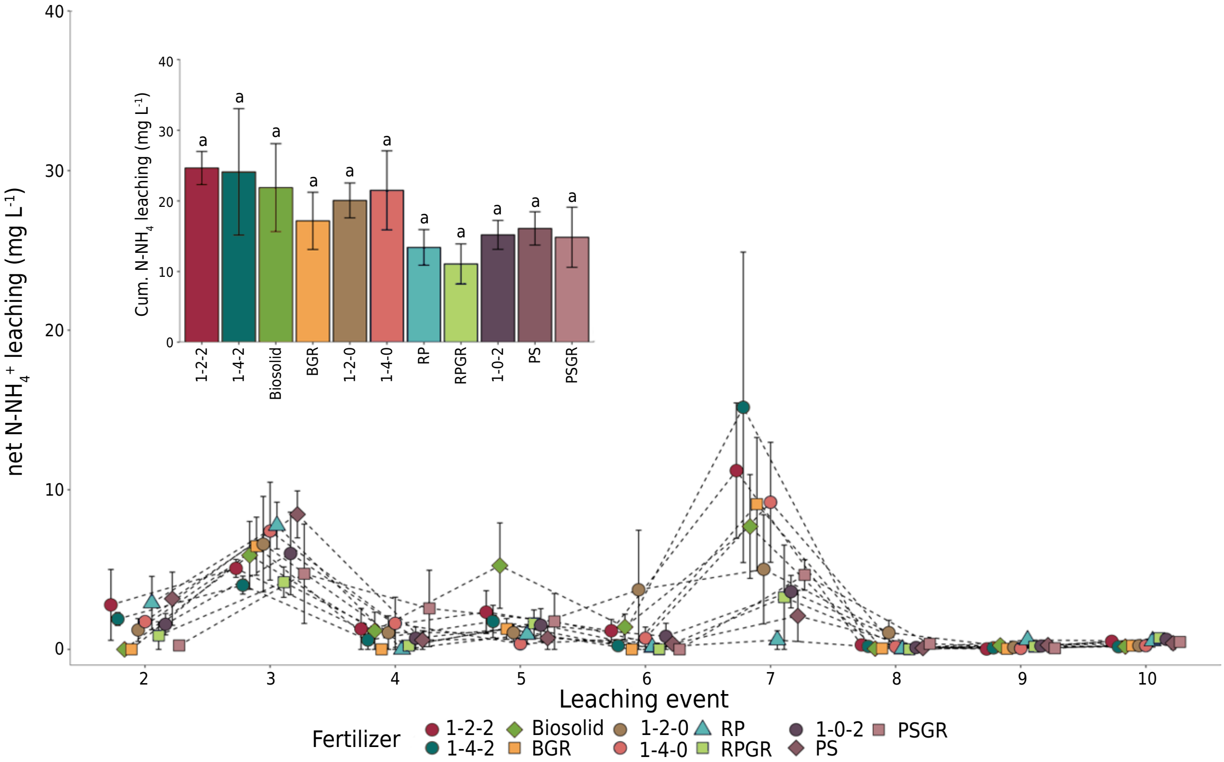

Figure 3. Mean (± standard error) of N-NH$_4^+$ leaching as a response to organomineral fertilizers. Treatments that share at least one letter do not differ when using a Bonferroni adjustment at $p<0.05$. BGR: Biosolid granulated; RP: Rock phosphate; RPGR: Rock phosphate granulated; PS: Potassium sulfate; PSGR: Potassium sulfate granulated. Some net leaching points at a given leaching event were moved slightly to avoid overlapping points and error bars, but all measurements for a given leaching event were taken on the same day. Points with no error bars had small standard errors (e.g., <1 mg L^{-1}).

K sources were granulated, a 70 % reduction compared to PS in its non-granulated form. Although there is a trend for lower K leaching with formulation (cumulative and net), these differences were not significant. Considering that all OMFs containing biosolid were granulated, reductions in leaching might have been due to the granulation of OMFs instead of adding an organic source in the formulation. While there were trends of lower leaching of N-NO$_3^-$ + NO$_2^-$ with granulation and formulation, these were not significant due to large variability in the effects of granulation and formulation (Figure 5).

DISCUSSION

Potassium leaching

Throughout the experiment, all fertilizers displayed comparable leaching patterns characterized by two peaks and a subsequent reduction in the net K leached. This could be explained by the dynamic interplay of vertical water mobility within the leaching column, as well as the regulatory effect of the Potassium Buffering Power (KBP) in governing the concentration of K$^+$ ions within the soil solution. Potassium buffering power denotes the soil's capacity to sustain a stable concentration of K$^+$ ions within the solution, accommodating both additions and removals of K from the soil matrix (Ernani et al., 2007). In this experiment, the soil was amended with limestone, elevating the pH, supplying calcium, and consequently augmenting the KBP by replacing exchangeable K (Kolahchi and Jalali, 2007). This liming-driven elevation of KBP could have facilitated the initial availability of K$^+$ ions in the solution, which was more pronounced in non-granulated PS.

Fluctuation in leachate concentrations may happen as K lost through leaching is effectively replenished by the soil itself through KBP. Equilibrium established by exchangeable K$^+$ guides this replenishment process and can be affected either by adding K sources or altering K forms (e.g., soluble vs. adsorbed) (Ernani et al., 2007). As all treatments containing K

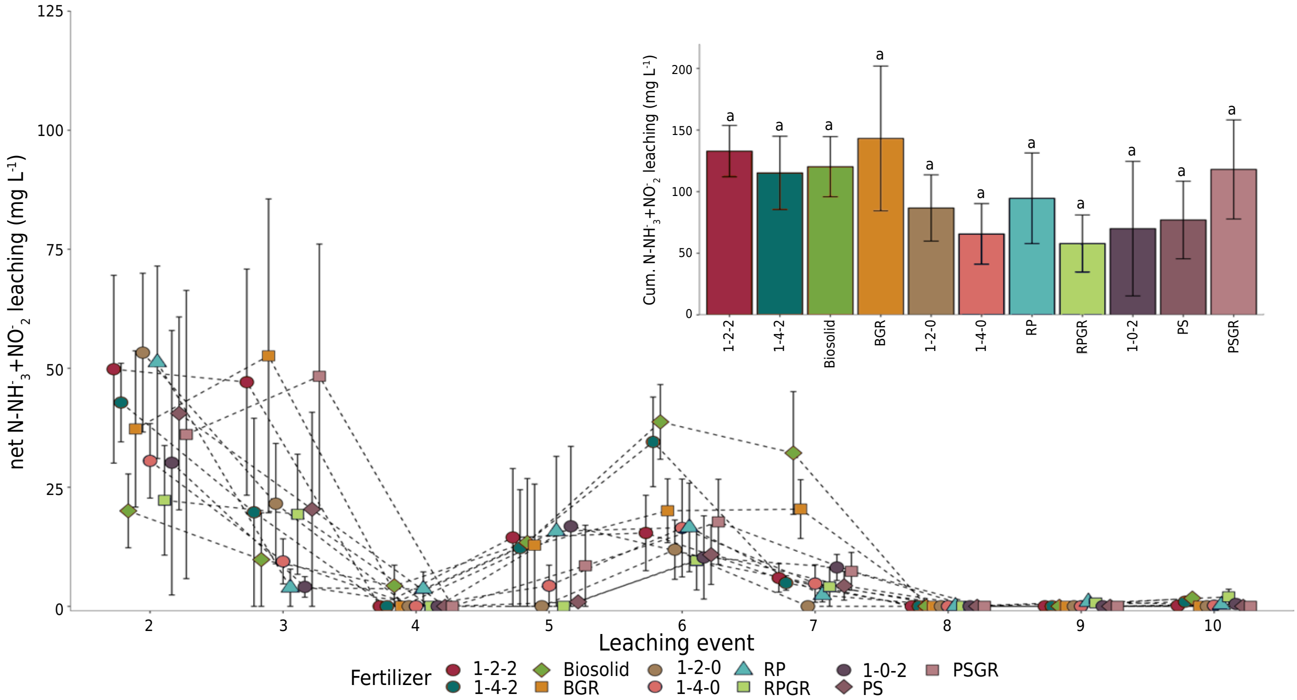

Figure 4. Mean (± standard error) of N-(NO_3^- + NO_2^-) leaching as a response to organomineral fertilizers. Treatments that share at least one letter do not differ when using a Bonferroni adjustment at p<0.05. BGR: Biosolid granulated; RP: Rock phosphate; RPGR: Rock phosphate granulated; PS: Potassium sulfate; PSGR: Potassium sulfate granulated. Some net leaching points at a given leaching event were moved slightly to avoid overlapping points and error bars, but all measurements for a given leaching event were taken on the same day. Points with no error bars had small standard errors (e.g., <7 mg L^{-1}) to be displayed on the plot.

were applied at the same rate, this distinct fluctuating behavior in the leachate can be reasonably attributed to the interplay with KBP, given that the addition of K via fertilizer enhances the quantity of the ion in the soil complex and the addition of water per week would not promote a complete flush of the soil column (85 % pore space).

As PS was the most soluble form of K applied, it increased KBP by enhancing K quantity. However, an increase in K leaching is also observed with other forms of K (PSGR, 1-0-2, 1-4-2, and 1-2-2) after the sixth leaching event, possibly reflecting the effect of granulation in reducing K solubility and delaying the supply of K to the soil solution. When the effect of treatments is isolated to granulation vs. formulation (i.e., presence of biosolid), formulation showed a trend in reducing leaching, but this effect was not significant. Therefore, the trend observed for the effect of formulation in minimizing leaching is possibly more related to the granulation of the OMFs rather than the presence of biosolid in their composition. In contrast, the granulation of the fertilizer significantly decreased cumulative and net leaching, indicating that the trend observed with formulation minimizing leaching is possibly more related to the granulation of the OMFs rather than the presence of biosolid in their composition.

Our results suggest that granulation effectively reduces K leaching, as it reduced cumulative leaching by 70 % compared to using K fertilizer in its natural form. Consistent with our results, other studies reported that granulation could affect the solubility and availability of K directly. For example, Pesonen et al. (2017) observed that K availability was reduced by 16 % when peat and wood ashes were granulated. Similarly, Fachini et al. (2022) observed a reduction of 13 % in K release and a 31 mg K L^{-1} reduction in cumulative leaching when comparing potassium chloride (KCl) granulated with biochar to only KCl. Reduction in K availability and leaching with granulation is due to changes in the physical structure of the fertilizer, which decreases its surface area (Fachini et al., 2022). A smaller surface area decreases the diffusion of water into the fertilizer and its contact

Table 5. Planned contrasts coefficients and significance with Bonferroni adjustment (α = 0.05)

Fertilizer	K		$N-NH_4^+$		$N-(NO_3^- + NO_2^-)$	
	Formulation	Granulation	Formulation	Granulation	Formulation	Granulation
BGR	0	0	-0.5	0.1666	-0.5	0.1666
RPGR	0	0	0	0	0	0
SPGR	-0.5	0.25	0	0	0	0
1-2-0	0	0	0.2	0.1666	0.2	0.1666
1-4-0	0	0	0.2	0.1666	0.2	0.1666
1-0-2	0.333	0.25	0.2	0.1666	0.2	0.1666
1-2-2	0.333	0.25	0.2	0.1666	0.2	0.1666
1-4-2	0.333	0.25	0.2	0.1666	0.2	0.1666
Biosolid	0	0	-0.5	-1	-0.5	-1
RP	0	0	0	0	0	0
PS	-0.5	-1	0	0	0	0
			P-value			
Cum. leaching	0.236	0.008	1	1	0.366	1
Net leaching	0.263	0.008	1	1	0.542	1

BGR: Biosolid granulated; RP: Rock phosphate; RPGR: Rock phosphate granulated; PS: Potassium sulfate; PSGR: Potassium sulfate granulated.

with the soil surface, reducing the solubilization of the inorganic source and contributing to a slower release of the nutrients they contain (Ernani et al., 2007; Kim et al., 2014).

Reducing fertilizer solubility can lead to both favorable and unfavorable outcomes, depending on the temporal synchrony between the nutrient requirements of the crop and soil nutrient availability. Therefore, lower solubility can be a desirable fertilizer property when conditions are favorable for leaching (Bocuti et al., 2020). Due to the observed relationship between effective hydraulic conductivity and soil sand content, sandy soils are more prone to K leaching (Bocuti et al., 2020).

When studying K leaching from KCl in soils with contrasting textures, Mendes et al. (2016) found a 13.5-fold increase in the amount of K leached in sandy soil compared to clay soil, depending on the water volume applied. According to the authors, the low CEC observed in sandy soils favored K dissolution in the soil solution and subsequent losses. Additionally, Flores et al. (2022) found that, in soils with a sandy loam texture (16 % clay, 14 % silt, and 70 % sand), the leaching of K intensified at higher K_2O application rates, with a more pronounced response with greater irrigation volumes. In contrast, altering irrigation volume did not affect K leaching in soils with higher clay content.

Sandy soils, especially those abundant in coarse sand, often have greater macroporosity, leading to enhanced drainage but reduced microporosity, which limits water retention within the soil (Viana et al., 2023). As water percolation is facilitated in sandy soils, this can cause the removal of most elements dissolved in the soil solution via leaching, including K. In our study, the soil used had 800 g kg^{-1} of sand, i.e., an environment conducive to K leaching, and fertilizer granulation was quite effective at mitigating K losses in this soil.

Nitrogen leaching

The interaction between fertilizer and leaching events for NH_4^+ shows that all treatments had a peak in leaching at the third event, whereas only fertilizers with biosolids peaked after the seventh leaching. This pattern in NH_4^+ leaching indicates the first peak is likely driven more by soil organic matter mineralization (SOM), and the second by biosolids mineralization. Moisture and pH adjustment in the soil columns and fertilizer application could have favored optimal conditions for SOM mineralization (Havlin et al., 2017). The subsequent increase in net NH_4^+ is significantly higher in fertilizers containing N in their composition. This rise occurs around 50 days, consistent with the peak in biosolid and

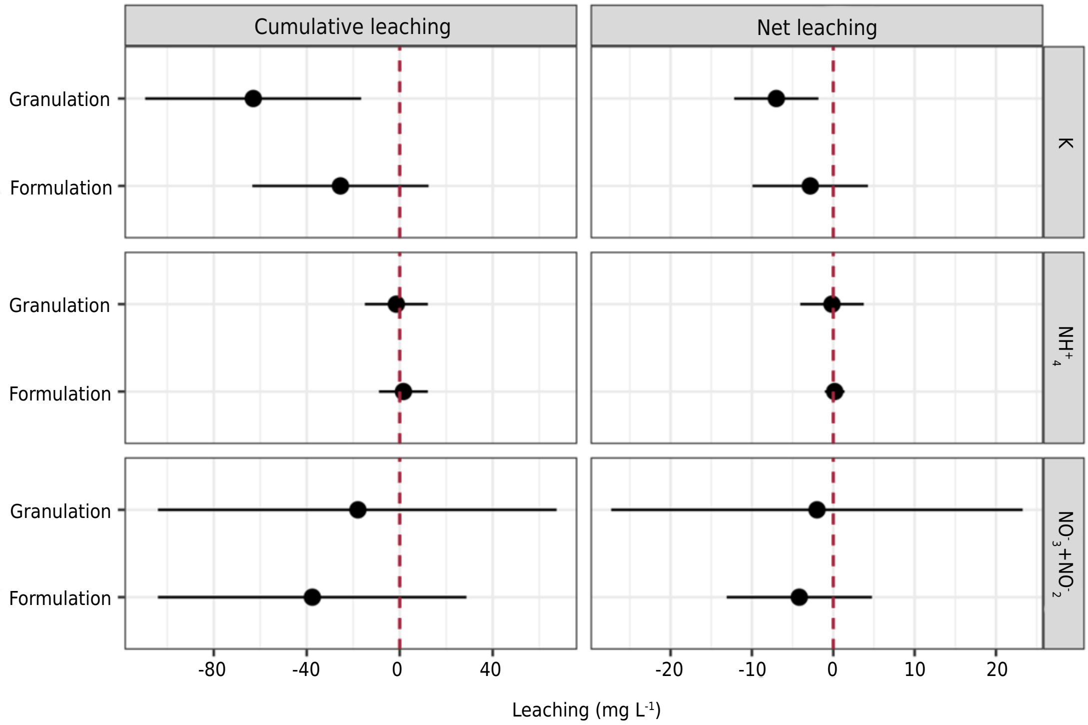

Figure 5. Effects of granulation and formulation on K, N-NH_4^+, and N-(NO_3^- + NO_2^-) leaching. Solid circles represent the estimated mean of each contrast group. Error bars are 95 % confidence intervals (CIs). Mean estimate sizes were considered significantly different only when the 95 % CIs did not overlap with zero.

OMF mineralization that was observed primarily between 28 and 56 days during an incubation study conducted with the same biosolid material and OMFs (Netto-Ferreira et al., 2023). Nitrogen mineralization of organic sources is dependent on N concentration, but mineralization also varies based on the quality of the C and N in their composition (Cassity-Duffey et al., 2020).

Although nutrient release from fertilizers is normally proportional to the applied rate, the actual availability in organic and organomineral sources may differ depending on nutrient interaction with organic and inorganic compounds in their composition (Fachini et al., 2022; Morais et al., 2023). In a previous study, the combination of P and K sources influenced N availability and the nutrient efficiency index of the same OMFs studied herein (Netto-Ferreira et al., 2023). Our results show that formulation may influence N mineralization and availability, but this behavior does not significantly reduce N leaching.

Even though the OMFs and biosolids can reduce leaching due to their potential to enhance CEC and soil structure, the lack of N leaching differences among biosolids and OMFs may be related to the supply of N being provided from the same source (e.g., biosolid) and applied at the same rate. When the same N rate and the same rate of struvite as P source were supplied, no differences were found in NH_4^+ leaching across different air-drying processes of the organic material (Reza et al., 2019). Similarly, providing the same N rate with variable OMF rates based on chicken manure, coffee husks, and monoammonium phosphate (MAP) did not influence N leaching in sandy soils (Morais et al., 2023).

In a meta-analysis evaluating the combined effect of soluble synthetic and organic N fertilizers, the presence of organic fertilizers reduced leaching by 44 % (Liu et al., 2021). These authors also show that a significant leaching reduction happened if 70 % of total N was supplied as organic N, but no significant effects were found when less than 70 % of total N was provided as organic N. Moreover, combining organic forms with synthetic fertilizers is most effective at reducing leaching when N fertilizer is applied at high rates that favor leaching, such as 150 to 300 kg ha^{-1} of N (Borchard et al., 2019). In our study, the biosolid was the N source that was applied at a rate equivalent to 80 kg ha^{-1} for all N-containing treatments, which was gradually released due to OMFs properties (Kominko et al., 2017; Fachini et al., 2022). The slow-release property of granulated OMFs combined with the low rate of N supplied could have been the reasons for the lack of effect of OMFs on N leaching. A better evaluation of the effect of organic sources and their granulation on N leaching could be achieved by comparing biosolid to an inorganic N source or multiple organic N sources.

CONCLUSIONS

Contrary to our initial hypothesis, the granulation and formulation of fertilizers did not exhibit a comparable impact on N leaching, although granulation contributed to reduced K leaching. Decoupling formulation and granulation more completely was essential to isolating them as independent factors that can be tested for both N and K leaching. This approach emphasizes that granulation had a more pronounced effect on K leaching inhibition than combining biosolid and inorganic sources in OMFs (organomineral fertilizers). Hence, considering the physical composition of OMFs is particularly relevant to employ them for effective nutrient management.

Effects on N leaching, however, were not affected by formulation or granulation. Limited influence from these factors could be due to the lack of differences between N rates and sources applied. A better understanding of the impacts of biosolids on N leaching dynamics could be observed when comparing biosolids to other organic sources or considering biosolids solely as the organic fraction of OMFs in combination with inorganic sources of N. Absence of P leaching could be attributed to the immobilization conditions of the highly weathered soils used in our experiment, although P leaching dynamics should be further explored in similar conditions as those we propose for N (i.e., inorganic and organic sources).

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