Distribution of nutrients from controlled-release polymers in sandy soil[1]

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ABSTRACT - Techniques such as fertilization can be used more frequently to achieve higher crop yields. The problem of excessive use of fertilizers is related to losses of nutrients by leaching contaminating water bodies. Adopting controlled-release fertilizers may favor the reduction of losses and the total cost of fertilization as the number of applications is reduced. This research aimed to study the dynamics of nutrients from polymer-coated fertilizers (KNO_3 controlled-release polymers) in sandy soil and analyze the spatial variation of soil solution storage at different depths when drip irrigation is adopted. Four soil boxes filled with Quartzipsamment and equipped with a mesh of 19 TDR probes were used to monitor soil moisture and electrical conductivity. Three soil boxes received the treatment of 50 g of the controlled-release polymer, and one soil box was used as a control. One liter of water was applied weekly through a drip irrigation system for ten weeks, followed by readings from the TDR probes to monitor the spatial variation of the soil solution storage in the soil profile. This monitoring allowed verifying that the release of nutrients happened slowly and gradually over the period without water retention by the material. The solution was displaced by advection (mass flow) and dispersion along with the irrigation water, which advanced more than the nutrients within the profile, reducing their leaching. Thus, controlled-release polymers maintained a slow and variable release over time.

Key words: Soil electrical conductivity. Drip irrigation. Potassium nitrate. TDR.

INTRODUCTION

Food security is defined by the Food and Agriculture Organization of the United Nations as the possibility of access to safe and nutritious food by anyone to make a healthy life possible (FAO; WWC, 2015). This agency states that food security can be achieved even in 2050 through investments in sustainable management techniques in producing crops, livestock, and fish on a large or small scale. It will enable the economy to reach the pillars of food security: availability, access, stability, and use of food.

Fertilizers have been used to increase yield since the Green Revolution. When performed consciously, the supply of external nutrients can increase by up to 50% of the total yield (CHEN *et al.*, 2018).

The biggest problem occurs when fertilizers are misused, with doses that go far beyond what the plant needs for its development. In this case, the production cost is increased due to a loss of efficiency of the applied product, and the environment is threatened due to the possibility of the degradation of water bodies through eutrophication (CHEN *et al.*, 2018).

Using controlled-release fertilizers can reduce nutrient losses due to leaching or volatilization and reduce environmental impacts. According to González *et al.* (2015), these products consist of coated fertilizers with polymeric materials of inorganic or organic origin, such as waxes, cellulose, or lignin. They have a certain amount of nutrients inside and release them continuously and gradually as the plant develops.

The mechanism of action of these fertilizers can mainly occur by diffusion, through the passage of water through the material that covers the granules (CHIAREGATO; FAEZ, 2021). According to Trinh and Kushaari (2016), water has an intimate relationship with the release because it is through it that the release begins; that is, the nutrient is released as the water enters the granule.

The materials that cover the granules can have the most diverse origins and ways of coating or encapsulating the nutrients (YANG *et al.*, 2018). Qiao *et al.* (2016) highlighted the importance of this coating material being biodegradable, as it will remain in the soil longer and cannot become a residue. Chitosan (CS) has stood out among biodegradable materials for being a polymer of natural origin and being quickly processed to form controlled-release materials. Biopolymers are considered low-cost materials (MENEGATTI *et al.*, 2017) and have the same degree of efficiency in releasing nutrients compared to synthetic polymers, showing advantages such as ease of environmental degradation (PEREZ; FRANCOIS, 2016).

The search for criteria that can help farmers properly use this fertilizer application concept in the field, especially in sandy soils, is essential. Moreover, the lack of information on the relationship dynamics between water-soil-solute has promoted discouragement of using controlled-release fertilizers for not extracting the potentialities related to the concept (SANTOS *et al.*, 2015; SOUZA *et al.*, 2017). In this context, this research aimed to study the dynamics of nutrients from a controlled-release polymer applied in sandy soil and analyze the spatial variation of soil solution storage at different depths when adopting drip irrigation.

MATERIAL AND METHODS

Characterization of the experimental area

The experiment was conducted in an agricultural greenhouse with 115 m² belonging to the Agricultural Sciences Center (CCA) of the Federal University of São Carlos (UFSCar). The CCA is located in the municipality of Araras, SP, at the geographic coordinates 22°18′ S and 47°23′ W. The average altitude of the experimental area is 698 m, with the climate characterized as Cwa, according to Köppen. The region has a predominant mesothermal climate with dry winters and hot and humid summers.

The soil used in the test was a Quartzipsamment collected in the municipality of Leme, SP, at 0 – 30 cm depth. The soil was analyzed at the Laboratories of Soil Physics and Fertility of UFSCar to determine physical and chemical attributes (Table 1).

Preparation of containers and installation of the irrigation system

The soil was air-dried and passed through a 2-mm opening sieve to obtain the air-dried fine earth, according to the methodology used by Bizari, Grecco, and Souza (2016). Subsequently, the soil was placed in four boxes of PVC with dimensions of 0.65 m in height and 1.10 m in diameter, totaling 500 L each. The soil in the boxes was slightly compacted with light taps on the surface and around the box to approximate the soil density to the field conditions.

Concomitantly, TDR probes were installed in each box to form a mesh with a spacing of 0.10 m from each other (vertically and horizontally) (Figure 1).

The TDR probes consisted of three 0.20 m stainless steel rods connected through a coaxial cable to a Campbell Scientific TDR 100 reflectometer, which collected moisture and electrical conductivity readings from the soil simultaneously through a multiplexer controlled by a datalogger.

Table 1 - Soil physical and chemical characterization at a depth of 0 – 0.20 m

Soil characteristic	Units	Value
Physical attributes		
Sand	%	93.5
Silt	%	1.4
Clay	%	5.1
Field capacity	$m^3 \, m^{-3}$	0.26
Porosity	$m^3 \, m^{-3}$	0.38
Soil density	$kg \, m^{-3}$	1670
Particle density	$kg \, m^{-3}$	2710
Infiltration capacity	$m \, h^{-1}$	0.41
Chemical attributes		
pH (CaCl$_2$)	–	4.90
Phosphorus	$mg \, dm^{-3}$	3.00
Organic matter	%	4
Potential acidity	$mmol_c \, dm^{-3}$	33.00
Potassium	$mmol_c \, dm^{-3}$	0.60
Calcium	$mmol_c \, dm^{-3}$	9.00
Magnesium	$mmol_c \, dm^{-3}$	3.00
Sum of bases	$mmol_c \, dm^{-3}$	13.20
Cation exchange capacity	$mmol_c \, dm^{-3}$	27.20
Base saturation	%	49
Sulfur	$mg \, dm^{-3}$	28.00
Boron	$mg \, dm^{-3}$	0.17
Copper	$mg \, dm^{-3}$	0.40
Iron	$mg \, dm^{-3}$	6.00
Manganese	$mg \, dm^{-3}$	1.60
Zinc	$mg \, dm^{-3}$	0.30

The probes provided the soil apparent dielectric constant (Ka), which was converted into volumetric moisture (Θ) by Equation (1). Similarly, the probes provided the soil apparent electrical conductivity, being converted into saturated soil-paste electrical conductivity by Equation (2) (BIZARI *et al.*, 2014; FRANÇA *et al.*, 2018).

$$\theta = -0.0007 K\alpha^2 + 0.036 K\alpha - 0.0403 \qquad (1)$$

Where Θ is the volumetric moisture ($m^3 \, m^{-3}$) and Ka is the apparent dielectric constant (dimensionless).

$$EC_{Paste} = 1.14720 + 1.5191 EC_{TDR} + 0.041 \qquad (2)$$

Where EC_{Paste} is the saturated soil-paste electrical conductivity (dS m^{-1}) and EC_{TDR} is the apparent electrical conductivity (dS m^{-1}).

The soil boxes were positioned inside the agricultural greenhouse. One of the boxes was used as a control and did not receive the treatment. The other boxes were replications of the treatment and received the controlled-release polymer.

The controlled-release polymer was based on chitosan, montmorillonite clay (MMt), and potassium nitrate (KNO$_3$, 47%) in microparticles. Briefly, KNO$_3$ was added to MMt in clay to the fertilizer ratio of 1:3 (mass mass^{-1}) by a mechanical-chemical process in a mortar, followed by drying (MESSA *et al.*, 2016). The MMt/KNO$_3$ mixture (32 g) was added to the CS solution (2 wt.% acetic acid) and stirred for 1 h in a Turrax stirrer (12,000 rpm) to ensure homogeneity (MESSA; SOUZA; FAEZ, 2020). Sodium tripolyphosphate was added to this mixture for the crosslinking of the chitosan (KAŠPAR; JAKUBEC; ŠTĚPÁNEK, 2013). Finally, the dispersion of CS/MMt/KNO$_3$ was dried in a mini spray-dryer (B-290, BUCHI) with a double fluid

atomizer nozzle (1.5 mm) at 180 °C inlet temperature, 40% flow rate of the peristaltic pump (7 mL min^{-1}), 36 m^3 h^{-1} aspiration rate, and 0.41 bar airflow.

Each soil box (except for the control) received 50 g of controlled-release polymer containing 47% KNO$_3$ in a mixture with sandy soil to increase the contact surface of the fertilizer with the soil particles. The material was introduced using a tube with dimensions of 0.05 m in diameter and 0.1 m in height, positioning the polymer up to 0.1 m deep, and the tube was removed from the soil after material addition (Figure 2).

A surface drip irrigation system was installed in the soil box (Figure 1C). It consisted of self-compensating drippers with a flow rate of 4 L h^{-1}, microtubes, and stakes for directing the water flow to assist in the application of water directly in the center of the box and maintain adequate moisture close to the concentration zone of the controlled-release polymer.

The irrigation system was evaluated based on a uniformity test with the application of Christiansen's uniformity coefficient (CUC) for all drippers. The final value was 98%, which is considered excellent according to Bernardo, Soares, and Mantovani (2009).

An application volume of 1 L of water was applied weekly based on previous tests to achieve the highest wetted soil volume (wet bulb) without hitting the soil box ends, totaling a value of 10 L of water at the end of the experiment (BIZARI *et al.*, 2014). Readings of soil moisture and electrical conductivity parameters were always obtained on the first and

Figure 1 - (A) Layout of the TDR probes inside the soil boxes, (B) soil volume monitored by a single probe (adapted from Souza, Favaro, and Folegatti (2007)), and (C) installation of the irrigation system with detail for the line positioning
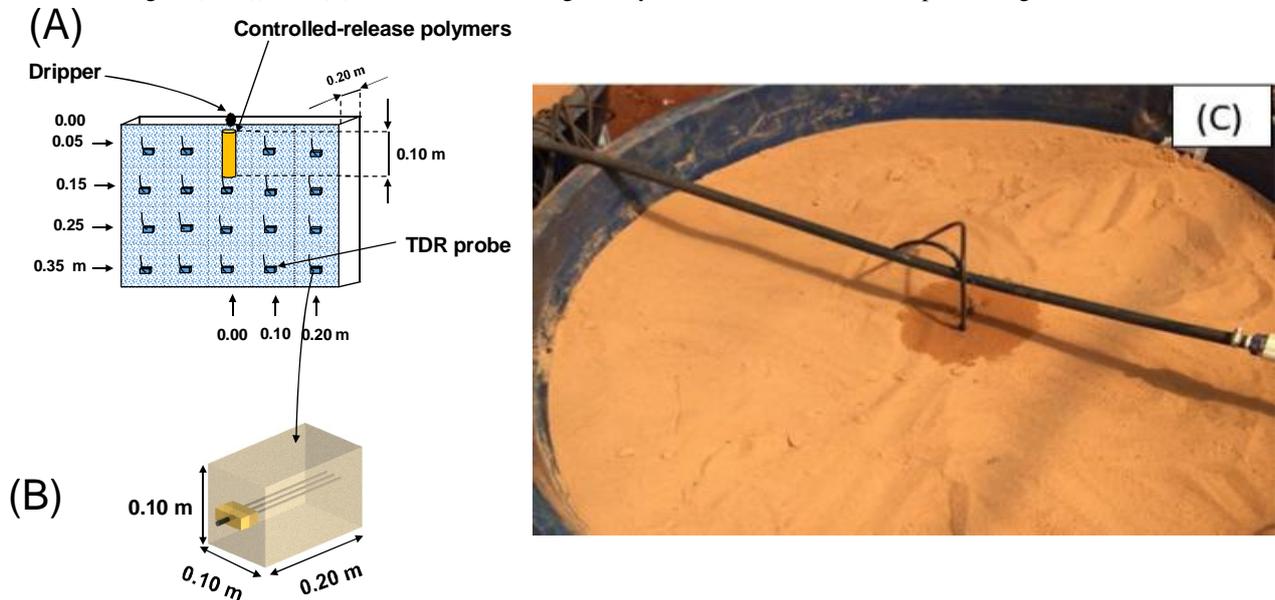
Figure 2 - (A) Controlled-release polymer used in the test, illustrating the laboratory preparation of the samples inserted into each container, (B) detail of the polymers mixed with soil, and (C and D) method of inserting the polymers into the containers

fourth days after irrigation. The readings allowed composing the distribution of water and solutes inside the soil boxes.

Means were calculated between the soil moisture estimates and electrical conductivity registered in the three soil boxes. These mean values were analyzed by a 3D surface mapping program, which presented the nutrient distribution profile as a function of soil moisture and electrical conductivity, providing essential information on the dynamics of prolonged release of fertilizers. The same analysis was applied to the control soil box.

The storage of nutrients was also compared in the different soil layers inside the box (0 – 0.10, 0.10 – 0.20, 0.20 – 0.30, and 0.30 – 0.40 m). Thus, an analysis of the storage variation over water applications was performed.

RESULTS AND DISCUSSION

Soil water retention

The possibility of increasing soil water retention due to the use of polymer was initially evaluated considering the polymer release mechanism and the fact that the material studied in the experiment has a chitosan and montmorillonite clay-based coating. Some studies with controlled-release fertilizers have reported an increase in soil water retention due to the dissolution of the material and later diffusion into the medium (TRINH; KUSHAARI, 2016).

Azeem *et al*. (2014) advocate using coated urea to confer the controlled release characteristic and improve the issue of weak water retention in soils from regions with water limitations. Narayanan *et al*. (2018) prepared a superabsorbent polymer with chitosan, citric acid, and urea in its composition and found a maximum water absorption of about 1250 g g^{-1}. Wu and Liu (2008) also developed a controlled-release fertilizer with chitosan in its coating layer. They observed that water absorption capacity increased by 10% compared to soils that did not receive the material.

In contrast, KNO_3 controlled-release microparticles with chitosan and clay as a coating layer showed reduced swelling capacity ($307.8 \pm 16.1\%$) compared to chitosan microparticles ($1523.3 \pm 238.2\%$) and, mainly, after polymer crosslinking ($159.1 \pm 19.1\%$) (MESSA; SOUZA; FAEZ, 2020). However, in contrast to what was found in the literature and starting from a linear regression between the mean soil moisture found in the soil boxes with and without polymers, soil moisture in both cases had the same behavior (Figure 3A). The visual comparison (Figure 3B) shows the wet bulb in the soil for the soil box without and with polymers after the first, sixth, and tenth water applications.

Figure 3 - (A) Linear regression of the means of moisture for the control and polymer containers and (B) comparison between the wet bulbs formed for the different treatments (with and without polymer) in the soil boxes
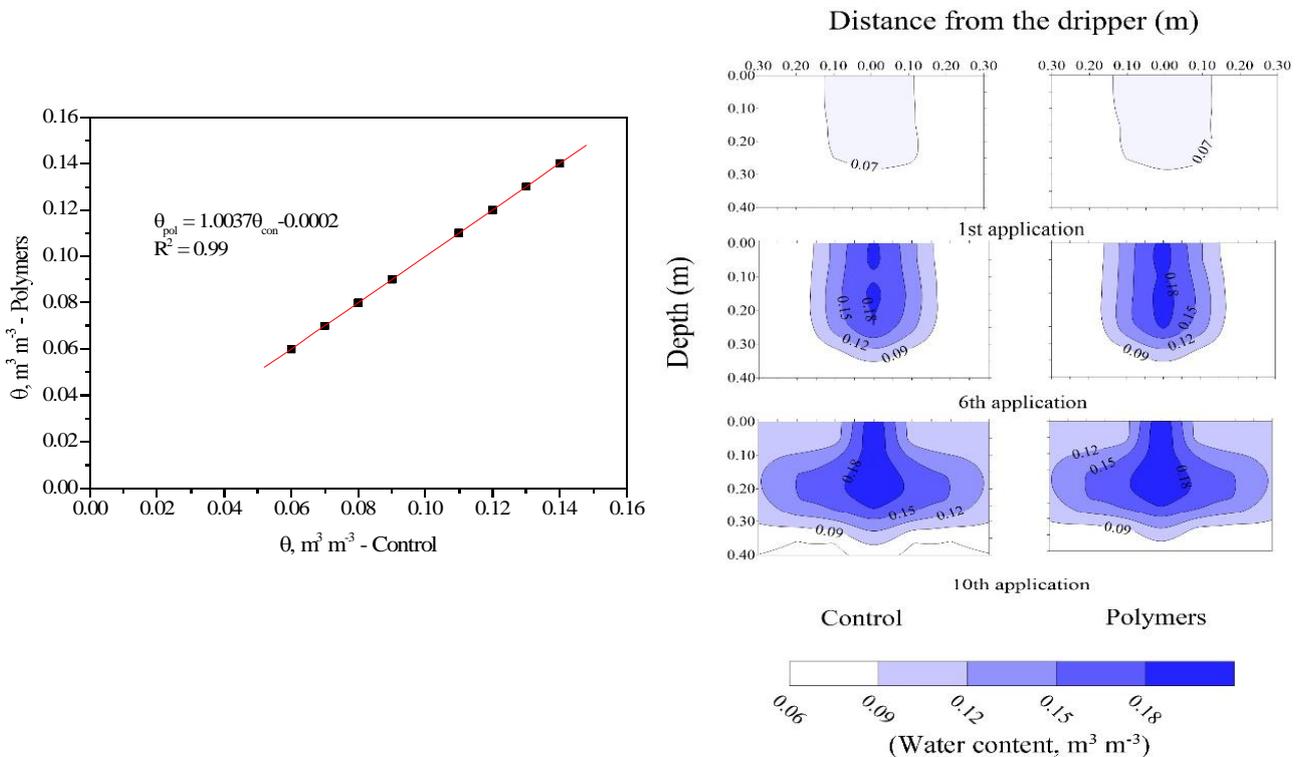
Corroborating with the result of the linear regression with an $R^2 = 0.99$, the bulbs for both cases showed the same conformation and the exact dimensions at the beginning, during the most intense release phase, and at the end of the experiment, with a coincidence between the region of polymers and the location of water source (Figure 3B). The material has a low swelling capacity and, therefore, could not provide a change in the soil water retention characteristic.

Release of nutrients in the soil

Figure 4 shows the monitoring of soil electrical conductivity (EC) together with soil moisture estimates. The ten EC profiles show the dynamics of the release of nutrients via polymer during weekly water applications. The orange isolines are spaced at 0.5 dS m^{-1} and demonstrate the variation in the arrangement of the ions inside the soil box. The EC measurements in the control soil box were spatial and temporal, observing the values of 0.09 dS m^{-1}.

The first point to be analyzed is the beginning of the nutrient release around the experiment's fourth week. The soil showed an electrical conductivity of 0.20 dS m^{-1} during the first three weeks, with no release. The release started subtly from the fourth week, increasing considerably between the sixth and seventh applications, marked by a variation in concentration in the region where the material is found. The nutrient release was facilitated due to the water added to the system, as the mechanism occurs from the entry of water through the polymer coating wall, nutrient dissolution inside, and its release in ionic form by the pressure difference between the internal and external environment of the material, as suggested by Trinh and Kushaari (2016).

The movement of ions inside a wet bulb occurs by mass diffusion and transport processes, related to differences in the concentration gradient and drag due to water flow through a laminar movement, respectively (SILVA, 2014; VENÂNCIO *et al.*, 2017). Silva (2014) explained that the direction of ions in the soil is governed by the interaction between the soil-water-plant systems and that the displacement considers the percolation intensity and fixation and adsorption reactions in the medium.

The controlled-release polymer positioned at 0.10 m from the soil surface led to a displacement of ions by the box extension up to 0.25 m deep with a 0.20 m lateral radius, reaching a difference of 1.5 to 3.5 dS m^{-1} between the polymer concentration regions and close to the bulb wetting front. It showed that the fertilizer was concentrated in the upper soil layers, even in the sandy soil.

This characteristic shows the efficiency of using the polymer of controlled-release fertilizers in soils with low cation exchange capacity, as in sandy soil (SANTANA *et al.*, 2015). This soil type has a considerable loss of nutrients through leaching because

there is less binding energy between the nutrients and soil colloids (DA ROS *et al.*, 2017).

The addition of nutrients via controlled-release material led to a decrease in the concentration of nutrients disposed of in the soil, which may be susceptible to leaching. As previously mentioned, the nutrients did not exceed the 0.25-m depth, which may favor crops with surface roots. Li *et al.* (2017) found advantages in tomato quality when working with slow-release fertilizers as a nitrogen source. The authors observed that this fertilizer class not only reduces leachate losses to deeper layers but also assists in the delay of root absorption, ensuring a more efficient conversion of nitrogen by plants.

Figure 4 - Evolution of the positioning of nutrients in the soil measured through electrical conductivity (dS m^{-1}) at each 1.0-L application
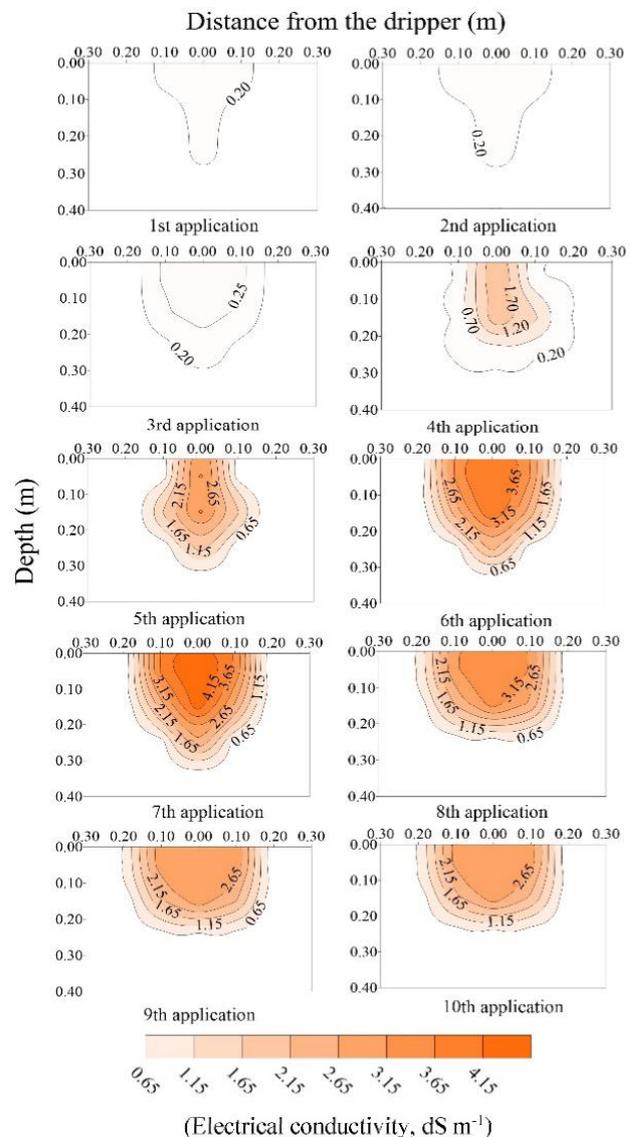
Bley *et al*. (2017) found that potassium losses in this type of soil using conventional KCl sources are 5 to 7%. These losses decrease to 1 to 2% using slow-release fertilizers with coated KCl.

Considering that the salt applied was KNO_3 and, although both nutrients (nitrate and potassium) have mobility in the soil, the movement of nutrients inside the wet bulb differed. Thus, nitrogen is assumed to be positioned close to the wetting front region of the bulb. At the same time, potassium is concentrated more inside and in the surface layers, close to the polymer. This assumption is based on studies like Donagemma *et al*. (2008), who found lower K^+ levels in-depth than NH_4^+ and NO_3^-. In this study, the cation K^+ still had a higher content in the surface ring in all treatments, indicating a lower mobility rate.

Donagemma *et al*. (2008) and Basso, Soto, and Chang (2017) consider the hydrated radius and valence of particles to explain the differences in the dispersion of ions in the soil. Potassium has a smaller ionic radius than ammonium, allowing higher attraction to soil colloids. In this sense, potassium is the cation that is most liable to leach in soils with low CEC and better drained, as in the case of sandy soils (MENDES *et al*., 2015). Regarding valence, opposite charges repel each other, which explains nitrate mobility in the soil solution, as its negative charge will be repelled by its CEC.

The comparison between the wet bulb and the soil electrical conductivity showed that the most distant region occupied by nutrients did not exceed 0.25 m in depth, while the wetting front for the wet bulb reached 0.35 m in depth. It shows that the nutrients moved through advective-dispersive transport but always in a retracted manner compared to water, which favors a reduction of leaching in the soil profile. Moreover, considering the concepts of solute transport and the nutrients potassium and nitrogen, Mendes *et al*. (2015, 2016) and Venâncio *et al*. (2017) explained that potassium can be transported radially via diffusion or advection and vertically via advection. Nitrogen in the form of nitrate is dissolved in the soil solution and moves via advection.

Fertilizer concentration decreased and remained constant at 0.65 dS m^{-1} at the end of the formed profile after the eighth week. The concentration of fertilizer along the soil profile varied from 1.70 to 4.15 dS m^{-1} and decreased to 2.65 dS m^{-1} during the ten weeks, which indicates a characteristic prolonged polymer release.

The data corroborates with the bulbs generated by the 2D mapping software (Figure 5). The controlled-release polymer presented values between 5.2 and 102 mmol L^{-1} and had a variable performance regarding the release of nutrients during the experiment.

Figure 5 - Variation of the KNO_3 concentration in the different soil layers throughout the applications
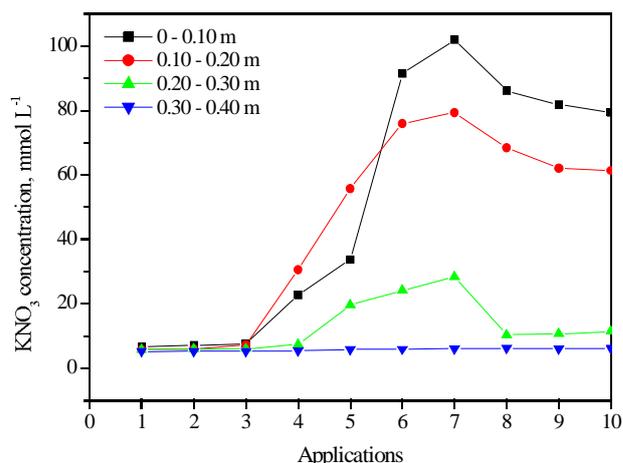
In general, the nutrient release presented three main phases: (i) initial phase, marked by an increase in the concentration of salts in the 0 – 0.10 and 0.10 – 0.20 m layers after the fourth application; (ii) release intensification in the sixth and seventh applications, marked by concentration peaks (Figure 5); and (iii) decreased release in the final phase of the experiment, between the eighth and tenth applications. The release of nutrients in controlled-release systems depends on the coating material type (YANG *et al*., 2018). SOUZA *et al*. (2017) also found a difference when working with a controlled-release fertilizer coated with chitosan in lettuce. The authors observed a peak of release at the beginning of the experiment, which declined in a short time (1 week), followed by a gradual release.

Considering the polymer location (0.10 m deep), most surface layers (0 – 0.10 and 0.10 – 0.20 m) maintained a higher concentration than the 0.20 – 0.30 m layer even though it received a certain amount of nutrients during the seventh application. The deepest layer (0.30 – 0.40 m) remained constant during the ten applications, expressing a much lower concentration than the other layers. Additionally, the percentage of variation in concentration can be related between one layer and another (Table 2). The first three weeks showed no significant change in the nutrient concentration in the soil, contrary to what was observed in the following weeks.

Table 2 shows that the highest nutrient storage rate was 50.9% in the ninth application's 0 – 0.10 m layer, while the lowest was 2.7% in the 0.30 – 0.40 m layer in the seventh application. This interesting second point shows that the nutrients did not reach the deepest layer even at the release peak.

Table 2 - Variation in the spatial distribution of nutrient storage between layers of the soil profile

Application	Layer				Total (mmol L⁻¹)
	0 – 0.10	0.10 – 0.20	0.20 – 0.30	0.30 – 0.40	
	Nutrient distribution (%)				
1	28.3	24.7	24.8	22.2	20.12
2	29.1	24.8	24.6	21.5	20.92
3	29.1	27.7	23.1	20.0	22.61
4	34.9	46.5	10.7	7.9	59.39
5	30.0	48.0	17.2	4.8	103.93
6	46.2	38.4	12.5	2.9	177.13
7	47.2	36.8	13.4	2.7	193.57
8	50.3	40.3	5.9	3.5	151.78
9	50.9	38.9	6.5	3.7	142.16
10	50.2	39.0	7.0	3.8	139.84

These variation results, together with the generated wet bulbs, showed the polymer efficiency not only in continuing to release nutrients even after ten weeks of the experiment but also in a lower concentration than that observed at the starting region, indicating a lower incidence of leaching.

This reduction in nutrient loss, especially nitrogen due to its high mobility, could be confirmed by Zhang *et al.* (2018) in a study with conventional and controlled-release urea in rice. The authors observed higher nitrogen concentrations in nitrate and ammonium in solutions subjected to conventional urea, showing leaching when compared to the concentrations observed in the solution treated with controlled-release fertilizers.

CONCLUSION

Controlled-release polymers did not retain water but maintained a slow and variable release over time. The nutrients from them were displaced *via* advective-dispersive transport inside the wet bulb formed by the drip irrigation. The spatial variation in the distribution of nutrients occurred mainly on the upper layers, retaining a higher amount of salts than the lower layers.

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