UNIVERSIDADE ESTADUAL PAULISTA
"JÚLIO DE MESQUITA FILHO"
Campus de Botucatu

FACULDADE DE CIÊNCIAS AGRONÔMICAS
UNESP | BOTUCATU

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ROTATION SOYBEAN-FORAGE AND THE CONTRIBUTION OF NON-EXCHANGEABLE K IN PLANT NUTRITION IN A CERRADO SOIL

Botucatu
2019

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Thesis presented to São Paulo State University, School of Agriculture, to obtain Doctor degree in Agronomy (Agriculture).

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Botucatu
2019

V914r

Volf, Marcelo Raphael
Rotation soybean-forage and the contribution of non-exchangeable k in plant nutrition in a cerrado soil / Marcelo Raphael Volf. -- Botucatu, 2019
128 p. : il., tabs.

Tese (doutorado) - Universidade Estadual Paulista (Unesp), Faculdade de Ciências Agronômicas, Botucatu
Orientador: Carlos Alexandre Costa Crusciol
Coorientador: Ciro Antonio Rosolem

1. Soybean-Yield. 2. Soybean - Grasses. 3. Crop rotation. 4. Potassium content. I. Título.

Sistema de geração automática de fichas catalográficas da Unesp. Biblioteca da Faculdade de Ciências Agronômicas, Botucatu. Dados fornecidos pelo autor(a).

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Título: **ROTATION SOYBEAN-FORAGE AND THE CONTRIBUTION OF NON-EXCHANGEABLE K IN PLANT NUTRITION IN A CERRADO SOIL**

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Botucatu, 17 de setembro de 2019.

I dedicate this thesis
to my family

ACKNOWLEDGMENT

To God for giving me health, strength, and courage to overcome difficulties in this arduous and challenging journey. "Without you Lord, none of this would be possible."

To my mother Rolaci Maria Volf and my aunt Cleunice Volf de Oliveira (Teti), I say thank you with all my heart, for all the deprivation that you had passed, so that I could get where I got, I know it was not easy. "I am who I am, thanks to you".

To my Uncle Sidinei and my Aunt Geni (in memoriam), for the support and for having believed in my capacity. - You were very important in my professional and personal life, I will never forget everything that you did, and nothing would be possible without their interference.

To my wife's family, Antonio Fumagalli (father in law); Neusa Balbino de Amorim (mother in law); Janine and Cassio (brother in law); who welcomed me and entrusted to me, my wife. And my beloved nephews, Vitoria and Vicenzo.

To my wife, Jaqueline, my greatest supporter, always present and my great mainstay, nothing in my life would have felt without her presence by my side, I love you forever and ever. To our daughter, Theodora, truly a gift from God. Without the sacrifice, which I know both have done, none of this would be possible, I will always do everything so that all this is not in vain. Thank you so much for everything.

To my advisor, Prof. Dr. Carlos Alexandre Costa Crusciol for having trusted me, it had believed, that although the resume was not the best, it could work, and I know it was a bet, in a project of the student that could do this. It will always be an honor to say, "I have been guided by Prof. Crusciol" I hope never to let you down - Thank you for having always worried about me and my family, the support in hard moments certainly made the difference.

To my co-adviser Prof. Dr. Ciro Antonio Rosolem, I would like to express my grateful thanks for all the help over all the project, certainly, the corrections and tips were decisive to the successful the of this journey. But the care that my wife, my daughter and I received from you and your wife, Monica, gave us the strength to face the challenges and made me believe that it would be possible to win.

To Prof. Dr. Antonio Carlos Azevedo, I would like to say thank you so much for opening the doors of his office and laboratory to an unknown student and full of doubts, thank you for having believed in the project and for all the teachings, I have sure that without these, my results would not be complete.

To Prof. Dr. Michael L. Thompson of Iowa State University and researcher Dr. Jhon Kovar of the National Laboratory for Agriculture and the Environment - USDA, for accepting me, for help me and giving me the opportunity to make my analyzes in your lab in the United States. I thank you for the care, attention, and support of my dream accomplishment. Thank you so much.

To Prof. Dr. Vinicius de Melo Benites, my advisor in the Master's degree, which started me at research, showed me the way to build the sequence in the Ph.D. Thank you so much.

Special thanks to my, biggest friends; Agnaldo Cecatto and Thiago Neves, because they were always very helpful both inside the company (Dalcin) and to help me with my project. It would not be able to further this step without your help.

To my customer, but much more than this a big friend and example, Carlos Guimarães, I say thank you so much for everything, all the scold and for trusting in my work, besides making it possible to conduct the project on your farm - "everything started from your questionings and stimulus (moral, professional and funding). "

To the State University of São Paulo, that through the program of its graduate program, which can give me the opportunity of a quality graduate. And to the laboratory of Solo and leaf of this university and all his team to help me.

To my friends, Cleiton José Alves, Amanda Prado Gilabel, Jorge Martinelli, Murilo de Souza, Luiz Moretti, Gabriela Siqueira, Letusa Momesso, Sueko Tanaka, Ariani Garcia, Nídia Costa, Daniele Scudeletti, Murilo de Campos, João Bossolani, and Vitor Rodrigues, all contributed in some way to my walk.

To the professors, and staff of the Faculty of Agronomic Sciences (FCA) - Unesp, Botucatu Campus, for the contribution in my learning and activities throughout these years.

To all the Brazilians I met in Iowa, Arthur, Joana, Jofran, Conceição, Laura, Edicarlos, Henrique, Ivair, Tanise, Vitoe, Flavia, Anderson, Grazi, Francisco, Marcio, Andreia, Gustavo and Andresa. You were more than friends, certainly a big family, with you everything became less difficult.

To the Coordination of Superior Level Staff Improvement (CAPES) for the financial support, awarded through a doctorate scholarship.

Thank you so much.

"The Insanity is to keep doing the same thing and expect different results".
(Albert Einstein)

RESUMO

As frações solúveis e trocáveis do K, podem não ser as únicas disponíveis para as plantas, uma vez que a fração não trocável também pode ser utilizada. As plantas de cobertura são eficientes na reciclagem deste nutriente e podem absorver tanto as formas trocáveis como as não trocáveis do K. Portanto, o objetivo deste estudo é identificar a localização do K não-trocável nas frações argila e silte após exaustão causada por sucessivos cultivos de milho, soja ou braquiária ruziziensis (*Urochloa ruziziensis*), e verificar a eficiência em suprir a demanda de K a partir do K não trocável. O estudo envolveu dois experimentos: 1) Cultivo de soja, milho ou capim-braquiária em vasos por três ciclos sucessivos em solos com alta concentração de K não-trocável, com e sem adubação potássica. As plantas foram removidas dos vasos no momento do pico de acúmulo de K para determinar o K trocável e não-trocável nas plantas e a mineralogia do solo. 2) Um experimento de campo foi realizado envolvendo três sistemas de produção: cultivo de soja com o solo deixado em pousio na entresafra; soja com *U. ruziziensis* entre culturas; e rotação com *U. ruziziensis*, com plantio de soja após crescimento da forrageira por 30 meses. O comportamento do K não-trocável foi analisado medindo-se o balanço entre K no solo e nas plantas de cobertura e analisando-se a composição mineralógica do solo. Determinou-se a eficiência das plantas de cobertura, em sucessão ou rotação, sobre a transformação não trocável em K trocável e a capacidade dessas plantas em atuar como biofertilizante para a soja, proporcionando fornecimento de potássio. Para a determinação da mineralogia do solo foi feita a separação das frações areia, silte e argila. Por meio da difração de raios-X foram identificados, em cada fração, quais minerais estavam presentes. Os minerais contidos na argila foram quantificados com a associação das técnicas de difração de raios-X, análise semiquantitativa de acordo com a área de pico de cada mineral e análises térmicas, quantitativas por meio de perda de peso dos minerais devido à temperatura, utilizando caulinita nativa determinada por métodos térmicos como padrão interno. A partir disso, foi possível medir cada mineral contido na argila e a mudança destes devido aos tratamentos. A rotação de culturas é um sistema de produção que proporciona maior disponibilidade de potássio para plantas de soja, bem como melhora a eficiência no uso de fertilizantes K. As plantas Ruzigrass têm maior capacidade de absorver K não-trocável do que soja e milho, embora a soja também use este K, ambas as culturas promovem mudanças na quantidade de vermiculita. O uso de forragem e soja em solo tropical, sem aplicação de K, é um dos agentes de intemperismo dos minerais potássicos presentes neste solo. Sistema e a exaustão de K devido a sucessivas culturas interferem na capacidade tampão de K deste solo a qual é coordenado pela adsorção de K na fração trocável e liberação pela fração não-trocável.

Palavra-chave: Potássio não trocável. potássio trocável. Intemperismo dos minerais potássicos em solos tropicais. Sistemas de produção soja-forrageira.

ABSTRACT

Some plants can utilize the non-exchangeable fraction of K and thus are not limited to the soluble and exchangeable fractions. In particular, cover plants are very efficient in recycling K and can absorb both the exchangeable and non-exchangeable forms. In the present study, the location of non-exchangeable K in the clay and silt fractions of a tropical soil after depletion by successive cropping with maize, soybean or ruzigrass (*Urochloa ruziziensis*) was identified, and the efficiency of non-exchangeable K in supplying the demand of these plants in soils with high concentrations of non-exchangeable K was determined in soybean-grass crop systems with succession or rotation. The study involved two experiments. In the first experiment, soybean, maize or ruzigrass was cultivated in pots for three successive cycles in soils with a high concentration of non-exchangeable K with and without potassium fertilization. The plants were removed from the pots at the moment of peak accumulation of K to determine the exchangeable and non-exchangeable K in the plants and the soil mineralogy. Second, a field experiment involving three production systems was carried out: soybean cultivation with fallow soil between crops; soybean with *U. ruziziensis* in the off-season; and rotation of *U. ruziziensis* with soybean planting after growth of the grass for 30 months. The behavior of non-exchangeable K was analyzed by measuring the balance between K in the soil and in the cover plants and by analyzing the soil mineralogical composition. The efficiency of the cover plants, in succession or rotation, in transforming non-exchangeable to exchangeable K and the ability of these plants to act as a bio-fertilizer for soybean by providing potassium nourishment were determined. For mineralogical analyses, the soil was separated into the sand, silt and clay fractions, and the minerals present in each fraction were identified by X-ray diffraction. The minerals present in the clay fraction were quantified by employing a combination of X-ray diffraction techniques, semiquantitative analysis of the peak area of each mineral, and thermogravimetric analyses using native kaolinite an internal standard. These analyses revealed the effects of each treatment on the content of each mineral in the clay fraction. The results showed that crop rotation improves K availability for soybean plants and the efficiency of K fertilizer utilization. Ruzigrass has a greater ability to absorb non-exchangeable K than soybean and maize, but both ruzigrass and soybean promote changes in the amount of vermiculite. The cultivation of forage grasses and soybean in tropical soil without application of K represents a weathering agent for the minerals present in this soil. Moreover, this cropping system and K exhaustion due to successive cultivation reduce the buffer capacity of K in this soil, which is regulated by the adsorption of K in the exchangeable fraction and release by the non-exchangeable fraction.

Keywords: Non-exchangeable potassium. Exchangeable potassium. Weathering of potassium minerals in tropical soils. Crop system, Soybean-forage.

SUMÁRIO

INTRODUCTION

Brazil is one of the largest producers of grains such as soybean and maize. These crops have high potential for potassium (K) extraction (OLTMANS; MALLARINO, 2014) because of their high requirements for K, behind only nitrogen (CALONEGO; FOLONI; ROSOLEM, 2005). This demand and weathering of the soil due to the tropical environment cause low soil levels of K (BENITES et al., 2010), resulting in high dependence on the import of this element, since the country produces only 10.8% of the demand (LOPES, 2005). To improve the efficiency of the use of K decrease dependence on K in the form of fertilizer (BENITES et al., 2010), many Brazilian regions have adopted strategies of grain and forage intercropping (PACHECO et al., 2008; BORGHI et al., 2012; CORREIA; GOMES, 2016; MATEUS et al., 2016) due to the ability of these plants to cycle K (GARCIA et al., 2008; ROSOLEM; VICENTINI; STEINER, 2012).

K is a constituent of some primary and secondary minerals and thus may be present in soils without anthropization (HUANG, 1977; THOMPSON; UKRAINCZYK, 2005). K is 7th in elemental abundance, representing approximately 2.1 to 2.3% of Earth's crust. Despite this abundance, large cropped areas are characterized by low or deficient levels of immediately available K (ZÖRB; SENBAYRAM; PEITER, 2014), particularly in highly weathered soils (MELO et al., 2003; KAMINSKI et al., 2010; ROSOLEM; STEINER, 2017). However, this shortfall may reflect a lack of availability of the nutrient to the plant rather than a deficiency of K in the soil.

The primary minerals with the highest presence of K are micas/illites and feldspars (THOMPSON; UKRAINCZYK, 2005), temperate environments have a greater presence and greater preservation of these minerals in the soils. Brazil's soils are more weathered with this cause loss of K of minerals. The main soil classes found in Brazil are Ferralsols / Oxisols, in about 39% of the area, Argisols with 24%, Entisols with 15% and Plintosols / Haplic Plinthosol in 6% of the territory (EMBRAPA 2006 / FAO-2014). This soil has the presence of source material (plintite) in the profile, low drainage and moderate deposition of clay along with the profile (EMBRAPA 2006), thus, although with less representativeness than the other three soil classes, it can be a source of K from minerals.

The arable layer of soil (0-20 cm) contains approximately 0.04-3% K, of which 90-98% is in structural form (ZÖRB; SENBAYRAM; PEITER, 2014) and is at least temporarily unavailable to plants. This structural K is found in primary and secondary minerals such as micas and feldspars (BORTOLUZZI et al., 2005). Feldspar tends to be more abundant in coarser fractions of the soil, such as sand and silt (HUANG, 1977). Although it is possible to find micas in both silt and clay, those with the highest potential to provide K are found in clay and are called illites (CHAVES et al., 2015). The main clay K-rich minerals are trioctahedral (biotite and phlogopite) and dioctahedral (muscovite) (KÄMPF et al., 2009; EGUCHI et al., 2015). Secondary minerals, such as vermiculite and smectite, can also be a source of K, and the presence of these minerals indicates the leaching of K from illites (MELO et al., 2009).

As a result of chemical, physical or biological weathering (MELO et al., 2009), these minerals can become important sources of K for plants. However, biochemical weathering caused by the action of plant roots, which is extremely important in soils with structural K, is more effective in removing this element from micas and/or illites (clay minerals 2:1) (BARRÉ; BERGER; VELDE, 2009). The K that becomes bio-available via the action of plant roots is referred to as non-exchangeable K, and in many cases, its extraction causes a change in mineral structure (HINSINGER; JAILLARD, 1993; GOMMERS et al., 2005; BARRÉ et al., 2007; MOTERLE et al., 2016). Consequently, K dynamics depend on the agricultural production system, which can potentially increase the efficiency of the use of this nutrient.

The term "non-exchangeable K" reflects its slow availability and the fact that it is not bound to cation exchange sites. This fraction can represent 1 to 10% of the K present in the tilling layer of the soil (COELHO, VERLENGIA, 1988). Micas/illites with greater weathering capacity, such as trioctahedral (biotite or phlogopite), can be considered direct sources of "non-exchangeable K" because they are more easily affected by biochemical weathering (MOJALLALI; WEED, 1978; BERTHELIN; LEYVAL, 1982; EGUCHI et al., 2015). However, in tropical regions, non-exchangeable K in soil can also come from secondary minerals such as vermiculite or smectite or can be adsorbed to soil colloids or the edges of clays under different binding forces (MOTERLE et al., 2016). This fraction cannot undergo cation exchange unless this binding is disrupted.

Even though this fraction of K is an important source of K for plants, the main parameter used to evaluate the availability of K in soil is the exchangeable fraction. However, even when K levels are below the critical soil level, plants frequently do not exhibit a response to the application of this nutrient (ZÖRB; SENBAYRAM; PEITER, 2014). The readily available fractions, represented by exchangeable K and soil solution K, represent 1-2% and 0.1-0.2%, respectively, of the K in the surface layer (0-20 cm) of the soil (ZÖRB; SENBAYRAM; PEITER, 2014).

The exchangeable K (adsorbed) and K of the soil solution are strongly influenced by other cations, such as Ca^{2+} and Mg^{2+} (ROSOLEM; CALONEGO; FOLONI, 2005). These cations bind negatively charged soil colloids. The attraction by colloids is determined by the valence and radius of hydration and decreases in the order $Ca^{2+} > Mg^{2+} > K^{+}$ (BENITES et al., 2010). The level of K in the soil solution depends on the levels of Ca^{2+} and Mg^{2+} and the soil-plant relationship because after K is taken up by the plant, replenishment by the solid phase occurs. As a result, exchangeable K is the main source of K in the soil solution. The soil solution contains dissolved organic and mineral compounds at concentrations of 1 to 20 mg L^{-1} and is the most readily available source of K (ERNANI et al., 2007; MELO et al., 2009).

Plant nutrition is related to the fractions of the K-solution, which is the form absorbed by plants, and the exchangeable fraction is responsible for buffering these fractions (MELO et al., 2003). This fraction is particularly important in soils with a high content of non-exchangeable K (WANG; HARRELL; BELL, 2004). However, Kaminski et al. (2007) observed that under successive cultivation, exchangeable K can remain at adequate levels even without the addition of K.

The soil-plant relationship depends on all fractions of K, which are interconnected. Consequently, knowledge of the environment and linked edaphic and climatic characteristics is needed, and situations and management practices should not be generalized (BENITES et al., 2010). According to Benites et al. (2010), the growth of cover plants, especially grasses, in the winter without the addition of fertilization will force the depletion of nutrients from their non-exchangeable forms to solution. This shift is the result of acidification of the rhizosphere due to the release of protons to chemical equilibrium and is responsible for the conversion of non-exchangeable K (HINSINGER; JAILLARD, 1993; GOMMERS et al., 2005). Garcia et al. (2008) observed that when *Urochloa brizantha* is intercropped with maize, conversion of non-exchangeable to exchangeable K occurs, allowing plant absorption of K in the non-exchangeable fraction (VAN RAIJ; QUAGGIO, 1984; ROSOLEM; VICENTINI; STEINER, 2012).

The use of this fraction by plants, including by soybean (MOJALLALI; WEED, 1978; ROSOLEM; BESSA; MARQUES, 1993), may be responsible for the poor relationship between the application of K fertilizer and increased productivity (ZÖRB; SENBAYRAM; PEITER, 2014). Thus, the properties of the soil have a greater long-term influence on the K supply than fertilization. As a result, non-exchangeable K can be classified as the K reserve (CURI et al., 2005; ROSOLEM; STEINER, 2017) and can be continuously supplied by potassium fertilization or leaching of K by cover crops (ROSOLEM et al., 2006; ROSOLEM; VICENTINI; STEINER, 2012; ROSOLEM; STEINER, 2017). In no-tillage systems, the K soil-plant relationship becomes more dynamic due to the ability of the cover crops to cycle K and supply the next crop. Grasses with deep root systems take up exchangeable and non-exchangeable K from the deeper layers of the soil profile, resulting in an increase in the exchangeable K in the upper layers of the soil (ECKERT, 1991; CRUSCIOL et al., 2015; ROSOLEM; STEINER, 2017). Cover crops can reduce soil and nutrient loss. Crop rotation under no-tillage reduces the K losses of the system from 4% to 23%, and the introduction of plants with high potential for K accumulation in the system results in lower K losses (CALONEGO; ROSOLEM, 2013). Consequently, crop systems in which different plants occupy the soil over the year can improve fertilizer efficiency and may increase soil fertility (CRUSCIOL; SORATTO, 2010).

In summary, soil, like Haplic Plinthosol (FAO – 2014), can be a natural source of K through its mineralogical matrix, and the biphasic dynamics of this element between the fractions are influenced by both the environment and the plant-environment relationship. In this context, we will explore what is known about the effect of cover crops on K management, based on the principle that cover crops can improve the efficiency of the use of K via cycling of this nutrient. Little is known about this effect in tropical soils when non-exchangeable K comes from potassium minerals or is bound under greater binding forces to the edges of clay. Moreover, the impact of the use of *Urochloa* as a cover plant on this fraction of K and mineral sources of K are unclear. It is also necessary to understand how to use cover plants to improve K use and increase productivity.

CHAPTER 1 - BIOAVAILABILITY OF EXCHANGEABLE K FOR SOYBEAN IN TROPICAL CROPPING SYSTEMS

ABSTRACT

Solution and exchangeable K (Ke) are not the only forms of K available to plants; non-exchangeable (Kne) can also be taken up. Crop rotation systems that include plant species efficient in acquiring this K fraction can impact K use efficiency in the system. This study aimed to assess how cropping systems differ in utilizing Kne. Six cropping systems in Central West Brazil were studied over three years: soybean (Glycine max L. Merr.) with a fallow offseason; soybean intercropped with ruzigrass (*Urochloa ruziziensis*) in the offseason; and ruzigrass without soybean, all with and without K fertilization. Early and late cultivars of soybean were used. Soybean grain yields were not decreased in the absence of K fertilizer when soybean was cropped in rotation with ruzigrass. K cycling was improved when ruzigrass was included in rotation with soybean because ruzigrass can explore soil Kne, which is then washed back in available forms such as soil solution K and Kne. The late cultivar took up more Kne than the early cultivar. No response to K was observed for soybean cropped in rotation with ruzigrass, indicating that this practice can lead to soil K depletion in the absence of fertilizer application. Therefore, even though an immediate response was not observed, K fertilizer must be used in this system to avoid exploitation of Kne.

Keywords: Rotation crop system. Nutrient cycling. Non-Exchangeable K. Exchangeable K.

1.1 INTRODUCTION

The Brazilian Cerrado is widely occupied by grain crops, primarily soybean. Soybean-maize succession is the main cropping system in this region (MATEUS et al., 2016; FRANCISCO and CÂMARA, 2013), although large areas are also grown with soybean or maize in rotation with forage (PACHECO et al., 2008; FRANCISCO; CÂMARA, 2013; CRUSCIOL et al., 2015; CORREIA; GOMES, 2016; MATEUS et al., 2016). The use of forage in this type of succession or intercropping can be important for both environmental management, by improving fertilizer use efficiency and soil conservation, and cash income from pasture grazing (CRUSCIOL et al., 2010, 2013, 2016; BENITES et al., 2010; CRUSCIOL; SORATTO 2010; ALMEIDA; ROSOLEM, 2016; ALMEIDA et al., 2018; TANAKA et al., 2019).

Rainfall in the Brazilian Cerrado is concentrated in the spring and summer, and there are long periods in fall and winter without precipitation (BORGHI et al., 2013; CRUSCIOL et al., 2014; MATEUS et al., 2016). As a result, the second crop faces water restrictions. The use of early-cycle soybean cultivars provides better conditions for succession cultivation (FORNAZZA et al., 2018). Continuous occupation of the soil by cropping with different species throughout the year improves fertilizer efficiency and increases soil fertility (BENITES et al., 2010; CRUSCIOL; SORATTO 2010; CALONEGO et al., 2011).

Grass species such as brachiaria (*Urochloa* spp.) from the African continent have high adaptability to the Brazilian Cerrado region and have buches or decumbent growth (BRAZ et al., 2004; CRUSCIOL et al., 2010, 2013, 2015b; PACHECO et al., 2011), with deep root systems. These grasses provide large amounts of biomass in the form of ground cover and improve grazing (CRUSCIOL et al., 2013, 2016). Brachiaria and millet are intensively used in rotation with main crops and typically can recycle several amount of K (BENITES et al., 2010).

Ruzigrass (*Urochloa ruziziensis*) is a forage grass with great adaptability to crop succession or rotation systems and good K cycling potential. Studies have confirmed that rotation with ruzigrass increases soybean grain yield (VAN RAIJ; QUAGGIO, 1984; CRUSCIOL et al., 2012; ROSOLEM et al., 2012; CALONEGO; ROSOLEM, 2013; ALMEIDA et al., 2018; TANAKA et al., 2019), although there is a risk of unavailability of P for soybean, which can reduce yields (ALMEIDA et al., 2018).

Ruzigrass improves the cycling and efficiency of K use in the production system due to the high accumulation of K in plant tissues, its ability to absorb nutrients at greater depths and the exudation of weak acids capable of removing K tightly bound to clays. (ERNANI et al., 2007; GARCIA et al., 2008; VOLF et al., 2018). Little of this absorbed K is exported (MARTINS et al., 2015; VILELA et al., 2007), and it is quickly returned to the soil after death or senescence of the forage leaves, thus promoting a high rate of recycling of this nutrient (MIELNICZUK, 2005; GIACOMINI et al., 2003; ROSOLEM et al., 2003; CALONEGO et al., 2005; GARCIA et al., 2008). Because these cover crops can improve the efficiency and use of K, in many cases, K application does not cause an increase in crop yield (ZÖRB t al.; 2014), especially for soybean, which can also harness Kne, albeit with impaired productivity (ROSOLEM et al., 1988).

Soybean cultivars belonging to different relative maturity groups tend to absorb different amounts of soil nutrients (PEDRINHO JUNIOR et al., 2004; OLIVEIRA JUNIOR et al., 2014; ARAUJO, 2018). The absorption of higher amounts of K by plants may force the depletion of Ke in the soil, resulting in the use of Kne (KAMINSKI et al., 2007). As a result, late cultivars may absorb more Ke and Kne.

Cerrado soils, with presence of non-exchangeable K, can provide K for soybean plants to eliminate the need for K addition, and the use of ruzigrass as a cover crop can improve the availability of this element. The objective of this study was to evaluate the effect of cultivation systems using ruzigrass in rotation or succession with soybean on the soil availability of K for soybean and consequently soybean yield.

1.2 MATERIAL AND METHODS

The field experiment was carried out from 2015 to 2018 in Nova Xavantina, State of Mato Grosso, Midwest Brazil (14º63'99 "S and 52º14'81" W, 300 m asl). The soil is classified as a Haplic Plinthosol, with 67% kaolinite, 3.4% hydroxyl interlayer vermiculite (HIV), 11.6% illite and 18% goethite in the clay fraction. The X-ray diffraction pattern showed a peak at 0.154 nm in the 060 (hkl) plane, indicating that this illite is trioctahedral. According to the Köppen classification, the climate is Aw, with a wet summer and dry winter. The amount of rainfall and the mean, maximum, and minimum temperatures during the experiment are shown in Figure 1. Before the start of the experiment, the soil had been managed for 5 years under NT (no tillage), with cropping of soybean in rotation with ruzigrass in the offseason. In November 2015, soil samples were taken for chemical and physical analysis as described by Raij et al. (2001) (Table 1).

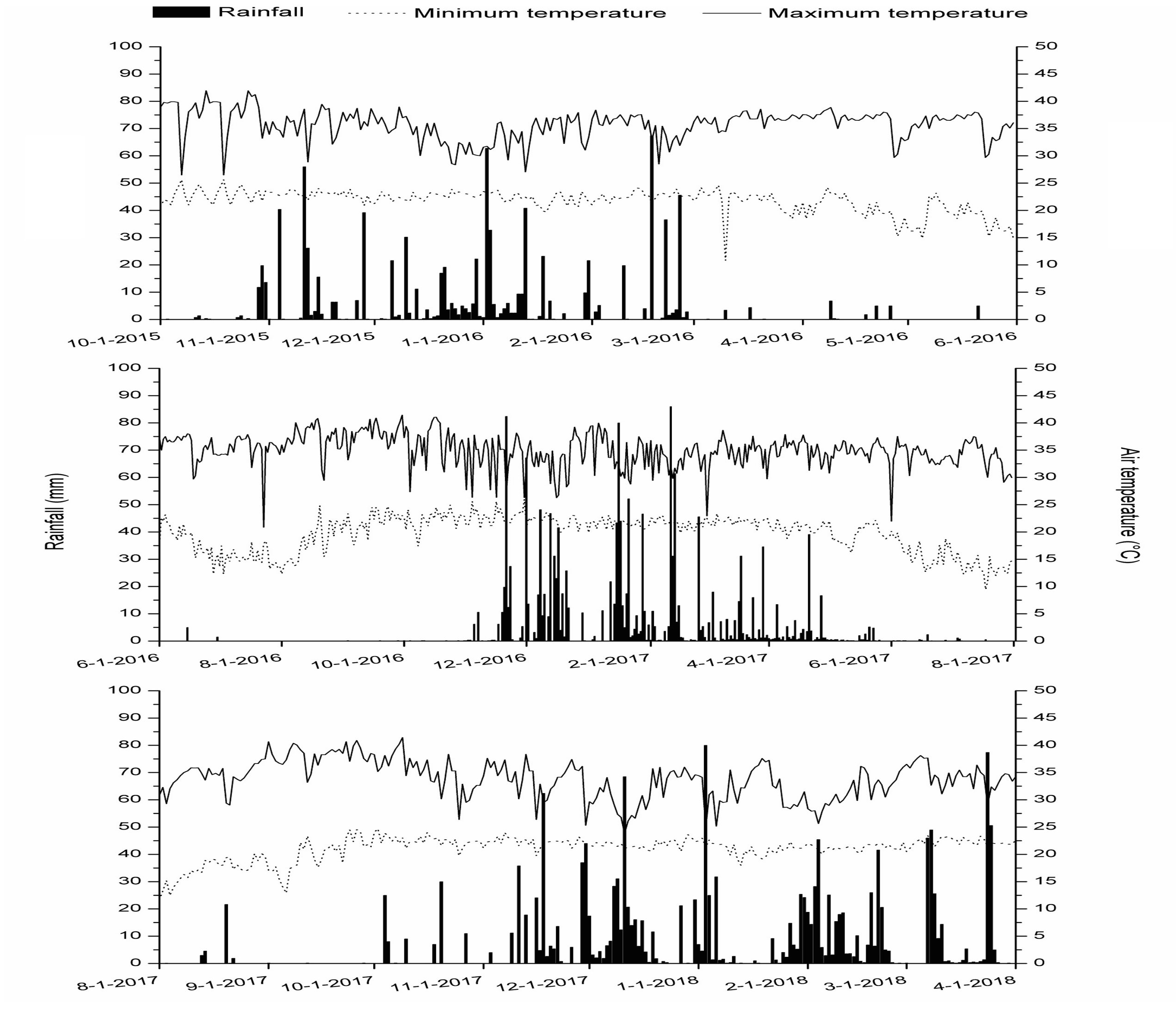
Table 1 – Selected soil chemical characteristics (Raij et al. 2001) and particle size distribution

Soils	pH[1]	P[2]	Exc K[3]	Non-exc K[4]	Ca	Mg	M.O.	Clay	Silt	Sand
		(mg dm^{-3})	(mg kg^{-1})		(cmolc dm^{-3})		(g dm^{-3})	(g kg^{-1})		
0-10	5.2	16.8	146.2	61.5	3.12	1.14	19.8	180	100	720
10-	4.4	4.4	43.0	95.4	1.46	0.77	10.6	230	125	645
20-	4.0	1.3	23.5	128.0	0.81	0.53	8.8	280	100	620
40-	3.9	0.8	19.8	178.2	0.70	0.45	7.5	370	90	540

[1] In $CaCl_2$ [2.3] extracted with resin [4] extracted with boiling HNO_3

The experimental design was a 6 X 2 factorial (six treatments and two soybean cultivars, early and late maturation groups) in completely randomized blocks and four replicates. The treatments are described in Table 2. The treatments consisted of ruzigrass in rotation with soybean and succession crop systems (soybean in the spring/summer and ruzigrass or fallow in the fall/winter). All systems included treatments with and without K fertilization. In the rotation treatments (ruzigrass/soybean), ruzigrass was overseeded in February 2015 and remained until October 2017 (30 months), when soybean was planted in all plots. In the succession treatments, ruzigrass seeds were overseeded in February each year in phenological stage R5.5 (FEHR; CAVINESS, 1977) according to the methodology of Pacheco et al. (2008).

Figure 1 - Rainfall and maximum and minimum temperatures during the study period and in the long-term at Nova Xavantina, Mato Grosso State, Brazil.



After the soybean harvest, herbicide was applied to the fallow plots for weed control. These plots were kept free of any plant throughout the winter season (Figure 2). Glyphosate (1.8 kg acid equivalent ha^{-1}) was applied using a spray volume of 250 L ha^{-1} 15 days before soybean sowing. The basic fertilization in the sowing furrows consisted of 100 kg ha^{-1} of P_2O_5 as triple superphosphate. Nitrogen was supplied via biological fixation by inoculating the soybean seeds with 6 mL kg^{-1} of *Bradyrhizobium japonicum* (SEMIA 5079-CPAC 15 and SEMIA 5080-CPAC7). The early cultivar was M-soy 8210 IPROâ at a population of 333,000 plants ha^{-1}, and the late cultivar was M-Soy 8644 IPROâ with 200,000 plants ha^{-1}, at a row spacing of 0.45 cm. Ruzigrass was overseeded at a density of 8.0 kg ha^{-1} of pure live seeds.

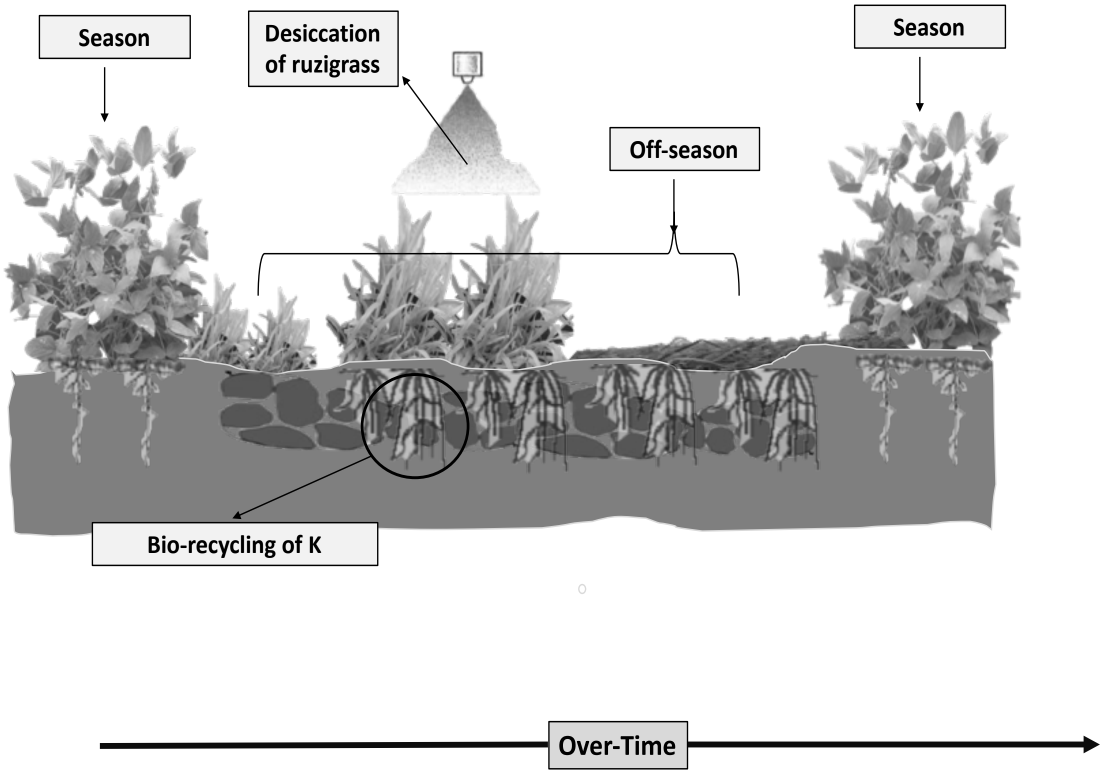
Table 2 - Description of treatments

Production Systems	Fall-winter 2015	Spring-summer 2015-2016	Fall-winter 2016	Spring-summer 2016-2017	Fall-winter 2017	Spring-summer 2017-2018
Early cultivars						
Soybean/Ruzigrass+K[1]	Ruzigrass	Early cultivars	Ruzigrass	Early cultivars	Ruzigrass	Early cultivars
Soybean/Ruzigrass[2]	Ruzigrass	Early cultivars	Ruzigrass	Early cultivars	Ruzigrass	Early cultivars
Soybean/Fallow+ K	w/o[2] plants	Early cultivars	w/o plants	Early cultivars	w/o plants	Early cultivars
Soybean/Fallow	w/o plants	Early cultivars	w/o plants	Early cultivars	w/o plants	Early cultivars
Ruzigrass/soybean+ K	Ruzigrass	Ruzigrass	Ruzigrass	ruzigrass	Ruzigrass	Early cultivars
Ruzigrass/soybean	Ruzigrass	Ruzigrass	Ruzigrass	ruzigrass	Ruzigrass	Early cultivars
Late cultivars						
Soybean/Ruzigrass+ K	Ruzigrass	Late cultivars	Ruzigrass	Late cultivars	Ruzigrass	Late cultivars
Soybean/Ruzigrass	Ruzigrass	Late cultivars	Ruzigrass	Late cultivars	Ruzigrass	Late cultivars
Soybean/Fallow+ K	w/o plants	Late cultivars	w/o plants	Late cultivars	w/o plants	Late cultivars
Soybean/Fallow	w/o plants	Late cultivars	w/o plants	Late cultivars	w/o plants	Late cultivars
Ruzigrass/soybean+ K	Ruzigrass	Ruzigrass	Ruzigrass	Ruzigrass	Ruzigrass	Late cultivars
Ruzigrass/soybean	Ruzigrass	Ruzigrass	Ruzigrass	Ruzigrass	Ruzigrass	Late cultivars

[1] with K application at 100 kg K_2O ha^{-1}. [2] no K applied

In November 2017, five soil subsamples were collected per plot with a 5-cm-diameter auger from depths of 00-10, 10-20, 20-40 and 40-60 cm. Samples from the same depth were combined in one composite sample per plot. In February 2018, when soybean reached phenological stage R6 (K accumulation peak) (OLIVEIRA JUNIOR et al. 2014), another set of soil samples was taken. The soil samples were air-dried, ground to pass through a 2-mm mesh screen, and analyzed as follows: Ke was extracted with ion exchange resin (RAIJ et al., 2001), and Kne was obtained from the difference between the amount of K extracted with boiling 1.0 mol l^{-1} HNO_3 and Ke (KNUDSEN et al., 1982).

Figure 2 - Representative design of soybean crop in the season and cover crops in the off-season.



Soon after soil sampling, the standing vegetation was collected by cutting the plants close to the ground, and samples were taken with the aid of a 0.25-m^2 metal square. Samples of soybean plants were taken in February at the same time points as soil collection from two lines along 1-m-long rows. Biomass was measured for the uptake of K after each sampling of soil and plants. Soybean leaves were collected for nutrient concentration analysis when stage R2 was reached (FEHR; CAVINESS, 1977); 30 leaves of the third node from the top were collected per plot (AMBROSANO et al., 1997). The samples were dried to constant weight (ROSOLEM et al., 1993) in a forced circulation oven at 60 °C for 72 hours, weighed, ground and wet digested with nitroperchloric acid to determine the K concentration (MALAVOLTA et al., 1997).

The K output from the systems, K balance, and the contribution of Kne to plant uptake were calculated using the following equations.

$$Bal = (Ke_{nov\text{-}2017} + K_{fert} + K_{covplant}) - Ke_{Fev\ 2018} - K_{soy\ plant} \quad (1)$$

where $K_{enov\text{-}2017}$ is Ke in the soil in November 2017, K_{fert} is the amount of K applied, $K_{covplant}$ is the amount of K in cover crop plants, $Ke_{Fev\text{-}2018}$ is Ke in soil in February 2018, and $K_{soy\ plant}$ is the K exported in the harvested soybean biomass.

$$Kne\ contribution\ (\%) = (Budget\ balance * K_{soy\ plant}) *100. \quad (2)$$

where Kne contribution is the contribution of Kne to the total K uptake by soybean, Budget balance is the K balance determined from Equation 1, and $K_{soy\ plant}$ is the K uptake by soybean.

Soybean was harvested manually, the grain weight was determined, and the yield was calculated at a moisture content of 130 g kg^{-1}. The weight of 100 grains was determined from eight randomly collected samples per plot.

Data from each year were analyzed separately. The plant and yield data were subjected to analysis of variance (ANOVA) using the statistical package SISVAR (FERREIRA 2011) considering a 2 × 6 factorial in a randomized complete block design with four replications, and means were compared using Fisher's protected LSD test at a probability level of 0.05.

1.3 RESULTS

1.3.1 Yield of dry matter and K absorbed in the 2017-2018 season

The dry matter yield of cover crops was affected by the production system, while the production of dry matter from soybeans was affected by both factors (production system and cultivar) (Table 3).

The production of cover crops was highest in the grass rotation system with soybeans, i.e. ruzigrass / soybean (Rz / Sy), while the lowest yields were obtained in the system containing only residues of the previous cultivation with soybeans and without cover plants in the off-season, i.e. soybean / fallow (Sy / Flw). The application of K did not influence the production of cover plant dry matter. The production of the rotation system (Rz / Sy system) was approximately 63% greater than that of the system with ruzigrass grown in succession to soy (soybean / ruzigrass system - Sy / Rz).

The soybean dry matter yield was higher for the late cultivar than for the early cultivar in all production systems (Table 3) except the RZ / Sy system, in which there was no difference in soybean dry matter yield between cultivars.

The production of dry matter of the early cultivar was highest in the system with cover crops in the off-season (Sy / Rz + K; Rz / Sy; Rz / Sy + K) and lowest in the fallow system (Sy / Flw). K application increased the soybean dry matter production in the fallow (Sy / Flw) and succession systems (Sy / Rz), but no increase was observed in the rotation system (Rz / Sy). Applying K in the Sy / Flw system resulted in the same soybean dry matter production as in the Sy / Rz system without application of K. The rotation system (Rz / Sy) without application of K increased the dry matter mass of the plants of the early cultivar by approximately 13.5% and 26.4% compared with the fallow system (Sy / Flw) with and without addition of K, respectively.

By contrast, the dry matter yield of the late cultivar was highest in the fallow system (Sy / Flw) and lowest in the Rz / Sy system. The use of K only influenced dry matter production in the Sy / Flw system, with an increase of approximately 10%. Dry matter production decreased severely in the rotation system (Rz / Sy) by approximately 32% compared with the fallow system (Sy / Flw). Under the succession system (Sy / Rz), a decrease in dry matter production of less than 14% was observed.

Similar to the dry matter results, the accumulation of K in the cover crops was only affected by production system, whereas the accumulation of K in soybean plants was affected by both factors (production system and cultivar) (Table 3).

The accumulation (content) of K in the cover plants followed the same trend as the accumulation of dry matter. Specifically, there was no influence of the application of K, and the accumulation was greatest in the cover plants in the rotation system (Rz / Sy) and lowest in the plants in the fallow system. Compared with the Rz / Sy system, K accumulation in the cover plant biomass was approximately 25% higher in the Sy / Rz system.

With little variation, the trend of K accumulation in soybean cultivars followed the same pattern as dry matter accumulation. Thus, with the exception of the Rz / Sy system, in which there was no difference between cultivars, the accumulation of K was greater in the late cultivar in all systems (Table 3).

The accumulation of K by plants of the early cultivar was highest in the Rz / Sy, Rz / Sy + K, and Sy / Rz systems but lowest in the Sy / Flw and Sy / Rz systems. The addition of K only increased K accumulation in the early cultivar in the succession system (Sy / Rz + K). Even with the addition of K, K accumulation in the fallow system (Sy / Flw) did not exceed that in the Sy / Rz system without K. The ruzigrass / soybean rotation increased the accumulation of K in the early cultivar by approximately 25% compared with the system without cover plants (Sy / Flw).

In the late cultivar, the highest K accumulation occurred in the Sy / Flw + K system, with a severe decrease of 19% in the system with the lowest K accumulation, Rz / Sy. The application of K increased K accumulation when this cultivar was grown in the Rz / Sy and Sy / Flw systems.

Table 3 - Dry matter (Mg ha^{-1}) of cover crop and soybean, and K content in plants of cover crops and soybean, as affected by crop systems with cover crop species with or without K. Data were collected at the end of the off-season of 2017 (November) and season 2018 (February – peak absorption of K) in Nova Xavantina, State of Mato Grosso, Brazil

Production Systems [1]	Dry matter Mg ha^{-1}					
	Off-season – Cover crops[2]		Average	Season - Soybean crops[3]		Average
	Early cultivars	Late cultivars		Early cultivars	Late cultivars	
Soybean/Ruzigrass	5.50	5.15	5.30 b	5.90 Bb	7.60 Ac	6.80
Soybean/Ruzigrass + K	5.75	5.10	5.50 b	6.50 Ba	7.40 Ac	6.90
Soybean/Fallow	1.50	1.50	1.48 c	5.30 Bc	8.20 Ab	7.50
Soybean/Fallow+K	1.40	1.60	1.50 c	5.90 Bb	9.00 Aa	6.70
Ruzigrass/Soybean	8.70	8.40	8.55 a	6.70 Aa	6.30 Ad	6.40
Ruzigrass/Soybean+K	8.80	8.60	8.70 a	6.60 Aa	6.70 Ad	6.20
Average	5.30A	4.95A		6.15	7.50	
ANOVA (Pr > *F*)						
Treatments		< 0.0001			< 0.0001	
Cultivation		0.0889			< 0.0001	
Treatments x Cultivation		0.0886			< 0.0001	
			K uptake. kg ha^{-1}			
Soybean/Ruzigrass	33.50	36.20	35.00 b	114.00 Bb	147.00 Acd	130.00
Soybean/Ruzigrass + K	31.80	35.80	34.00 b	138.00 Ba	154.00 Abc	146.00
Soybean/Fallow	6.20	5.90	6.10 c	113.00 Bb	153.00 Abc	133.00
Soybean/Fallow+K	6.00	6.50	6.30 c	115.00 Bb	169.00 Aa	142.00
Ruzigrass/Soybean	42.80	40.50	41.65 a	145.00 Aa	142.00 Ad	144.00
Ruzigrass/Soybean+K	47.00	43.60	45.00 a	121.00 Bb	160.00 Aab	140.00
Average	27.80	27.60		124.00	154.00	
ANOVA (Pr > *F*)						
Treatments		< 0.0001			0.0019	
Cultivation		0.7494			<0.0001	
Treatments x Cultivation		0.3351			<0.0001	

[1] Soybean/Ruzigrass - in succession Soybean in summer and ruzigrass (*Urochloa ruziziensis*) a grown as cover crop in the off-season. Soybean/Fallow - Soybean in summer without plants as cover crop in the off-season. Ruzigrass/Soybean - Rotation Ruzigrass during 3 years with soy after.

The K2O was application on September of each year

[2] The cover crops will remain over time in the off-season (autumn/winter) and sampling was done in November at the time of desiccation of the cover plants.

[3] Soybean plants were sampled at the time of nutrient absorption peak phenolic stage R5.5.

Means followed by different lowercase letters in columns and uppercase in lines are significantly different. LSD – Least Significant Difference (P<0.05)

1.3.2 Leaf K concentration and soybean yield

In the 2017-2018 season, there was an interaction between factors (cultivar and production systems). With the exception of the Sy / Flw + K and Rz / Sy + K systems, in which there was no difference between cultivars, the K concentration was higher in the early cultivar (Table 4).

The concentrations of K in the early cultivar were highest in the Sy / Rz + K, Rz / Sy and Rz / Sy + K production systems but lowest in the Sy / Rz system. The application of K increased the concentration of K in the Sy / Rz system but not in the Rz / Sy system. The concentration of K in the leaves of the late cultivar also varied. The concentration of K in leaves was highest in the Rz / Sy + K system but lowest in the Sy / Rz and Sy / Flw systems. In all systems, the concentration of K was higher with the application of K than without. The concentration of K was similar in the Rz / Sy and Sy / Flw + K systems (Table 4).

Table 4 - Soybean leaves K concentration as a function of productions systems during season 2016-2017 e 2017-2018 in soybeans cultivars Early and Late in Nova Xavantina, State of Mato Grosso, Brazil

Production Systems [1]	K concentration $g\ kg^{-1}$		
	Early cultivars	Late cultivars	Average
	Crop 2016.2017		
Soybean/Ruzigrass	17.10	16.70	17.0 a
Soybean/Ruzigrass + K	15.80	16.30	16.5 ab
Soybean/Fallow	15.50	15.90	15.4 c
Soybean/Fallow+K	14.50	17.20	15.7 bc
Ruzigrass/Soybean	-------	-------	
Ruzigrass/Soybean+K	-------	-------	
Average	15.75 B	16.5 A	
ANOVA (Pr > *F*)			
Treatments		0.0235	
Cultivation		0.0478	
Treatments x Cultivation		0.1623	
	Crop 2017.2018		
Soybean/Ruzigrass	12.50 Ac	9.30 Bc	10.90
Soybean/Ruzigrass + K2O	14.70 Aa	12.35 Bb	13.56
Soybean/Fallow	13.20 Abc	9.90 Bc	11.60
Soybean/Fallow+K2O	14.20 Aab	13.30 Ab	13.75
Ruzigrass/Soybean	14.40 Aa	12.45 Bb	13.42
Ruzigrass/Soybean+K2O	14.50 Aa	14.62 Aa	14.60
Average	14.0	12.0	
ANOVA (Pr > *F*)			
Treatments		< 0.0001	
Cultivation		< 0.0001	
Treatments x Cultivation		0.0001	

[1] Soybean/Ruzigrass - in succession Soybean in summer and ruzigrass (*Urochloa ruziziensis*) a grown as cover crop in the off-season. Soybean/Fallow - Soybean in summer without plants as cover crop in the off-season. Ruzigrass/Soybean - Rotation Ruzigrass during 3 years with soy after.

Means followed by different lowercase letters in lines and uppercase in columns are significantl different. LSD – Least Significant Difference (P<0.05).

In the 2015-2016 and 2016-2017 seasons, soybean grain yield was influenced by the interaction of factors (cultivar and production systems). However, in the 2017-2018 season, there was no influence of production system or cultivar (Table 5).

In the 2015-2016 season, the early cultivar had higher grain productivity than the late cultivar except in the Sy / Rz + K system, where the grain productivities of the two cultivars were similar.

The yield of the early cultivar was highest in the Sy / Rz + K system. The application of K to the succession system (Sy / Rz) resulted in increases of 340 kg ha-1 (12%) and 480 kg ha-1 (18%) compared with the system without cover plants in the offseason, even when this latter system received application of K.

For the late cultivar, the yield was highest in the production systems with cover crops in the offseason, i.e. Sy / Rz and Sy / Rz + K, and lowest in the Sy / Flw system. A response to K application was observed only when there were no cover plants in the offseason (Table 5).

In the 2016-2017 crop, compared with the late cultivar, the productivity of the early cultivar was lower in the Sy / Flw + K production system, similar in the Sy / Rz system, and greater in all other systems.

The yield of the early cultivar was highest in the Sy / Rz + K and Sy / Flw systems. The addition of K to the ruzigrass system in succession increased the yield by 9% compared with no application of K. For the late cultivar, productivity was highest in the Sy / Flw + K system but lowest in the Sy / Flw and Sy / Rz systems (Table 5). Productivity increased with the addition of K in the Sy / Flw system but not the Sy / Rz system.

In the 2015-2016 and 2017-2018 seasons, the 100 grain weight was affected by the interaction between the factors; however, in the 2016-2017 harvest, only the production system affected the 100 grain weight (Table 5).

In the 2015-2016 season, the 100 grain weight was higher for the late cultivar than the early cultivar, regardless of the cultivation system. With respect to the production system, the 100 grain weight of the early cultivar was highest in the Sy / Rz system and smallest in the Sy / Flw system. The addition of K did not influence the 100 grain weight. By contrast, the 100 grain weight of the late cultivar was higher when K was applied and was highest in the Sy / Rz + K system and lowest in the Sy / Flw system. The 100 grain weights in the Sy / Rz and Sy / Flw + K systems were similar, and the addition of K to the Sy / Flw system did not increase the 100 grain weight compared with the Sy / Rz system.

In the 2016-2017 season, the 100 grain weight was higher in the Sy / Rz + K and Sy / Flw + K systems than in the Sy / Rz and Sy / Flw systems. For the late cultivar, the application of K increased the 100 grain weight.

In the 2017-2018 season, the 100 grain weight of the early cultivar was higher than that of the late cultivar in all production systems. Moreover, the 100 grain weight of the early cultivar was higher in the rotation production system (Rz / Sy + K) than in the Sy / Rz and Sy / Flw systems, with a greater 100 grain weight in the Sy / Rz system than in the Sy / Flw system.

The late cultivar produced the largest 100 grain weight in the Rz / Sy + K production system, followed successively by the Rz / Sy system and then all other systems. The application of K (Rz / Sy + K) increased the 100 grain weight by 10% compared with both the Sy / Rz and Sy / Flw systems, even when K was applied to the latter systems.

Table 5 - Soybean grain yield, and 100-grain weight in function of productions systems and soybeans cultivars Early or Late, in seasons 2015-2016, 2016-2017 e 2017-2018, in Nova Xavantina, State of Mato Grosso, Brazil

Production Systems[1]	Soy Yield (Mg ha^{-1})			100 - grain weight (g)		
	Early cultivars	Late cultivars	Average	Early cultivars	Late	Average
			Crop 2015.2016			
Soybean/Ruzigrass	2.84 Ab	2.95 Aa	2.90	10.30 Ba	12.10 Ab	11.2
Soybean/Ruzigrass + K	3.18 Ba	3.00 Aa	3.10	10.10 Bab	12.60 Aa	11.3
Soybean/Fallow	2.70 Ab	2.60 Ac	2.60	10.30 Bb	11.40 Ac	10.7
Soybean/Fallow+K	2.75 Ab	2.80 Ab	2.80	9.90 Bb	12.10 Ab	11.0
Ruzigrass/Soybean	-------	-------	-------	-------	-------	-------
Ruzigrass/Soybean+K	-------	-------	-------	-------	-------	-------
Average	2.80	2.90		10.90	12.00	
ANOVA (Pr > *F*)						
Treatments		<0.0001			<0.0001	
Cultivation		0.2557			<0.0001	
Treatments x Cultivation		0.0003			<0.0001	
			Crop 2016.2017			
Soybean/Ruzigrass	3.30 Ab	3.10 Ab	2.99	15.90	16.60	16.3 b
Soybean/Ruzigrass + K	3.60 Aa	3.30 Bab	3.45	16.90	16.90	16.9 a
Soybean/Fallow	3.70 Aa	3.40 Bb	3.60	16.40	16.40	16.4 b
Soybean/Fallow+K	3.50 Bab	3.80 Aa	3.70	16.70	17.00	16.8 a
Ruzigrass/Soybean	-------	-------	-------	-------	-------	-------
Ruzigrass/Soybean+K	-------	-------	-------	-------	-------	-------
Average	3.50	3.40		16.50	16.70	
ANOVA (Pr > *F*)						
Treatments		0.0014			0.0210	
Cultivation		0.0407			0.0647	
Treatments x Cultivation		0.0016			0.2161	
			Crop 2017.2018			
Soybean/Ruzigrass	3.60	3.65	3.59	16.60 Ac	13.80 Bd	15.21
Soybean/Ruzigrass + K	3.70	3.80	3.95	17.17 Aab	14.50 Bc	15.80
Soybean/Fallow	3.75	3.90	3.83	16.30 Ad	14.40 Bc	15.30
Soybean/Fallow+K2O	3.90	3.80	3.85	17.30 Aab	14.50 Bc	15.90
Ruzigrass/Soybean	3.65	4.00	3.87	16.90 Abc	15.00 Bb	15.90
Ruzigrass/Soybean+K	3.65	4.20	3.91	17.53 Aa	15.90 Ba	16.73
Average	3.75	3.90		16.93	14.70	
ANOVA (Pr > *F*)						
Treatments		0.2340			<0.0001	
Cultivation		0.0858			<0.0001	
Treatments x Cultivation		0.3314			0.0015	

[1] Soybean/Ruzigrass - in succession Soybean in summer and ruzigrass (*Urochloa ruziziensis*) a grown as cover crop in the off-season. Soybean/Fallow - Soybean in summer without plants as cover crop in the off-season. Ruzigrass/Soybean - Rotation Ruzigrass during 3 years with soybean after.

Means followed by different lowercase letters in lines and uppercase in columns are significantly different. LSD – Least Significant Difference (P<0.05).

1.3.3 K balance in the soil-plant system and the contribution of non-exchangeable K

Both the production system and soybean cultivar influenced the K balance between soil and plant absorption as well as the percentage contribution of nonexchangeable K to the K accumulated by the soybean plants (Figure 3).

With the exception of the Rz / Sy system, the K balance of the early cultivar was higher than that of the late cultivar. However, only systems with K application had a positive K balance. The late cultivar showed a positive K balance only when cultivated in the Sy / RZ + K and Rz / Sy + K production systems (Figure 3a).

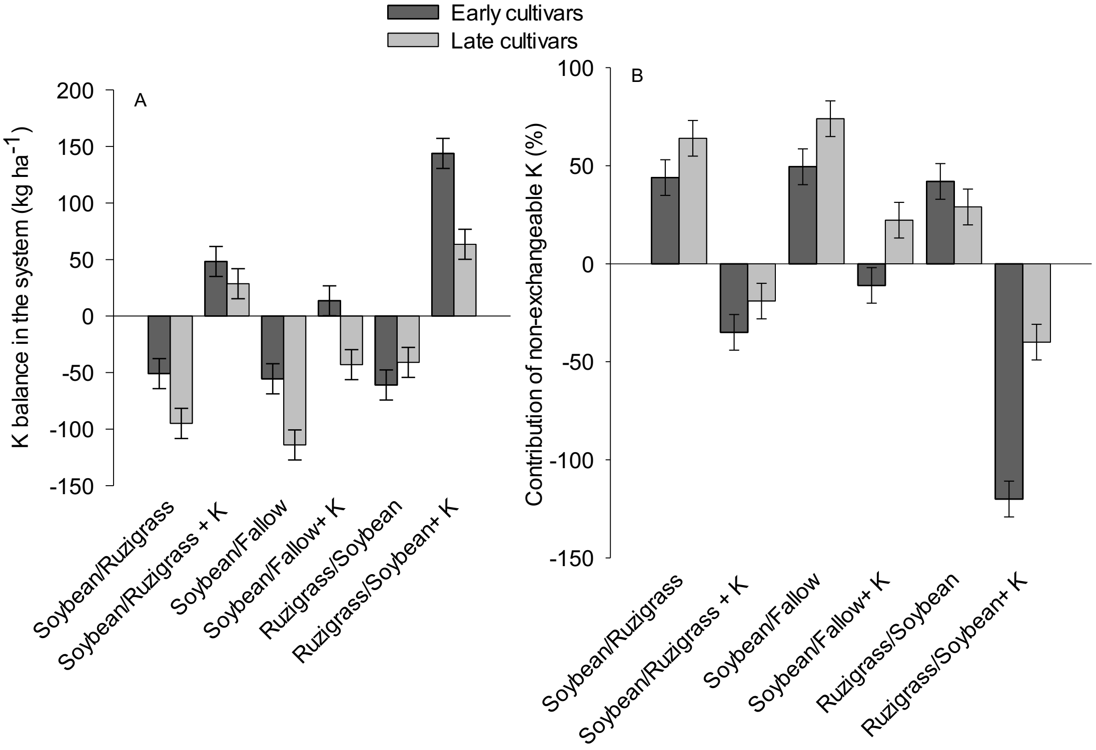
The K balance of the early cultivar was negative and similar in the Sy / Rz, Sy / Flw and Rz / Sy production systems. However, when cultivated in these systems with the application of K, the K balance became positive and was highest in the Rz / Sy + K system, followed successively by the Sy / Rz + K system and Sy / Flw + K system. The combination of K application and cover plants provided a positive K balance that was superior to that in the fallow system plus K.

In the late cultivar, the K balance was most positive in the Rz / Sy + K production system, followed by the Sy / Rz + K system. Due to greater K output from the soil, the K balance of the late cultivar was most negative in the Sy / Flw system, followed successively by the Sy / Rz and Rz / Sy systems (Figure 3a).

The contribution of nonexchangeable K was the same for the early and late cultivars in the Rz / Sy system, whereas in all other systems the contribution of nonexchangeable K was higher in the late cultivar than in the early cultivar (Figure 3b).

For the early cultivar, similar contributions of nonexchangeable K were observed for the production systems without added K (Sy / Rz, Sy / Flw and Rz / Sy); by contrast, in the systems with added K (Sy / Rz + K, Sy / Flw + K and Rz / Sy + K), there was no contribution of nonexchangeable K. Similar results were obtained for the late cultivar, except that a contribution of nonexchangeable K was also observed in the Sy / Flw + K production system. The largest contribution of nonexchangeable K was observed in the Sy / Rz and Sy / Flw production systems (Figure 3b).

Figure 3 - Potassium balance in the soil – plant system [(Bal = ($Ke_{nov\text{-}2017}$ +K_{fert} + $K_{cov\ plant}$) – $Ke_{Fev\ 2018}$) - $K_{soy\ plant}$] of exchangeable K to a depth of 0.6 m, plus relation to total K applied and K in cover crop plant in off-season 2017 (A) contribution of the non-exchangeable K [$K_{ne\ contribution}$ (%) = ($Budget_{balance}$ * $K_{soy\ plant}$) *100] in the nutrition of soybean plants in season 2017-2018, due to the systems of production and soybean cultivars Early or Late. Vertical bars show the LSD – Least Significant Difference (P<0.05).

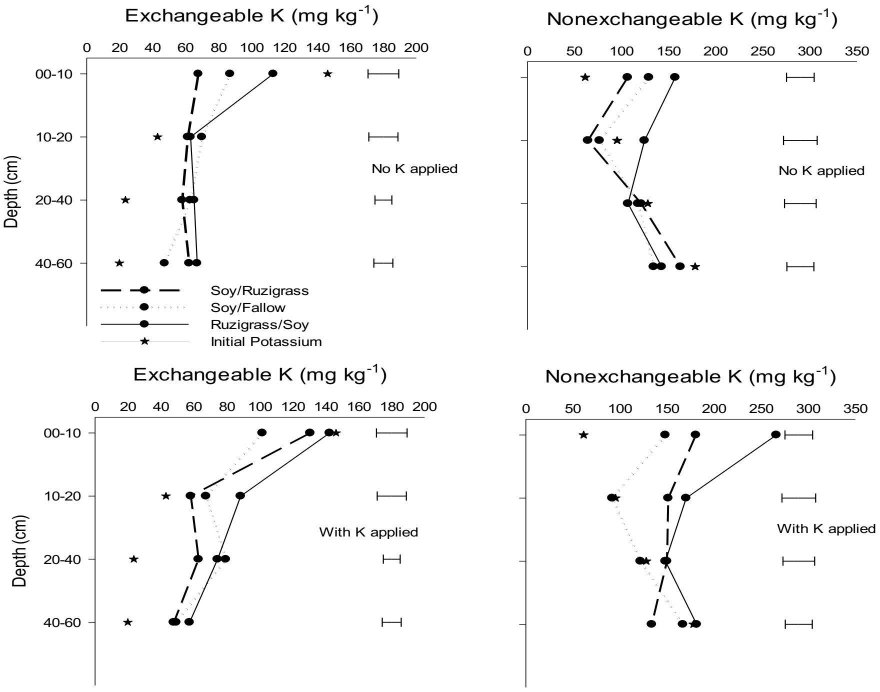


Exchangeable K in the soil decreased over time at a sampling depth of 0-10 cm, While nonexchangeable K increased, in all systems without K application. However, at all other depths, exchangeable K in the soil increased, and the nonexchangeable K maintained the content in depth. (Figure 4). The rotation system (Rz / Sy) showed both the highest increase in nonexchangeable K and the lowest decrease in exchangeable K (Figure 4).

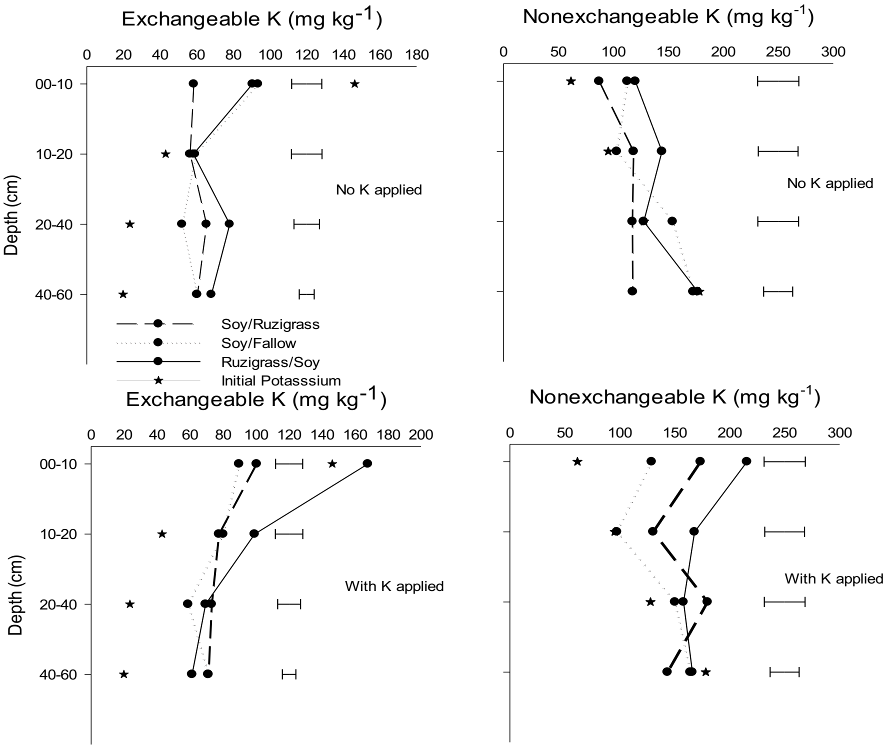
Without K application, the level of exchangeable K on the surface was lower in the succession system (Sy / Rz) than in the other two systems. By contrast, when K was applied, the level of exchangeable K on the surface was highest in the rotation system. Up to a depth of 20 cm, nonexchangeable K was greater in the rotation system with K (Figure 4).

Figure 4 - Soil exchangeable K and nonexchangeable K. Sampling in November of 2007, ★ represent contents of K in the start of experiment (November 2015). Horizontal bars show LSD in each depth (P < 0.05).

Early Cultivars



Late Cultivars



1.4 DISCUSSION

1.4.1 Ruzigrass

The cover crop produced different amounts of dry matter when grown in the different systems. In the Rz/Sy rotation system, ruzigrass produced approximately 60% more dry biomass than when in succession to soybean in the Sy/Rz system (Table 3). The season of growth of the genus *Urochloa* influences dry matter production (Figure 1); more dry matter is produced in the rainy season than in other seasons (SOBRINHO et al., 2009). During the period that ruzigrass was grown in the Sy/Rz system, rainfall was only 250 mm (Figure 1), which may have limited ruzigrass development.

The biomass production of the cover crop did not respond to K application (Table 3). When grown in succession or rotation with a grain crop, plants of the genus *Urochloa* tend not to respond to the addition of fertilizers (GARCIA et al., 2008; TANAKA et al., 2019).

Even though late-cycle plants tend to absorb more nutrients than early ones (CRUSCIOL et al., 2015a), thus leaving more soil residues for the successor crop, there was no effect of soybean cultivar on ruzigrass dry matter production when the cover crop was grown in succession to soybean (Sy/Rz).

K accumulation in the cover crop (Rz/Sy and Sy/Rz) as well as in the soybean plant residue (Sy/Flw) followed the trend of dry matter yield and also did not respond to K application (Table 3). The largest accumulations were observed in the Rz/Sy system, followed successively by Sy/Rz and Sy/Flw. The amount of K accumulation in the cover crop corresponded to approximately 60% and 73% of the recommended maintenance fertilizer K (SOUSA and LOBATO, 1996) in the Sy/Rz and Rz/Sy systems, respectively, thus showing the capacity of these plants to recycle K and supply subsequent crops. However, dry matter K accumulation was lowest in the Sy/Flw system, as K was not observed in any cellular constituent (ROSOLEM et al., 2006; MARSCHNER, 2011). These results indicate that K was quickly leached and deposited in the soil in this system, as sampling occurred 9 months after the soybean harvest.

1.4.2 Soybean

The late cultivar produced more dry matter than the early cultivar, reflecting its higher capacity to accumulate K (Table 3). Plants with a later cycle tend to have a longer juvenile period and thus produce higher amounts of biomass as well as higher leaf area indices than early cultivars (ZANON et al., 2015), leading to higher nutrient accumulation. The dry matter yield of the early cultivar was influenced by the addition of K when grown in succession systems: Sy/Rz + K > Sy/Rz. The ability of cover crops to minimize applied K losses (ROSOLEM et al., 2006; ROSOLEM; STEINER, 2017) is associated with the ability of plants of the genus *Urochloa* to cycle K and make it available for the successor crop (ROSOLEM et al., 2004; GARCIA et al., 2008; ROSOLEM et al., 2012), thus improving the efficiency of the use of K by soybean. The use of K without a cover crop (Sy/Flw + K) resulted in a lower dry matter yield of soybean compared with the rotation system without the use of K (Rz/Sy), as well as higher accumulated K. These results illustrate the effect of forage on K biofertilization (Table 3).

However, the late cultivar had lower dry matter yields and K accumulation under the Rz/Sy or Sy/Rz system than when cultivated under the Sy/Flw + K system. The higher production of biomass of the cover crop in the Rz/Sy and Sy/Rz systems (Table 3), which was deposited under the soil after desiccation, may have hampered the initial development of the soybean plants. Restricting radiation of seedlings may promote seed breakage when grown in a consortium (SILVA et al., 2013; CRUSCIOL et al., 2014), resulting in excessive energy expenditure by the soybean seedlings and subsequent developmental losses. Similarly, soybeans grown in rotation with ruzigrass may have reduced availability of nutrients such as N and P (ALMEIDA; ROSOLEM, 2016; ALMEIDA et al., 2018), thus impairing their growth.

The soybean plants exhibited high shoot dry matter yield from shoots and, as a consequence, a high capacity to accumulate K (Table 3), although approximately 53% of this accumulated K was exported via grain production (EMBRAPA, 2014). On average, approximately 59 kg ha^{-1} and 73 kg ha^{-1} of K were returned to the soil by the early and late cultivar, respectively. If this K is absorbed by cover crops grown in succession or rotation with soybean, loss due to leaching may be avoided (ROSOLEM et al., 2006; ROSOLEM et al., 2012; ROSOLEM; CALONEGO, 2013; ROSOLEM; STEINER, 2017), particularly in soils with a sandy surface texture (Table 1).

The K leaf concentration in soybean was within a sufficient range (KURIHARA et al., 2008) in the 2016-2017 crop, although higher leaf K content was observed when soybean plants were grown with the cover crop than when cultivated with a fallow period. By contrast, in 2017-2018, the K concentration in the early cultivar was in the low range when grown under the Sy/Rz and Sy/Flw systems (KURIHARA et al., 2008); in the late cultivar, this index was in the sufficient range only when cultivated under Rz/Sy + K (Table 4). The K concentration in soybean leaves can be influenced by the K application rate (ANTONANGELO et al., 2019).

For both cultivars, the leaf K concentration was higher when soybean was grown in rotation with the cover crop (Sy/Rz or Rz/Sy) than when grown with a fallow period (Sy/Flw). The continuous presence of cover crops helps to maintain soil moisture (TORRES et al., 2006; CALONEGO et al., 2011). For K, the main mechanisms responsible for root ion contact are mass flow and diffusion (MARSCHNER, 2011; VOLF et al., 2018); consequently, higher soil water content can improve K absorption and thus influence the leaf K concentration.

In the 2015-2016 season, soybean grain yield was influenced by the presence of the cover crop (Table 5), although the early cultivar showed a response to K application when cultivated in the fallow system. This shows that cover crops can provide the required K for soybean plants; in the absence of cover crops, K addition is required (FOLONI; ROSOLEM, 2008). Early cultivars tend to be more sensitive than late cultivars to the low-rainfall environment of Cerrado soils (Figure 1), but the difference in soybean yield is due to different levels of K addition rather than the maturity group (PARVEJ et al., 2015).

For the late cultivar, the yields were highest in rotation with the cover crop and followed the tendency of leaf K concentration (Table 4), with no response to K application. Without the cover crop, this cultivar had higher productivity when grown with K application (Sy/Flw + K > Sy/Flw). Cover plant biomass, especially of the genus *Urochloa*, can prevent productivity losses in response to climatic adversity (TANAKA et al., 2019), such as the periods of water deficit in November/December and January/February (Figure 1).

In the 2016-2017 season, soybean grain yield was lower in the Sy/Rz system than in the fallow system (Sy/Flw) (Table 5). Soybean rotated with ruzigrass had adequate leaf K content (Table 4) but lower yields compared with fallow rotation, suggesting a negative effect of ruzigrass on N and P (MERLIN et al., 2013; ALMEIDA et al., 2018). Further supporting this hypothesis, the grain yield of the early cultivar in the Sy/Flw system was similar to those in the Sy/Flw + K and Sy/Rz systems, showing that the addition of K was not the factor limiting productivity in all systems.

In the 2017-2018 season, there was no difference in yield with or without the addition of fertilizer K, thus demonstrating that the soil can provide K to soybean plants without the need for K addition (ROSOLEM et al., 1993) and that application of K may not improve productivity (ZÖRB et al., 2014). The climatic regularity in this season may also have played a role in the reduced effect of cover plants on productivity (Figure 1).

On the other hand, the effect of K application on the 100-grain weight, which was mainly observed in the 2017-2018 season (Table 5), as well as the higher K accumulation in the whole plant in the 2017-2018 season, may indicate that K is one of the nutrients involved in the translocation of photoassimilates to the grain (MARSCHNER, 2011). The resulting higher grain weight indicates luxury consumption of this nutrient by soybean plants (KAMINSKI et al., 2007; OLTMANS; MALLARINO, 2014; ANTONANGELO et al., 2019). Consistent with the results of the present study, Carpentieri-Pípolo et al. (2005) observed no correlation between the 100-grain weight and the productivity of various soybean cultivars.

1.4.3 Balance of K

The balance of K in the soil-plant system, obtained by relating the accumulated K in the shoots of soybean plants to the Ke content in the soil (ROSOLEM et al., 2012; NIU et al., 2013), shows the capacity of the soybean production system and plants to use K from other fractions, such as Kne (ROSOLEM; STEINER, 2017), and reduce their dependence on applied or exchangeable K, although in some cases plants may absorb Kne even with the addition of fertilizer K (ROSOLEM et al., 2012; NIU et al., 2013). When no K was added, the soybean plants absorbed Kne in addition to Ke (ROSOLEM et al., 1993), resulting in a negative balance regardless of cultivar (Figure 3a) and a contribution from Kne (Figure 3b). However, a negative balance was also observed for the late cultivar in the Sy/Flw + K system, despite the application of K (Figure 3a), in addition to a contribution of Kne to the total absorbed K. In all other systems with K, the balance was positive, and there was no contribution of Kne (Figure 3b). Soil containing trioctahedral micas, as in the case under study, may be a source of Kne for soybean (MOJALLALI; WEED, 1978), and this fraction may be important for the K balance in the soil-plant system (ROSOLEM; STEINER, 2017).

The Sy/Flw and Sy/Rz succession systems provided the same negative balance index and generated similar K contributions. However, for the late cultivar cultivated under the rotation system (Rz/Sy), the negative balance was smaller, since plants of the genus *Urochloa* absorb K and deposit it on the surface as Ke (GARCIA et al., 2008). This result shows that the Rz/Sy system had a greater ability to cycle K than the Sy/Rz system and thus made K available in more available forms for subsequent cultivation. Cover plants grown only in the offseason, which is characterized by restricted rainfall (Figure 2), may have lower dry matter accumulation compared with plants cultivated in summer (Table 3), resulting in lower K accumulation (Table 3) and less cycling.

Due to the high demand of soybean for K (OLTMANS; MALLARINO, 2014), the high capacity of *Urochloa* plants to cycle K (VAN RAIJ; QUAGGIO, 1984; GARCIA et al., 2008; ROSOLEM et al., 2012) is not sufficient to prevent soybean plants from using the Kne fraction when there is no K addition. However, under K application, succession or rotation became superior to the Sy/Flw system (Figure 3a), showing that it is possible to avoid system exhaustion. Care is therefore needed in the use of K in production systems involving high-demand plants, as withholding K application can damage the soil K reserve, depleting both Ke and Kne (BENIPAL; PASRICHA, 2002; KAMINSKI et al., 2007; ROSOLEM; STEINER, 2017). On the other hand, because soybeans accumulate a large amount of K (Table 3) and export only approximately 53% (EMBRAPA, 2014), there will be a return balance of this K. Thus, strategies to prevent the loss of this nutrient are needed.

The increase in exchangeable K along the soil profile (Figure 4) shows that K can leach even when there is no application of K. In addition to causing a negative balance in the system (Figure 3a), sandy soils subjected to long cultivation periods suffer K losses (STAINER; ROSOLEM, 2017). There is greater absorption by plant roots up to a depth of 20 cm (Figure 4), and thus the depletion of exchangeable K is greater at shallower depths.

Due to the ability of ruzigrass plants to cycle K, even in the nonexchangeable fraction (VOLF et al., 2018), and the deposition of this K on the surface (GARCIA et al., 2008), nonexchangeable K increased up to a depth of 20 cm (Figure 4), since the leachate from the straw of the cover plants deposited on the soil surface was converted to the Kne fraction (ROSOLEM et al., 2006).

The increase in nonexchangeable K was smaller in systems without K application than in systems with K application (Figure 4). Thus, in this type of soil, there is a negative balance of K in the absence of K fertilizer (Figure 3a), and the contribution of nonexchangeable K (ROSOLEM et al., 2003; 2006) to K accumulation by plants may increase to greater than 50% (Figure 3b).

1.5 CONCLUSION

The use of Urochloa ruzigrass in the soybean rotation system (Rz/Sy) provided a longer period of residence of this species compared with the soybean-ruzigrass (Sy/Rz) succession system. Therefore, the result, the Rz/Sy system provided higher K uptake and, in turn, a greater ability to recycle this nutrient.

The use of *Urochloa* ruzigrass as a cover crop provided sufficient K to avoid a decrease in grain yield for soybean grown with a fallow period and K application.

Soybean plants with higher K absorption capacity and K accumulation, such as the late cultivar, resulted in higher absorption of Kne.

The forage-soybean rotation systems decreased Kne uptake by the soybean crop due to the K cycling ability provided by the cover crop.

The use of ruzigrass only in the offseason, due to the water restrictions in this period, did not reduce the need for soybean plants to use Kne compared with soybean plants cultivated with a fallow period.

The association of K application with cover crops grown in rotation or the offseason improved the efficiency of K use by the system.

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CHAPTER 2- NONEXCHANGEABLE K USE BY CROPS GROWN IN A TROPICAL ULTISOL

ABSTRACT

Purpose: Some plant species are effective in utilizing nonexchangeable forms of K These species differences lead to improved K cycling in cropping systems and justify the lack of crop response to K in some environments. The aim of this study was to assess nonexchangeable K use by three selected crop species grown in a tropical Haplic Plinthosol with low exchangeable K.

Methods: A greenhouse experiment was conducted with soybean (Glycine max L., Merr.), maize (Zea mays L.), and ruzigrass (Urochloa ruziziensis) with or without K fertilization for three growing cycles. The cropped soil was compared with a control without plants.

Results: Nonexchangeable K contributed more than 80% of the K demand of the plants. In the first growing cycle, soybean and maize took up more nonexchangeable K than ruzigrass. However, in the second and third cycles, ruzigrass took up more nonexchangeable K than the other plant species. In general, the contribution of nonexchangeable K to plant nutrition decreased from the first through third cycles.

Conclusions: Nonexchangeable K was increasingly important for plant nutrition as exchangeable K was depleted. As plants began to depend on nonexchangeable K, both K uptake and biomass yields decreased. Ruzigrass showed a higher ability to acquire nonexchangeable K than maize and soybean. Therefore, the introduction of this species in production systems may enhance K cycling, improving the use efficiency of soil and fertilizer K.

Keywords: Exchangeable K uptake, Nonexchangeable K uptake, Nonexchangeable K, Exchangeable K, K in tropical soil, plant K availability.

2.1 INTRODUCTION

Potassium (K) is second only to nitrogen as an essential nutrient required by plants. Potassium is found in large concentrations in the vacuoles and cytoplasm of plant cells. It is a major enzyme activator, although it is not strongly bound by any cellular molecules (Marschner 2012). In soil, K is transported to roots primarily by diffusion in the soil solution; however, under some conditions, mass flow may also be an important transport mechanism (Barber 1995; Rosolem and Steiner 2017). The K concentration in soil solution is primarily buffered by K ions adsorbed electrostatically at the surfaces of soil mineral and organic colloids. When these ions move into the soil solution and are taken up by plants, it is normally assumed that they can be replenished by K from nonexchangeable interlayer sites in secondary layer silicates, and perhaps by structural K in primary minerals (micas and feldspars), where most soil K typically occurs (Barré et al. 2007). Interlayer K is retained in the soil solid phase.

Soil K that could be bioavailable over a growing season is commonly estimated in standardized soil tests by extraction with 1 M ammonium acetate or with Na-saturated anionic resin beads. This fraction of soil K may include both electrostatically adsorbed ions at mineral surfaces and an indeterminate fraction of interlayer K. This fraction is often termed “exchangeable K,” although the extraction is strongly driven by the carboxylate functional groups of the acetate or the resin, in addition to displacement of K by NH_4^+ or Na^+. In contrast, most interlayer K is not readily extracted by typical soil tests and is often referred to as “nonexchangeable K”, although a preferred term would be non-extractable K (since the soil test method involves more than simply cation exchange). In soils, the movement of K ions between surface-adsorbed and nonexchangeable interlayer fractions depends on coupled biological, chemical, and physical mechanisms in the rhizosphere (Kerbauy 2013). Plant uptake of K generates a strong concentration gradient in the rhizosphere solution that leads to release of electrostatically adsorbed K and some K from the interlayers of 2:1-layer silicate minerals.

Release of interlayer K may also be accelerated by the presence organic anions, exuded by roots and microorganisms, that promote mineral weathering and K dissolution. It is usually assumed that charged sites near mineral edges (also called wedge sites) are the most likely locations from which interlayer K is released to the soil solution (Hinsinger and Jaillard 1993; Volf et al 2018).

Although most tropical soils are low in soil-test K (Benites et al. 2010; Darunsontaya et al. 2012; Rosolem and Steiner 2017), crop response to K fertilization can be inconsistent (Oliveira et al. 2001; Castilhos et al. 2002; Melo et al. 2003; Khan et al. 2014), perhaps because some of the added K has been adsorbed at interlayer sites or because some interlayer K can be accessed by plants. When surface-adsorbed K concentrations are very low, plants depend on interlayer K, and there is often a concomitant yield penalty (Villa et al. 2004; Hosseinpur and Motaghian 2013). The dynamics of surface K and nonexchangeable interlayer K in soil have primarily been studied in soils of temperate climates where 2:1 clay minerals are dominant. Potassium in tropical soils, which are often dominated by 1:1 clay minerals and Fe or Al oxides, has received much less attention, and the importance of nonexchangeable interlayer forms of K to plant uptake and growth in these soils is not well understood.

Some plants, such as those of the genus Urochloa ssp. and pearl millet (Pennisetum americanum, L), are able to take up K from both the surface-adsorbed fraction and nonexchangeable interlayer fractions (Rosolem et al. 2003; Benites et al. 2010). Soybean is also able to take up interlayer K (Rosolem et al. 1993), albeit less efficiently than Urochloa ssp. (Garcia et al. 2008). The drivers and mechanisms vary from species to species and are related to root morphology (Barré et al. 2009), the compounds that the roots exude, and the concentrations of soluble K. Hence, plants such as ruzigrass (Urochloa ruziziensis), maize, and soybean grown in soils with low soil-test K may exhibit different responses to K fertilizer. The uptake of interlayer K has been observed mainly when fertilizer K is not applied, i.e., when nonexchangeable surface-adsorbed K is present (Benites et al. 2010).

In this paper, we define exchangeable K as that which is extractable by a Na-saturated exchange resin. We define nonexchangeable K as the difference between K extracted by boiling 1 M HNO$_3$ and exchangeable K. Exchangeable K roughly corresponds to surface-adsorbed K, and nonexchangeable K represents some but not all interlayer K.

The objectives of this study were to: (i) evaluate the growth of ruzigrass, maize, and soybean in a highly weathered soil with low exchangeable K content, and (ii) measure the capacity of these plants to take up and deplete nonexchangeable K over three successive crop cycles.

2.2 MATERIAL AND METHODS

A pot experiment was carried out in a greenhouse in Botucatu, Sao Paulo, Brazil. The soil was collected from a depth of 0.20–0.40 m of a Haplic Plinthosol (FAO – 2014) under native forest in Nova Xavantina, Mato Grosso, Brazil. The soil was air-dried and passed through a 5-mm sieve. Selected initial soil physical and chemical properties (Raij et al., 1986; Walkley and Black, 1934) are shown in Table 1. Plastic pots (12 L) were each filled with 14 kg of air-dried soil. Soil pH (CaCl$_2$) was raised to pH by applying dolomitic limestone (Rosolem et al. 2003). Subsequently, the soil was fertilized with 175 mg kg^{-1} of P as triple superphosphate, 13 mg kg^{-1} of S as calcium sulfate, 1.75 mg kg^{-1} of Cu as copper sulfate, and 1.75 mg kg^{-1} Zn as zinc sulfate.

Table 1. Table 1 - Selected initial soil5 chemical* characteristics and particle size distribution

pH^1	P^2	Exc K^3	Non-exc K^4	Ca^3	Mg^3	O.M.	Clay	Silt	Sand
(1:1)	(mg kg^{-}1)	------------ (mg kg$^{-1)}$ -------------				(g dm^{-3})		(g kg^{-1})	
3.8	7,2	52	230	120	30	12.0	275	175	550

1 In 0.01 mol L-1 CaCl2 ; 2, 3 extracted with exchange resin; 4 NAE – nitric-acid-extractable K, extracted with boiling HNO3– 5 Soil before amendment with limestone. O.M = Organic Matter – Walkley and Black, (1934).

Nitrogen supply varied with crop species. For soybean, nitrogen was supplied via biological fixation. Soybean seeds were inoculated with 5 mL kg^{-1} of Bradyrhizobium japonicum (strains SEMINA 5079-CPAC 15 and SEMINA 5080-CPAC7). For maize, nitrogen was applied at a rate of 80 mg kg^{-1} of N as urea 15 and 21 days after transplanting (Andreotti et al. 2001). For ruzigrass, 25 mg kg^{-1} of N were applied (Rosolem et al. 2012) at each application.

Potassium, as potassium chloride, was applied in each planting cycle 15 days after transplanting at the rates shown in Table 2. The amount of K applied was calculated both to replenish the K taken up by the plants in the preceding crop and to maintain soil exchangeable K at a minimum of 70 mg kg^{-1}. For this purpose, the total K in plant shoots and soil exchangeable K (K extracted with resin at planting and harvest) were determined. The amount of K replenished was the sum of K contained in the plants plus the amount of K in the soil minus 70 mg kg^{-1}. This strategy ensured that plants would not need to use K sources other than the applied fertilizer. In pots without plants, exchangeable K concentrations were maintained at 150 mg kg^{-1} by applying potassium chloride.

Before starting the experiment, soil exchangeable K was 75 mg kg^{-1}. Ruzigrass (Urochloa ruziziensis) was grown in all pots up to 45 days after plant emergence to deplete soil K, which decreased to 21 mg kg^{-1}. The aboveground biomass of ruzigrass was then removed from the pots, and germinated seeds of each species (soybean, maize and ruzigrass) were sown. Soil moisture was maintained at 80% of field capacity by applying deionized water every other day. To avoid water loss by percolation, the bottom of the pots were sealed. The greenhouse was maintained with 13 h of artificial light per day and temperature between 23°C and 35°C.

The experimental design was randomized complete blocks with four replications. The treatments included three crop species grown with or without K fertilization, as described in Table 2. Control pots without plants and with (control + K) or without (control) K were also included. The experiment was run for three successive growing cycles.

Table 2 - Potassium (K) application rate

Treatments	Rates mg kg^{-1} Soil		
	First Crop	Second Crop	Third Crop
Ruzigrass + K	70	115	70
Ruzigrass	0	0	0
Soy + K	70	130	70
Soy	0	0	0
Maize+ K	70	200	140
Maize	0	0	0
Soil + K	140	140	140
Soil	0	0	0

Before planting, seeds of soybean and maize were germinated for 48 h on paper towels moistened with distilled water and then transplanted into the soil. For ruzigrass, transplanting was performed only once, and the plants were allowed to regrow after cutting. Five soybean plants, six ruzigrass plants, or three maize plants were transplanted per pot. After seven days, the plants were thinned to two soybean plants, three ruzigrass plants, or one maize plant. The peak accumulation of K occurs at approximately 90 days for soybean (Oliveira Junior et al 2014) and maize (Vilela and Büll 2014), and thus these crops were grown for 90 days in each of the three successive cycles. By contrast, ruzigrass was grown for three successive cycles of 45 days each (Rosolem et al. 2012).

The entire aboveground portions of the plants in each pot were harvested by cutting at the soil surface and dried to constant weight in a forced-air oven at 60°C for 72 hours. The dry plant tissues were digested using a double-acid solution (HNO_3 + $HClO_4$, at a ratio of 2:1, v/v, Malavolta et al. 1997). K in roots and root residues was assumed to be readily extractable, so it was included in the exchangeable K fraction when calculating the K balance.

After shoot harvest, the soil in each pot was sampled with a small-core auger (1.27 cm diameter), long enough to reach the bottom of the pot. Three samples were taken per pot and combined into one replication. Soil samples were air-dried and ground to pass a 2-mm mesh screen. Resin-extractable (i.e., exchangeable) K was extracted with an ion-exchange resin saturated with Na (Raij et al. 1986), and nonexchangeable K was calculated as the difference between the amount of K extracted with boiling 1.0 mol L^{-1} HNO_3 (Schmitz and Pratt 1953) and exchangeable K (extracted with resin). The fraction of total K uptake attributable to nonexchangeable K was determined after three crop cycles as the difference between the total K accumulated by the plants and the cumulative decrease in exchangeable K.

The K output from the systems, K balance, and the contribution of nonexchangeable K to plant uptake were calculated using the following equations, as in Rosolem et al. (2012, 2017):

$$\Delta Ke = Ke_{final} - Ke_{initial}, \qquad [1]$$

and

$$\Delta Kne = Kne_{final} - Kne_{initial}, \qquad [2]$$

where Ke represents exchangeable K and Kne represents nonexchangeable K, ΔK is the change in the amount of each fraction of soil K (g pot^{-1}), K_{final} is the Ke or Kne in the soil after three crop cycles, and $K_{initial}$ is the Ke or Kne in the soil before starting the experiment. .

To estimate K balance in the soil-plant system, equation [3] was used:

$$Balance = (K_{fertilizer} + \Delta Ke) - K_{plant}, \qquad [3]$$

where $K_{fertilizer}$ is the mass of K applied per pot, K_{plant} is the mass of K exported in the harvested plant biomass per pot, and ΔK_e is the difference in the masses of exchangeable K in the pot before and after each crop cycle. A negative value was interpreted as a release of nonexchangeable K, or depletion of soil reserves, whereas a positive value indicates that K was bound by the soil and root residues.

To estimate the contribution of nonexchangeable K to plant uptake, we used equation [

$$Kne_{tk\text{-}up} = K_{AbsT} - [(Ke_{Bfc} - Ke_{Afc}) + K_{Fert}], \qquad [4]$$

where $Kne_{tk\text{-}up}$ is K absorbed from nonexchangeable forms; K_{AbsT} is total absorbed K; Ke_{Bfc} is exchangeable K before planting; Ke_{Afc} is exchangeable K after harvest, and K_{Fert} is K applied in fertilizer, all calculated on the basis of mass of K per pot.

The data were subjected to homogeneity testing, and if appropriate, ANOVA was run on data of exchangeable K, nonexchangeable K, plant dry matter yield and K balance in the system. When the effects were significant, means were compared by the least significant difference (LSD. $p<0.05$).

Table 3 - Summary of the ANOVA results (*p* value) and CV, exchangeable and non-exchangeable K, K uptake by plant and dry matter of plants

Source of variation	Non-exchangeable K	Exchangeable K	K uptake by plants	Dry matter
Treatment	0.0001	0.0001	0.0001	0.0001
Block	0.1994	0.5355	0.1738	0.6642
Season	0.0001	0.0001	0.0001	0.0001
Treatment X Season	0.0001	0.0001	0.0001	0.0001
CV*	4.25	12.06	6.88	9.7

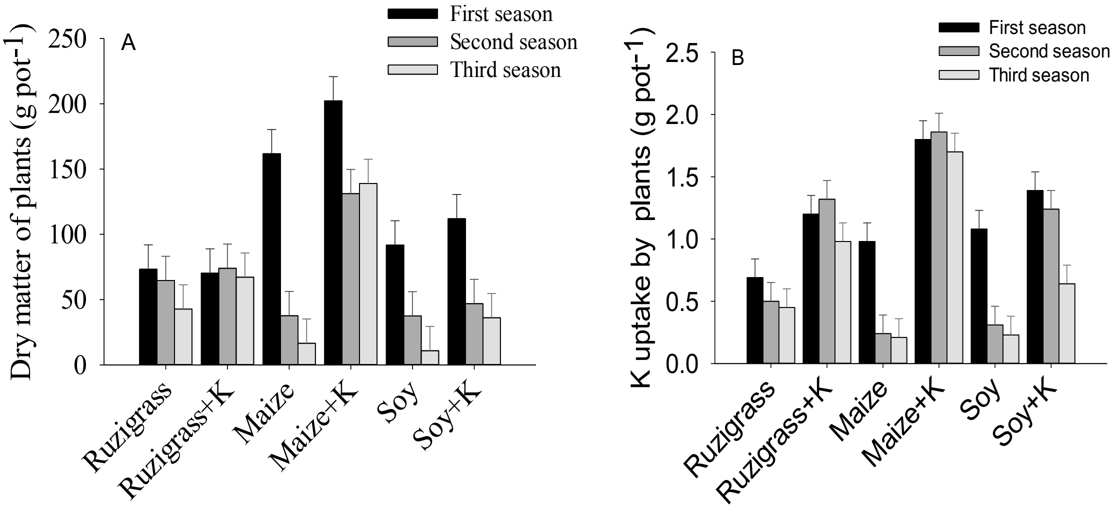
*CV Coefficient of variation

2.3 RESULTS

2.3.1 Potassium in soil and plants

Maize and soybean aboveground dry matter yields (DMY) decreased from the first through the third crop cycles, whereas for ruzigrass the decrease was significant only in the third crop (Fig. 1). Maize responded to K fertilizer in all crop cycles, but the response by soybean and ruzigrass was statistically significant only in the third cycle. When K fertilizer was applied, maize DMY was higher than that of the other species. Soybean DMY was higher than ruzigrass only in the first crop, even without K application. After the second crop, ruzigrass without K showed higher DMY than did maize and soybean. (Fig. 1A).

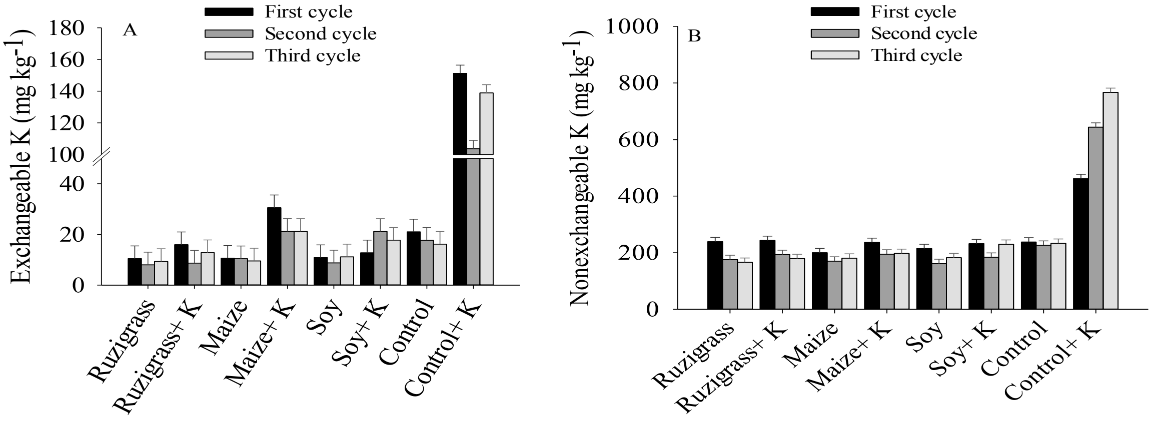
Figure 1 - Plant dry matter production (A) and K uptake (B) in three successive ruzigrass, maize, and soybean crops with and without application of K fertilizer. The absence of overlap by vertical bars show significantly different at p ≤ 0.05 according to the LSD test.



Potassium uptake decreased from the first through the third crop cycles, even when K fertilizer was applied (Fig. 1B). In the presence of K fertilizer, the plants absorbed larger quantities of K, as expected. Potassium uptake was highest in the maize + K treatment in the first and third crop cycles compared with the other crops with or without K fertilization (Fig. 1B). Without K application, soybean and maize accumulated more K than ruzigrass in the first crop cycle, whereas maize accumulated more K in the third crop cycle, consistent with the greater decreases observed in soil Ke and Kne (Fig. 2).

Soil Ke concentration in the control without plants and without K was 21 mg kg^{-1} (Fig. 2A), which is considered low (Sousa and Lobato, 1996). In the fertilized treatments without plants (control + K), soil Ke increased. Without K application, successive crops depleted Ke, which decreased compared with the control. However, in the third crop cycle, the Ke under fertilized ruzigrass was equivalent to that in the treatment without K application (Fig. 2A). Fertilization of maize and soybean with K kept the K concentration equal to or greater than that in the control, indicating that the amount applied was enough to supply the plant without increasing the soil concentration.

Figure 2 - Soil exchangeable K (A), and nonexchangeable K (B), in three successive ruzigrass, maize, and soybean crop growth cycles with or without application of K fertilizer. The absence of overlap by vertical bars show significantly different at p ≤ 0.05 according to the LSD test.

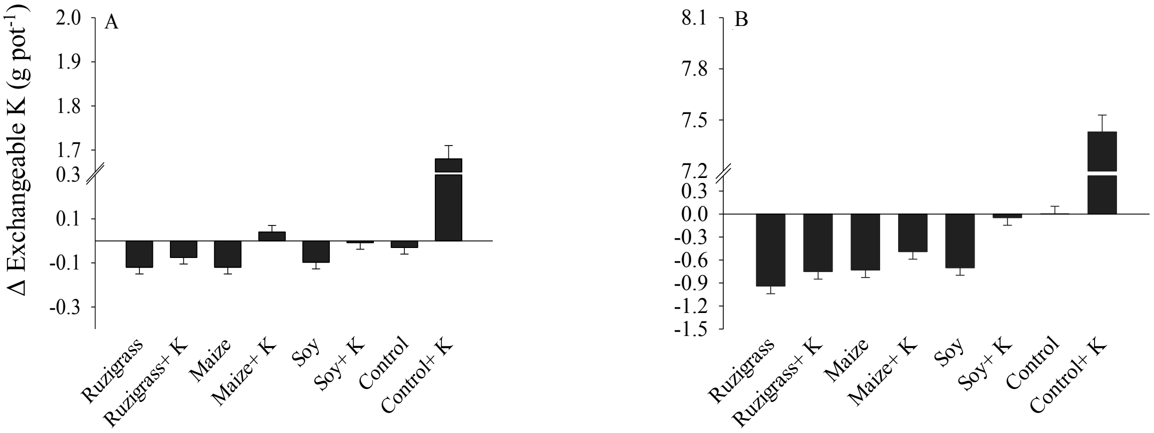


The soil Kne concentration by the end of the first crop cycle was similar to that in the control in all treatments except in soybean and maize without K, where a decrease was observed (Fig. 2b). After the second crop cycle, Kne concentrations were lower in all treatments without K compared with the fertilized treatments. The soil cropped with soybean without fertilizer had the lowest Kne concentration. After the third crop cycle, the greatest soil Kne decreases were observed in treatments with unfertilized ruzigrass. Regardless of K application, Kne decreased from the first to the second crop for all species. From the second to the third crop, the soil Kne concentration did not change, except for an increase in the treatment with K fertilized soybean. The application of K to pots without plants (control + K) resulted in increased soil Kne over time (Fig. 2B). The unfertilized ruzigrass showed the largest decrease in soil Kne following the first cycle.

Under soybean and maize, K fertilization was sufficient to maintain soil Ke at levels similar to the control treatment even after the three successive crops. Ruzigrass depleted soil

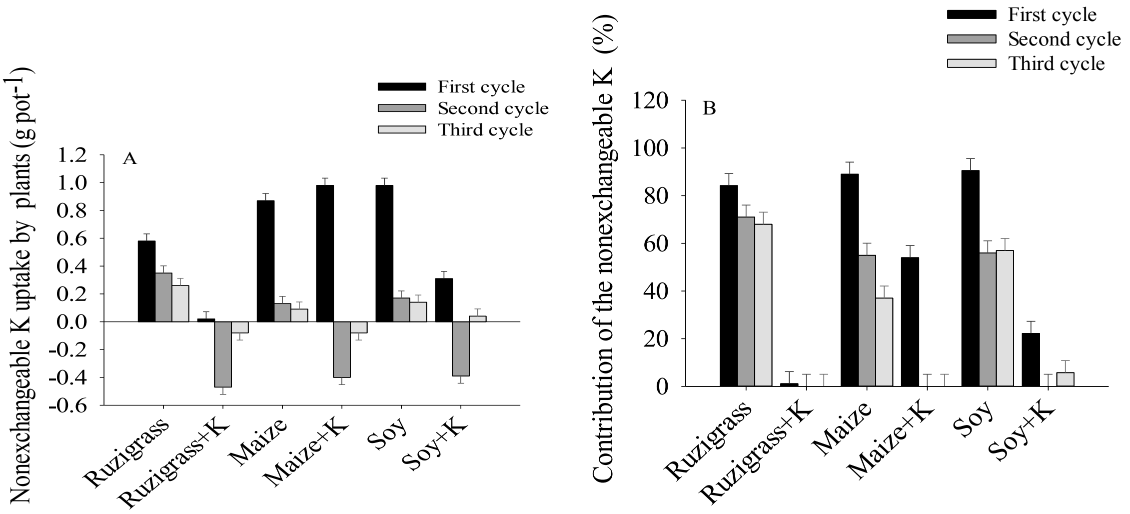
Ke, even with K fertilizer application. Furthermore, without K application, maize and ruzigrass depleted soil Ke more than did soybean (Fig. 3A).

Figure 3 - Difference between soil exchangeable K (A) and nonexchangeable K (B) contents of the third crop growth cycle and the first crop growth cycle ΔKsoil = Ksoil final – Ksoil initial). Vertical bars show the 95% confidence interval. No overlap of confidence intervals indicates a statistically significant difference (α = 0.05).



Depletion of Kne was observed in all treatments, although Kne was similar to the control in the fertilized soybean treatment. Increased Kne was observed in the control + K (Fig. 3B). The greatest depletion of Kne throughout the experiment was observed for the unfertilized treatments (Fig. 3B).

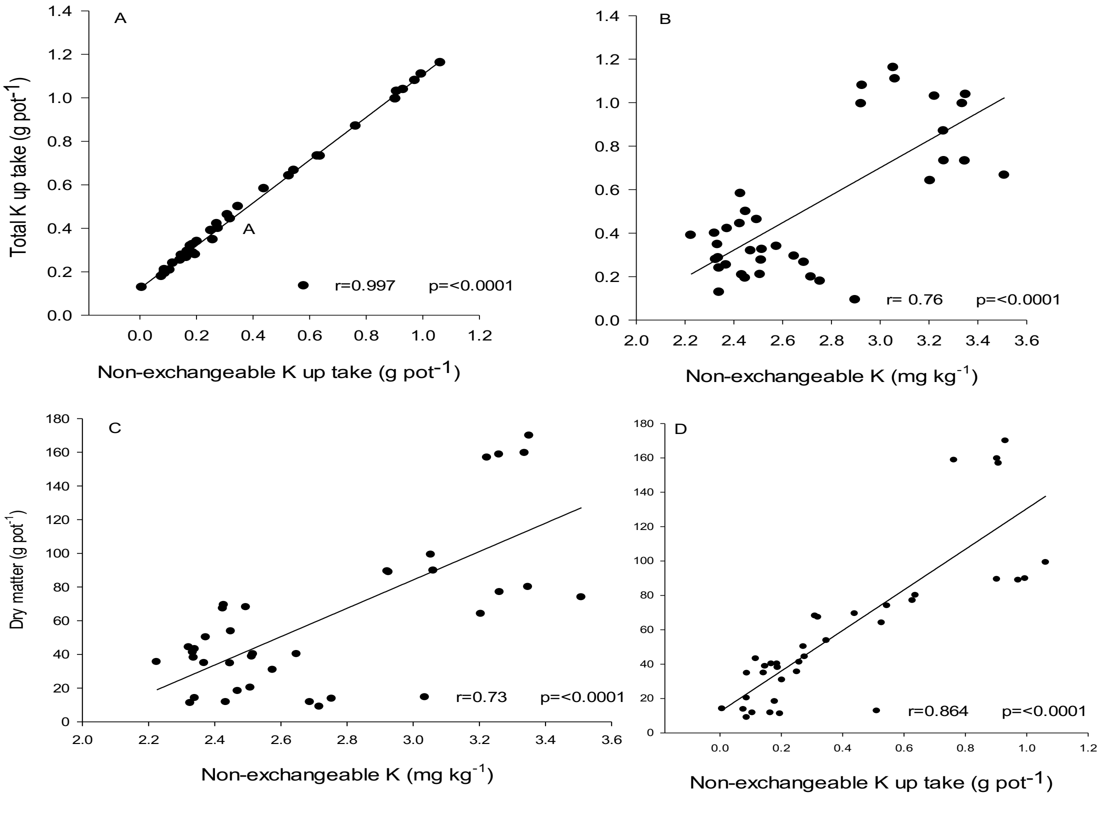
Figure 4 - Nonexchangeable K uptake by plants during each crop growth cycle (A), and the contribution of nonexchangeable K to total K uptake. Maize and soybean were grown for 90 days, and ruzigrass was grown for 45 days. The absence of overlap by vertical bars show significantly different at p ≤ 0.05 according to the LSD test.



Maize plants with and without K and soybean without K showed the greatest uptake of Kne in the first crop cycle (Fig. 4A). Uptake of Kne was highest in the first growing cycle and decreased as the experiment progressed (Fig. 4B). We hypothesize that the Kne taken up by plants in the first cycle was more accesible, perhaps because it was bound at interlayer sites near the edges of layer silicate minerals. After depletion of this Kne fraction, the plants may have had more difficulty in taking up Kne from interlayer positions This suggests that Kne was bound at different energy levels, and not all Kne extracted with HNO_3 was taken up by plants.

A linear relationship was observed between dry matter yield and soil Kne concentration in all non-fertilized crops over all crop cycles (Fig. 5). The higher the concentration of Kne in the soil, the higher the dry matter yield (Fig. 5A). Dry matter yield was also correlated with the uptake of Kne (Fig. 5B).

Figure 5 - Simple linear correlation between total K uptake by plants and non-exchangeable K uptake by plants (A), total K uptake by plants and soil non-exchangeable K (B), dry matter of plants and soil non-exchangeable K (C); and dry matter of plants and non-exchangeable K uptake by plants (D). Only data from treatments without K application are included.

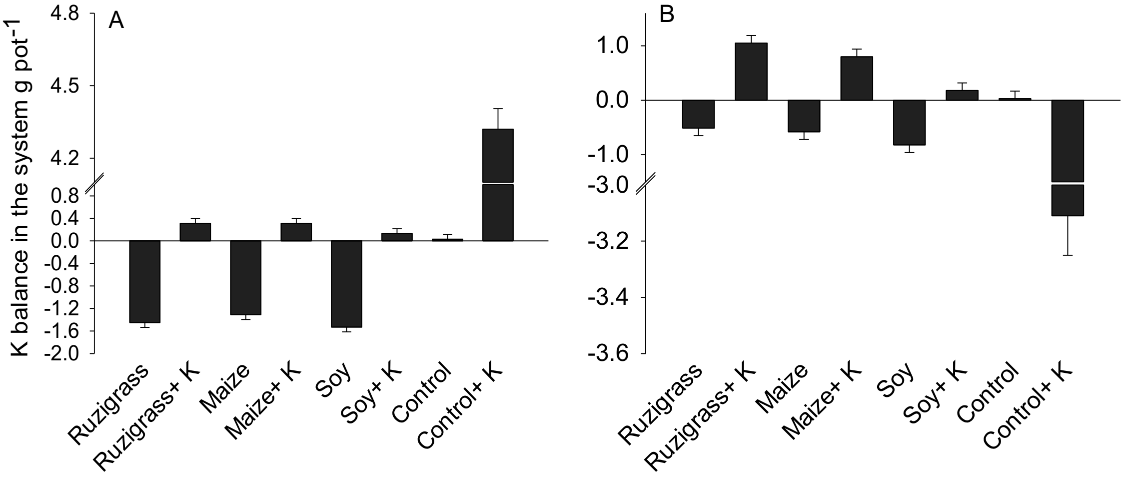


2.3.2 Potassium balance in the system

The mass balance of K in the system considering only ΔKe from Eq. 1 [Bal = (K_{fert} + ΔKe) –K_{plant}] was positive whenever fertilizer was applied and negative without K fertilization (Fig. 6A). Soybean cultivation did not change the soil K balance. When ΔKne was considered in the calculation (Eq. 2) [Bal = (K_{fert} + ΔKe + ΔKne) – K_{plant}], the negative mass balance decreased, and the positive balance increased, except for the K-fertilized soybean treatment (Fig. 6B). The K balance in soil in the control with fertilizer application became negative.

The more negative the K balance, the higher the K uptake from less soluble fractions such as Kne. Without K application, the soil K balance was more negative for soybean than for ruzigrass and maize. However, when K was applied, a positive balance was observed for the all three crops (Fig. 6).

Figure 6 - Potassium balance in the soil – plant system [(Bal = Kplant – (K fert + ΔKsoil)] in relation to total K uptake after three crop growth cycles for (A) exchangeable K and (B) exchangeable + non-exchangeable K. Error bars are Standard Error – (P<0.05).



2.4 DISCUSSION

Dry matter yields and K uptake decreased as the experiment progressed, even when K fertilizer was applied (Fig. 1). Successive crops, even when fertilized, tend to exhaust soil nutrients, eventually compromising yields (Moterle et al. 2016). However, in this study, diagnostic leaf analysis did not show insufficient levels of any nutrient (data not shown) that could explain the decrease in dry mass production. Despite plant uptake of K from less soluble fractions, soil K extraction over successive crop cycles can eventually lead to a decrease in K absorption and dry matter production (Li et al. 2015, 2017; Islam et al. 2018). Dry matter yields over the course of three growing cycles could have declined for reasons other than K fertility, too.

Since ruzigrass responded to K application only in the third crop cycle and was not affected by the low Ke levels in the first and second crop cycles, it is possible to infer that ruzigrass is able to use less soluble fractions of K (Van Raij and Quaggio 1984; Garcia et al. 2008; Rosolem et al. 2012). Ruzigrass yields without K were higher than those of maize and soybean (without K) in the second and third crop cycles (Fig. 1A), an indication that this species is better able to acquire K than are maize and soybean. This difference in K utilization was reflected in the Kne uptake by the plants (Fig. 4A): in the first crop cycle, maize and soybean without K absorbed more Kne than ruzigrass, whereas in the second and third cycles, ruzigrass absorbed more Kne than maize or soybean.

Because the soil initially had low Ke, i.e., below the minimal critical level (Sousa and Lobato, 1996), the plants had to rely on soil Kne release to meet K demand even when K fertilizer was applied to replace the amount taken up by the plants in the previous cycle. Plant demand for K is an important driver of Kne exploration (Rosolem et al. 2012; White 2013). The high dry matter yields of soybean and maize (Fig. 1B) were responsible for the larger decrease in Kne compared with the control in the first cycle (Fig. 2B). The increase in soil Kne in the third cycle with fertilized soybean (Fig. 2B) may have been due to the conversion of Ke to Kne, as shown in Fig. 2A. For this crop, Ke decreased even in the presence of K fertilizer, as did dry matter yield (Fig. 3B). This suggests that not all Ke was utilized, and the surplus was converted to Kne.

For maize and soybean, there was a contribution of the soil Kne fraction even with K application (Fig. 4). These plants showed higher dry matter yields than ruzigrass (Fig. 1B). The K demand of plants is the limiting factor favoring the use of Kne (Rosolem et al. 2012; White 2013), and maize and soybean had higher demand for K than ruzigrass. As reported by Rosolem et al. (2012), K application did not prevent the use of Kne by ruzigrass, which releases organic acids in the rhizosphere that can solubilize K from less available forms (Meurer and Castilhos 2001).

Although Ke in the soil was low, the plants were able to take up K. Ruzigrass has been reported to have a higher capacity to acquire nonexchangeable K than maize or soybean (Garcia et al. 2008). Therefore, maize and soybean could explore some of the K that is considered nonexchangeable according to routine chemical extractions, whereas it can be speculated that ruzigrass root exudates were able to solubilize additional nonexchangeable K bound more tightly to soil colloids. It can be inferred that most of the K taken up initially came from the fraction bound to the clay edges with intermediate solubility; even when HNO_3-extracted K was high, less and less was acquired by the plants over time (successive crop cycles) (Fig. 4).

Even with the sequential application of K and without plants (control + K), Ke increased only after the first application and decreased with time. Because CEC was low in this soil (Table 1), the exchange sites were probably saturated after the first K application. Consequently, the K from the subsequent applications was converted to Kne, which increased after each fertilization (Fig. 2B). Over time, as there were no plants to take up Ke, it may have been converted to Kne, and Ke decreased. Conversion of applied K to Kne is possible and may occur quickly (Rosolem et al. 2006; Moterle et al. 2016). This conversion may explain the decrease in Ke (Fig. 2A) with a corresponding increase in Kne over time (Fig. 2B), in the control. Increased Kne after K application to soils without plants has been observed in tropical soils (Moterle et al. 2016). In soil with low Ke, a balance will occur between adsorption and release in the soil solution after K application. After this equilibrium is reached, increased K rates result in a linear increase in Kne (Wang et al. 2004). The increase in the nonexchangeable fraction after K fertilization in the control + K treatment (Fig. 2B) suggests that some of the added K was adsorbed in interlayer positions of 2:1 minerals in the soil (Fig. 2).

The responses of the three species to soil Ke and Kne depletion clearly demonstrated the differences in K utilization among them and the importance of Kne for K supply (Fig. 3) in systems with ruzigrass. More importantly, the results suggest that Kne can be used by plants without complete depletion of the Ke fraction. As shown in Fig. 3, Kne extraction is related not only to the equilibrium between the fractions, but also to the uptake capacity and the plant demand for K. Thus, in K-fertilized maize, which did not deplete Ke, Kne still decreased, perhaps due to the high K demand by maize. For soybean, however, the applied K was sufficient, and there was no need to use Kne. By contrast, due to its lower K demand and greater cycling efficiency, ruzigrass depleted soil Ke and Kne (Fig. 3), even when fertilized with K. Plants with more aggressive root systems are more efficient in absorbing K from less soluble fractions, and root systems with a larger surface area, like those of grasses (Pereira et al., 2004), may exude organic acids in a larger volume of the soil (Singh et al. 2002; Barré et al. 2009) and thus remove more K from these fractions.

In treatments without K, dry matter yields were similar to those in the treatment with K application in the first and second crop cycles of ruzigrass and the second crop cycle of soybean (Fig. 1B). Hence, cultivation of soybean and ruzigrass without K caused a greater decrease in Kne throughout the experiment, showing that both plants can use Kne, as has been observed in other studies (Mojallali and Weed 1978; Rosolem et al. 1993; Garcia et al. 2008).

The highest Kne depletion was observed when K was not applied. Unfertilized plants were dependent on Kne, with a direct relationship between productivity and K released from this fraction (Fig. 5). Furthermore, the contribution of Kne to plant absorption increased (Fig.

4B) as soil Ke decreased (Fig. 2A). The higher uptake of Kne by maize and soybean without K in the first crop cycle (Fig. 4A) reflected the lower Kne compared with the control. However, this pattern was not observed in the following crop cycles (Fig. 4B), showing once more that the first K to be taken up originates from soil pools more soluble than the interlayer K and that these plants (soybean and maize) have greater difficulty in acquiring Kne. The application of K to maize in the first crop cycle promoted higher uptake of both Ke (Fig. 2A) and Kne (Fig. 4A) that soybean and ruzigrass. Consequently, production of dry matter was higher. This greater uptake may have occurred because the uptake of K favors acidification; that is, H^+ release occurs when K is taken up by the root (Marschner, 2012). This acidification may have increased the release of Kne as well as its absorption.

The K balance in soil/plant systems is important, especially considering the different fractions of K that plants may access and the K inputs, removals, and losses from the system. The balance will always tend to be negative for both Ke and Kne when K fertilizer is not applied and will be positive as the amount of K applied increases (Moterle et al. 2016). However, when considering Kne in the balance, there is a tendency for the amount of K in soil, mainly in the upper layers, to increase with higher clay content (Rosolem and Steiner 2017). Therefore, if the balance is still negative when Kne is taken into account, it is clear that plants are using K from less soluble pools than the K extracted with resin or even HNO_3. This K may originate from trioctahedral phyllosilicates, such as biotite, or it may occur in hydroxy-interlayered vermiculite. X-ray diffraction studies of the soil in this study indicate the presence of both of these minerals in the clay fraction (data not shown). Maize and soybean have been shown to be capable of removing more K from biotite than from muscovite (Mojallali and Weed 1978; Berthelin and Leyval 1982). The higher positive K balance indicated an uptake of such forms of K.

2.5 CONCLUSIONS

In a tropical agricultural system with limited exchangeable K, HNO3-extractable K contributed 80% to 100% of the amount taken up by plants. Uptake depended on the plant species, since ruzigrass apparently has a greater ability to acquire NHO3-extractable K than soybean and maize. Therefore, this soil K fraction is bioavailable to some extent. Soil K extraction by the resin method is not able to show all of the K taken up by the plants, indicating that soil K bound to different sites with different binding energies is bioavailable. This is an important point to be considered in recommending K fertilization for cropping systems where ruzigrass, maize and soybean are included.

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CHAPTER 3 - RELATIONSHIP BETWEEN POTASSIUM QUANTITY/INTENSITY (Q/I) AND POTENTIAL BUFFERING CAPACITY IN A TROPICAL SOIL UNDER POTASSIUM DEPLETION.

ABSTRACT

The dynamics of soil potassium (K) is driven by the relationships among its fractions, which are affected by weathering, soil management and plant species. The aim of this study was to assess the effect of successive crops and K fertilization on the K potential buffering capacity (PBCk) of the soil and the role of K fractions in the soil solution equilibrium. Samples were taken from a soil under Cerrado vegetation or cropped with soybean in spring/summer and ruzigrass (*Urochloa ruziziensis*) as a cover crop in fall/winter. The Cerrado soil was submitted to successive cropping to deplete soil K, and the agricultural soil was sampled at depths of 0-0.1 m and 0.4-0.6 m. Isothermal analyses of both soils were performed to determine the PBCk and the quantity/intensity (Q/I) ratio. Soils with low K depletion or low K content tended to undergo greater K release than adsorption. Under K exhaustion, the non-exchangeable fraction of K (Kne) can supply and adsorb K. Soils with a predominance of 1:1 minerals initially have a higher potential for K release than adsorption. This K may be adsorbed to planar sites or to the edges of clay. When K is depleted in tropical soils with a low content of K-source minerals, the exchangeable sites must be saturated before K can be released to the soil solution. The PBC of this soil is a function of the exchangeable K (Ke). Since the Kne fraction is always releasing K, it can be considered a recharger of the other soil K pools.

Keywords: Non-Exchangeable K. Potassium dynamics; Quantity – Intensity. Potential Buffering capacity.

3.1 INTRODUCTION

Potassium is one of the main elements in Earth's crust and is found in soil in structural, non-exchangeable, exchangeable and soil solution forms (ROSOLEM et al., 2003; CALONEGO et al., 2005). The concentration of each form/fraction and their relative proportions have important implications for the dynamics of K in the plant-soil system. The relationships among soil K fractions are influenced by the environment through weathering, fertilizer application, and plant exudates. Weathered tropical soils usually contain low levels of readily available K (BENITES et al. 2010; ROSOLEM; STEINER, 2017), although soils with high silt content and in semi-arid regions may contain K bound to primary minerals (K or non-exchangeable K) (AZEVEDO; VIDAL-TORRADO, 2009; GHIRI et al., 2012).

The structural fraction of feldspars contains K and can be a direct source of soil solution K. K can also originate from 2:1 micas via weathering, which is an important source of K through the non-exchangeable fraction (Kne) (HUANG, 1977; THOMPSON; UKRAINZCYK, 2002; AZEVEDO; VIDAL-TORRADO, 2009; KÄMPF et al., 2009; ZÖRB et al., 2014; CHAVES et al., 2015). In tropical regions with a predominance of 1:1 clay minerals (kaolinitic soils), Kne can be adsorbed to the edges of the clay under different retention forces (NACHTIGALL; RAIJ, 2005; ZÖRB et al., 2014; MOTERLE et al., 2016) and will not be affected by cationic exchange unless this connection is broken. In this way, Kne is considered a source of K with slow availability.

The equilibrium between soil K pools or the dynamics of K in the soil is interdependent and biphasic. The introduction or withdrawal of K in soil occurs through the soil solution (JALALI et al., 2006; LAKARIA et al., 2012; SARKAR et al., 2014), which is in equilibrium with exchangeable K (Ke) (WIHARDJAKA et al., 1999; SINGH et al., 2002). The extraction of K by crops depletes the K in these pools, leading to the release of mineral Kne and K, which are bound with higher strength to the soil colloids (SINGH et al., 2002; MOTERLE et al., 2016). Furthermore, the solid fractions of soil K (Ke, Kne, and structural K) and thus soil K dynamics are also modified by the addition of K via fertilizer, K leached from plant residues, or even by the weathering of primary minerals via the action of plant roots (HINSINGER; JAILLARD, 1993; GOMMERS et al., 2005; ROSOLEM et al., 2006; BARRÉ et al., 2007; EGUCHI et al., 2015; VOLF et al., 2018).

The K potential buffering capacity (PBCk) is an indicator of the soil K equilibrium and is regulated by the ratio of the quantity (Q) of Ke to the intensity (I) of K in the soil solution (Q/I), which are in equilibrium (BILIAS; BARBAYIANNIS, 2018)

The use of plants capable of taking up large quantities of K from less available fractions, such as Kne, and recycling this K to the soil as Ke will affect the PBCk (GARCIA et al., 2008; ROSOLEM et al., 2012). For example, Ke content is higher in the rhizosphere of ruzigrass (*Urochloa ruziziensis*) than in regions farther from the root, probably due to K replenishment from the Kne reservoir (VOLF et al., 2018).

In soils with low levels of Ke-bearing micaceous minerals, Q may also be influenced by Kne, which is at least partially responsible for maintaining soil PBCk (WANG et al., 2014). In this case, Kne would be the main reservoir responsible for supplying K and maintaining equilibrium with K in solution (readily available to plants). Soils with micas have a Ke equilibrium point of approximately 30 mg kg^{-1} (BORTOLUZZI et al., 2005), whereas 3 mg kg^{-1} is required to release K from the illite interlayers (HINSINGER; JAILLARD 1993). In soils without micas, the balance is close to 6 mg kg^{-1} (ROSOLEM et al., 2012). We hypothesized that since the depletion of K by successive crops may affect PBCk (MIELNICZUK; SELBACH, 1978), partitioning of the Q/I ratio may indicate which fraction is responsible for the equilibrium with the soil solution K (WANG et al., 2014). Soil management practices such as acidity remediation or K application can also affect the partitioned fraction indexes.

The effects of soil use and fertilization on PBCk in tropical soils with low Ke and a small proportion of 2:1 micas are poorly understood. Therefore, the aim of this study was to assess the effect of successive crops or the addition of K on the behavior of PBCk and whether Q/I ratio partitioning could be an indicator of which soil K pool is the driver of the soil solution equilibrium.

3.2 MATERIAL AND METHODS

3.2.1 Soil sampling

Samples of a Haplic Plinthosol (FAO – 2014) were taken in Nova Xavantina, State of Mato Grosso, Brazil. Soil samples were collected from two environments: A) under native forest (Cerrado), collected at a depth of 0.20 - 0.40 m; B) a 2-year field experiment planted with soybean in spring/summer and ruzigrass (*Urochloa ruziziensis*) as the cover crop in fall/winter, without K application. The Cerrado soil was used for the successive cultivation of ruzigrass and maize (*Zea mays*) in pots with the aim of reaching soil K exhaustion. Since the soil in the field experiment had been cropped for five years without K application before the experiment, it represented a K exhaustion experiment under field conditions. Selected soil characteristics are presented in Table 1. The sampling depth of the Cerrado soil was selected with the aim of collecting a less-disturbed sample that was closer to the matrix state.

Table 1 - Selected soil chemical characteristics, particle size distribution and clay mineralogy parameters

Soils	TRT											Clay mineralogy [3]				
		pH	Exc K[1]	Non-exc K[2]	CEC	Ca[1]	Mg[1]	M.O.	Clay	Silt	Sand	Klt	Ver	HIV	Ilt	Ght
		Ca	(mg kg^{-1})		---- (cmolc dm^{-3}) -----			(g dm^{-3})	(g kg^{-1})			% of each minerals[4]				
Forest	1	3,7	77	260	8.3	0.03	0.2	120	280	100	620	61	1,47		22	15
Forest + CaCo3	2	5,5	63	236		2.71	0.5						1,33			
Forest + CaCo3 + 1st	3	5,1	33	245		2.52	0.5						1,60			
Forest + CaCo3 + 1st Corn	4	5,2	17	228		2.23	0.5						1,20			
Forest + CaCo3 + 3rd Corn	5	4,4	18	169		1.88	0.3						1,60			
Forest + CaCo3 + 1st KCl	6	5,3	160	462		3.1	0.8						2,00			
Forest + CaCo3 + 2nd KCL	7	5,2	234	644		3.15	0.7						1,20			
Forest + CaCo3 + 3rd KCl	8	5,3	255	733		2,57	0.6						1,50			
Field Experiment 0-10 cm	9	4,9	56	170	8.0	1.11	0.5	19.8	180	100	720	67		3,5	11,5	18
Field Experiment 40-60 cm	10	3,9	22	190	6.8	0.23	0.2	8.8	370	90	540			3,4		

[1] In $CaCl_2$ [2], extracted with mehlich 1 [3] extracted with boiling HNO_3, quantification of the peak area of the minerals, by X-ray diffraction XRD, and thermal analysis to determine the percentage of each mineral. TRT treatments. Klt = Kaolinite, Ver = Vermiculite, HIV = Hydroxide interlayer Vermiculite, Ilt = Ilite e Ght = Goethite.

Limestone (28% CaO, 12% MgO) was applied to the Cerrado soil to raise the base saturation to 70% (ROSOLEM et al., 1993), followed by the application of 175 mg kg^{-1} of P (as triple superphosphate), 13 mg kg^{-1} S (calcium sulfate), 1.75 mg kg^{-1} Cu (copper sulfate), and 1.75 mg kg^{-1} Zn (as zinc sulfate).

In the field, soybean had been cropped for 5 years under no-till management in rotation with ruzigrass (*Urochloa ruziziensis*) in the offseason. In November 2015, soil samples were taken for selected chemical and physical analyses as described by RAIJ et al. (2001). At a depth of 0-0.1 m, the pH ($CaCl_2$) was 5.2, with 19.8 g dm^{-3} of O.M., 16.8 mg dm^{-3} of P (resin), 146.22 g kg^{-1} of Ke, 56.3 g kg^{-1} of Kne, 31.2 cmolc dm^{-3} of Ca, 11.4 cmolc dm^{-3} of Mg, 280 g dm^{-3} of clay, 100 g dm^{-3} of sand, and 620 g dm^{-3} of silt; at a depth of 0.4-0.6 m, the soil had a pH ($CaCl_2$) of 3.9, with 7.5 g dm^{-3} of O.M., 0.8 mg dm^{-3} of P (resin), 39.4 g kg^{-1} of Ke, 178.3 g kg^{-1} of Kne, 7.0 cmolc dm^{-3} of Ca, 4.5 cmolc dm^{-3} of Mg, 370 g dm^{-3} of clay, 90 g dm^{-3} of sand, and 540 g dm^{-3} of silt. Application of 100 kg ha^{-1} of P_2O_5 as triple superphosphate was performed annually, and N was supplied via biological fixation.

In the greenhouse, ruzigrass was first grown for 45 days, and then maize was grown for cycles of 90 days each. K fertilizer was applied at 200 mg kg^{-1} every 90 days according to the treatments, with the aim of saturating the soil.

For this work, we used soil samples from both environments, with the following treatments: T1) Cerrado soil; T2) T1 after limestone application; T3) T2 after ruzigrass, T4) T3 after first successive maize crops; T5) T3 after three successive maize crops T6) T3 after the first application of K; T7) T3 after the second application of K; T8) T3 after the third application of K; T9) soil from environment B at a depth of 0.0-0.1 m; T10) soil from environment B at a depth of 0.4-0.6 m. The greenhouse experiment was conducted in quadruplicate.

3.2.2 Isothermal analysis of Q/I and PBCk

The soil samples were air-dried and passed through a 2-mm sieve. For T1-T3, samples of 50 g were collected, since the soil was not potted. The soil from environment A was sampled after harvest using a pot core sampler. In the field experiment (environment B), soil samples were collected in the second year of the experiment, and five soil subsamples were collected with a 5-cm-diameter auger at the time of desiccation of the cover crops.

For isothermal analysis, 2.5 g of soil was added to 30-mL pre-weighed centrifuge tubes. Then, 25 mL of 0.01 M $CaCl_2$ solution with a KNO_3 concentration of 0.0, 0.5, 1.0, 2.0, 3.5 or 5.0 mM was added, and the suspensions were shaken for 30 min, allowed to rest for 18 h, and then centrifuged. The supernatant was collected for analysis of K, Ca and Mg by inductively coupled plasma analysis (ICP). Soil samples of 0.62 and 1.25 g were mixed with 25 mL of 0.01 M $CaCl_2$ solution without KNO_3 to facilitate the construction of the K release curve. After removal of the supernatant by decanting, the tubes with soil on the bottom were weighed. Then, 25 mL of NH_4OAc was added to each centrifuge tube, and the tubes were shaken for 30 min and centrifuged. The supernatant was collected to analyze K, and the tubes were weighed after removal of the supernatant (WANG et al., 2004).

3.2.3 Partitioning of Q/I and potential buffering capacity of K (PBCk)

The determination of I and Q as related to total buffer capacity (PBCt) was partitioned into a) PBCe: potential buffering capacity due to K; b) PBCn: potential buffering capacity due to Kne; and c) PBCt, as suggested by Wang et al. (2004), using the following equation:

$$\Delta K=(CK_i - Ck_f)\ (v/w) \qquad (1)$$

where ΔK is the change in solution K, CKi is the initial (K added) concentration of solution K, CKf is the final concentration of solution K, and v and w are the solution volume and the soil mass, respectively.

The potassium concentration (CR) ratio was used to describe the intensity of K in the presence of Ca and Mg:

$$CR = CK_f/(Ca_f - Mg_f)^{1/2} \qquad (2)$$

where C_{af} and M_{gf} are the final concentrations of Ca and Mg in the final equilibrium solutions. According to Wang et al. (2004), CR can represent AR_k, which is correlated with the intensity of K.

ΔK is usually used to identify the proportion of K that has been adsorbed or released by the solid phase of the soil. Positive values of ΔK indicate K adsorption, and negative values mean that K was released from the solid phase to the solution.

Then, to measure the contribution of each fraction of K in this equilibrium, such as Ke and Kne, the following equations were used:

$$\Delta Exch\ K = (EK_f - EK_0) \tag{3}$$

$$\Delta Non\text{-}Exch\ K = \Delta K - (EK_f - EK_0) \tag{4}$$

where EK_0 is the Ke corresponding to $\Delta K = 0$, which is estimated from the linear regression equation of EK_f (final exchangeable K) vs. ΔK.

PBC was obtained by fitting a linear regression of CR against ΔK using the coefficient “b” for PBCt (MIELNICZUK; SELBACH, 1978). The same procedure was followed for PBCe and PBCn using ΔExch K and ΔNon-Exch K, respectively.

Regression analysis and other statistical studies were conducted using Sigma Plot software version 12.5. For the statistical study, the mathematical equations were adjusted to adequately express the behavior of the obtained results, and the significant equations with the best fit were chosen (ROSOLEM et al., 2003b). The first derivative of the data-adjusted equation was used to represent CR_0, and "b" of the linear equation was used to represent PBC.

3.3 RESULTS AND DISCUSSION

3.3.1 Release and adsorption of K

The Cerrado soil (Figure 1a) and the soil with the K application sequences (Figures 1e) showed high K release. K adsorption was observed when CR was above 0.03 $(mol\ L^{-1})^{1/2}$, although in T8 (Figure 1e), K adsorption was observed at CR=0.04. In this treatment, total K did not change, resulting in $CR_0=0$ because of K release from the total pool to the solid fractions. These results indicated that the equilibrium point had been reached and all K had been released since the second application (Table 2). Acidity amelioration and fertilizer application resulted in saturation of the solution K; since there were no plants to use it, K adsorption and release were low (Figure 1e).

Table 2 - Parameters used to describe the K dynamics, in soil under the exhaustion of K

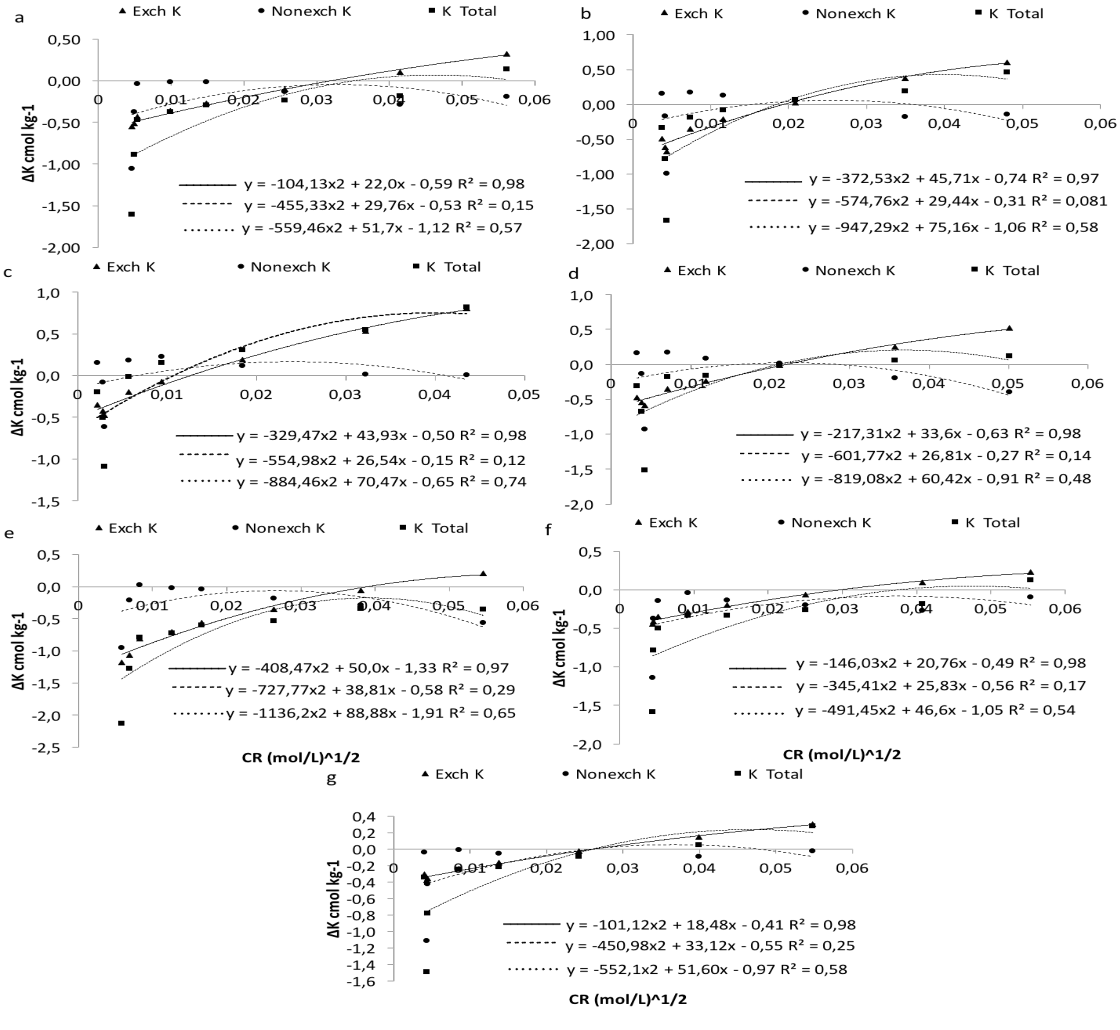
Soil	Treatments	CR_0	α	$\mathcal{B}$	PBC_e	PBC_n	PBC_t
		(mol/L)^1/2			cmol kg^{-1}/(mol/L)$^{\wedge 1/2}$		
Forest	**1**	0,034	1,34	-0,48	14,81	-4,71	10,10
Forest + CaCO3	**2**	0,018	1,48	-0,75	24,67	-8,43	16,24
Forest + CaCO3 + 1ft Ruzigrass	**3**	0,017	1,26	-0,56	26,65	-6,77	19,88
Forest + CaCO3 + 1ft Corn	**4**	0,010	1,20	-0,40	27,35	-4,85	22,50
Forest + CaCO3 + 3rd Corn	**5**	0,021	2,24	-1,16	20,81	-12,29	8,52
Forest + CaCO3 + 1 ft KCl	**6**	0,031	1,19	-0,49	22,88	-5,87	17,02
Forest + CaCO3 + 2 nd KCL	**7**		2,26	-1,30	23,15	-14,26	8,90
Forest + CaCO3 + 3 rd KCl	**8**		2,05	-1,13	22,25	-12,47	9,78
Field Experiment 0-10 cm	**9**	0,037	0,99	-0,17	11,34	-1,23	10,16
Field Experiment 40-60 cm	**10**	0,026	1,02	-0,04	11,93	-0,36	11,57

CR_0, equilibrium concentration ratio; α is slope of linear regression between final exchangeable K (EKf) and change in solution K; ß is slope of linear regression between change in non-exchangeable K (Non-Exchangeable K) and initial constraint (Φ); PBCe, potential buffering capacity due to exchangeable K; PBCn, potential buffering capacity due to non-exchangeable K; PBCt, total potential buffering capacity.

In soils with low CEC and/or high acidity, exchange sites tend to be occupied by aluminum (Al_3^+) and hydrogen (H^+) (ALLEONI et al., 2009), resulting in low K adsorption (Figure 1a). Cerrado soils are mainly composed of minerals whose charges vary as a function of pH (ALLEONI et al., 2009). Thus, after the application of limestone and high amounts of K, the exchange sites were saturated with basic catio.

After one crop with ruzigrass and another with maize (T4), the soil started to adsorb more K than it released, as evidenced by the decrease in CR_0 from 0.02 (mol $L^{-1})^{1/2}$ to 0.015 (mol $L^{-1})^{1/2}$. Successive crops clearly decreased the soil's ability to release K (Figure 1b and 1c), and adsorption of the nutrient began. The CR corresponds to the I of K in solution, and CR_0 is the equilibrium point at which K is neither adsorbed nor released (WANG et al., 2004). The decrease in CR_0 suggests that Kne is responsible for maintaining the equilibrium of solution K or the I of solution K (MIELNICZUK; SELBACH, 1978). Supporting this conclusion, Ke was depleted after ruzigrass and maize growth (Table 1). Since Kne was lower at the end of the experiment regardless of CR_0, it can be inferred that Kne was released to Ke (Figure 1).

Figure 1 - Partitioned of K-solution (quantity/intensity - Q/I) about relation of the different fractions of K, represented by the pool ΔK Total, Δ Exchangeable K, Δ non-exchangeable K, with the CR (mol/L)$^{\wedge 1/2}$. The treatments are represented by: a) Forest, b) Forest + CaCo3, c) Forest + CaCo3 + 1^{ft} Maize, d) Forest + CaCo3 + 3rd Maize, e) Forest + CaCo3 + 3rd K, f) Field Experiment 0-10 cm, g) Field Experiment 40-60 cm.



The succession of three maize crops without K application and the low Ke in the soil resulted in an increase in CR_0 compared with the first crop (Table 2). This increase is evidence of K adsorption in all soil K pools up to the first cycle; after the third cycle, the Kne fraction started to release K up to a CR of approximately 0.023 (mol L -1)$^{1/2}$ (Figure 1d). Decreases in soil Ke (Table 1) below 30 mg kg^{-1} (BORTOLUZZI et al., 2005) induced the absorption of Kne by plants (ROSOLEM et al., 1988), potentially leading to an increase in vermiculite (Table 1) with K release from the illite interlayers (HINSINGER; JAILLARD, 1993; GOMMERS et al., 2005; BARRÉ et al., 2008; BORTOLUZZI et al., 2012). Some of this K may have been taken up, and some was bound to the preferential regions, i.e., K bound with higher strength to the clay edges (MOTERLE et al., 2016). Thus, the CR increased between the first and third maize crops because the non-exchangeable pool started to release K to replenish the other soil K fractions (Figure 1d).

Crop succession decreased soil pH (Table 1), which may explain the release of structural K (Figure 1d), since at lower pH, H^+ and Al^{3+} bind to planar exchange sites. Then, the K released is adsorbed at the edges of the clay under higher binding forces (preferential position), causing an increase in the equilibrium point (CR_0).

In the field experiment, the soil content of K and the CEC may have affected the release and adsorption of K. Ke was 2.5 times higher in the surface layer than in the subsurface (Table 1). Since Ke correlates with planar K (MIELNICZUK; SELBACH, 1978), there was likely more planar K in the surface layer acting as a driver of solution K equilibrium. As a result, K was released from the total pool at a depth of 0.0–0.1 m, while at a depth of 0.4–0.6 m, K was adsorbed into the total and exchangeable K pools. At both soil depths, K was released from Kne (Figure 1f and 1g).

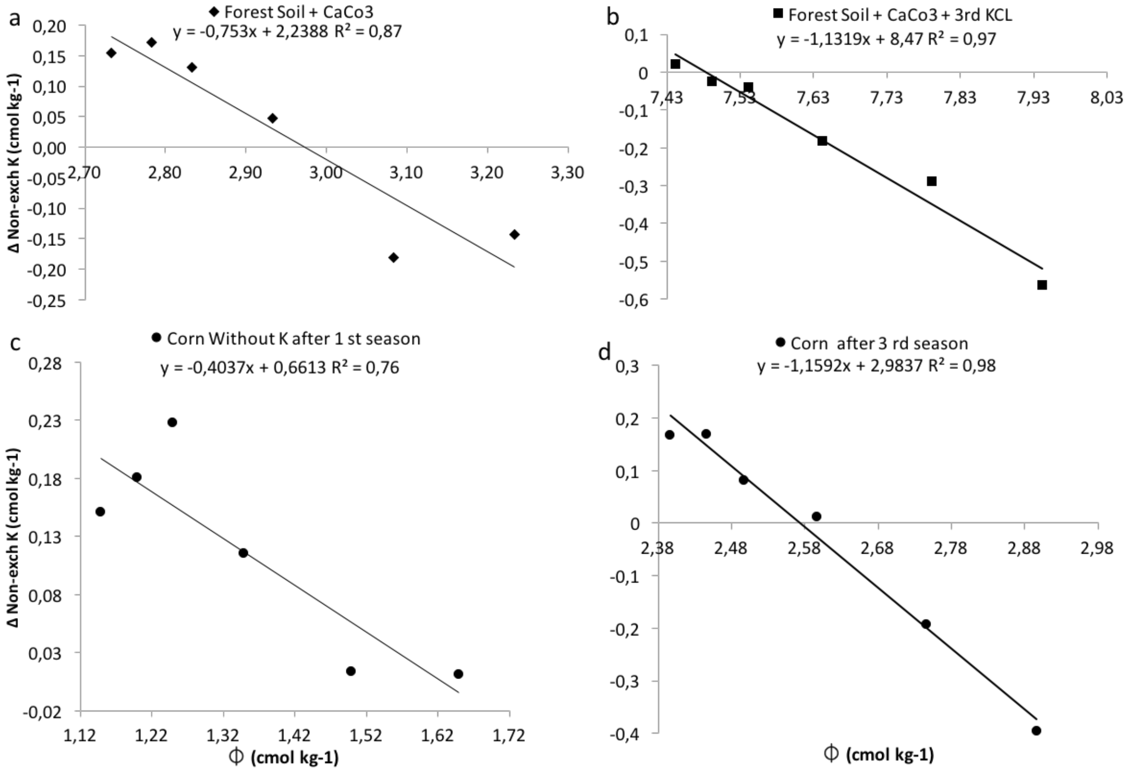
Plants take up K from the Ke and Kne pools throughout the soil profile (GARCIA et al., 2008; ROSOLEM; STEINER, 2017), and cover crop desiccation or plant senescence returns this K to the soil, first in the soil solution and Ke fraction (GARCIA et al., 2008). The increased K (Fig. 1f) may induce migration to preferential K as in the case of T10 (Fig. 1g) if some leaching occurs (WERLE et al., 2008). Supporting this result, ß and PBCn were higher at 0.4-0.6 m than at 0-0.1 m (Table 2), indicating retention of the applied K in the Kne fraction and a contribution of Kne to the solution balance (WANG et al., 2004). When cropped under no-till management, the soil surface experiences constant K input from cover crop senescence or desiccation (CALONEGO et al., 2005).

3.3.2 Partitioning of the total K and K fraction dynamics

Partitioning the Q/I curve revealed that, in general, all sowed soils had a low capacity to adsorb K (Figure 1). The change in total K was not greater than that in the Ke fraction, and low K adsorption was observed after three maize growing cycles. Considering that ß represents the K adsorbed in the Kne fraction and PBCn the potential to maintain solution equilibrium through Kne (Wang et al., 2004), the negative values of these parameters (Table 2) indicate that Kne is a source of K in this soil, which has a low K holding potential when K is applied. Thus, successive crops deplete Ke as well as the K bound to colloids more strongly or at preferential positions (MOTERLE et al., 2016). Moreover, the equilibrium dynamics of Q/I will equilibrate the solution K and Ke with Kne. Tropical soils are usually poor in K-fixing minerals (Azevedo and Vital-Torrado, 2009), and due to their low Ke levels (BENITES et al., 2010; ROSOLEM; STEINER, 2017), the Kne pool can be a source of K to plants (ROSOLEM et al., 1988; BORTOLUZZI et al., 2005). Since K fertilizer recommendations are based on Ke levels, its adsorption, as shown in Figure 1, is important.

The low ability of this soil to adsorb K in the Kne fraction is evident in Figure 2, which shows the relationship between K added, represented by the initial constant (Φ), and its adsorption as Kne. The higher the initial equilibrium point, i.e., the amount of K needed to reach a steady K concentration ($\Delta K=0$), the lower the K adsorption ability in Kne (Figure 2). Thus, in this soil, when K is adsorbed as Kne, it is not fixed into minerals, and the bonding sites must be saturated, indicating that this K is adsorbed into the clay edges with higher strength. This conclusion is supported by the increase in Kne with sequential fertilizer application (Table 1). Studies have shown that nutrients from fertilizer or leached from plant residues left on the soil surface can end up in the non-exchangeable soil fraction (ROSOLEM et al., 2003; CALONEGO et al., 2005; ROSOLEM; STEINER, 2017).

Figure 2 - Change in non-exchangeable K as a function of initial concentration Φ.

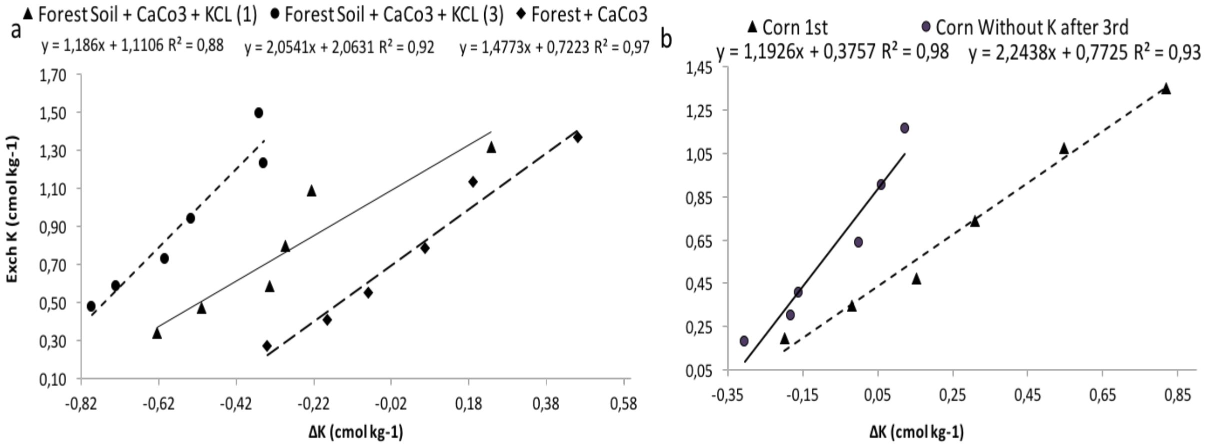


The amelioration of acidity as well as the three K applications without plants resulted in an increase in Φ (Figure 2a, b). In the treatment with lime, which had a negative non-exchangeable ΔK, K release from the non-exchangeable pool occurred as Φincreased (Figure 2a). Conversely, K application increased soil K and Kne, and K was released regardless of the value of Φ (Figure 2b).

While low K adsorption was observed after the first crop (Figure 1c) regardless of the equilibrium point (Figure 2c), after three maize crops, adsorption was observed only at the lower equilibrium points (Figure 2d). These results indicate that at the first maize cycle, Kne taken up was adsorbed to the clay edges, leaving free sites to be filled by K from less-soluble fractions. With the decreases in Ke and Kne after three maize crops (Table 1), K was released from Kne to the other pools (Figure 1d). The increase in vermiculite (Table 1) from the first to third maize cycles shows that K was dissolved from the 2:1 mineral (GOMMERS et al., 2005; BARRÉ et al., 2007; BARRÉ et al., 2009). Crop succession and this K release led to an increase in Ke adsorption (Figure 1c, d) and a decrease in Kne, indicating that the K source was mineral.

The slope of the linear regression between the final exchangeable K (Exch K) and total K (ΔK) (Figure 3), represented by α (Table 2), indicates the magnitude of the migration of solution K to Ke. Since all α values were above 1.0, the change in ΔK was preferentially to Ke, thus confirming the K fraction behavior shown by partitioning (Figure 1) discussed previously.

Figure 3 - Final exchangeable K (EKf) as a function of K in solution or K total (Δ K).



The conversion from Ke to solution K was clearly influenced by the successive crops and continuous addition of K. Liming and the accumulation of K through fertilizer application and successive cropping resulted in an increase in α (Table 2). As soil Ca and Mg increased due to liming (Table 1), the K/Ca + Mg ratio decreased, and K was released from the planar positions, as supported by the decrease in CR_0 (Figure 1b) compared with the treatments without limestone.

In the absence of plant uptake, continued K application results in an equilibrium between the solid and liquid phases (JALALI, 2006). According to this equilibrium, excess K is retained in the non-exchangeable fraction or leached (FOLONI; ROSOLEM, 2008; ROSOLEM et al., 2010).Therefore, soil with low CEC and high Ke and Kne tends to adsorb K in the Ke fraction without generating CR_0 (Figure 1e and Table 2) because the fractions are saturated and in equilibrium.

The depletion of soil K caused by cropping maize also influenced the migration of total K to Ke, as shown in Figure 3b by the negative trend of total K after the first crop. The successive maize crops increased preferential K (Figure 1c, d) due to the increase in CR (MIELNICZUK; SELBACH, 1978), as supported by the data in Figures 2c, 2d and 3b. With the successive crops, the soil Ke and Kne were depleted (Table 1), resulting in K dissolution from the total pool (Figure 3b). Consistent with this observation, vermiculite content increased by 33% due to loss of K from the illite interlayers (BARRÉ et al., 2008; BORTOLUZZI et al., 2012) as the crop sequence progressed and due to the presence of some feldspar in the silt fraction of this soil. Weathering of feldspar through hydrolysis or indirect effects of soil oxireduction (THOMPSON; UKRAINCZYK, 2002) could have resulted in K release directly to the soil solution (Huang, 1977).

In the soil sampled in the field experiment, conversion of both Ke and Kne to solution K occurred at both depths. Thus, approximately 99% and 100% of the change in ΔK was converted into Ke at 0-0.1 m and 0.4-0.6 m, respectively. The conversion rates were expected to differ between the layers due to the lower values of Ke and pH in the subsurface layer; the similar conversions may reflect the greater amount of planar K in the deeper layer due to the lower CR_0 (Figures 1h and 1i). The increased clay and vermiculite content with depth (Table 1) may generate stronger binding due to the higher charge of the 2:1 micaceous minerals in the presence of clay (CHAVES et al., 2015).

3.3.3 Potential buffering capacity (PBC)

The application of lime (T2) and the exhaustion of K (T3 and T4) resulted in increases in PBCt (Table 2) for different reasons. PBC is defined by the ratio of quantity to intensity (Q/I) (WANG et al., 2004). When lime is applied or K is taken up by plants, the proportion of K in solution decreases due to the increases in Ca and Mg, resulting in increased PBC and a decrease in CR_0 (Table 2).

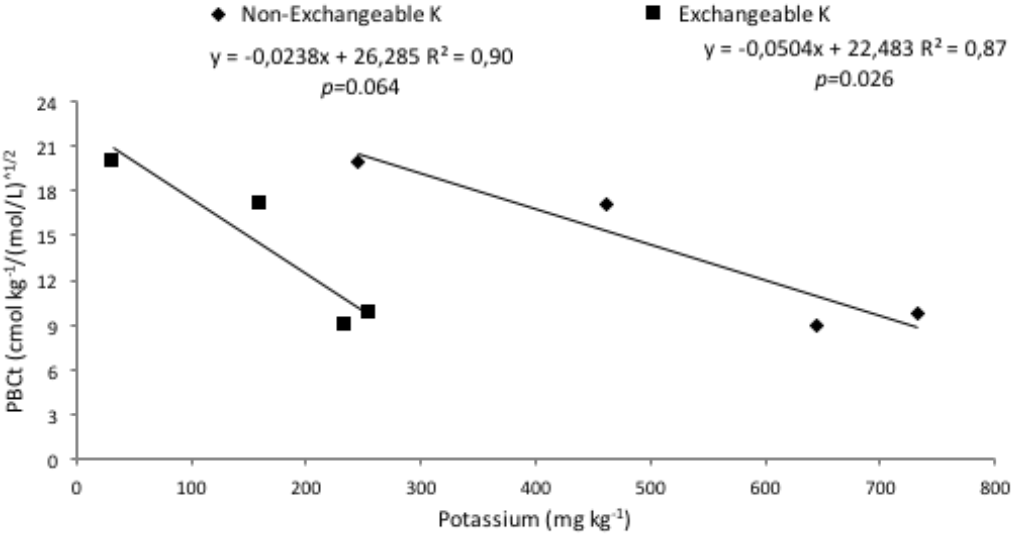
The decrease in CR_0 from T3 to T4, the beginning of K exhaustion, showed that with successive crops, Kne started to release K to maintain the solution K concentration. PBCt was similar in these two treatments despite the difference in CR_0 (Table 2), indicating slower release of the Kne fraction as the solution K concentration decreased (BILIAS; BARBAYIANNIS, 2018). Exhaustion of K by successive crops led to a decrease in I and, consequently, Q/I (equivalent to CR_0) and an increase in PBCk (MIELNICZUK; SELBACH, 1978).

With continuous K depletion by maize (T5) the soil Ke and Kne contents decreased while vermiculite increased (Table 2), showing that K was released from the illite interlayers (BARRÉ et al., 2007) directly to solution. The resulting increase in the Q/I ratio and decrease in PBCt are reflected in the increase in CR_0 (Table 2). The decrease in pH (Table 1) may have contributed to the adsorption of K at preferential sites (Figure 1d) and the migration of K to solution, since the planar sites were likely occupied by H^+ and Al^{3+}.

In the field experiment, trends of higher PBCt and lower CR_0 were observed at both depths of the soil sampled. PBCt was expected to be higher at the surface layer due to the higher content of organic matter (Table 1), which is directly related to CEC (MOTTA; MELO, 2009), Ke and pH. However, CEC may not be the main driver of PBCt, since the CEC was similar between treatments in the soil of environment A. Therefore, for this kind of soil or soils with 2:1 minerals or other charge sources, CEC should not be used as a parameter for estimating PBC or as a basis for K fertilizer recommendation (WANG et al., 2004). However, the increase in clay content with depth may partially explain the increase in PBCt (WANG et al., 2004); decreases in pH can also result in increased PBC (MIELNICZUK; SELBACH, 1978).

The application of K caused the Ke content in the soil to rise to apparent stability or saturation of the exchange sites based on the results for T3 (before K application) and T6, T7, and T8 (three successive applications without plants) (Table 1). The increases were significant from T3 to T6 (80%) and from T6 to T7 (32%), but the increase in Ke from T7 to T8 was only 9%. The responses of PBCn and PBCt were similar, with decreases up to the second K application, whereas PBC was stable from the first application (T6). PBCt decreased as Ke and Kne in the soil increased (Figure 4). As PBC increased, CR_0 decreased to zero in soil with higher levels of Ke and Kne, resulting in the release of K from total K or Kne instead of adsorption. Adsorption of K from Ke only was observed above 0.04 $(mol\ L^{-1})^{1/2}$ (Figure 1e); as a result, PBCn was negative, while PBCe was higher than PBCt. Both PBCe and PBCt were positive because these are sources of K for the Kne fraction. The increase in the Kne fraction was not due solely to liberation from minerals but was also the result of both K fertilization and leaching of K from decaying plants (ROSOLEM et al., 2006; EGUCHI et al., 2015; ROSOLEM; STEINER, 2017).

Figure 4 - Simple correlation between soil K content and PBCt (total potential buffering capacity) with the treatments 3 (representing the exchangeable K in equilibrium with the non-exchangeable K, Bortoluzzi et al., 2005) and treatments where the soil had received K applications (treatments 6, 7 and 8).



High levels of soil Ke and Kne led to a decrease in PBCt (Figure 4) due to the increase in intensity factor (I). Consistent with this result, migration to the Ke fraction occurred when ΔK was negative (Figure 3a) as well as when α was greater than 1 and ß was negative (Table 2), indicating that Ke was supplying K to the Kne fraction.

In the Cerrado soil without treatment and the soil with three applications of K, PBCt increased and increasing vermiculite (Table 1 and 2). Soils with more vermiculite tend to have higher charge and more activity (AZEVEDO; VIDAL-TORRADO, 2009) and thus have a greater capacity to retain K in the solid fractions (Figures 3a and 1g). The increase in PBCt and decrease in CR_0 (Table 2) can increase the probability that the soil will preferentially accumulate K (Kne).

3.4 CONCLUSION

Soils with a predominance of 1:1 minerals initially have greater potential to release than to adsorb K. However, higher adsorption may be observed after acidity amelioration and after successive crops. This K may be adsorbed to planar sites or to clay edges. The decrease in CR with successive crops alters the K dynamics, and the soil solid phase starts to adsorb K due to the low I of the solution K. As Ke is depleted, more charges become available. When K is depleted in tropical soils with a low content of K-source minerals, the exchangeable sites must be saturated before K can be released to the soil solution. Conversely, in soils high in Ke and Kne, the soil solution is saturated, and K is adsorbed into less-soluble pools or leached. After the saturation of the soil solution and exchangeable sites, K will be adsorbed with higher strength in the clay edges and become Kne, thus reducing its availability to plants. The PBC of this soil is a function of Ke. Since the Kne is always releasing K, it can be considered a recharger of the other soil K pools.

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CHAPTER 4 - WEATHERING OF POTASSIUM MINERALS IN TROPICAL SOILS UNDER A LONG-TERM INTENSIVE PRODUCTION SYSTEM.

ABSTRACT

Weathering of minerals can be a significant source of K to plants. Exudation of organic acids by plant roots plays a central role in the intensity of mineral sources of K, resulting in feedback for plant nutrition. However, some minerals may immobilize human-applied K fertilizers by binding with greater strength than regular adsorption to clay minerals. Since Brazilian agriculture is heavily dependent on imported K (more than 90% of raw material for KCl fertilizer production is imported), improvements in the use of natural and sequestered sources of K in soil are urgently needed. The aim of this work was to study the behavior of soil minerals at the limit of K exhaustion in a tropical soil in which the clay fraction is dominated by kaolinite. Two experiments were carried out: 1) a 33-month-long field trial (in soil previously cultivated for more than 15 years) using three treatments: soybean/ruzigrass (succession), soybean/fallow (succession) and ruzigrass (monoculture) and 2) a greenhouse experiment with soil samples collected at a depth of 20-40 cm in a forest area (Cerrado biome) neighboring the field experiment. The soil in pots was planted with ruzigrass and then subjected to successive crops of soybean, corn or ruzigrass or a control treatment without plants, all with and without K application. The forest soil contained a small amount of illite (a micaceous 2:1 phyllosilicate) with the ability to release non-exchangeable K (Kne), minerals with greater susceptibility to weathering (trioctahedral mica), and vermiculite (Vm). Illite was also present in the cropped field soil, but the Vm had been transformed into hydroxy-interlayered vermiculite (HIV), possibly due to the acceleration of weathering due to K export by crop harvesting and other agricultural practices. The ruzigrass-soybean rotation affected the partitioning of K into different levels of plant availability but did not rapidly modify the K minerals. However, raising annual crops over the previous 15 years resulted in mineralogical modifications of the soil.

Keywords: Weathering tropical soil. Nonexchangeable K. Thermal analysis. XRD analysis.

4.1 INTRODUCTION

Soil is constantly evolving, and the evolutionary process may have an important influence on soil fertility. This process includes weathering of soil minerals by chemical, physical or biological mechanisms (MELO et al., 2009; KÄMPF et al., 2009). The intensity of mineral weathering is determined by the environment and can be characterized by assessing the mineralogical constituents of the soil.

Primary minerals that contain K in their structure, such as micas (FANNING; FANNING, 1989) and K-feldspars (CURI et al., 2005; BORTOLUZZI et al., 2005), are of great agricultural interest, particularly 2:1 phyllosilicates, which can be reservoirs of both exchangeable (Ke) and non-exchangeable (Kne) K. Feldspars, by contrast, irreversibly release K during weathering. In addition, feldspars tend to be concentrated in coarse soil fractions such as sand and silt (HUANG, 1977). These particles have small specific surface areas and thus have lower rates of weathering compared to the clay fraction. K-bearing phyllosilicates can be found in all soil fractions, but the minerals found in the clay fraction are the most reactive (FANNING; KERAMIDAS, 1977; CHAVES et al., 2015). In tropical soils, both chemical and biological drivers of hydrolysis and oxidation are active throughout the year and may continuously influence K concentrations in the soil solution (HUANG, 1977; THOMPSON; UKRANCZYK, 2002; KÄMPF et al., 2009; ZÖRB et al., 2014; CHAVES et al., 2015).

The most important group of K-rich clay minerals is 2:1 phyllosilicates, such as trioctahedral (biotite and phlogopite) and dioctahedral (e.g., muscovite and illite) micas (KÄMPF et al., 2009; EGUCHI et al., 2015). A considerable portion of these minerals are considered “non-exchangeable” because they are not detected by the standard laboratory ion exchange methods used to extract K. However, under specific conditions, soil Kne can be accessed by some plant species.

Tropical soils are typically poor in 2:1 phyllosilicates and rich in kaolinite and oxides (MELO et al., 2003; KAMINSKI et al., 2010; BENITES et al., 2010; ROSOLEM; STEINER, 2017). However, even small amounts of 2:1 minerals can play a significant role in K dynamics for crops (ROSOLEM; STEINER, 2017). K dynamics at the interface between phyllosilicates and the soil solution may be very complex if multiple 2:1 phyllosilicates are present in the soil (e.g., micas like illite, vermiculite (Vm), smectites, hydroxy-interlayered vermiculite or smectite (HIV or HIS)).

Agricultural management systems in which plants are actively growing throughout the year also undergo constant chemical and biological processes in the soil. Biochemical processes such as root exudation of organic anions and ion absorption from the soil solution may promote weathering of primary minerals, significantly impacting the dynamics of structural K (HINSINGER; JAILLARD, 1993; GOMMERS et al., 2005; BARRÉ et al., 2007; GARCIA et al., 2008; BARRÉ et al., 2009; MOTERLE et al., 2016; ROSOLEM; STEINER, 2017). Some plants, such as *Brachiaria* and soybean, can access soil Kne (MOJALLALI; WEED, 1978; ROSOLEM et al., 1993; ROSOLEM et al., 2012) and therefore may intensify the weathering of mica through vermiculitization and further formation of HIV (HINSINGER et al., 1993; HINSINGER; JAILLARD, 1993; GOMMERS et al., 2005).

Highly weathered tropical soils may provide K to plants by depleting this element from the layers of 2:1 minerals. The loss of K from these minerals may intensify weathering. The aim of this work was to study the availability of K in a tropical soil to successive crops without application of K in order to provide a scenario of K exhaustion to properly observe the changes in 2:1 minerals and their impact on K dynamics in the soil-plant system.

4.2 MATERIALS AND METHODS

The soil studied was a Haplic Plinthosol (FAO – 2014) in the municipality of Nova Xavantina, Mato Grosso State, Midwest Brazil, 14º63'99"S and 52º14'81"W, 300 m. a.s.l. The climate, according to Köppen's classification, is Aw type, with rains in summer and a dry winter. The initial soil chemical analyses are shown in Table 1 and were carried out in the Soil and Plant Laboratory at UNESP (Botucatu State University - Faculty of Agrarian Sciences), Brazil. The mineralogical analyses were conducted at Iowa State University and the National Laboratory for Agriculture and the Environment in Ames, Iowa, USA.

Table 1 - Selected soil chemical characteristics and particle size distributio

Soils depth cm	pH[1]	P[2]	Exc K[3]	Non-exc K[4]	Ca	Mg	M.O.	Clay	Silt	Sand
		(mg dm^{-}	------------	(mmolc dm$^{-3)}$	--------------		(g dm^{-3})		(g kg^{-1})	
Forest Soil	3.9	7.0	1.32	5.9	5	2	12	275	175	550
0-10	5.2	16.8	3.4	1.2	31.2	11.4	19.8	180	100	720
10-20	4.4	4.4	1	2.17	14.6	7.7	10.6	230	125	645
20-40	4	1.3	0.5	2.73	8.1	5.3	8.8	280	100	620
40-60	3.9	0.8	0.10	3.8	7	4.5	7.5	370	90	540

Analyses done according to (Raij et al. 2001)[1] In $CaCl_2$;[2,3] extracted with resin [4] extracted with boiling HNO_3

4.2.1 Field experiment

The experimental site was covered by native Cerrado (savannah) until 1997, when the vegetation was cleared, the soil was amended, and pasture species were sown. The area was maintained as pasture with intensive cattle grazing without fertilization until 2008, when the forage was eliminated and the soil was amended with limestone. Then, soybean cultivation was started in a no-tillage system in the summer, followed by pasture with ruzigrass in the offseason (Fig. 2). In the first three years of soybean cultivation, 60 kg ha^{-1} of K_2O was applied per year. No K was applied while the soil was occupied by pasture or after 2011.

In April 2015, the experiment was started with the following treatments: soybean/ruzigrass (soybean in spring-summer and ruzigrass in autumn-winter), soybean/fallow (soybean in spring-summer and absence of plants in autumn-winter) and ruzigrass without soybean for 33 months (monoculture). In the treatment with ruzigrass in the autumn-winter period, ruzigrass was sown in February of each year, when soybean was at the R 5.5 phenological stage (FEHR; CAVINESS, 1977), according to the methodology of Pacheco et al. (2008). In the treatment designated as ruzigrass (monoculture), ruzigrass was sown in February 2015 and remained until November 2017. In all treatments, no K was applied. The experiment was carried out in randomized complete blocks, with four replicate plots per block.

After the soybean harvest, the fallow plots received herbicide application to control weeds in the offseason and keep the plots free of any plants during the winter. Soybean sowing was always performed in November, 15 days after desiccation of the cover plants. The basic fertilization in the sowing furrows consisted of 100 kg ha^{-1} of P_2O_5 via monophosphate, and nitrogen was supplied via biological fixation.

Soil samples were collected in February and November 2017 at depths of 0-10, 10-20, 20-40 and 40-60 cm with the aid of a 5.0-cm-diameter metal probe. Three samples were collected for each depth interval and combined as a composite sample for the plot. In February, soybean and ruzigrass plants were collected at the same time points as the soil samples, and in November, the aboveground biomass and residues of the previous crops were sampled in the fallow plots. All vegetation inside a metal frame with dimensions of 0.5 m x 0.5 m was harvested. After drying to constant weight in a forced air oven at 60°C for 72 hours, the plant tissues were ground using a hammer mill and then wet digested to determine the K concentration (MALAVOLTA et al. 1997).

Figure 2 - Evolution of the environment in the area of the experiment for over 15 years. Before the beginning of the crops (1998), all the soil under native forest (a), area under forage crop (2010) (b), Beginning of the field experiment (February 2016) (c).

Source: the Landsat – 5 and 8 EarthExplorer.

4.2.2 Greenhouse experiment

Soil for the greenhouse experiment at the UNESP campus at Botucatu-SP was collected from a forested area (Fig. 2a) next to the field experiment at a depth of 20-40 cm. After the soil was collected, the acidity was corrected by applying dolomitic limestone (CaO = 28%, MgO = 12%, with an effective calcium carbonate equivalent (ECCE) of 96%) to raise the base cation saturation to 70% (ROSOLEM et al., 1993). Then, the soil was conditioned in pots with a capacity of 12 L. Subsequently, the soil was fertilized with 175 mg kg^{-1} of P as triple superphosphate, 13 mg kg $^{-1}$ of S as calcium sulfate, 1.75 mg kg^{-1} of Cu as copper sulfate, and 1.75 mg kg^{-1} Zn as zinc sulfate. The experiment was conducted from November 2016 to November 2017 and included successive corn, soybean or ruzigrass crops, without application of K fertilizer.

Three ruzigrass seedlings were transplanted per pot, and after 45 days, they were cut close to the soil surface. After cutting the grass, two soybean seedlings, one maize seedling or three ruzigrass seedlings that had been germinated in a sand tray for 7 days were transplanted to the pots. The soybean was inoculated with *Bradyrhizobium japonicum* (SEMINA 5079-CPAC 15 and SEIMNA 5080-CPAC7) to supply nitrogen. In the corn and ruzigrass treatments, urea was applied 15 and 21 days after transplanting at a dose of 80 mg N kg $^{-1}$ for maize and 25 mg N kg $^{-1}$ for ruzigrass.

The soybean and corn plants were cultivated for approximately 90 days i.e., to the peak K accumulation in the plants, for three sucessive cycles. The ruzigrass was cut after 45 days and cultivated for four additional 45-day cycles. Pots without plants were used as a control. All treatments were performed with and without K application. K was applied at a rate of 200 mg kg^{-1} of K. The soil was sampled after each harvest of the shoots. Soil moisture was maintained at approximately 80% of field capacity through the application of deionized water. The plants were grown with a cycle of 13 h of light per day and temperatures between 23 and 35°C.

4.2.3 Chemical analyses of soil and plants

The soil samples were air dried and ground to pass a 2-mm sieve. Ke was extracted with an ion exchange resin (RAIJ et al., 2001), and Kne was obtained from the difference between the amount of K extracted with boiling 1.0 mol L^{-1} HNO_3 and Ke (KNUDSEN et al., 1982).

4.2.4 XRD analysis

Before mineralogical analysis, organic matter was removed from the fine earth fraction of the samples by treatment with hydrogen peroxide and heating in a water bath. Then, the samples were dispersed in NaOH with shaking. Sand was separated by wet sieving at 53 µm. Silt and clay were separated by sedimentation based on Stokes' law. An aliquot of the clay suspension was washed with 1 mol L^{-1} $MgCl_2$ by shaking and centrifugation, washed with deionized water and 95% ethanol until the supernatant was Cl^- free by the $AgNO_3$ test, and then freeze-dried. Then, 15 mg of each Mg-saturated clay sample was suspended in 25 mL of deionized water by using a microtip sonicator probe for 30 seconds at a power level of 4. A similar aliquot of each clay sample was saturated with 1 mol L^{-1} KCl, washed with deionized water to remove excess Cl^-, and suspended in 25 mL of deionized water as before. Each clay suspension was filtered through a 0.45-µm membrane filter and transferred to a glass slide as an oriented sample for X-ray diffraction (XRD) analysis (a modification of the method of (DREVER, 1973)). Each oriented, Mg-saturated sample was sprayed with a 1:1 glycerol:water solution and placed in a desiccator for 24 h. Air-dried sand and silt fractions were prepared for XRD analysis as random powder mounts and oriented samples, respectively.

XRD analysis was conducted on the oriented samples using CuKα radiation generated at 30 mA and 40 kV using a Siemens D5000 diffractometer (Bruker AXS, Inc., Madison, WI). The scanning speed was 0.05° 2θ min^{-1}. To estimate the mass percentage of the clay minerals from the XRD patterns, the mineral intensity factors (MIFs) were determined for model layer silicates (Vm, mica, and kaolinite) by using NEWMOD (MOORE; REYNOLDS, 1997) and the procedures of Harris and White (2008).

4.2.5 Thermal analysis

Freeze-dried clay samples were analyzed by thermogravimetry using a TA Instruments 2960 SDT (TA Instruments, New Castle, DE). Approximately 10 mg of each sample was heated in air from 25°C to 1000°C and monitored. The thermogram was analyzed by TA Instruments Universal Analysis 2000 software. Mass loss between 300 and 400°C was attributed to goethite, and mass loss between 400 and 600°C was attributed to kaolinite.

4.2.6 Quantification of clay minerals

To quantify the clay minerals in the soil samples, the TA (thermal analysis) technique was used. A standard curve was first created althrough TA analysis of samples with known mixtures of kaolinite and quartz of 100% and 0%; 50% and 50%; 25% and 75%; and 0% and 100%, respectively. The resulting linear equation was used to quantify the percentage kaolinite content in the soil samples as described previously (KARATHANASIS; HAJEK, 1982). Four samples from the field experiment and three from the greenhouse experiment were chosen for TA, and the kaolinite contents estimated from these treatments were extrapolated to the other samples because the kaolinite content should not be affected by the treatments used. These calculations are presented in the appendix.

4.2.7 Non-exchangeable K absorption

As proposed by Rosolem et al. (2012), the K absorbed from Kne after cultivation was calculated from the difference between the total accumulated by the plants after cultivation and the decrease in Ke after the cycles of cultivation without application of K using the following equation:

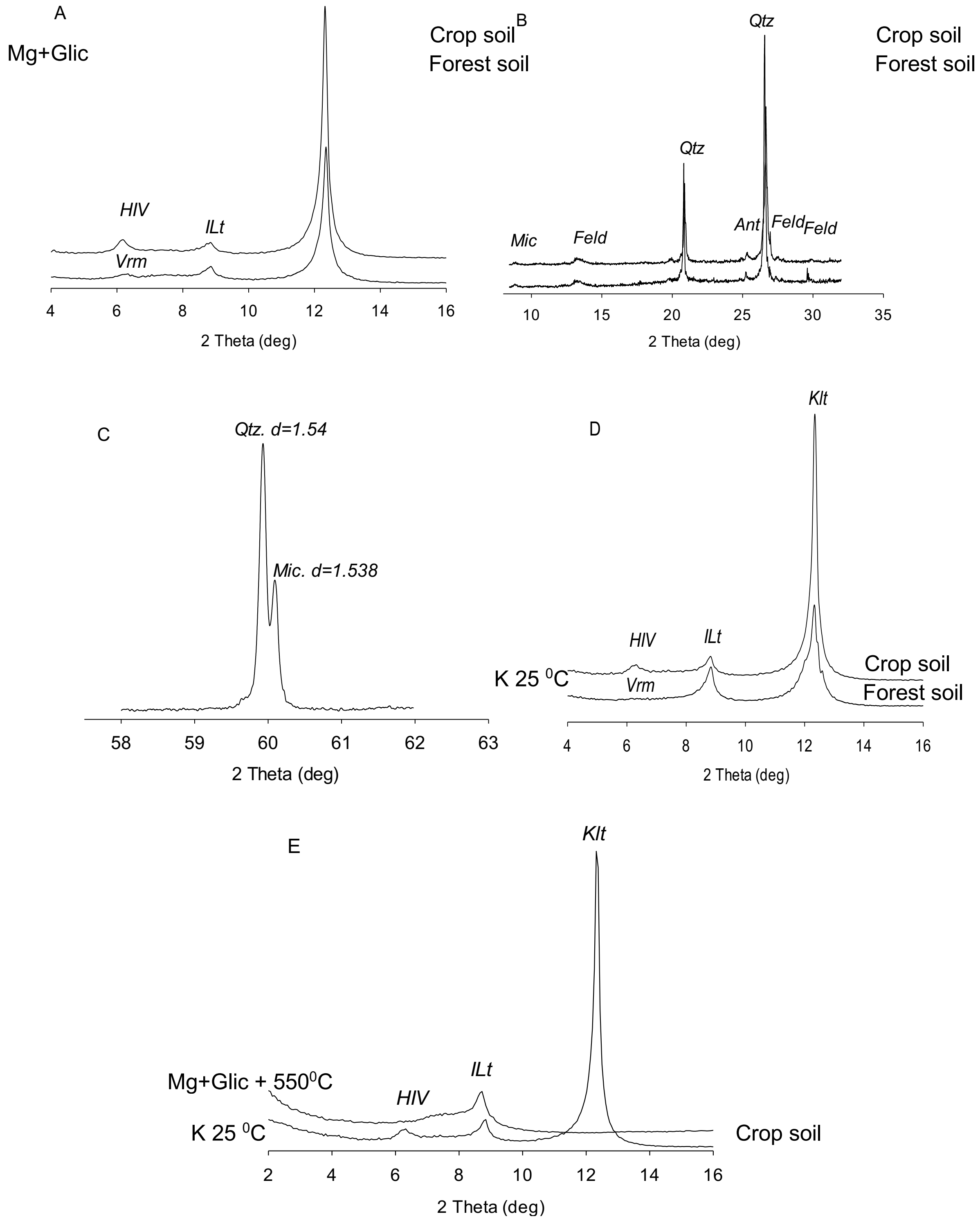
$$Kne_{Abs} = K_{AbsT} - [(Ke_{Bfc} – Ke_{Afc}) + K_{Fert}] \qquad (1)$$

where Kne_{Abs} is K absorbed from non-exchangeable forms; K_{AbsT} is total K absorbed; Ke_{Bfc} is exchangeable K before cultivation; Ke_{Afc} is exchangeable K after cultivation; and K_{Fert} is K applied as fertilizer.

4.3 RESULTS

The XRD analyses showed that quartz, feldspar, and hematite were predominant in the sand and silt fractions of both soils (forest and crop), while anatase and mica were present in the silt fraction (Fig. 1b). The mica in the silt fraction was trioctahedral (peak (060) at 1.538 nm) (Fig. 1c). In the crop soil, the clay fraction was composed of Vm (forest soil) and vermiculite with interlayered hydroxy Al (HIV). Illite, kaolinite, and goethite were identified in both soils (Fig. 1a). The XRD pattern showed a peak at 0.154 nm in the 060 (hkl) plane, indicating trioctahedral illite.

Figure 1 - The x-ray diffraction patterns of clay and silt fractions of a soil sample under intensive grain and forage cultivation (crop soil) and a comparable soil under forest (Cerrado) (forest soil). Silt (b, c), Clay (a, d, e). HIV Hydroxy interlayered vermiculite, Vm- Vermiculite, ILt- Illiteillite, Klt-Kaolinite.



The Vm peaks in the forest soil were confirmed after saturation with K, as collapse of this peak occurred at 25°C, whereas Vm with HIV did not collapse in cultivated soil (Fig. 1d). Heating of the sample (Mg 550°C) confirmed the presence of kaolinite based on the disappearance of the corresponding peaks (Fig. 1e). In addition, the formation of a shoulder between 7 and 8°2θ demonstrated the incomplete collapse of Vm to 1.0 nm, indicating the presence of hydroxy polymers as expected in HIV (Fig. 1e). Modification of the environment due to use of the soil over the last 15 years (Fig. 2) caused a change in Vm to HIV (Fig. 1d), as well as an increase in illite, compared to the native soil (Tables 2 and 3).

Table 2 - Quantitative analysis of the clay minerals in the forest soil in the greenhouse experiment and exchangeable K (Ke) and non-exchangeable K (Kne) and or non-exchangeable K (Kne uptake) taken up by plants

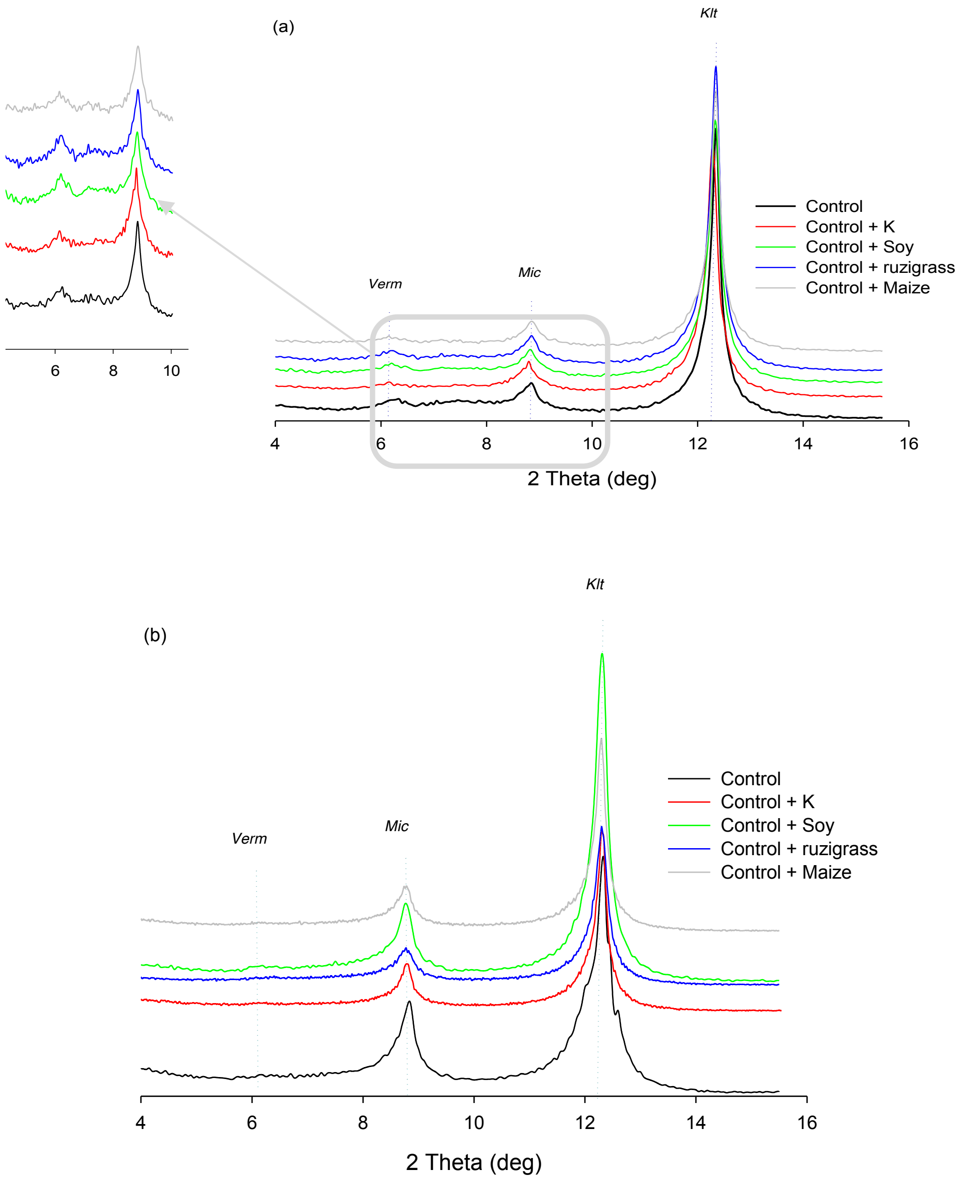
Minerals	% of the minerals								
	Control	Ruzigrass 1st Crop	Ruzigrass 5th Crop	Control+ 1st K	Control+ 3rd K	Soy 1st Crop	Soy 3rd Crop	Maize 1st Crop	Maize 3rd Crop
Kaolinite	67	67	67	67	67	67	67	67	67
Vermiculite	1.47	1.73	2.31	2.21	1.26	1.29	2.87	1.3	1.78
Goethite	15	15	15	15	15	15	15	15	15
Ilita	16.53	16.27	15.69	15.79	16.74	16.71	15.13	16.7	16.22
Contents of K in soil									
Ke (mg kg^{-1})	20.99	10.44	9.33	151.26	138.9	10.84	11.13	10.58	9.53
Kne (mg kg^{-1})	237.78	239	166	462	766.59	214	182.37	200	180.7
Kne Uptake (g pot^{-1})	-	0.58	0.37	-	-	0.99	0.22	0.87	0.48

Table 3 - Quantitative analysis clay minerals in the field soil at the depths sampled and the content of the exchangeable K (Ke) and non-exchangeable K (Kne) in soil

Mineral	% of the minerals									
	Control field	Ruzigrass	Soy/Fallow	Soy/Ruzigrass	Average	Ruzigrass	Soy/Fallow	Soy/Ruzigrass	Average	Average
	February					November				
0-10 cm										
Kaolinite	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00
HIV	2.12	2.88	3.17	3.36	3.14	2.49	2.96	1.97	2.47	2.81
Goethite	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00
Ilita	18.88	18.12	17.83	17.64	17.86	18.51	18.04	19.03	18.53	18.20
Contents of										
Ke (mg kg-	133.00	70.00	193.00	44.00	102.33	91.00	94.00	59.00	81.33	91.83
Kne (mg	52.00	166.00	145.00	183.00	164.67	120.00	113.00	87.00	106.67	135.67
10-20 cm										
Kaolinite	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00
HIV	3.42	3.68	3.24	3.03	3.32	1.97	3.24	3.08	2.76	3.04
Goethite	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00
Ilita	17.58	17.32	17.97	17.76	17.68	19.03	17.92	17.76	18.24	17.96
Contents of										0.00
Ke (mg kg-	43.00	40.00	12.00	33.00	28.33	58.00	59.00	57.00	58.00	43.17
Kne (mg	94.00	203.00	198.00	163.00	188.00	144.00	103.00	118.00	121.67	154.83
20-40 cm										
Kaolinite	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00
HIV	3.80	3.50	3.29	3.05	3.28	3.17	3.35	2.62	3.05	3.16
Goethite	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00
Ilita	17.20	17.50	17.95	17.71	17.72	17.83	17.65	18.38	17.95	17.84
Contents of										
Ke (mg kg-	39.00	32.00	21.00	25.00	26.00	83.00	44.00	65.00	64.00	45.00
Kne (mg	118.00	158.00	200.00	190.00	182.67	127.00	154.00	117.00	132.67	157.67
40-60 cm										
Kaolinite	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00	61.00
HIV	3.14	4.20	3.77	3.15	3.71	2.75	3.02	2.45	2.74	3.22
Goethite	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00	18.00
Ilita	17.86	16.80	17.23	17.85	17.29	18.25	17.98	18.55	18.26	17.78
Contents of										
Ke (mg kg-	28.00	39.00	28.00	21.00	29.33	53.00	60.00	60.00	57.67	43.50
Kne (mg	238.00	163.00	193.00	209.00	188.33	177.00	172.00	118.00	155.67	172.00

No change was observed in the symmetry of the peaks in the XRD patterns for the forest soil after successive crops of ruzigrass, soybean, and corn (Fig. 2). There was no change in Vm to HIV (Fig. 3b), but Vm increased by 125%, 40% and 82% after the last crop of soybean, corn, and ruzigrass, respectively, compared to the control (Table 2).

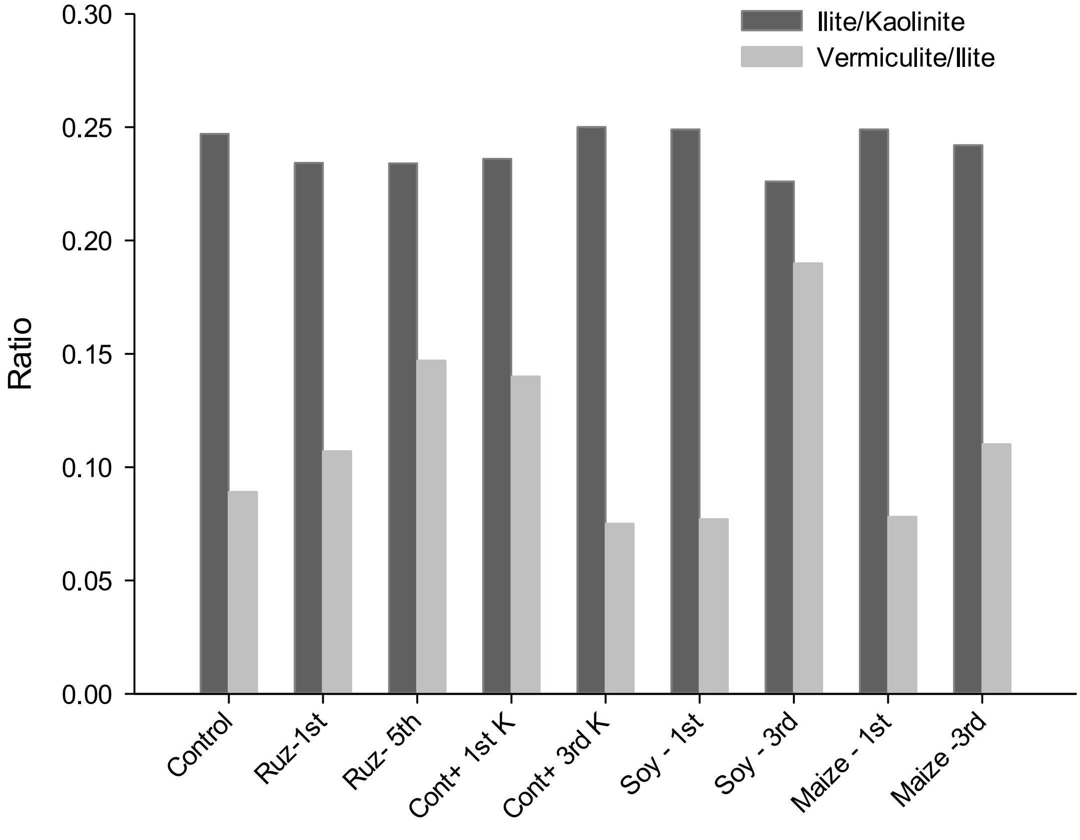
Figure 3 - The X-ray diffraction pattern of clay, of a soil sample under forest soil after crops after successive potting cultures *. Saturated with Mg and solvated with glycerol (a); saturated with K (b). Vm- vermiculite, ILt- illite, Klt- kaolinite.



This result suggests that illite was transformed to Vm because continuous cropping resulted in exploitation of Kne by the plants, resulting in an increase in the Vm/illite ratio (Fig. 4) and a concurrent decrease in Kne content compared to the control after the last crop of each species. The changes in the percentage of Vm, the

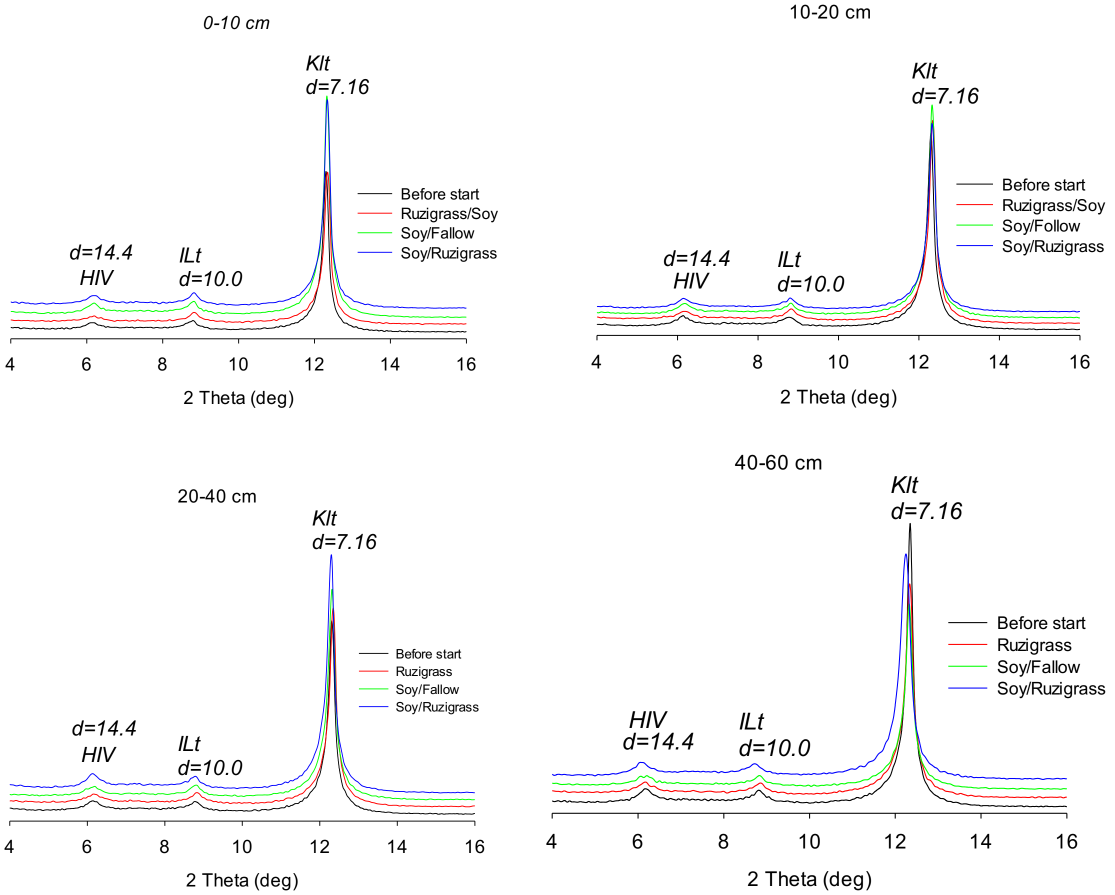
Vm/illite ratio and the Kne content in the soil were greater after the last crop of each species compared with the first crop (Table 2). The change in this ratio was greatest for soybean and smallest for maize. By contrast, the application of K in the pots without plants caused a decrease in the percentage of Vm and an increase in illite (Table 2). Thus, the Vm/illite ratio depends on the K dynamics/availability in soil solution (Fig. 4).

Figure 4 - Quantitative thermal analysis of clay minerals, forest soil under successive greenhouse crops. Sampling after cultivation 1ft e 5th (ruzigrass) e 1st 3rd (soybean and maize) and in control with application of K, after 1st 3rd application . Ratio Illite/Kaolinite and Illite/vermiculite.



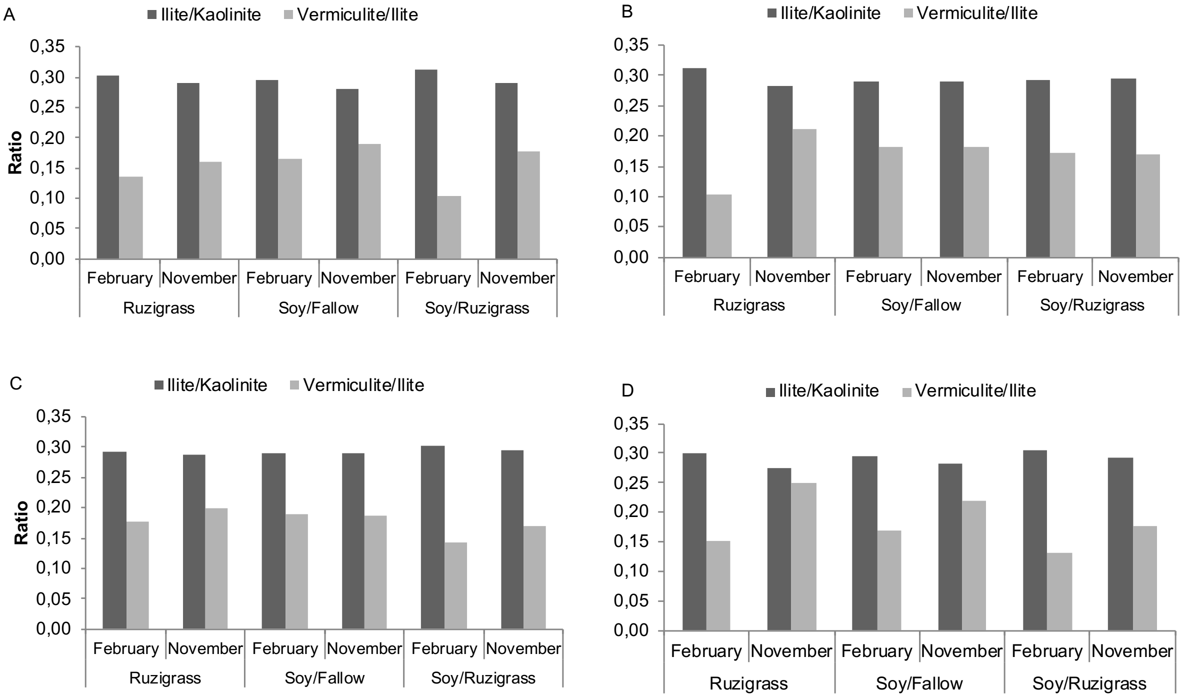
In the field experiment, after two harvests without the use of K fertilizer, no modification of the XRD patterns was observed at any depth of sampling (Fig. 5), although there was a change in Kne over time (Table 3). Likewise, the HIV/illite ratio did not undergo significant modifications (Fig. 6). However, at a depth of 0-10 cm, there was a positive correlation between Kne and HIV (Fig. 7). At this depth, in the soybean/ruzigrass treatment, there was an increase in the HIV/illite ratio between the February and November samplings (Fig. 6a). At the 10-20 cm depth, a decrease in this ratio occurred in the treatment with ruzigrass (Fig. 6b).

Figure 5 - The X-ray diffraction pattern of clay, soil samples at depth of 0-10, 10-20, 20-40 and 40-60 cm soil of the field experiment (crop soil). Saturated with Mg and solvate with glycerol. HIV-Hidroxy interlayer vermiculite, ILt- Illite, Klt- Kaolinte.



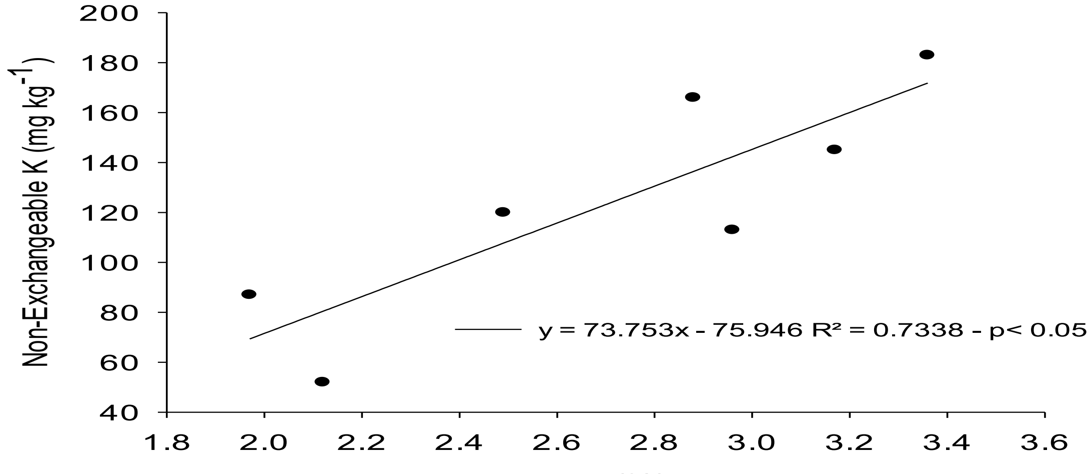
At a depth of 40-60 cm (Fig. 6d), the HIV/illite ratio decreased in all treatments during the same period. Thus, influences of depth and plant species were evident. In the treatment with ruzigrass, the HIV/illite ratio increased with depth in the soil profile sampled in February, with an increase of 50% at 40-60 cm compared with 0-10 cm. No changes were observed in the other treatments (Fig. 8).

Figure 6 - Quantitative thermal analysis of clay minerals in soil from the field experiment. Sampling in February and November 2017 at depths of 0-10 cm (a), 10-20 cm (b), 20-40 cm (c) and 40-60 cm (d). Ratio between Illite / kaolinite and Illite / vermiculite.



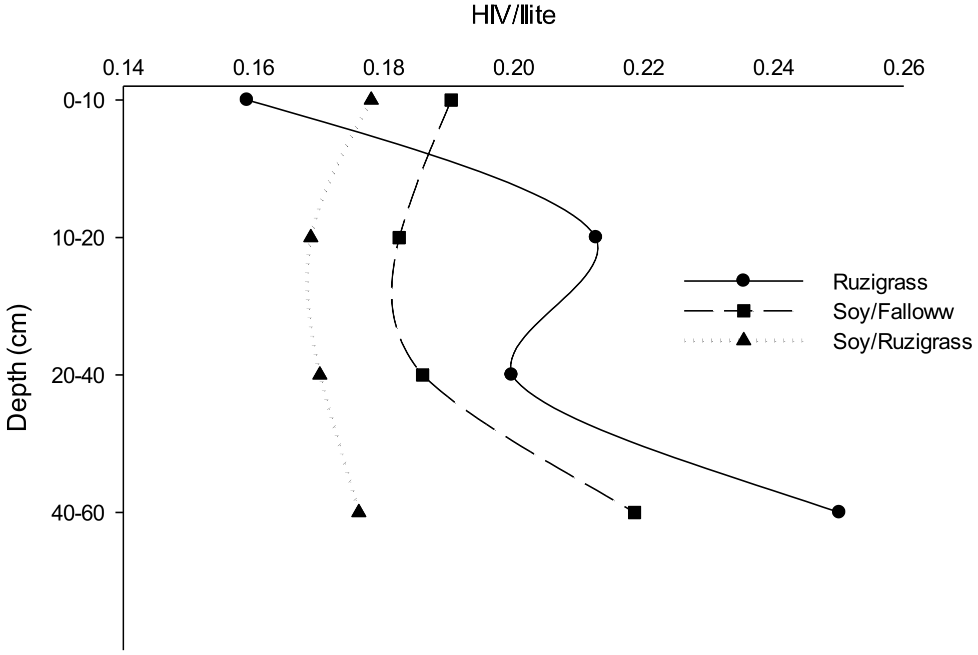
The Ke and Kne levels in the soil changed over time compared with the control soil. Ke mainly decreased on the surface (31%) (Table 3). By contrast, Kne increased by 160% at 0-10 cm, 64% at 10-20 cm, and 33% at 20-40 cm but decreased by 28% at 40-60 cm. When the two sampling times were compared (February and November), Ke levels increased at all depths except 0-10 cm, whereas Kne decreased.

Figure 7 - Simple correlation between non-exchangeable K content in soil and percentage of Hydroxy interlayer vermiculite in the field experiment.



In the crops without K application, the percentage of HIV changed only at a depth of 0-10 cm, with an increase of 32% compared to the control field. The time of sampling (February and November) also influenced the percentages of HIV. In the November sampling, the mean percentages of HIV were smaller than those at the February sampling throughout the soil profile (Table 3). However, at 10-20 cm, ruzigrass generated a significant decrease in HIV on the order of 55%.

Figure 8 - Ratio HIV/Illite with the behavior along the depth in the field experiment in the sampling of February 2017.



4.4 DISCUSSION

Soils on mature surfaces and in tropical environments tend to present only very resistant primary minerals due to the intensity and duration of weathering. The presence of feldspar in both the silt and clay fractions (Fig. 1a and 1b) indicates that these soils are still in the initial/intermediate desilication stage, with conditions for 2:1 mineral formation and/or permanence. The parent material of this soil must contain micaceous minerals, which can still be found in the silt fraction (Fig. 1c). The presence of Vm is likely associated with the weathering of trioctahedral mica (probably biotite). This transformation has long been known (MORTLAND et al., 1956) and studied in detail (BANFIELD; EGGLETON, 1988).

In addition, K-feldspars can be indirectly altered to 2:1 phyllosilicates or may contribute to the maintenance of the bissialitization stage (and preservation of altered Vm and biotite) by maintaining high silica activity in solution (DANESHVAR; WORDEN, 2018). Due to the surrounding conditions and the fluctuation of silica and aluminum activity, illite neogenesis may also occur, as indicated by the disappearance of the peak in the X-ray diffractograms at 7 nm after heating at 550°C, which also ruled out the presence of geogenic chlorite (Fig. 1e).

The soil under study contained at least two potential K sources (K-micas and K-feldspars) and two routes of formation of 2:1 phyllosilicates: by transformation of micas into illite and subsequent transformation into Vm and by neoformation of 2:1 phyllosilicates from the elements made available by the dissolution of feldspars and other intermediate-solubility silicates present in the source material. However, the details of these processes are not the objective of this work.

The action of plants is an important agent of weathering and involves several mechanisms, such as the exudation of organic acids by roots, the absorption of ions from the soil solution, and the sheltering of microbial communities in the rhizosphere (VELDE and BARRÉ, 2010). The conditions created by these mechanisms alter the structure of the minerals, such as by favoring the exchange of K in the interlayers via H^+ exchange (BARRÉ et al., 2007; KÄMPF et al., 2009). The consumption of K by plants may force its release from the mica/illite structure, resulting in modification of the mineral (MOTERLE et al., 2016). In agricultural fields, this K is partially exported by the crop, accelerating the process of weathering by decreasing biocycling of K.

In the forest soil, Vm was present in the clay fraction (Fig. 1a), and trioctahedral mica was present in the silt fraction (Fig. 1b). The presence of these minerals indicates the undisturbed weathering stage of the soil as well as the extent of K extraction from the interlayers of illite. The extent of this extraction was smaller than that in the cultivated soil, based on the smaller amount of Vm (Table 2). In addition, trioctahedral mica was present in the silt and clay fractions (Fig. 3a). The depletion of K from the interlayers was accelerated by the action of the crop plants and caused the conversion of illite to Vm (HINSINGER et al., 1993; HINSINGER; JAILLARD, 1993) as well as further acid weathering via partial filling of the interlayers of Vm by Al polymers (MOTERLE et al., 2016).

The use of the soil for agriculture in the last 15 years (Fig. 2) seems to have accelerated weathering, since a change of Vm to HIV was observed (Fig. 1d).

Increases in kaolinite and goethite (Tables 2 and 3), minerals typical of the next stages of soil weathering, monosialization and alitization, were also observed. Therefore, in this environment, there is an overlap of the stages of bissialitization (formation/maintenance of 2:1 phyllosilicates), monosialitization (formation/maintenance of 1:1 phyllosilicates) and even alitisation (presence of oxides and goethite). This co-existence is partly due to the seasonality of the climate, with alternating states of humidity and leaching during the year (KARATHANASIS, 1991; NORFLEET et al., 1993). The cultivation of this area during this period without the application of K via fertilizer, even when it was cultivated with soybean (in 2011), favored the exit of K from Vm to HIV, as well as a decrease in illite (Tables 2 and 3).

The presence of HIV and/or higher amounts of Vm in soils differing only in agricultural use, i.e., crop soil or forest soil, respectively (Fig. 1d), suggests that the causative agent of the acceleration of weathering is the export of K and other agriculture-induced modifications, such as increased acidity and aluminum in solution, over 15 years without addition of K (Fig. 2). The continuous cultivation of plants such as soybean and/or grasses of the genus *Urochloa spp.*, which are able to utilize soil Kne (ROSOLEM et al., 1993; GARCIA et al., 2008), may cause depletion of K (BENIPAL; PASRICHA, 2002; BARRÉ et al., 2007; BILIAS; BARBAYIANNIS, 2017), resulting in mineral modification.

Although weathering is considered intense in tropical soils, changes in minerals over time spans as short as 15 years are not typically captured by routine techniques, such as XRD patterns (AZEVEDO et al., 1996; INDA et al., 2010; BAKKER et al., 2019). Quantitative analysis techniques, such as thermal analysis, can be used to quantify the extent of these changes.

Two sub-fractions of Kne were observed: the K retained on the edges of the clays at higher bond strength (1st subfraction) than Ke and the K of the interlayers of the 2:1 minerals (2nd subfraction). In the first crop, the plants probably absorbed Kne from the 1st subfraction since no change was observed in the percentage of Vm compared with the control. However, in the last crop, there was a decrease in the absorption of Kne and a significant increase in the percentage of Vm compared with the first crop (Table 2).

The 2nd subfraction of K is less soluble and comes from 2:1 minerals, since exhaustion of the first subfraction must have occurred. The existence of this fraction (attached to the edges of clay) may have contributed to the lack of formation of HIV.

Although the plants absorbed Kne, the amount derived from the interlayers was low and only apparent in the last crop. Thus, several cycles of cultivation would be necessary for the formation of this mineral, as shown in Fig. 1d.

The Kne from the 2:1 mineral interlayers is released slowly and has low plant availability (CAREY; METHERELL, 2003; GHIRI et al., 2012; ZÖRB et al., 2014; LI et al., 2017). When high and rapid absorption of K from this fraction occurs without modifications of mineral sources of K, such as illite (Fig. 5 and Table 3), the soils may contain either K bound to wedges or edges of these clays under forces greater than those of Ke (MOTERLE et al., 2016). This K is the source of K when minerals such as illite are not modified.

Because the soil only contained traces of Ke (Table 2), K must have been output from other fractions to maintain equilibrium in the soil solution (BORTOLUZZI et al., 2005; JALALI, 2006; ZÖRB et al., 2014; EGUCHI et al., 2015). This K could have come from feldspars (Fig. 1b) through hydrolysis (HUANG, 1977) or from the effects of the soil oxidation process (increased acidity and complexation) and trioctahedral micas in the silt fraction (Fig. 1c). Iron oxidation occurs in trioctahedral mica-containing media (THOMPSON; UKRAINCZYK, 2002). The absence of a change in Kne content and the percentage of Vm compared with the control after the first crop indicates that, at first, the fraction that was "recharged" by K from these primary minerals was the 1st subfraction of K. Then, over time, as this recharge failed to meet the needs of the plant, dissolution of K of the interlayers occurred, causing a decrease in Kne and an increase in Vm, as observed in the last crop of the plant species (Table 2).

The transformation of illite into Vm is the first sign of biological weathering, with release of K from the interlayers. The resulting use of this K by plants over time and the proximity of the rhizosphere can cause total vermiculitization of the illite (HINSINGER; JAILLARD, 1993; GOMMERS et al., 2005).

The illite/Vm ratio can be used to characterize the origin of Kne (from the interlayers or bound to the clay edges). Transformation to Vm only occurs once K has completely exited the illite interlayers, as transformation cannot occur if the Kne is at the edges of the clay. As shown in Fig. 4, the plants were not able to cause this modification in the first crop, nor did they modify the Kne content in the soil, although the absorption of Kne was high. By contrast, in the last crop, the Vm/illite ratio increased, and the content and absorption of Kne decreased (Table 2). The effect of plants under 2:1 mineral weathering is also evidenced by the decrease in illite of the forest soil in crop soil after 15 years of forage and grain crops (Tables 2 and 3). Maize clearly had the lowest capacity to remove K from the interlayers, because even though the absorption of Kne was highest in the last crop, the contents of illite and Vm (Table 2) and the ratio between them remained unchanged (Fig. 4). By contrast, for soybean and ruzigrass, although less Kne was absorbed (Table 2), the contents of illite and Vm and their ratio were altered because these species absorbed K from the interlayers. Soybean and ruzigrass plants are able to absorb large amounts of Kne as well as K from trioctahedral micas (VAN RAIJ; QUAGGIO, 1984; ROSOLEM et al., 1993; GARCIA et al., 2008).

The chemical equilibrium between the rhizosphere and soil is regulated by the balance of ionic charges in the rhizosphere (SINGH et al., 2002; MARSCHNER, 2012). Thus, the rhizospheric environment will be alkaline or acid depending on the absorption of cations or anions. Acidification of the rhizosphere improves the K solubilization efficiency of the interlayers, whereas a more alkaline rhizosphere can modify aluminum speciation, unlocking the K of the edges of the clays and making it available to plants.

Upon the application of K, soils with micaceous minerals tend to exhibit an increase in illite (BARRÉ et al., 2007, 2008), which may cause a decrease in the illite/Vm ratio (Fig. 4). The adsorption of K by primary minerals is accompanied by an increase in Kne content (Table 2) because the K can be adsorbed at the edges or interlayers of 2:1 clays (ZÖRB et al., 2014; MOTERLE et al., 2016).

Cultivation under intensive production systems without the use of K fertilizer for short cropping periods in the field, such as two harvests and an offseason, was not able to modify the results of the qualitative analyses by XRD (Fig. 5). Soils with advanced weathering and HIV formation, such as soil from the field experiment (Fig. 5), tend to show increased charges in the interlayers and thus greater K stability (MELO et al., 2009). Rotation or succession of crops with grasses may cause changes in soil K fractions (GARCIA et al., 2008). Thus, the cycling of K by these plants may interfere with the mineral dynamics, resulting in the adsorption or dissolution of K.

Re-entry of K into the system may occur through the absorption of K from other fractions or from deeper soil layers (Fig. 8), followed by release onto the surface by washing from plant residues (ROSOLEM et al., 2006). Thus, it is possible to observe increases in the levels of Ke and Kne (Table 3, Fig. 6d and 7) in the superficial layers of the soil, as well as changes in the HIV/illite ratio (Fig. 6a and b). The cycling of the K to the superficial layers of the soil makes it difficult to exhaust the K of the more soluble fractions, which remained high (Table 3), and hinders weathering over short periods of time.

On the other hand, succession of crops (soybean/ruzigrass) tends to reduce the depletion of mineral sources of K compared with grass monoculture (ruzigrass) or soybean monoculture (soybean/fallow). The K dynamics in the ruzigrass/soybean treatment resulted in fewer changes in the HIV/illite ratio along the depth profile (Fig. 8) as well as increases in Kne and HIV in February compared with the control. Succession of grain crops with forage plants of the genus *Uruchola* can recycle sufficient quantities of K to supply the next crop and even raise the Kne content (GARCIA et al., 2008).

The influence of plants on 2:1 minerals as well as on the dynamics of K was primarily observed on the soil surface. This layer showed the largest and most representative changes, such as the correlation between HIV and Kne (Fig. 7), with a mean Kne increase of 160% compared to the beginning of the experiment (Table 3). This is evidence that HIV can be increased (Table 3) by withdrawing K from illite to increase Kne and decrease illite in the crop soil (Tables 2 and 3). As most of the root system is concentrated in the upper layers (BARRÉ et al., 2009), the depletion of K from illite, the increase in HIV (Fig. 7), and the depletion of Ke were greater at this depth (Table 3). These conditions facilitated the solubilization of Ke from the other fractions. The high cycling capacity of these systems, which cycle more K than the plants are able to absorb, causes the exit of K from the interlayers and its fixation at the edges of the clays, increasing the solubility of this K compared with the K in the interlayers (GARCIA et al., 2008; BARRÉ et al., 2009; ROSOLEM et al., 2012). Supporting this conclusion, Kne decreased between February and November, i.e., in a short period of time, in all treatments (Table 3), as a subfraction of Kne became more readily available to the plants.

The effects of the root system on 2:1 mineral dynamics were also indicated by the increase in the HIV/illite ratio with depth in the ruzigrass treatment, with an increase in HIV to the detriment of illite. This change in HIV was not accompanied by a similar change in Kne (Table 3). The higher root exploration in rotation or succession systems favors both the cycling of K in more soluble fractions and the solubilization of K in the 2:1 mineral interlayers by exudates (BARRÉ et al., 2009). These changes result in the absorption of K in deeper layers and relocation of K on the surface, both fixed in 2:1

minerals and in the edges of clays. Consequently, Kne increases on the surface, and the HIV/illite ratio increases with depth.

4.5 CONCLUSION

In the cropped field soil, although illite was present, Vm was transformed into HIV, possibly due to the acceleration of weathering due to K export by crop harvesting and other agricultural practices. The ruzigrass-soybean rotation affected the partitioning of K into different levels of plant availability without rapidly modifying K-containing minerals. Raising annual crops over the previous 15 years resulted in mineralogical modifications of the soil. Soil in tropical environments may contain 2:1 minerals that are both sources and a reservoir of K. The exhaustion caused by agricultural crops in this soil may alter these minerals due to the supply of K to plants.

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GENERAL CONSIDERATIONS

In tropical soils, especially in the Brazilian Cerrado region, cover crops are necessary to obtain the benefits of the no-tillage system and can determine crop productivity. The bio-availability of nutrients, especially K, is one of the main benefits of the use of cover crops. The genus *Urochloa* is particularly useful as a cover crop due to its high capacity to use K fractions of low availability and convert them to more available fractions, thus improving the absorption of this nutrient by plants grown in succession. These improvements in K utilization are enhanced when cover crops are used in rotation with soybean, which also improves the efficiency of the use of K fertilizer. The ability of ruzigrass to absorb non-exchangeable K is greater than that of soybean, although both plants can utilize this fraction and modify minerals 2:1, thereby increasing the proportion of vermiculite due to loss of K from illite. Despite the tropical environment and the strong influence of weathering, this soil contains primary minerals as a source of K, which are modified by long-term K depletion through the use of non-exchangeable K under successive cropping as well as by the crop system. This fraction is a potential source of K for maintaining potassium equilibrium dynamics. The production system and K exhaustion due to successive cropping alter the buffer capacity of the soil for K, which is regulated by the adsorption of K on the exchangeable fraction and release by the non-exchangeable fraction.

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APPENDIX CHAPTER - 2

Table 4 - Simple pairwise linear correlation coefficients between the levels of exchangeable K (Ke), non-exchangeable K (Kne) and dry matter of plants during three growth cycles

Variable	% contribution Kne		Kne soil		Ke Soil		K uptake		DMP	
	r	*P*	*r*	*P*	*r*	*P*	*r*	*P*	*r*	*P*
1st Season										
Kne uptake	0.83	<0,0001	-0.60	<0.0001	0.20	0.35	0.63	<0.0001	0.68	<0.0001
% contribution Kne			-0.23		-0.29	0.19	0.73	<0.0001	0.45	0.03
Kne soil					-0.66	<0.0001	-0.040	0.86	-0.36	0.080
Ke Soil							-040	0.050	0.28	<0.20
K uptake									0.74	<0.0001
2nd Season										
Kne uptake	0.92	<0.0001	-0.38	0.06	-0.61	0.001	0.83	<0.0001	0.50	0.012
% contribution Kne			-0.27	0.18	-0.65	<0.001	-0.89	<0.0001	0.546	0.002
Kne soil					0.33	0.14	-0.29	0.17	-0.27	0.21
Ke Soil							0.33		-0.50	0.016
K uptake									0.80	<0.0001
3rd Season										
Kne uptake	0.87	<0.0001	-0.16	0.44	-0.52	0.010	-0.60	0.001	-0.53	0.0083
% contribution Kne			-0.40	0.050	-0.63	<0.050	-0.79	<0.0001	-0.55	0.0053
Kne soil					0.65	<0.0001	0.67	<0.0001	0.06	0.93
Ke Soil							0.87	<0.0001	0.58	0.002
K uptake									0.64	<0.0001

Table 5 - Pearson correlation coefficients (*r*) and t probability (*P*) between the levels of exchangeable K (Ke), non-exchangeable K (Kne) and dry matter of plants with and without applied K

Variable	DMP		Kne uptake		% contribution Kne		Kne soil		Ke soil	
	r	*P*	*r*	*P*	*r*	*P*	*r*	*P*	*r*	*P*
					Without K					
K uptake	0.87	<0,0001	0.99	<0.0001	0.86	<0.0001	0.77	<0.0001	0.24	0.15
DMP			0.86	<0.0001	0.75	<0.0001	0.72	<0.0001	0.15	0.37
Kne uptake					0.86	<0.0001	0.77	<0.0001	0.26	0.09
% contribution Kne							-0.70	<0.0001	0.26	0.14
Kne soil									0.35	0.03
					With K					
K uptake	0.83	<0.0001	0.06	0.72	0.08	0.63	-0.22	0.21	0.36	0.031
DMP			0.45	0.005	0.46	0.0043	0.003	0.92	0.35	0.031
Kne uptake					0.98	<0.0001	0.34	0.038	0.22	0.20
% contribution Kne							0.43	0.009	0.18	0.30
Kne soil									-0.38	0.047

APPENDIX CHAPTER - 4

Figure A – Weight loss (%) using the Kaolinite standard of laboratory.

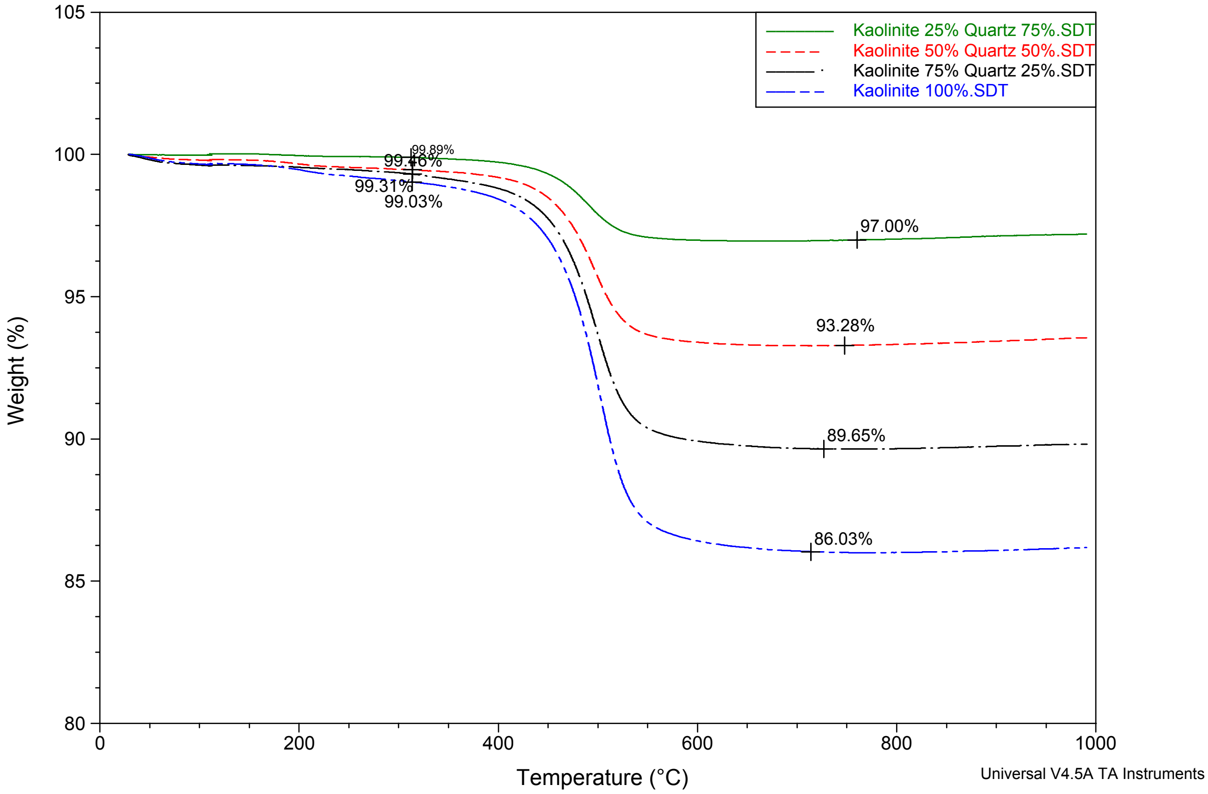

Figure B - Calibration of Kaolinite using 400 and 600 degree as the limits.

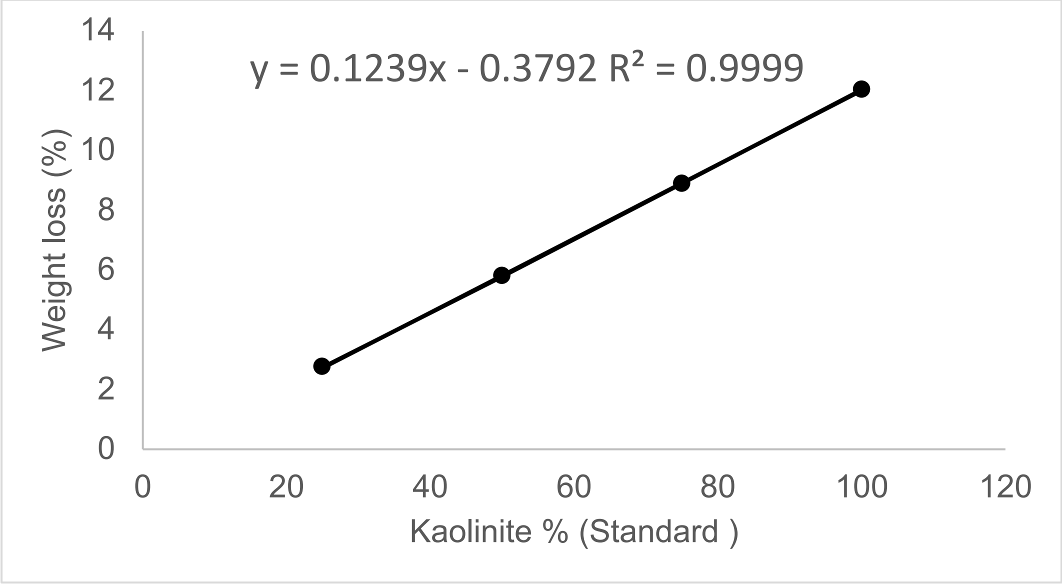

Table A – Kaolinite calculations for some clay samples of field experiment and green house experiment

Sample		Weight loss from 400 to 600 ^{0}C (%)*	kaolinite using calibrated standard equation (%)**
Field Experiment	Control - 3rd	7,152	61
	Soy - 1 st	7,072	60
	Control	7,008	60
Field Experiment (February Sy/Rz)	0-10 cm	7,926	67
	10_20 cm	7,762	66
	20-40 cm	7,632	65
	40-60 cm	7,715	65

* Weiqht loss determination of Kaolinite from Thermogravimetric analysis (TG)
**Mensure of the kaolinite (%) of samples using calibration standard through figure A.

Figure C - Weight loss (%) using the samples of field experiment. It determination of Kaolinite from Thermogravimetric analysis (TG)

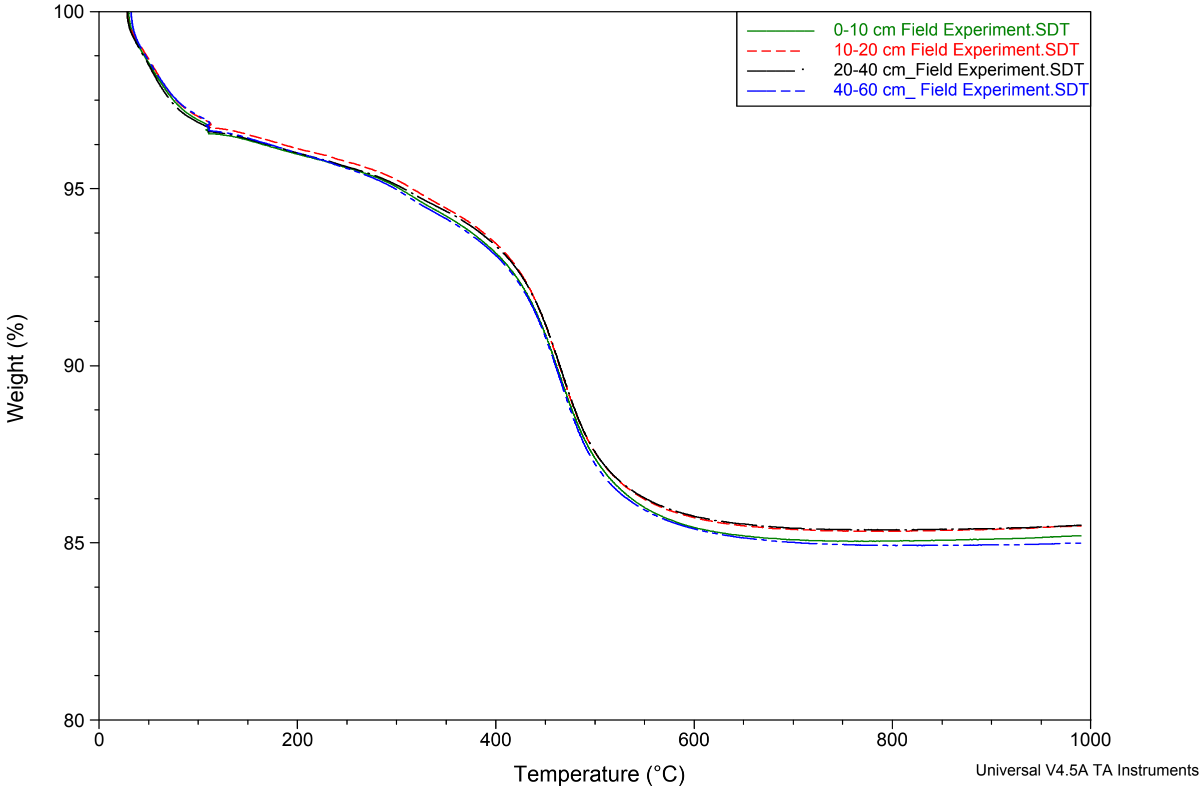

Figure D - Weight loss (%) using the samples of gree house experiment. It determination of Kaolinite from Thermogravimetric analysis (TG).

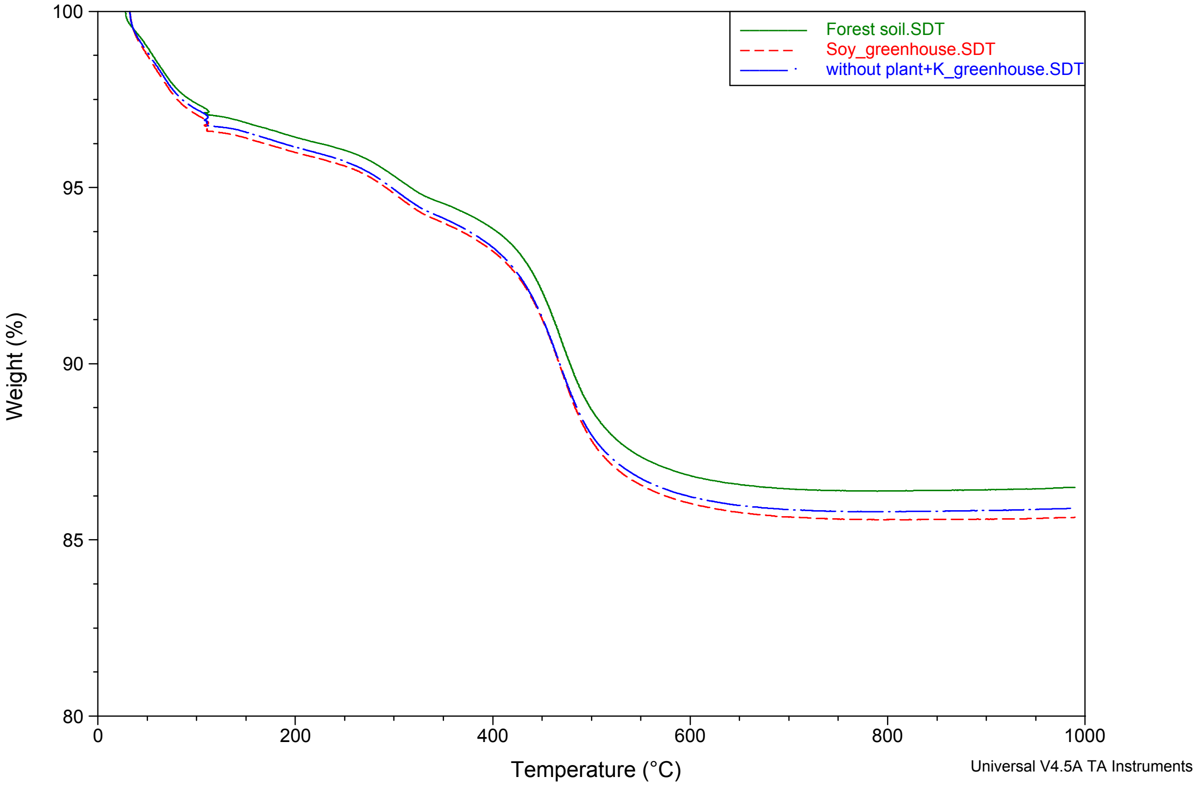

Table B – Goethite calculations for some clay samples of field experiment and green house experiment

Sample		Weight loss from 300 to 400 ^{0}C (%)*	Goethite (%)**
Field Experiment	Control - 3rd	1,65	16,3
	Soy - 1 st	1,35	13,4
	Control	1,51	15,0
Field Experiment (February Sy/Rz)	0-10 cm	1,89	18,7
	10_20 cm	1,82	18,0
	20-40 cm	1,71	16,9
	40-60 cm	1,88	18,6

* Weiqht loss determination of Goethite from Thermogravimetric analysis (TG)

**Mensure of the Goethite (%) of samples using the equation 1 (KARATHANASIS and HAJE, 1981).

$$\% \, Goeth = \frac{\% \, weight \ loss \ at \ 300-400°C}{0.101} = \qquad (1)$$

Where Assume 10.1% (0.101) mass loss of goethite between 300 and 400 deg.

Table C – Amount (%) of mica, kaolinite, and goethite in the clay of the field experiement and greenhouse samples

Samples	**Kaolinite (TG)**	**Goethite (TG) %**	**Vermiculite***	**Mica****
Control - 3rd	60,8	16,3	1,1	21,8
Soy - 1 st	60,1	13,4	1,1	25,4
Control	59,6	15,0	1,3	24,1
0-10 cm	67,0	18,7	2,1	12,1
10-20 cm	65,7	18,0	3,3	13,0
20-40 cm	64,7	16,9	2,7	15,7
40-60 cm	65,3	18,6	2,6	13,5

*Using the equation 2. (KARATHANASIS and HAJE, 1981)
** Using the equation 3.

Table D – Calculated using the ratio of vermiculite peak area to that of kaolinite with a mineral intensity factor (MIF) of 1.05 and tied to the kaolinite determined from thermal analysis

Samples	Vermiculite area*	Kaolinite area*	kaolinite (TA)	MIF
Control - 3rd	52,09	2969	60,8	1,05
Soy - 1 st	73,9	4107	60,1	1,05
Control	66,9	3271	59,6	1,05
0-10 cm	128	4236,8	67,0	1,05
10-20 cm	236,3	5012	65,7	1,05
20-40 cm	174	4334,9	64,7	1,05
40-60 cm	117,5	3124,2	65,3	1,05

*Using de XRD for measure peak area of each mineral.

$$\% \, Vermiculite = \frac{Peak \ area \ of \ Vermiculite \ from \ XRD}{Peak \ area \ of \ Kaolinite \ from \ XRD} \ x \ \% \ Kaolinite \ from \ TGA \ x \ 1.05 \qquad (2)$$

$$\% \, Mica = 100 - \Sigma \, (Kaolinite + Vermiculite + \ Goethite) \qquad (3)$$