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Nutrient Release, Plant Nutrition, and Potassium Leaching from Polymer-Coated Fertilizer

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ABSTRACT: The increase in food consumption and limitations in food production areas requires improved fertilizer efficiency. Slow- or controlled-release fertilizers are an alternative for synchronizing nutrient availability with the plant demands, reducing losses to the environment. The aim of this study was to evaluate the efficacy of polymer-coated KCl compared with conventional KCl. The products were incubated in soil under controlled conditions to evaluate the time required for nutrient release. A greenhouse experiment was performed with corn plants in pots with loamy sand- or clay-textured soil types to evaluate plant nutrition and losses due to leaching. The K application rates were 0, 18, 36, and 54 mg dm^{-3}. The pots were irrigated, and the percolated liquid was collected. The plants were harvested 30 days after sowing to quantify dry matter (DM) and its K content. In the incubation study, the K release from the coated fertilizer was found to be 42 % over 154 days. The data were fit to a linear function from which a period of 315 days was estimated as required for the release of 75 % of the nutrient. Meanwhile, conventional KCl releases 85 % of the K nutrient in the first 48h. In the cultivation of plants in pots, the coating reduced K losses due to leaching in the loamy sand soil; however, only the application rate of 54 mg dm^{-3} promoted DM production equivalent to conventional KCl. It is possible that the need for K in the early stages of corn development was not met by a coated KCl.

Keywords: enhanced efficiency, slow- or controlled-release, leaching.

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INTRODUCTION

The challenge of sustainable agriculture involves the production of economically viable and environmentally friendly food. The application of slow- or controlled-release technology in fertilizer is based on the gradual release of nutrients to meet plant demand and reduce loss to the environment. However, research needs to advance to enable an understanding of the pattern of release associated with this technology so as to properly regulate these products and provide consumer information.

In Brazil, Law No. 6894/1980, which is known as the "Fertilizer Act", is regulated by Decree No. 4954/2004 and provides for inspection of the production and trade of fertilizers and other plant nutrition inputs. The regulation entailed by this law lays down general rules regarding the registration and classification of such products. Production establishments, importers, and fertilizer dealers are required to register with the Ministry of Agriculture, Livestock and Food Supply (*Ministério da Agricultura Pecuária e Abastecimento* - MAPA), and registration of the products is also required. The criteria for registration and the limits of guarantees and specifications are established by normative rulings (IN) specific to each type of product (Brasil, 2004).

In the United States of America (USA), the terms "enhanced efficiency fertilizer" (EEF), "slow-release fertilizer" (SRF), and "controlled-release fertilizer" (CRF) have been defined by the Association of American Plant Food Control Officials (AAPFCO) (Trenkel, 2010). The SRF name is assigned when a nutrient is released at a rate slower than normal, but the pattern and length are not specified by the manufacturer. It is common to designate nitrogenous products that are degradable by microorganisms, including urea formaldehyde, as SRFs. The accepted use of the term CRF is for fertilizer in which the factors that determine the standard rate and release period are predictable and influenced by the manufacturing process (Shaviv, 2005; Trenkel, 2010). Controlled-release fertilizers are fertilizers in which the release is controlled by a physical barrier. The fertilizer may be in the form of pellets or beads that are coated with hydrophobic polymers or dispersed in matrices that restrict dissolution of the fertilizer. The technology used to produce the coating material and the thickness of the coating material determine the gradual release of the coated or encapsulated nutrient (Shaviv, 2005).

The two main resins used in such coatings are alkyd type resins and polyurethane. The pattern of nutrient release is determined by variations in the polymer composition and the coating thickness. Typical formulations contain urea, NPK (nitrogen, phosphorus, and potassium) compounds, and/or microelements. Various manufacturers use two layers of polymer to increase the efficiency of protection and resistance to attrition of the granules. In such cases, the first layer consists of sulfur, and the second is composed of an organic polymer (thermoplastic or resin) (Shaviv, 2005; Trenkel, 2010). In Europe, the European Committee for Standardization (CEN) has the authority to classify and define the conditions under which a product can be categorized as slow- or controlled-release fertilizer. The CEN criteria to establish a fertilizer as "slow release" (at 25 °C) are: (i) no more than 15 % release in 24 h; (ii) no more than 75 % release in 28 days; and (iii) at least 75 % release within the period prescribed by the manufacturer (Trenkel, 2010).

Expressions such as EEF, SRF, or CRF are not established in Brazilian standards, and the regulation of these products is enforced only in terms of chemical and physical guarantees because there is no official methodology for determining whether the percentages of nutrient release are within the limits guaranteed by the manufacturer. Standardization of tests for validating the release of nutrients from coated fertilizers is required.

Studies have been performed in Brazil and abroad using incubation methods in controlled environments to predict the release of nutrients and to evaluate different environmental parameters, such as temperature, moisture, microorganisms, and soil pH and texture (Golden et al., 2011; Adams et al., 2013; Mota, 2013; Medina et al., 2014). Some field studies such as Oosterhuis and Howard (2008) tested slow-release fertilizers N or K coated with polyolefin resin in the US cotton crop. The reference treatments were potassium chloride (KCl) and ammonium nitrate (AN). Results indicated that use of a coated N source did not reduce yield. Results were similar for treatments with coated K in an irrigation management system; however results were contradictory in tillage. The study presented comparison of means between them, but the response curves for each product were not shown. The conclusion of the researchers was that the use of technology for K was not as effective as for N in promoting responses in cotton fiber production. In another study (Blaylock, 2007), coated N-fertilizer in the US corn crop showed that reduction in N losses caused greater efficiency in its use. Reduction in the N application rate to 70 to 80 % of that commonly applied did not reduce yield. Both authors suggest prior evaluation of the economic viability of adopting this technology.

In Brazil, research projects regarding fertilizer use efficiency that include slow- or controlled-release technology have been intensified in recent years. The main research groups with publications in this area are the ESALQ/USP, the *Instituto Agronômico de Campinas* (IAC), and the Federal University of Lavras/MG. The common goal of these studies is to evaluate loss of N by volatilization from urea coated with inhibitors or with polymers.

The urea coated with a double layer of polymer did not reduce loss of ammonia by volatilization (Zavaschi et al., 2014). The application of conventional urea did not differ from coated urea for the variables of grain yield, N content in the leaves and grain, and chlorophyll content, all in corn. However, Nascimento et al. (2013) observed a reduction in N volatilization losses from urea coated with elemental sulfur or boric acid and copper sulfate compared to conventional urea, in sugarcane. Faria et al. (2014) also studied urea with inorganic coatings (S, B, and Cu), but with the objective of evaluating the hygroscopicity of the product in comparison to ammonium nitrate (AN). The coated urea is more susceptible to absorbing moisture from 75 % relative humidity, but remains lower than the AN.

The potential use of enhanced efficiency fertilizers is to reduce emission of nitrous oxide (N_2O) from degradation of N fertilizer and thereby lower the environmental impact of greenhouse gases (Soares et al., 2015). The study was conducted in two sugarcane crops. The application of two nitrification inhibitors (DCD - dicyandiamide and DMPP - dimethylpyrazol phosphate) resulted in a 90 % reduction in N_2O released and did not differ from the control without N. For the N-fertilizers with sulfur and polymer coating, the opposite effect occurred; the emission of N_2O gas was higher. This was not expected by the researchers and the reasons for this effect are not clear. Under laboratory conditions, gradual release of N avoided nutrient concentration peaks above the plant uptake capacity; it reduced the N available to microorganisms, as well as nitrification and denitrification processes that lead to N_2O emissions.

Beginning with the premise that coating the granules of a mineral fertilizer with polymers delays nutrient release, the hypothesis of this study is that nutrient release into the soil from such fertilizers occurs gradually and causes less leaching compared with conventional mineral fertilizers. The aim of this study was to evaluate the efficacy of a polymer-coated fertilizer in terms of three aspects: nutrient release over time, the reduction in losses due to leaching, and plant nutrition.

MATERIALS AND METHODS

Two studies were performed. The first was a soil fertilizer incubation study that was conducted at the soil laboratory. In the second study, plants were grown in pots in the greenhouse.

The products used in the experiments as K sources were conventional potassium chloride (KCl) with a 60 % K_2O content, and a coated KCl, called Producote®, from the

Produquímica Company from Brazil, with nominal concentrations of 51 % K_2O and 14 % S. The manufacturer indicated that the coating is composed of a layer of elemental S and another layer of organic polymers whose compositions are not specified to protect industrial secrets. The product in question has around 4 months of longevity (time to release), declared by manufacturer. Elapsed time to release nutrients (longevity) is determined by the thickness of the layer of the organic polymer that coats each granule and the temperature of the soil.

The fertilizers used were previously analyzed according to the official methodology (Brasil, 2007) in MAPA Fertilizer Laboratory (Lanagro) to confirm the contents of K_2O and S.

First study - Incubation

Fertilizer incubation was performed in a sandy loam *Argissolo Vermelho-Amarelo Distrófico* (Embrapa, 2006), an Ultisol (Soil Survey Staff, 2010). The bulk density was 1.48 Mg m^{-3} and the main soil properties were 160 g kg^{-1} clay, 50 g kg^{-1} silt, 790 g kg^{-1} sand, cation exchange capacity (CEC) 5.6 $cmol_c$ dm^{-3}, and K content of 33 mg dm^{-3} (Tedesco et al., 1995).

The soil was placed in 100-mL plastic bottles. The coated KCl fertilizer granules were selected by size (4 mm), each treatment were compound from 12 to 15 integer granules, to maintain de same dose. Conventional KCl granules were screened by weight to ensure a supply of 1,790 mg dm^{-3} of K in all treatments.

The fertilizer granules were placed in 55 mesh polyester called a “sachet” that measured 2.0 × 1.5 cm and was sealed at the ends. The sachets were placed in bottles containing 50 g soil and covered with 100 g of soil. The soil was saturated with 25mL distilled water, and the bottles were kept open inside an incubator at a constant temperature (25 °C).

Soil moisture was monitored at 3-day intervals based on the weight of the bottles and restored to 80 % of maximum capacity by the addition of distilled water. The bottles were left in the incubator for periods of 1, 2, 5, 7, 9, 14, 21, 28, 37, 48, 56, 72, 89, 124, and 154 days. After the incubation period, the bottle was removed from the incubator, and the sachet was separated from the soil. The soil was dried and homogenized and K was extracted through the Mehlich-1 method (Mehlich, 1953) as adapted by Tedesco et al. (1995); determination of K was performed by inductively coupled plasma optical emission spectrometry (ICP OES). The experiment was conducted in a completely randomized 2 × 15 factorial design, which corresponded to evaluation of two products at 15 points in time, with three replications.

The results were subjected to analyses of variance (Anova). The main effects were considered significant at 5 % of probability, and the interaction effects were considered significant at 25 %, as suggested by Perecin and Cargnelutti Filho (2008). Complementary interaction analyses were performed through the F and t tests. Analysis of the effects of the quantitative factors was complemented by regression fitted to linear, quadratic or cubic functions. The equation fitted to the data was used to determine the longevity of the product to release 75 % of the nutrient (CEN standard).

Second study – Plant cultivation in a greenhouse

Two soils were used to grow the plants in a greenhouse. A sandy loam *Argissolo Vermelho-Amarelo Distrófico* (Embrapa, 2006), an Ultisol (Soil Survey Staff, 2010) (160 g kg^{-1} clay, 70 g kg^{-1} silt, and 770 g kg^{-1} sand; CEC 6.9 $cmol_c$ dm^{-3}; K 13 mg dm^{-3}) and a clayey *Nitossolo Vermelho Distrófico* (Embrapa, 2006), a Rhodic Hapludox (Soil Survey Staff, 2010) (550 g kg^{-1} clay, 210 g kg^{-1} silt, and 240 g kg^{-1} sand; CEC 14 $cmol_c$ dm^{-3}; K 85 mg dm^{-3}) (Tedesco et al., 1995) were used. Soil acidity was corrected by applying a mixture of $CaCO_3$ and $MgCO_3$ at a molar ratio of 3:1 to achieve a pH of 6.0.

Pots of PVC of 0.15 m diameter and 0.35 m height were used. For soil drainage, the bases of the pots were filled with crushed stone (800 g) and four layers of nylon fabric (1 mm mesh) and coupled to a hose (8 mm diameter), one end of which was inserted into a bottle (1.5 L) for storage of the liquid leachate. The volume of soil in each pot was 5 dm^3. A total of 56 pots were set up [(two soils × two fertilizers × three application rates + one control for each soil for both products)× four replicates], and these pots were divided into four blocks with 14 randomized treatments.

Fertilization was calculated in accordance with the recommendations for corn (*Zea mays* L.) (CQFS-RS/SC, 2004). The K treatments were applied at three rates, plus a control treatment without K fertilizer (zero rate). The maximum rate of K_2O recommended for use in the planting row is 80 kg ha^{-1} (CQFS-RS/SC, 2004), and the rates defined for the treatments represented 55, 110, and 165 % of this reference limit. The rates of K were thus quantified for the volume of soil in the pots as follows: rate 1 = 18.3 mg dm^{-3}; rate 2 = 36.5 mg dm^{-3}; and rate 3 = 54.8 mg dm^{-3}. The N fertilizer used was ammonium sulfate, to provide S and to avoid the influence of the S present in the coating of the fertilizers tested. The P was supplied with triple superphosphate, and micronutrients were provided by a complex mineral fertilizer containing Zn, Mn, B, and Cu.

The experiment began on March 6, 2015 with incorporation of the fertilizers into the soil to a depth of 0.05 m. Four daily irrigations with 250 mL of distilled water were performed to promote solubilization of the salts and ensure sufficient depths in the soil profile. On March 10, 2015, four corn seeds (early cycle) were sown per pot at a depth of approximately 0.02 m. Irrigation was performed, and the next day the first accumulated liquid leachate from the beginning of the experiment was collected. Six days after sowing, the plants were 0.05-0.10 m tall, and thinning was performed to retain only two plants per pot.

The moisture of the soil was evaluated weekly by the weight of the pots to estimate the irrigation volume required. The total volume of irrigation over the 34 days from fertilization to cutting of the plants corresponded to 210 mm of rain. The percolated solutions from the pots were measured with beakers, and samples were taken for analysis by ICP OES. The temperature inside the greenhouse ranged from 16 to 50 °C.

The variable K leached was obtained by multiplying the amount of K in the collected sample (mg L^{-1}) by the total volume (L) of the four collections. The plants were harvested 30 days after sowing at a height of 1 cm from the soil and were dried at 65 °C for 48 h. The dry matter (DM) variable was calculated as the sum of the weight (g) of the two corn plants in each pot. The K was extracted from the DM through nitric perchloric digestion and determined by ICP OES. The “total K absorbed” variable was obtained as the K content multiplied by the DM produced.

The results were separately subjected to analyses of variance (Anova) for each type of soil. The main effects were considered significant at 5 % probability, and the interaction effects were considered significant at 25 %, as suggested by Perecin and Cargnelutti Filho (2008). Complementary interaction analyses were performed with the F and t tests. The analysis of the effects of the quantitative factors was complemented by regression fitted to linear, quadratic or cubic functions. For the qualitative factors, multiple comparisons of the averages were performed, with Tukey’s tests at 5 % probability, when necessary.

RESULTS AND DISCUSSION

First study - Incubation

The mean comparison test for the effects of the product in each period confirmed the superiority of the conventional KCl in terms of K release in all treatments (Figure 1). The conventional KCl released approximately 72 % of the K within 24 h, 85 % within two days, and 90 % within five days. These findings confirmed the immediate release from this source. The coated KCl exhibited wide variability in release across the replicates in the initial period. The release of K was approximately 20 % after 28 days, 30 % after 72 days, and 40 % at five months of incubation, and the coefficients of variation (CV) in these periods were 123, 40 and 26 %, respectively.

High variability (CV 10 to 40 %) in the release of a thermosetting resin-coated fertilizer (NPK) incubated in a liquid medium over 400 days was observed by Adams et al. (2013). These authors attributed the problem of uneven release to the coating manufacturing technology based on observation of dilation of the granules and disruption of the coating. In the present study, the variability in the initial period may have been due to the presence of air and the lack of water contact with the fertilizer. During the incubation period, the wetting and drying sequence minimized this problem.

The interaction effect in the Anova (product × days) confirmed that the effect of days was significant only for the coated product. Regression analysis of this product yielded a linear function ($\hat{y} = 165.59 + 3.65^{*}x$). Considering the K input of the fertilizer in the soil (1,790 mg dm^{-3}) minus the initial content (33 mg dm^{-3}), the estimated time up to release of 75 % of the nutrients from the coated fertilizer according to this equation was 315 days. Thus, the longevity of this coated fertilizer could reach 10 months, rather than the 4 months stated by the supplier. In practice, this product is offered in a mixture with conventional fertilizers to provide part of the nutrients immediately and to protect against leaching losses of the remainder of the nutrients, especially in sandy soils. The use of a pure coated product is recommended by the supplier in cases of transplanting seedlings of perennial crops. The results of this study differ from those obtained by Mota (2013) and Medina et al. (2014), who used similar incubation conditions on the ground at a temperature of 25 °C and observed the release of 80 to 90 % of the N within 40 to 180 days. Notably, these authors evaluated coated N-fertilizer. No other incubation studies that focused exclusively on K-fertilizer were identified.

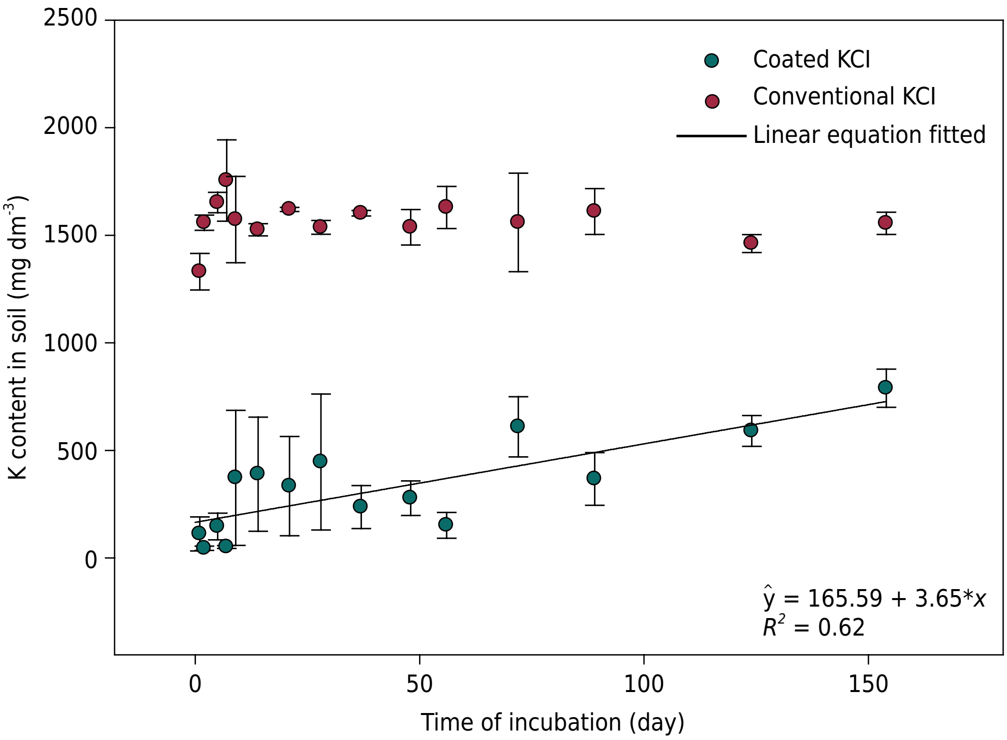

Figure 1. Exchangeable K contents over time in soil incubated with two KCl fertilizers. The average scores with the standard error bars and line of the equation fitted to the data are shown. *: significant at 5 %.

Second study – Plant cultivation in a greenhouse

Potassium leached

The results are presented by variable and soil type. The K leached variable refers to the total weight of K that leached from the soil (Figure 2). There were significant interaction effects (product × application rate) in both soils. However, an application rate effect on loss of K was only observed for the conventional KCl. Regression analysis of this relationship yielded quadratic functions for both soils. The K leaching in the soil with conventional KCl was greater than that in the soil with coated KCl at rates 2 and 3 in the sandy loam soil, and rate 3 in the clay soil. These losses occurred due to the immediate solubility of conventional KCl, whereas the coated KCl restricted the release of the nutrient into the soil solution, as noted in the incubation study presented earlier.

Leaching of K was observed in the control treatments without KCl (zero rate). The nutrient was lost from the soil-exchangeable K that was moved vertically by water flow. The treatments with conventional KCl in the sandy loam-texture soil revealed that the availability of K from this source was greater than the uptake capacity of the corn roots at this stage of development. This hypothesis was also mentioned by Werle et al. (2008). In contrast, in the clayey soil, the K losses were less than 1 % of the total available K in all treatments, regardless of the fertilizer and the rate applied. Therefore, soil properties, such as clayey texture, were more crucial for reducing mass flow to prevent loss of K than the initial content of the nutrient. The use of coated fertilizers to reduce leaching losses may be unnecessary for clay soils with high cation exchange capacities (for example, into South Brazil soil, CECs >15 $cmol_c\ dm^{-3}$).

However, comparison of this result with results obtained by Ernani et al. (2007) and Werle et al. (2008) indicate that the volume of water applied and the time elapsed in this experiment were insufficient to promote K leaching in the clay soil. The results of this study confirm the current knowledge regarding K dynamics in soils. K^+ ions move in the soil profile, and significant quantities can be lost by leaching, particularly when the K comes from soluble sources in medium or sandy soils with low CECs (<5 $cmol_c\ dm^{-3}$) (Sanzonowicz and Mielniczuk, 1985; Alfaro et al., 2004; Werle et al., 2008).

Dry matter (DM)

All treatments in both soils resulted in significant effects on DM production. The addition of fertilizers increased the values of this variable relative to the control. The treatments

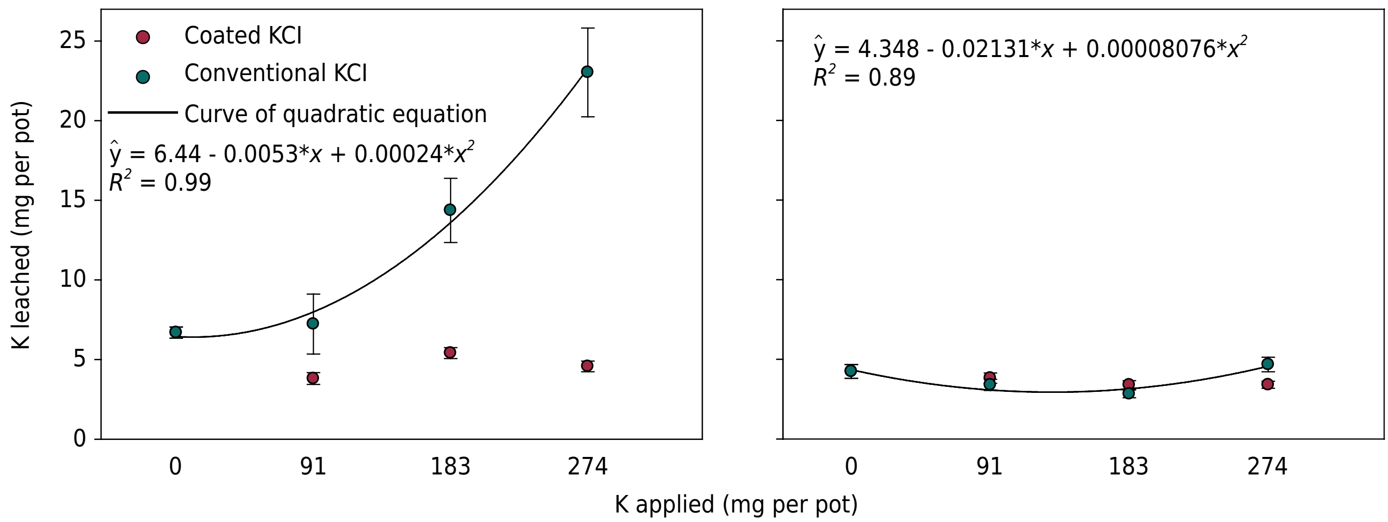

Figure 2. Rates of K leaching from the (a) sandy loam Ultisol and (b) clayey Rhodic Hapludox soils treated with conventional or coated KCl fertilizer. The average scores with standard error bars and curves fitted to quadratic functions are shown. *: significant at 5 %.

with conventional KCl promoted greater DM production than the treatments with coated KCl in both soils, and these differences were most notable in the sandy loam soil (Figure 3). The interaction effect (product × application rate) was significant only in the sandy loam soil. The details of Anova revealed the superiority of conventional KCl in terms of DM production, which occurred only at application rates 1 and 2. For the third rate, DM production did not differ between the fertilizers.

Considering that DM did not differ among the three rates of conventional KCl, it can be assumed that the first rate yielded a sufficient K level. Accordingly, complementary statistical tests (Tukey test, 5 %) revealed no significant differences between rate 3 (54.8 mg dm^{-3} K) with coated KCl and rate 1 (18.3 mg dm^{-3} K) with conventional KCl in terms of DM (p=0.35) or K taken up (p=0.65). Therefore, it can be inferred that the release of the coated KCl at rate 3 was approximately 30 %.

Potassium uptake by plants

The effects of K rates on K uptake by the plants were significant in both soils (Figure 4). The interaction effect (product × application rate) was significant in the sandy loam soil and fulfilled the assumptions of Perecin and Cargnelutti Filho (2008). For statistical

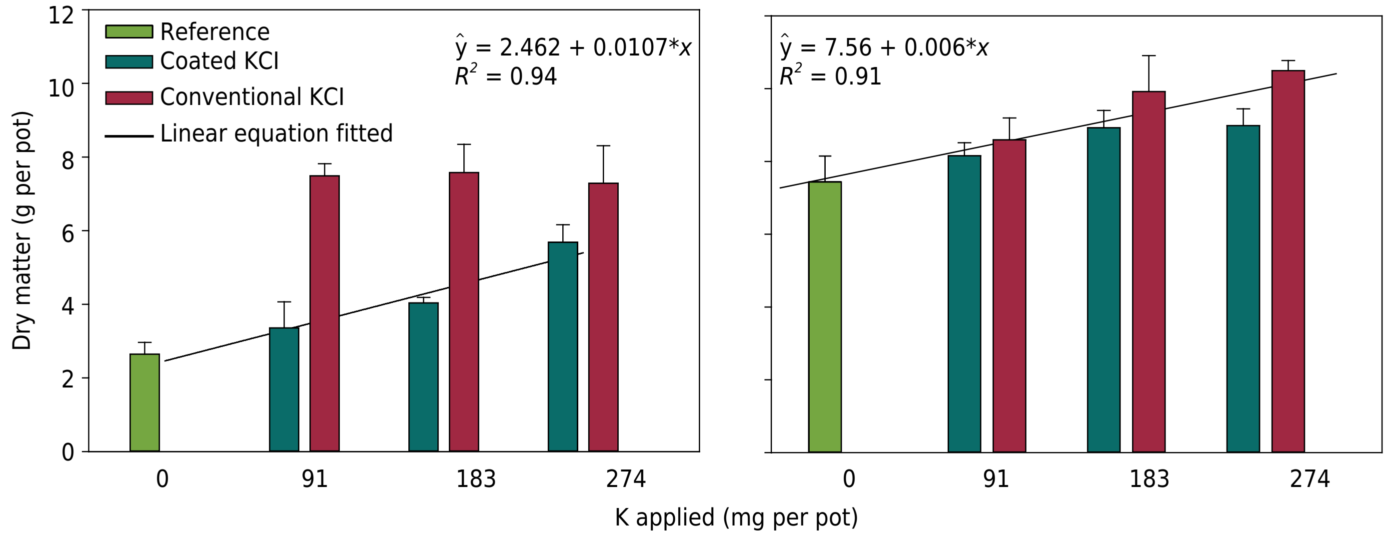

Figure 3. Dry matter produced by corn plants grown in pots in two soil types, i.e., (a) sandy loam Ultisol and (b) clayey Rhodic Hapludox, which received conventional or coated KCl fertilizers. The columns with the standard error bars indicate the averages, and the linear function equations are shown. *: significant at 5 %.

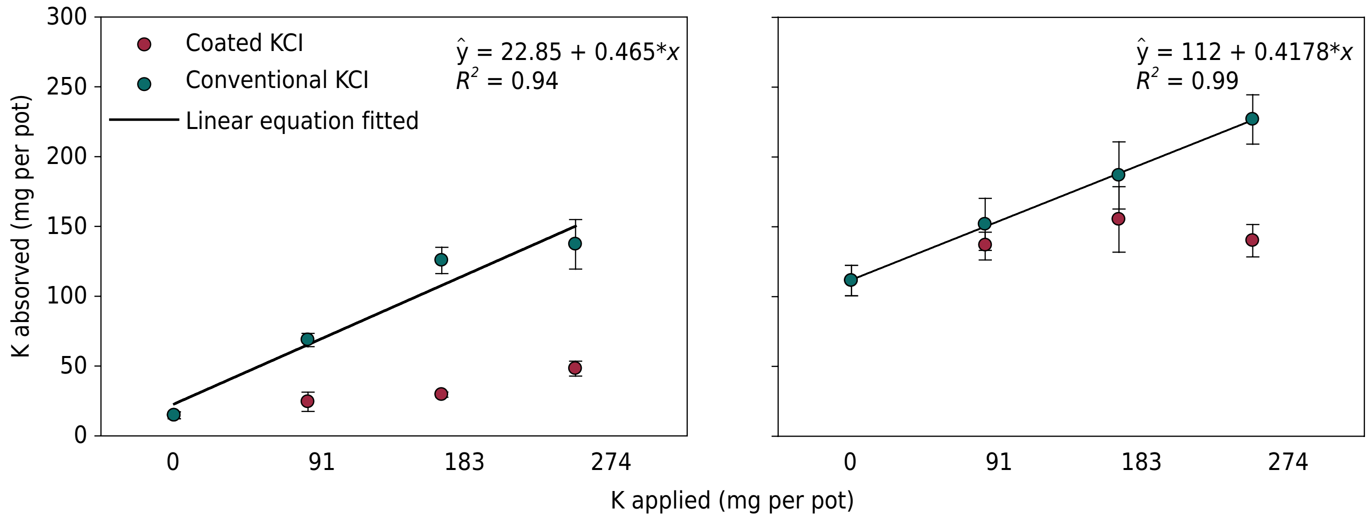

Figure 4. Total K absorbed by corn plants grown in (a) sandy loam Ultisol and (b) clayey Rhodic Hapludox soils that were treated with conventional or coated KCl fertilizer. The average scores with the standard error bars and the linear function equations are shown. *: significant at 5 %

analysis, the clay soil data were also split. The K uptakes of the plants were higher in the three conventional KCl treatments in the sandy loam soil and rate 3 in the clay soil. An effect of application rate on the K taken up was observed only in the treatment with conventional KCl, and the data from both soils were fit to linear functions.

The increased uptake of K at the higher rates of conventional KCl indicated that this nutrient was not limiting for DM production in the sandy loam soil. These results are similar to those observed by Kaminski et al. (2007), who studied plants (oats, soybeans, wheat, corn, and jack beans) grown in a sandy loam Ultisol. The K content in the DM was greater at higher rates, regardless of the original K content in the soil, and this difference did not necessarily result in increased DM production (Kaminski et al., 2007).

Plants can activate more than one uptake mechanism when K is available, which results in uptake that exceeds the physiological needs of the plant. Thus, K in the DM of some plant organs, with the exception of grain, has been defined by Marschner (1995) as luxury consumption. However, the addition of excessive K can affect the uptake of Ca and Mg and cause nutritional imbalances that are reflected in DM production (Barber, 1995).

Although immediate release from conventional fertilizer can result in losses due to leaching in sandy loam soil, significantly greater dry matter production and plant K uptake demonstrate the requirement of availability of this nutrient during the initial period of corn development. This was noted by Barber (1995), who stated that the period of maximum influx of nutrients in corn roots occurs in the first 20 days of the plant cycle. Thus, coated fertilizers should not be used as sole sources of nutrition in sandy loam-textured soils with low nutrient contents for short cycle crops because K deficiency in the early stage can compromise final yield. Instead, the amount applied and the release rate should be optimized to meet demand in the first weeks of plant growth. It should be noted that although the Hapludox soil has higher levels of K, the plants responded to the application of K.

CONCLUSIONS

The technology of coating KCl with sulfur and an organic polymer resulted in gradual release of K into the soil, with estimated longevity of 315 days.

The physical protection of the coating and the gradual release of the nutrients into the soil solution reduce the loss of K leaching into the sandy loam-texture soil.

Although the study results are restricted, the coated KCl fertilizer in soils with low K levels was not able to meet the K demands of the initial stage of the plants.

The use of coated KCL fertilizer in sandy loam soil with low K levels must be at higher rates than common KCl, or it must be combined with an immediately-available K source.

This coated KCL fertilizer, pending economic evaluation, may represent a technically possible alternative in clay soil and in conditions involving sufficient nutrient levels.

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