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IN SITU MONITORING OF A CONTROLLED RELEASE OF FERTILIZERS IN LETTUCE CROP

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ABSTRACT: Agriculture has sought ways to increase food production to meet global demands, intensifying dependence on natural resources. An alternative for intensive production has relied on the use of "smart" fertilizers that release nutrients in a controlled manner. Biodegradable polymers containing fertilizers has presented economic and environmental advantages when applied to the soil, as they release water and nutrients gradually to the environment without leaving residues. The objective of this study was to monitor the behavior of chitosan-clay hybrid microspheres, used as potassium nitrate soil ferti-releaser in lettuce cultivation. The experiment consisted of two treatments: (i) application of ferti-releasing chitosan-clay microspheres and (ii) use of conventional fertilization. Both treatments were monitored with TDR probes to measure electrical conductivity and soil moisture. The results indicate that both moisture and electrical conductivity are higher in soils treated with the ferti-releasing material. In addition, there was a gradual and homogenous release if compared to the conventional fertilization. The statistical analysis showed that the microspheres were efficient mainly for the controlled nitrogen release.

KEYWORDS: ferti-releasing biopolymer, clay, moisture, electrical conductivity

INTRODUCTION

Food production has increased following population growth due to increased food demand. However, one of the great challenges is to seek alternatives to food production by optimizing the use of agricultural inputs, making economic gains for the farmer, and reducing the activity's environmental impact.

In this context, irrigation combined with fertilization has helped producers to increase crop productivity within the same production area. However, the major problem related to the use of fertilizers is in the processes of losses such as leaching and volatilization, since the producer needs to know the best irrigation system to be used, as well as the process of splitting these inputs.

One of the alternatives available in the market is the use of hydrogels in agriculture, which are polymers capable of absorbing large amounts of water and retaining it for a certain period, in order to reduce irrigation frequency, as they contribute to increasing water retention capacity of the soil (Venturoli & Venturoli, 2011). However, some hydrogel types without polymer biodegradability characteristics have left residues in the soil, causing salinization of the medium with their increasing use (Mendonça et al., 2013).

Thus, the use of biodegradable polymers has been studied in this area. Among a wide class of biodegradable polymers, chitosan is a natural, inexpensive product with the ability to form films, fibers, gels, and microspheres. The existence of amine (NH_2) and hydroxyl (OH) groups in its chemical structure contribute to the interaction with several molecules and ions. It is soluble in an acid medium, so it can form a cationic polymer through the protonation of amine groups (NH_3+), giving interesting properties such as biocompatibility, biodegradability, and non-toxicity (Kumar, 2000; Messa et al., 2016). Due to its great compatibility with other materials, chitosan can be used from the pharmaceutical industry in the manufacturing of medicines, to the food industry in the protection against pathogens.

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Chitosan is very promising for fertilizer management in agriculture due to its biodegradability, and the study and adaptation of this polymer may contribute to the preparation of "smart" fertilizers. According to Santos et al. (2015), slow release fertilizers provide nutrients to plants gradually, thus requiring less frequency of inputs application, reducing labor costs for fertilizer installment, avoiding injuries to plant seeds and roots resulting from excessive applications, and being less susceptible to losses, which minimizes the risk of environmental pollution.

Electrical conductivity (EC) is an effective parameter to follow the release of these nutrients in the soil and, according to Queiroz et al. (2009), EC correlates with the total concentration of dissolved electrolytes (ions) in solution, since only pure water is a poor electric current conductor. There are laboratory methods for this, such as the conductivity meter, as well as indirect methods such as TDR (time-domain reflectometry) which stands out for estimating the medium electrical conductivity, allowing real time readings without deforming the sample (Pavão et al., 2014).

TDR has been seen as a useful tool for evaluating whether there is the release of encapsulated fertilizers (Santos et al., 2015). Messa et al. (2016) successfully used the TDR technique to determine the actual nutrient-release profile using chitosan-clay hybrids for the release of fertilizer potassium nitrate. However, literature presents little information for the analysis of these materials, since when subjected to evaluation, the samples need to be deformed, using more complex and time-consuming techniques such as aliquot removal and sample preparation for chemical analysis.

TDR is an electromagnetic technique for precise measurements of moisture and electrical conductivity in the soil. This tool measures soil physical parameters, enabling an in situ monitoring of fertilizer release into the soil, both in the laboratory and in the field. It has as advantages being precise, non-destructive, non-radiation use, ease to automate and coupling to multiplier reading devices, little influenced by soil texture, density, and salinity (Pavão et al., 2014; Souza et al., 2016).

In this context, the objective of this study was to monitor the behavior of chitosan-clay hybrid microspheres as nutrient ferti-releaser in lettuce cultivation, as a way of evaluating the controlled release of fertilizers in the soil for crop development.

MATERIAL AND METHODS

The experiment was carried out in a Quonset greenhouse (6.40 m wide x 20 m long, and 5 m high), covered with transparent polyethylene film (150 microns). This structure is installed at an experimental area belonging to the Agrarian Sciences Center, Federal University of São Carlos (UFSCar), in the city of Araras, São Paulo state (Brazil). It lies on the geographical coordinates of 22° 18' S and 47° 23' W, and at an average altitude of 600 m.

The climate of the region, according to the Köppen system, is of the Cwa type, mesothermic, with hot and humid summers and dry winters. The annual climatic conditions are as follows: average rainfall of 1414 mm; average temperature of 21.1 °C; evaporation of Class "A" tank, an average of 1443 mm, average wind speed of 1.44 m s^{-1}; relative humidity of about 70%, and average insolation of 2573 h.

The predominant soil in the experimental area is dystrophic Red Latosol, according to the Brazilian Soils Classification System (EMBRAPA, 2013). The analyses to characterize the soil physical and chemical attributes (Table 1) were performed in the Soil Fertility Laboratory and in the Soil Physics Laboratory, both belonging to UFSCar, according to the methods described by EMBRAPA (2011).

TABLE 1. Physical and chemical soil characteristics at a depth range 0-0.20 m.

Attributes	Content
Sand (%)	15
Silt (%)	14
Clay (%)	71
Field capacity (m^3 m^{-3})	0.33
Permanent wilting point (m^3 m^{-3})	0.17
Porosity (m^3 m^{-3})	0.50
Soil density (kg m^{-3})	1300
Particle density (kg m^{-3})	2580
pH in water	4.60
Phosphorus (mg dm^{-3})	10.00
Organic matter (%)	18
Potential acidity (mmol dm^{-3})	50.00
Potassium (mmol dm^{-3})	2.90
Calcium (mmol dm^{-3})	14.00
Magnesium (mmol dm^{-3})	6.00
Sum of bases (mmol dm^{-3})	23.10
Cation exchange capacity (mmol dm^{-3})	73.10
Base Saturation (%)	32

Eight seedbeds were built in the experimental area, with 0.30 m depth (Figure 1). The ferti-releasing microspheres of potassium nitrate (KNO_3) were applied in four intercropped beds, and the other four were fertilized with KNO_3 in its conventional form, with the cover mineral fertilization divided in 2 applications, 10 and 20 days after sowing (Figure 2). The following treatments were liming (dolomitic limestone), fertilization of planting and cover, according to the recommendation of Bulletin IAC 100, Raij et al. (1997). It is important to emphasize that the fertilization of planting and cover was applied in a single dose at planting for the treatment with ferti-liberating microspheres.

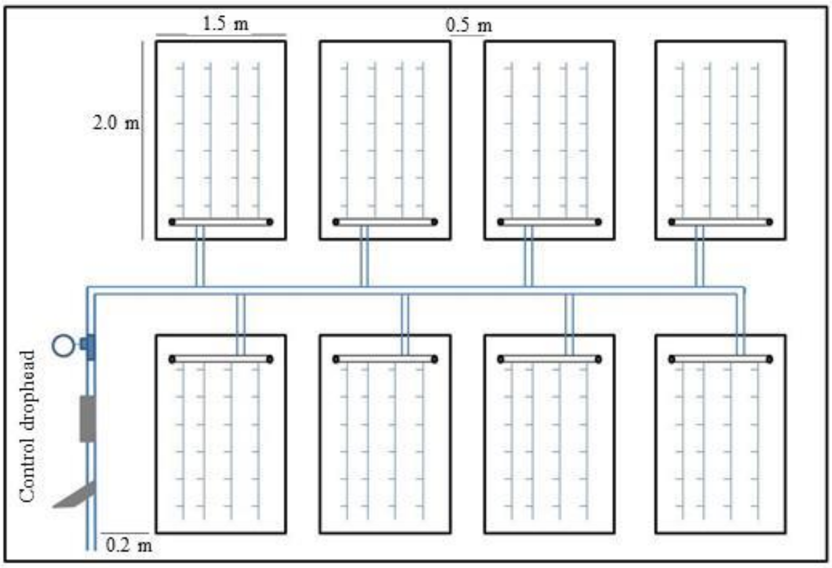

FIGURE 1. Distribution of seedbeds and drip irrigation system inside the greenhouse.

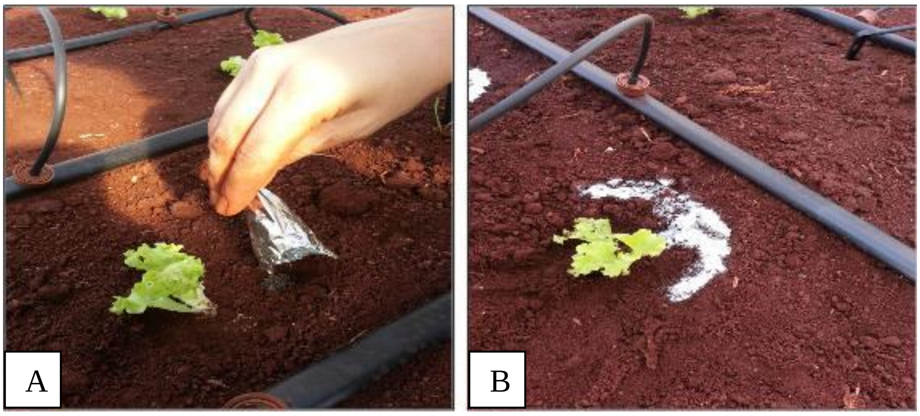

FIGURE 2. A - Treatment 1: KNO_3 ferti-releasing microspheres; B-Treatment 2: KNO_3 in the conventional form.

Lettuce seedlings of the "Vanda" variety were used. The microspheres were prepared according to Santos et al. (2015), with microsphere KNO_3 content of 18%. Simple superphosphate fertilizer was used for the basic phosphate fertilization. The same was applied to the total area of each seedbed in the same amounts for the two treatments.

Irrigation was performed by surface drip irrigation, using self-compensating emitters of 4.0 L h^{-1} (PC CNL-Naan Dan Jain - reference to the registered trademark is no endorsement by the authors), according to water consumption by plants. Then, four TDR probes were installed in each seedbed close to the lettuce seedlings, in order to monitor soil moisture for irrigation management during the lettuce development cycle, as well as the soil electrical conductivity to monitor the release of fertilizers into the soil.

For irrigation management, soil moisture was obtained in the 0 to 0.20 m layer from the soil apparent dielectric constant (Ka) provided by the TDR probes, considering the average between the results of each treatment probes. Readings were performed every two days, and soil moisture was calculated from Ka through [eq. (1)], obtained according to the methodology proposed by Souza et al., 2013. The water irrigation depth to raise soil moisture up to field capacity was 0.33 m^3 m^{-3}, being estimated by [eq. (2)], as follows:

$$\theta_{TDR} = 0.000005Ka^3 - 0.0003Ka^2 + 0.0161Ka + 0.0132 \tag{1}$$

In which,

θ_{TDR} - soil moisture, m^3 m^{-3}, and

Ka - soil apparent dielectric constant, dimensionless.

$$L = ((\theta_{CC} - \theta_{TDR})\, z) x 1000 \tag{2}$$

In which,

L water depth, mm;

θ_{CC} - soil moisture at field capacity, m^3 m^{-3};

θ_{TDR} - moisture obtained from [eq. (1)], m^3 m^{-3}, and

z – root system effective depth, m.

The mean values of each treatment's probes and the value adjusted through [eq. (3)] were also considered for soil electrical conductivity. Equation 3 was obtained through the methodology described by Souza et al. 2006, which performs an indirect relation with the methodology of saturated soil paste extraction.

$$\mathbf{EC_e} = \left(\mathbf{0.0303} + \left(\mathbf{4.602EC_{TDR}}\right) - \left(\mathbf{0.7x\,\theta}\right)\right) \tag{3}$$

In which,

EC_e - soil electrical conductivity of a saturated paste extract, dS m^{-1};

EC_{TDR} - apparent electrical conductivity, dS m^{-1}, and

θ - soil moisture, $m^3\ m^{-3}$.

The first lettuce monitoring cycle was 28 days, and then plants were harvested. The two central rows of the beds containing four plants each, totaling eight plants, were considered the useful area (1 m^2) for material evaluation. Thus, the plants of the useful area were weighed to identify the fresh weight, and freshly opened leaves were sent for laboratory chemical analysis.

The second lettuce cycle was then planted. For the second cycle, only lettuce seedlings were replanted and there was no replacement of mineral fertilizers, nor addition of potassium nitrate microspheres. The purpose of the second cycle was to observe whether the microspheres would continue to release fertilizers into the soil so that lettuce seedlings could absorb it and develop normally. The second cycle lasted 35 days after seedlings transplanting.

RESULTS AND DISCUSSION

Soil moisture was monitored for irrigation management during the lettuce development cycle. Soil solution electrical conductivity was also monitored for the release of fertilizers in the soil (Figure 3). Irrigation management followed a constant irrigation shift and started always close to 0.23 $m^3\ m^{-3}$. The availability of nutrients provided by traditional fertilization was uneven and concentrated at the beginning of the first cycle. However, in this treatment, the availability of fertilizer was always observed to remain below the concentrations of the treatment with microspheres throughout the two cycles, which shows a reflection of the losses occurred at the beginning of the first cycle.

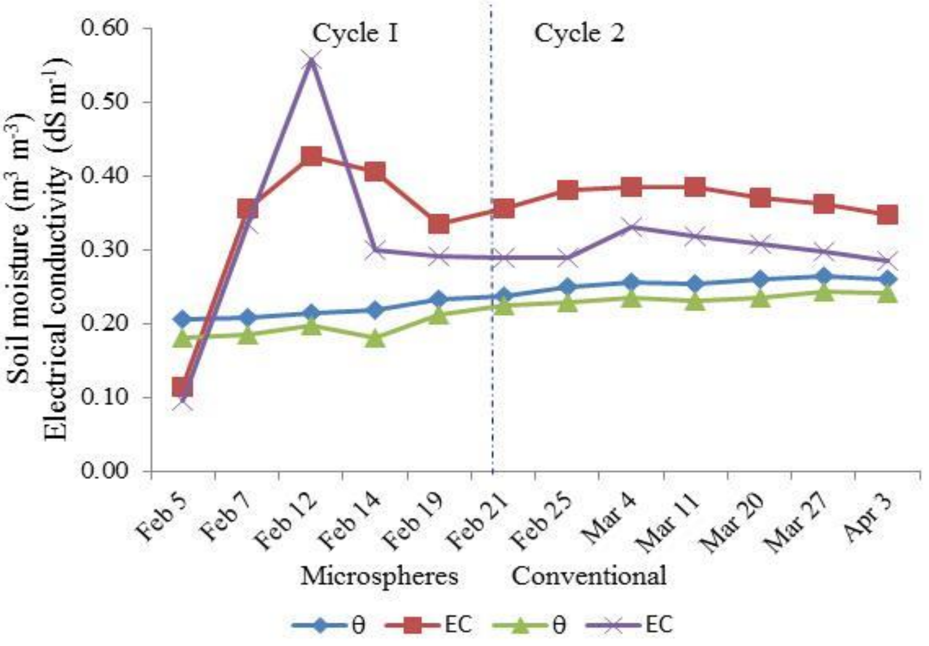

FIGURE 3. Monitoring of soil moisture and electrical conductivity for the different treatments, microspheres and conventional.

In the first cycle, the losses of the treatment with microspheres were lower at 0.132 dS m^{-1}, being justified by a nutrient release around the microspheres (Santos et al., 2015). After the losses, nutrient distribution behaved regularly and constantly during the different culture cycles. A comparison between averages using linear regression showed that the nutrient availability of microspheres in the two cycles is 12% higher than that observed in the conventional treatment.

Microspheres distribute nutrients regularly and constantly, which corroborates with this study's hypothesis. When studying the behavior for controlled release of coated fertilizers, Adams et al., 2013 observed slow and uniform release for one of the commercial products experimentally tested. On the other hand, in conventional fertilization, there is a greater use of work force for splitting the recommended topdressing dose for crop development.

First cycle harvesting was 28 days after sowing lettuce seedlings, assessing its productivity by weighing plants within the useful area of each plot. The average yield per treatment observed was 425 g m^{-2} and 477 g m^{-2} for the microspheres and conventional treatments, respectively (Table 2). The lettuce yield for conventional treatment was 1909 g of the total weight, and for treatment with microspheres, the total weight was 1701 g. These results have no statistical difference from each other.

TABLE 2. Comparative statistical analysis of the first cycle (C1) of lettuce planting for foliar nutrient contents and yield.

Analysis of variance	N	P	K	Ca	Mg	S	Yield
	g Kg^{-1}						g m^{-2}
GL Residue	3	3	3	3	3	3	3
F treatment	0.13	0.12	2.86	0.00	0.24	0.02	0.33
General mean	37.81	3.37	31.28	2.56	1.76	3.68	451.16
Standard dev;	6.33	0.56	5.90	0.40	0.20	0.81	128.40
DMS (5%)	14.25	1.25	13.28	0.89	0.46	1.82	288.96
CV (%)	16.75	16.51	18.86	15.48	11.62	21.92	28.46
Tukey at 5%							
Microspheres	37.00a	3.30a	27.75a	2.56a	1.72a	3.65a	425.18a
Conventional	38.63a	3.44a	34.80a	2.56a	1.79a	3.72a	477.15a

Level of significance: **: 1%; *: 5%.

GL: degrees of freedom; DMS: minimum significant difference; CV: coefficient of variation.

These results comparison were established for the nutrients absorbed by the crop, from leaf analysis. Table 2 shows the average concentrations found in lettuce leaves, which show no significant difference in nutrient contents absorbed by the crop for both conventional treatment and microspheres, reinforcing the observations regarding the productivity of lettuce plants.

As observed, the concentrations of nutrients absorbed by plants were proportional for the two treatments, and the nitrogen content represented the greater amount of absorption, followed by potassium. Magnesium was the one of smaller absorption.

The macronutrient values found in leaves can be compared with the appropriate ranges of macronutrients recommended by IAC Bulletin 100 (Raij et al., 1997). Thus, when comparing the average concentrations presented in Table 2 with the recommended ranges, phosphorus (4-7 g kg^{-1}), potassium (50-80 g kg^{-1}), calcium (15-25 g kg^{-1}), and magnesium (4-6 g kg^{-1}) were below the recommended range, and sulfur (1.5-2.5 g kg^{-1}) presented high levels. However, lettuce plants were healthy and had no nutritional deficiency. Sandri et al. (2006) and Urbano et al. (2017) describe the nutritional imbalance of macronutrients in lettuce leaves, but in none of the treatments performed by the authors were symptoms of toxicity or nutrient deficiency observed.

Evaluation of lettuce yield and analysis of leaf nutrient content were also performed for both treatments at the second sowing cycle. It is worth noting that there was no supplementation of mineral nutrients in this second cycle, in order to evaluate the gradual release of fertilizers by the microspheres for plant absorption over time, in this case totaling 63 days after the application of microspheres, and 43 days after the last portion of conventional fertilization.

Lettuce yield was equivalent for the two treatments, considering a total weight of 1238 g, and an average yield of 309 g m^{-2}. Nitrogen also presented higher value for the concentrations of nutrients absorbed by the plant, around 37.4 and 30.6 g kg^{-1} for conventional treatments and microspheres, respectively.

All values related to yield and leaf nutrient content were submitted to analysis of variance and Tukey test at 5%. Results showed no significant differences between the two treatments within the same parameters evaluated. The mean values found for leaf nutrient contents were 34, 4.2, 3.4, 6.6, 2.6, and 0.4 g kg^{-1}, respectively, for N, P, K, Ca, Mg, and S.

The comparative statistical analysis of the first (C1) and second (C2) lettuce planting cycles for leaf nutrient contents presented significant differences for potassium (31.3 and 3.4 g kg^{-1}), calcium (2.6 and 6.6 g kg^{-1}), magnesium (1.7 and 2.6 g kg^{-1}), and sulfur (3.6 and 0.4 g kg^{-1}). This response was already expected based on the procedure adopted to replant seedlings without a new fertilization.

A reduction of approximately 90% from the first cycle to the second one was observed for foliar potassium concentration. At this point, microspheres are observed to have no influence in supplying potassium to the soil solution gradually for more than 30 days. Results indicate a higher release of potassium in the first cycle. This result corroborates that without crop residues (Santos et al., 2015), in which a marked peak is observed at the start of the release test due to the presence of potassium on the microspheres outer surface. According to the authors, the potassium bound around the microspheres is released more easily when compared to the element present within the polymeric material.

These values increased for foliar concentrations of calcium and magnesium in the second cycle. Some studies report that the levels of Ca in both soil and plant are influenced by other elements (Salvador et al., 2011). According to Moreira et al. (1999), high levels of calcium and magnesium in the soil promote competitive inhibition against potassium. Clark et al. (1997) observed a calcium concentration reduction in maize shoot with an increasing application of magnesium, while potassium remained constant. Freiberger et al. (2014) reported an inhibitory effect of potassium on magnesium absorption in jatropha plants.

Nitrogen concentrations were according to the recommended range for macronutrient levels in lettuce leaves in both cycles, that is, between 30-50 g kg^{-1}. Studies reported that both irrigation depths and fertilization levels are generically recommended with the aim of reaching maximum yields. However, in this case, they are economically unviable since cost effectiveness rarely corresponds to technical efficiency. Pereira et al. (2003) evaluated the yield of crispy lettuce cv. Verônica in different water and nitrogen contents and concluded that plants grown in protected environments can undergo nitrogen deficit, with nitrogen varying only according to its content in the soil.

The chitosan-clay microspheres used as soil nutrient ferti-releaser in lettuce cultivation showed a behavior similar to the conventional application of fertilizers during monitoring and, consequently, evaluation in the lettuce crop development. Results highlight a better nitrogen nutrient response during controlled release, and the microspheres continued releasing it even after 30 days. Unlike what was seen for potassium, with leaf content values below the appropriate range for lettuce leaves, between 50-80 g kg^{-1}. Potassium is believed to be partially retained on the surface of microspheres, being consequently released without proper control at the beginning of the first cycle.

There was a release peak after 7 days, and a release reduction over the following week, showing evidence of leaching of the potassium present in the soil solution (Figure 3). In the aftermath, the nutrients within the center of the microspheres are released again in a controlled manner, thus remaining until the end of the monitoring. This result is extremely important for the improvement of ferti-releaser microspheres, which already demonstrate a practical potential for sustainable agriculture development.

CONCLUSIONS

The use of ferti-releasing microspheres was efficient for a controlled release of potassium nitrate, mainly for nitrogen, when compared to the conventional fertilizing form.

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