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Nitrogen, phosphorus, potassium, calcium and magnesium release from two compressed fertilizers: column experiments

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Abstract

We used soil columns to study nutrients release from two compressed NPK fertilizers. The columns were filled with soil material from the surface horizon of a granitic soil. Tablets of two slow-release NPK fertilizers (11-18-11 or 8-8-16) were placed into the soil, and then water was percolated through the columns in a saturated regime. Percolates were analyzed for N, P, K, Ca and Mg. These nutrients were also determined in soil and fertilizer tablets at the end of the trials. Nutrient concentrations were high in the first percolates, reaching a steady state when 1426 mm water have percolated, which is equivalent to approximately 1.5 years of rainfall in the geographic area. In the whole trial, both tablets lost more than 80 % of their initial N, P and K contents. However, K, Ca and Mg were the most leached, whereas N and P were lost in leachates to a lesser extent. Nutrient release was slower from the tablet with composition 8-8-16 than from the 11-18-11 fertilizer. In view of that, the 8-8-16 tablet can be considered more adequate for crops with a nutrient demand sustained over time. At the end of the trial, the effects of these fertilizers on soil chemical parameters were still evident.

1 Introduction

Conventional fertilizers supply plants quickly with nutrients, giving rise immediately to high nutrient availability. In some cases, this rapid contribution may be excessive, and nutrient excess, as well as nutrient deficiency, can have deleterious effects on plant growth. Moreover, nutrient excess may cause them to be transferred to surface and ground water, resulting in environmental problems (Khan et al., 2014). Therefore, a sound management of fertilization should reconcile the maintenance of high crop yields with reduced costs, resource economy and environmental issues.

Slow-release fertilizers may represent a solution to these problems. The behavior of “slow acting” fertilizers is close to that of an ideal fertilizer, since theoretically the release of nutrients takes place in the moment and the amount required by plants (Oertli,

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1980; Jimenez, 1992). Jiménez (1992) and Shaviv (2001) classify slow-release fertilizers according to the mechanism of delaying nutrient transfer to the substrate: materials coated by polymers or resins, low-solubility organic substances (urea formaldehyde, isobutylendiurea), nutrients in a carrier matrix (waxes, peat, vermiculite, lignin, etc.).

5 Most trials conducted to test the effectiveness of these fertilizers conclude that the amount of nutrients required is significantly reduced compared to conventional fertilizers, highlighting the energy savings and the improved use of N, minimizing its losses (Shoji and Kanno, 1994; Shaviv, 2001; Hangs et al., 2003; Chen et al., 2008; Sato et al., 2008; Entry and Sojka, 2008; Hyatt et al., 2010; Wilson et al., 2010). Another
10 reason for recommending the use of slow-release fertilizers is to prevent the emission of N₂O from N fertilization practices, due to its role in climate change (Cheng et al., 2006; Jingyan et al., 2010). However, the effectiveness of this type of fertilizer has not been extensively tested under a range of environmental conditions that may occur due to climatic variation and soil water content.

15 In Galicia (NW Spain), some studies were conducted in forest plots using tablet-type slow-release fertilizers, produced in the *Lourizán Forest Center* (Pontevedra) by compressing various mixtures of fertilizers without covers or binders. The results indicated that, compared to conventional fertilizers, slow-release fertilizers increased the height, diameter and survival of *E. globulus* and *P. pinaster*, whereas no significant differences
20 were observed in *P. radiata* (Bará and Morales, 1977). However, these studies are limited and focused on the effects on forest production, thus needing further research to test the behavior of such slow-release fertilizers and to investigate the dynamics of each nutrient release.

25 The objectives of this work are: (1) to study the dynamics of nutrient release by two different slow-release fertilizers, (2) to understand the behavior of nutrients released and their effects on the chemistry of the soil and the drainage water. For that purpose a laboratory experiment was conducted under controlled conditions using soil columns.

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2 Materials and methods

2.1 Soil material

The experiment was conducted on an acid sandy loam soil developed over granite, collected in an abandoned field. This soil has high organic matter content, low eCEC, and is classified as Cambic Umbrisol (Humic) (WRB, 2007). The surface soil layer (0–20 cm) was collected after removing the vegetation and the litter. The soil was oven-dried at 40°C and sieved through a 5 mm mesh prior to introduction in laboratory columns (50 cm long and 7.3 cm inner diameter).

2.2 Fertilizer tablets

One NPK compressed tablet, having an 11-18-11 or 8-8-16 composition, was placed in each soil column. Calcium phosphate, potassium sulfate, N as amide, and urea formaldehyde and magnesite (magnesium carbonate) were used in the manufacture of the tablets. The size of these tablets was 3.3 mm in diameter and 33.0 mm in thickness. Table 1 shows the weight and nutrient contents of fertilizer tablets.

2.3 Laboratory columns

The experimental design consisted of three replicates per treatment, including controls. The experimental device was described by Núñez-Delgado et al. (1997) and has been used in previous studies (Núñez-Delgado et al., 2002; Pousada-Ferradás et al., 2012). A soil sample (900 g) was introduced in each column, tapping the column to facilitate the settlement of the particles and to achieve a bulk density similar to that of natural soil. The experiment was conducted under saturation conditions, in order to avoid variability in moisture content and at the same time ensuring water-saturation conditions, thus ruling out the influence of redox processes.

After filling the columns, the soils were saturated with distilled water from the bottom by capillarity, to facilitate the removal of pore air and to guarantee wetting. When the

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wetting was completed, the soils were weighed to determine the water content at saturation. Then, distilled water started to flow through the columns by gravity. The flow rate and the pH and electrical conductivity of the leachates were measured for 18 days. By this time, the electrical conductivity was stabilized at around $9 \mu\text{S cm}^{-1}$, and one fertilizer tablet was placed in each column (excepting controls), introduced in the upper part of the soil (within the first centimeters). The water flow was resumed and, on average, six water samples were collected daily from each column for 15 days, preserving it at $4 \text{ }^\circ\text{C}$. pH and electrical conductivity were measured in freshly collected samples; when values for these parameters were very similar in successive samples, the sampling frequency was reduced to once a day. At the end of the columns experiment, the flow of distilled water was stopped, the samples corresponding to each day were mixed and homogenized and an aliquot reserved for analysis. The whole period of water flow was 80 days and the total water flow was 56.15 L. At the end of the experiment, the remaining of each tablet was collected and analyzed.

2.4 Chemical analysis

The following determinations were performed in leachates: electrical conductivity and pH (potentiometric methods), concentrations of NH_4^+ and NO_3^- (by steam distillation), P (by visible spectrophotometry; Olsen and Sommers, 1982), Ca, Mg, Na, K and Al (by atomic absorption or emission spectrometry).

Soil samples before and at the end of the experiment were subjected to the following determinations: pH in water (soil : water ratio 1 : 10), total carbon and nitrogen (using a LECO 2000 auto-analyzer), exchange Ca, Mg, Na, K, Al (extracted by $1 \text{ M NH}_4\text{Cl}$ and measured by a Perkin-Elmer AAnalyst 200 atomic absorption spectrometer), available phosphorus (Olsen and Sommers, 1982). The effective cation exchange capacity was calculated as the sum of Ca, Mg, Na, K and Al, extracted by $1 \text{ M NH}_4\text{Cl}$. NO_3^- and NH_4^+ were extracted by 2 M KCl and determined by steam distillation (Keeney and Nelson, 1982).

The differences between the two treatments regarding the amount and type of the elements that have been leached may be related to the quantity contributed by each treatment, as well as the different solubility of the compounds that form the tablets.

3.2 Change of soil parameters after percolation

The soil used in the experiment presented a very low initial pH, which had increased after the experiment, particularly in the fertilized columns (Table 2). In fertilized columns, cations contributed by fertilizers may replace acid exchange cations, which would result in soil alkalization. This seems to be particularly remarkable in treatment 8-8-16, which is richer in K and Mg; also leachates from this treatment, excepting the initial period, had higher pH values than those from treatment 11-18-11 (Fig. 1). In control columns, the pH increase might result from alkalizing reactions occurring in reducing conditions. The carbon concentration in soil had decreased slightly after the experiment in all columns (Table 2). This fact can result from leaching of soluble organic substances.

Ammonium concentrations in soil at the final stage were higher in the fertilized columns, particularly in treatment 8-8-16, compared to the initial stage and also to control columns, but the differences were not significant. These results were comparable to ammonium concentrations in leachates. Apparently, treatment 8-8-16 contributed more ammonium than treatment 11-18-11. With regard to nitrate, no significant differences were observed between treatments or with respect to the initial stage. Nitrogen contributed by fertilizers may have been leached, either as nitrate (although nitrate leaching was very similar from fertilized and unfertilized columns, Fig. 3) or as ammonium, or, more likely, lost through de-nitrification processes or immobilized in microbial biomass.

Unlike nitrogen, final available phosphorus concentrations in fertilized soil columns were notably higher than in the initial stage or in control columns, particularly in treatment 11-18-11, which provides more P (Table 2). These results are in agreement with the limited measured P leaching and may be related to the recognized low mobility

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of this element in soils, and particularly in acid soils (Gil-Sotres et al., 1982; Garcia-Rodeja and Gil-Sotres, 1997).

As for the exchange cations, the concentrations of calcium, magnesium and potassium increased in the fertilized columns. Calcium was significantly higher in treatment 11-18-11, while K and Mg were higher in treatment 8-8-16 (Table 2). The relative increases of Ca, Mg and K in both fertilized treatments were in agreement with their respective contributions (more K and Mg in 8-8-16, more Ca in 11-18-11). After a water flow equivalent to 13 years rainfall, and despite leaching losses, particularly of potassium, both fertilized soils were significantly enriched in these exchangeable cations. The remarkable decline of exchange aluminum in both fertilized treatments, compared to either initial stage or control columns, is related to the pH increase (Table 2) and the input of other cations with fertilizers.

The effective cation exchange capacity (eCEC) was very low both at the initial stage and in control soils at the final stage. In fertilized columns, the soil effective CEC at the final stage had significantly increased (Table 2), being moderately low (between 4 and 9 cmol(+) kg⁻¹), according to Buol et al. (1975). The increase of eCEC is related to the pH increase, given the variable-charge nature of the soils used in the experiment.

3.3 Nutrient balances during the experiment

The percentages of nutrients released from the tablets were calculated from the nutrient amounts contained initially in the fertilizer tablets and the amounts remaining at the end of the experiment (Table 3). Similarly, the percentages of leaching losses were calculated by comparing the accumulated leaching losses with the amounts of nutrients released (Table 3).

In general, the percentages of nutrients released at the end of the trial were very high, excepting Ca and Mg from tablet 8-8-16. However, the percentage of leaching loss was extremely low for nitrogen (< 4%). The nitrogen contributed by the tablets may be retained by soil in different ways: immobilized in microbial biomass, fixed by hydroxyaluminium vermiculites, etc. Part of the nitrogen may be lost from soil, either

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by leaching or through de-nitrification processes. De-nitrification is expected to play an important role in the reducing conditions prevailing during the experiment. Also Paramasivam and Alva (1997) reported low recovery of the applied N in the leachate (from 5 % to 28 %) in experiments with different urea-based controlled-release formulations (Meister, Osmocote, and Poly-S) added to soil columns, attributing it to the combination of loss of N through NH_3 volatilization, microbial assimilation of the applied N and de-nitrification processes. Phosphorus was leached at low rates (Table 3), as expected from its well-known low mobility and in agreement with the increases in soil available P. By contrast, potassium leaching was relatively high (more than 60 % of the total released). The potassium not leached can remain in the soil either as exchange cation or fixed by hydroxyaluminium vermiculites that are very common in these granitic acidic soils. Núñez-Delgado et al. (1997) also reported a nearly total P retention in soil and low NH_4^+ and K leaching in column experiments carried out with Galician soils after the addition of cattle slurry. In another study also using laboratory columns and different slow release fertilizers, but with a lower total water flow (21 L), Broschat and Moore (2007) obtained a P leaching between 47 and 80 %, lower than that of N and K (> 80 %). These percentages are clearly higher than those found in our study, probably because Broschat and Moore (2007) filled up their columns with washed sand, with much lower retention capacity for elements and compounds. Calcium and magnesium leaching, similarly to Ca and Mg release, were relatively high in treatment 11-18-11 and low in treatment 8-8-16 (Table 3). Besides the increase of exchange Ca and Mg (Table 2), immobilization by the microbial biomass and precipitation of insoluble Ca and Mg compounds may explain the differences in the percentage leached.

4 Conclusions

At the end of the trial, after the percolation of an amount of water equivalent to 13 years rainfall in the area, releases from fertilizer tablets were more than 80 % for most nutrients. Under the conditions of this study, Ca and Mg were usually released at lower

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rates, especially in the treatment 8-8-16 (less than 60%), while more than 99% of N was released from both tablets. Despite this, the amounts leached are generally low when compared with the total released. Most leaching occurred at the beginning of the experiment, within an interval of flow equivalent to 1.5 years rainfall. From that moment on, an increase of pH and a sharp decrease of nutrient concentrations were observed in leachates. The overall results indicate that most of the elements contained in the fertilizers remained in the soil or were lost by volatilization. At the end of the percolating study, the concentrations of available Ca, Mg, K and P had increased significantly in the soils into the fertilized columns, along with pH and effective CEC, showing at the same time a decrease of exchangeable Al. This means that, under the conditions of this study, the fertilizer treatments maintained their effects in these soils even after the passage of a water flow equivalent to 13 years rainfall. In these conditions, the formulation 8-8-16 underwent a lower overall nutrient loss, then being more suitable for crops having a nutrient demand sustained over time, also implying lower risks of water pollution, while the formulation 11-18-11 would be more suitable for crops with a strong initial demand.

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**Table 1.** Initial tablet weights (grams) and N, P, K, Mg and Ca amounts (grams) applied to each column with the treatments.

Treatment	Initial weight	N	P	K	Mg	Ca
8-8-16	38.03	5.04	1.74	4.49	2.89	1.27
11-18-11	30.83	5.29	2.68	2.50	0.29	1.79

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Table 2. Chemical characteristics of the soils used in the laboratory column trials.

	Initial stage	Final stage		
		Control	11-18-11	8-8-16
pH	4.13 ^a	4.92 ^b	5.70 ^b	6.19 ^c
C (g kg ⁻¹)	19.2 ^a	17.1 ^b	16.6 ^b	16.8 ^b
NH ₄ ⁺ (mg kg ⁻¹)	45.51 ^a	44.0 ^a	51.9 ^a	58.7 ^a
NO ₃ ⁻ (mg kg ⁻¹)	187.11 ^b	174.8 ^b	170.9 ^b	194.2 ^b
Available P (mg kg ⁻¹)	8.96 ^a	17.7 ^a	113.4 ^c	86.4 ^b
Exchange K (cmol ₍₊₎ kg ⁻¹)	0.24 ^{a,b}	0.11 ^a	0.31 ^b	1.03 ^c
Exchange Ca (cmol ₍₊₎ kg ⁻¹)	0.11 ^a	0.21 ^a	5.50 ^c	1.86 ^b
Exchange Mg (cmol ₍₊₎ kg ⁻¹)	0.13 ^a	0.11 ^a	0.33 ^a	4.24 ^b
Exchange Al (cmol ₍₊₎ kg ⁻¹)	0.92 ^b	0.80 ^b	0.08 ^a	0.01 ^a
Effective CEC (cmol ₍₊₎ kg ⁻¹)	1.40 ^a	1.27 ^a	6.23 ^b	7.20 ^b
Exchange Al (%)	66 ^b	63 ^b	1.3 ^a	0.1 ^a

^{a,b,c} Indicate significant differences ($p < 0.001$).



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**Table 3.** Percentages of nutrients released and leached from the tablets at the end of the experiment.

Treatment		N	P	K	Ca	Mg
11-18-11	Released, %	99.87	81.09	98.57	94.31	81.40
	Leached, %	2.82	28.76	70.61	53.53	78.11
8-8-16	Released, %	99.78	99.99	98.95	18.51	58.37
	Leached, %	3.86	8.76	63.96	19.61	18.85

Released: amount lost from the fertilizer tablet referred to the initial amount.

Leached: accumulated leaching loss referred to nutrient release.

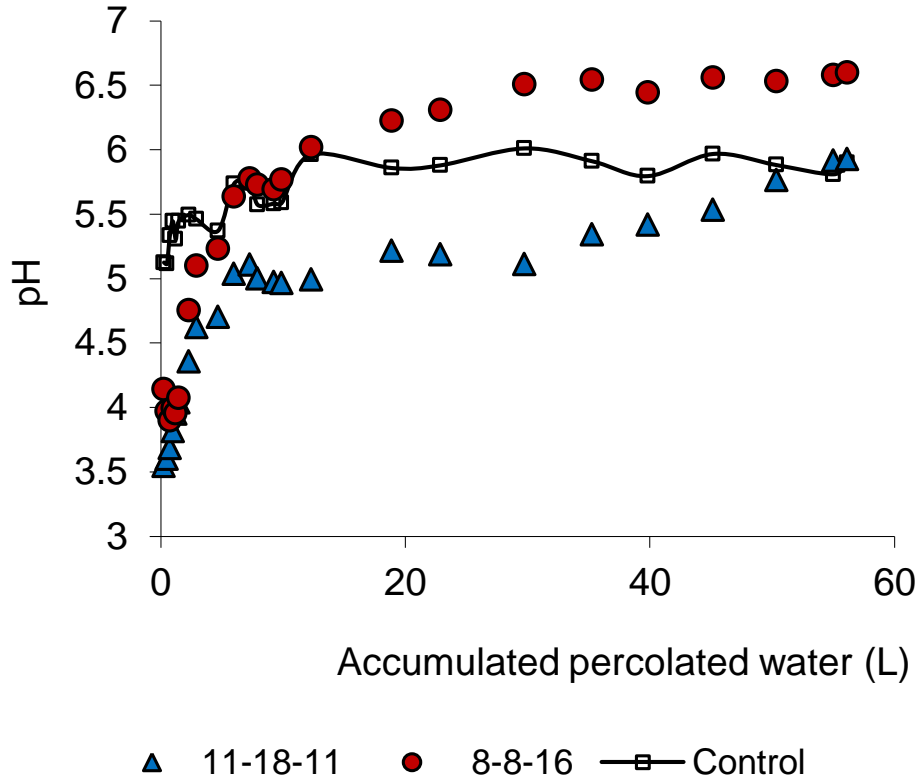


Figure 1. pH of leachates from fertilized and control columns as a function of the volume of percolated water.

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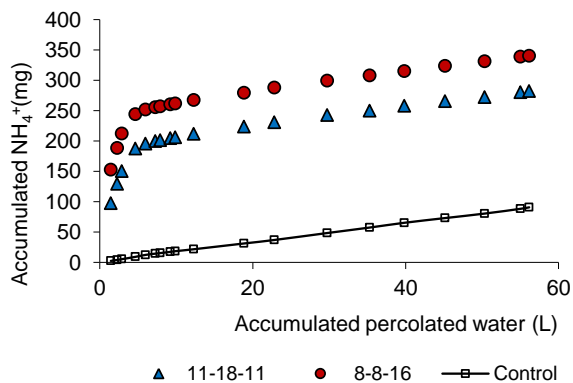
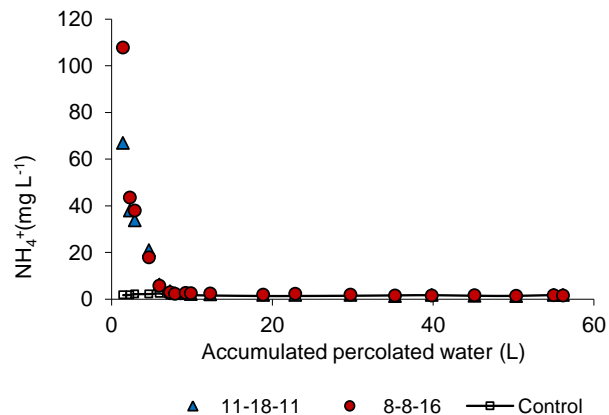


Figure 2. Ammonium concentrations in leachates and accumulated NH₄⁺ losses from fertilized and control columns along the experiment.

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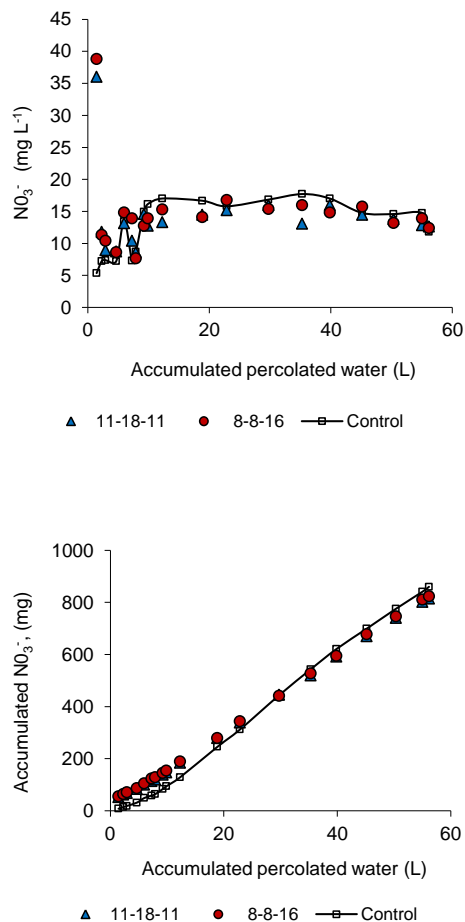


Figure 3. Nitrate concentrations in leachates and accumulated NO_3^- losses from fertilized and control columns along the experiment.

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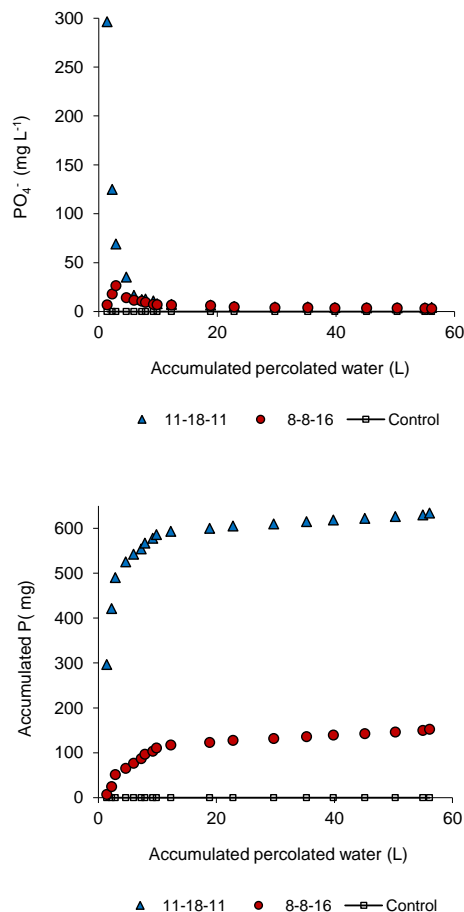


Figure 4. Phosphorus concentrations in leachates and accumulated P losses from fertilized and control columns along the experiment.

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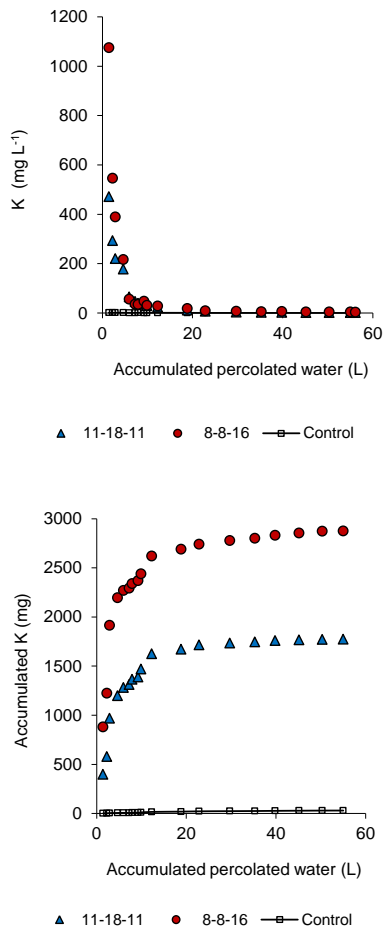


Figure 5. Potassium concentrations in leachates and accumulated K losses from fertilized and control columns along the experiment.

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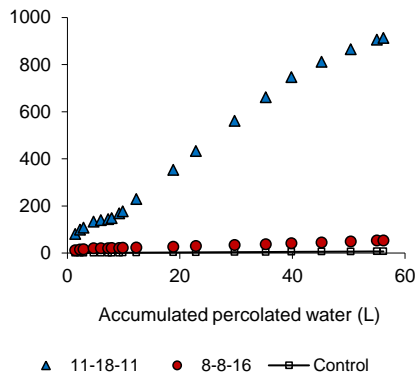
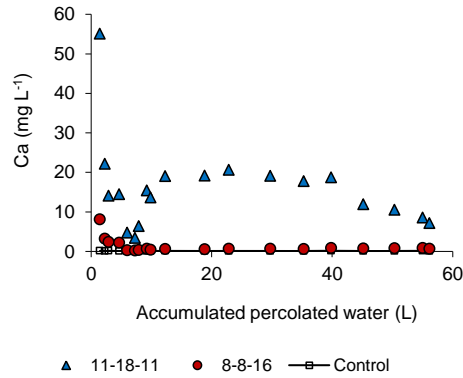


Figure 6. Calcium concentrations in leachates and accumulated Ca losses from fertilized and control columns along the experiment.

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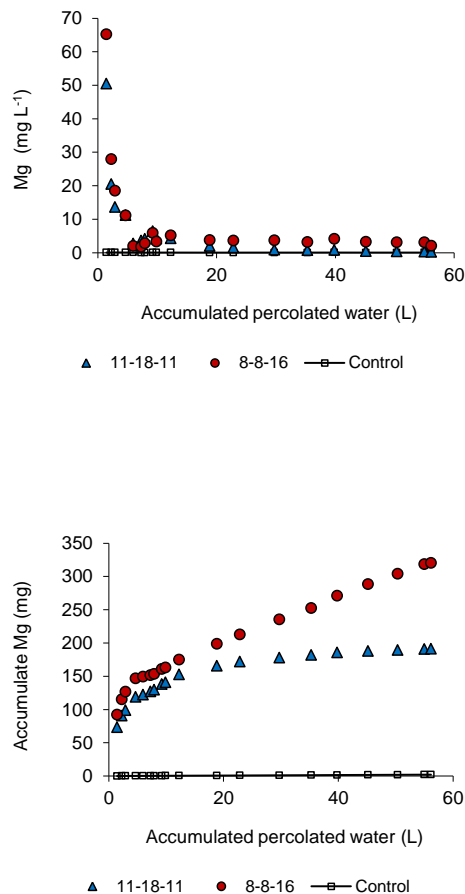


Figure 7. Magnesium concentrations in leachates and accumulated Mg losses from fertilized and control columns along the experiment.

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