UNIVERSIDADE DE SÃO PAULO CENTRO DE ENERGIA NUCLEAR NA AGRICULTURA

PEDRO LOPES GARCIA

Nitrogen (¹⁵N) use efficiency for fertilization managements of maize and common bean using mixtures of polymer-sulfur coated urea and conventional urea

> Piracicaba 2021

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Revised version according to the Resolution CoPGr 6018 at 2011

Thesis presented to the Center for Nuclear Energy in Agriculture from University of São Paulo to obtain the degree of Doctor in Science

Concentration Area: Nuclear Energy in Agriculture and Environment

Advisor: Prof. Dr. Paulo Cesar Ocheuze Trivelin

Piracicaba 2021 AUTORIZO A DIVULGAÇÃO TOTAL OU PARCIAL DESTE TRABALHO, POR QUALQUER MEIO CONVENCIONAL OU ELETRÔNICO, PARA FINS DE ESTUDO E PESQUISA, DESDE QUE CITADA A FONTE.

Dados Internacionais de Catalogação na Publicação (CIP)

Seção Técnica de Biblioteca - CENA/USP

Garcia, Pedro Lopes

Eficiência de uso de nitrogênio (¹⁵N) em manejos de adubação de milho e feijão com o uso de misturas de ureia revestida com enxofre e polímero e ureia convencional / Nitrogen (¹⁵N) use efficiency for fertilization managements of maize and common bean using mixtures of polymer-sulfur coated urea and conventional urea / Pedro Lopes Garcia; orientador Paulo Cesar Ocheuze Trivelin. - - Versão revisada de acordo com a Resolução CoPGr 6018 de 2011. -- Piracicaba, 2021.

96 p.

Tese (Doutorado – Programa de Pós-Graduação em Ciências. Área de concentração: Energia Nuclear na Agricultura e no Ambiente) – Centro de Energia Nuclear na Agricultura da Universidade de São Paulo, Piracicaba, 2021.

Adubação 2. Feijão 3. Fertilizantes 4. Isótopos estáveis 5. Latossolos 6. Macronutrientes
 Milho 8. Nitrogênio 15 9. Ureia I. Título.

CDU (631.811+631.84)

Elaborada por: Marilia Ribeiro Garcia Henyei CRB-8/3631 Resolução CFB Nº 184 de 29 de setembro de 2017

I would like to dedicate this thesis to my parents Sílvia M. Lopes de Mello and José Carlos B. Garcia. I offer to my brothers Theo, Dario, and Júlia, my nephew Gabriel, and my wife Shirlane O. Garcia.

ACKNOWLEDGMENTS

To my family for their love, support and encouragement.

To my wife for always unconditionally sticking by my side.

To my advisor prof. Dr. Paulo Cesar Ocheuze Trivelin for his support and guidance throughout my graduate studies, and the confidence in my work. You have been a tremendous mentor and friend for me and I will forever be thankful.

To the Center for Nuclear Energy in Agriculture (CENA) from University of Sao Paulo (USP) and the Laboratory of Stable Isotopes (LIE) for the infrastructure during my graduate studies. Thanks to the technicians: Hugo, Ana, Miguel, Pingin, Clélber and Bento. Interns: Cátia, Henrique, Matheus, Carla, Pablo, Leonardo, Monique, Maick, Bianca, Everton, Maria Roberta and Magrão (in memoriam). Graduate students: Nicole, Bruno, Grabriela, Beatriz and Saulo. Professors: José Lavres Junior, José Albertino Bendassolli and Renata Alcarde Sermarini. All these members were essential to making possible my doctorate research with their strong help in the field and laboratory activities.

To the São Paulo Research Foundation (FAPESP) for the granted scholarship (grant # 2017/25813-5) and the financial support (grant # 2017/24516-7) during my doctorate program.

To the Compass Minerals Plant Nutrition for the financial support for my doctorate research, and for the areas provided in the South American Innovation Center for the field experiments (from 2017 to 2020) during two maize and two common bean growing seasons. Thanks to the Compass Minerals South America research team (Ithamar Prada, Michel Castellani, Carlos Roberto de Sant Ana Filho, Daniella Vitti, Douglas, Linker, Bruno Saito, José Marcos Leite, Robson Mauri, Gabriel Uehara, interns and the previous research manager Fabio Scudeler) for the strong field assistance.

To the researcher Caue Ribeiro, Paulo Lasso, EMBRAPA Intrumentation and Rede Agronano for the X-ray microtomography analyses.

To the New Ag International (NAI) and the chief editor Luke Hutson for selecting me for presentation in the event in Dublin/Ireland in 2019 organized in partnership with International Fertilizer Association (IFA), and in an international online event in 2020 which I presented results of this thesis that were published in the Agronomy journal from MDPI.

To my relatives Luiz Antonio, Delma and Zito Garcia (in memoriam). Marlene, Maria de Lourdes, Mary, Pedro Angelo, Joseane, Juquinha, Márcia, Simone, Cíntia, Íris, and Brisa.

To my friends for the enjoyable moments during the journey: Izaias Lisboa, Michel Colmanetti, Anderson, Júnior Damian, Marcelo Gomes, Felipe Hermínio, Luis Henrique, Nikolas Mateus, Antonio Florentino, Ismael Meurer, João Cardoso, José Lucas, Lucas Satiro, Nicole Cheng, Bruno Moschini, Felipe Rinaldi, Hugo Villalba, Eduardo Mariano, Hugo Batagello, Gabriela Salgado, Beatriz Nastaro, Saulo Quassi, Marcel Góes, Leonardo Pinto, Douglas Brecht, Fábio Manetti, Rodrigo Magalhães, Renato Rocha, Marcel Oliveira, Diana Oliveira and Meris Oliveira.

Thank you all!

"One man can be a crucial ingredient on a team, but one man cannot make a team." Kareem Abdul-Jabbar

ABSTRACT

GARCIA, P. L. Nitrogen (¹⁵N) use efficiency for fertilization managements of maize and common bean using mixtures of polymer-sulfur coated urea and conventional urea. 2021.
96 p. Thesis (Doctorate in Science) – Center for Nuclear Energy in Agriculture, University of São Paulo, Piracicaba, 2021.

Nitrogen (N) is the most demanded nutrient by maize (Zea mays L.) and common bean (Phaseolus vulgaris L.) being necessary in small quantities at the beginning and at appropriate times and quantities during the growth cycle of these crops. It is desired that blends of polymersulfur coated urea (PSCU) and urea (U) treated with NBPT (N- (n-butyl) thiophosphoric triamide) meet the need for N of these crops with a single N application at sowing. In order to accurately recommend these blends in the current cropping systems, it is necessary to know the ¹⁵N-fertilizer recovery by plants of each N source in the blend, as well as the N dynamics in the soil - plant - atmosphere system. Thus, the objective of this study was to evaluate the ¹⁵N recovery from PSCU and/or U by plants, the ammonia volatilization from N-fertilizers applied on the soil surface, the macronutrient (N, P, K, Ca, Mg and S) uptake by plants, as well as the soil mineral N content, and grain yield. The ¹⁵N-fertilizer recovery and ammonia volatilization were evaluated in one growing season and the other analyses in two growing seasons for each crop. The experiments were conducted in a Rhodic Eutrustox soil (clayey texture), with straw on the soil surface, during the 2017-2018 and 2019-2020 maize growing seasons, and the common bean during the 2018 and 2019 irrigated growing seasons. Two blends (70% PSCU + 30% U and 30% PSCU + 70% U) were applied in three ways (incorporated application at sowing; broadcast application at sowing; and split application). The N rate was 180 kg ha⁻¹ (maize) and 90 kg ha⁻¹ (common bean). Control treatment (without N-fertilizer) was included. In 2019-2020, PSCU was the main N-fertilizer supplier in maize at V4 (fourth-leaf) applying 70% PSCU + 30% U, and both blends provided 73.8% of N recovery at the physiological maturity (47.9% in the grain). There was not difference in yield (12.1 Mg ha⁻¹) and macronutrient (N, K, S, Ca and Mg) uptake among treatments in 2019-2020. In 2017-2018, 70% PSCU + 30% U provided higher yield (8.3 Mg ha⁻¹), and the broadcast application provided higher total N and K uptake than split application and control. In the common bean, more than 50% of N from U was recovered from the blends, and the broadcast application provided lower N recovery in the grain than split and incorporated applications in 2019. In addition, the broadcast application provided lower yield (3.3 Mg ha⁻¹) than split application and control (3.6 Mg ha⁻¹) in the average of years. There was no difference in macronutrient uptake by common bean in both years (except N). The unrecovered N by maize and common bean can be attributed to the ammonia volatilization (~12% of the applied N), and the likely N percolation below the root zone. Based on the results, it is possible to recommend the most cost-effective treatment for maize, and opt for split or incorporated application in the common bean with the less expensive blend in the soil of the experiments.

Keywords: Controlled-release fertilizer. NBPT-treated urea. Fertilizer recovery. ¹⁵N isotope. *Zea mays* L. *Phaseolus vulgaris* L.

RESUMO

GARCIA, P. L. Eficiência de uso de nitrogênio (¹⁵N) em manejos de adubação de milho e feijão com o uso de misturas de ureia revestida com enxofre e polímero e ureia convencional. 2021. 96 p. Tese (Doutorado em Ciências) - Centro de Energia Nuclear na Agricultura, Universidade de São Paulo, Piracicaba, 2021.

O nitrogênio (N) é o nutriente mais exigido pelo milho (Zea mays L.) e feijão (Phaseolus vulgaris L.), sendo necessário o seu fornecimento em menor quantidade no início e em momentos e quantidades adequadas durante o desenvolvimento dessas culturas. É desejado que misturas de ureia (U) tratada com NBPT (N-(n-butil) triamida tiofosfórica) e ureia revestida com enxofre e polímero (PSCU) supram a necessidade de N dessas culturas com uma única aplicação na semeadura. Para recomendar de forma precisa essas misturas nos atuais sistemas de cultivo é necessário conhecer quanto as plantas recuperam de N das fontes da mistura, bem como a dinâmica de N no sistema solo - planta - atmosfera. Dessa forma, o objetivo do trabalho foi avaliar a recuperação de 15N do PSCU e/ou do U pelas plantas, a volatilização de amônia do N-fertilizante aplicado na superfície do solo, o acúmulo de N e dos outros macronutrientes (P, K, Ca, Mg e S) pelas plantas, bem como o N mineral no solo durante o ciclo, e a produtividade de grãos. A recuperação de ¹⁵N-fertilizante e a volatilização foram avaliados em uma safra e o restante em duas, em cada cultura. Os experimentos foram desenvolvidos nas safras de milho 2017/18 e 2019/20 e nas de feijão de inverno irrigado em 2018 e 2019 em um Latossolo Vermelho eutrófico de textura argilosa, manejado com palha na superfície. Duas misturas (70% PSCU + 30% U e 30% PSCU + 70% U) foram aplicadas de três maneiras (incorporado na semeadura; superfície na semeadura; e parcelado), com dose de N de 180 kg ha⁻¹ (milho) e 90 kg ha⁻¹ (feijão). Tratamento controle (sem N-fertilizante) foi incluído. Na safra 2019/20, PSCU foi o principal fornecedor de N-fertilizante no milho em V4 (quarta folha), aplicando 70% PSCU + 30%U, e as duas misturas garantiram 73,8% de recuperação de N na maturidade fisiológica (47,9% nos grãos). Não houve diferença de produtividade (12,1 Mg ha⁻¹) e de acúmulo total de N, K, S, Ca e Mg entre os tratamentos (safra 2019/20). Na safra 2017/18, 70% PSCU + 30% U apresentou maior produtividade (8,3 Mg ha⁻¹), e a aplicação em superfície maior acúmulo total de N e K comparado com a parcelada e o controle. No feijão, mais de 50% do N proveniente do U foi recuperado das misturas, a aplicação em superfície recuperou menos N do U nos grãos comparado com a parcelada e a incorporada em 2019, e produziu menos (3,3 Mg ha⁻¹) que a parcelada e o controle (3,6 Mg ha⁻¹) na média dos anos. Não houve diferença de acúmulo total dos macronutrientes em 2018 e 2019 (exceto N). O N não recuperado, no milho e no feijão, pode ser atribuído a volatilização de amônia (~12% do N aplicado) e o possível movimento vertical do N no solo abaixo da zona radicular (percolação). Baseado nos resultados, é possível recomendar o tratamento menos oneroso para o milho e optar pelo parcelamento ou incorporação com a mistura menos onerosa no feijão no solo do experimento.

Palavras-chave: Fertilizante de liberação controlada. Ureia tratada com NBPT. Recuperação do fertilizante. Isótopo ¹⁵N. *Zea mays* L. *Phaseolus vulgaris* L.

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1 GENERAL INTRODUCTION

The nutrient most required by maize (*Zea mays* L.) and common bean (*Phaseolus vulgaris* L.) is nitrogen (N), being a constituent of amino acids, enzymes, and chlorophyll, which can interfere in photosynthesis and plant growth (MALAVOLTA, 1997). In maize, more than 70% of the total N uptake occurs after V8 (eighth-leaf), the maximum rate of N uptake occurs between V10 (tenth-leaf) and V14 (fourteen-leaf) (GARCIA et al., 2018; MUELLER; VYN, 2016; BENDER et al., 2013), and the yield potential is determined between V4 (fourth-leaf) and V6 (sixth-leaf) (FANCELLI; DOURADO NETO, 2004). In common bean, the N needs to be supplied aiming to increase the number of pods per plant, i.e. before flowering (SORATTO et al., 2006; ROSOLEM, 1996); and, there is a high demand for N at flowering and grain filling. In addition, N is needed at the beginning of the common bean growth cycle, especially with the straw of the previous crop left on the soil surface (SORATTO et al., 2001). Thus, in both crops, there is a low N demand at the beginning, and it increases during the crop growth cycle, mostly at the beginning of the reproductive stages.

In Brazil, the main N source in maize and common bean is urea because of its low acquisition costs and high N content compared to other N sources (RAIJ; CANTARELLA, 1997). However, urea has low nitrogen use efficiency (NUE) (DOBERMANN, 2007; CANTARELLA, 2007) in agricultural crops, due to several factors, to consider: losses by ammonia volatilization after the application of urea on the soil surface (LARA CABEZAS et al., 2000), nitrate leaching (HONG et al., 2007), and denitrification (DUSENBURY et al., 2008). In addition, the immobilization by the soil microorganisms can make N temporarily unavailable for plant uptake and resulting in low yield and/or low agronomic efficiency (DOBERMANN, 2007). The split N-urea application is an alternative to reduce N losses and improving NUE in agricultural crops but it increases cost with additional tractor operations.

Controlled-release urea (CRU) can be another option to reduce N losses by nitrate leaching (WILSON et al., 2009), ammonia volatilization (CANCELLIER et al., 2016), and denitrification (HALVORSON; DEL GROSSO, 2012), consequently improving NUE in agriculture crops (GUO et al., 2016). In addition, CRU can be considered an "ideal" fertilizer (SHOJI; KANNO, 1994) because can improve the synchronism of nutrient release and crop needs (SHAPIRO et al., 2016) with a single application, and reduce the salt toxicity in plants caused by high N rates of urea applied at sowing (GARCIA et al., 2019b; OYANG; MACKENZIE; FAN, 1998). However, CRU can provide a low soil mineral N content at the

beginning of the agricultural crop growth and consequently reduction in dry matter, N uptake, and yield as observed by Grant et al. (2012). In addition, the acquisition costs of CRU are normally higher than conventional urea.

Aiming to provide N at the beginning of the agricultural crop growth and to reduce the acquisition costs of CRU, blends of CRU and conventional urea can be a promising alternative. In Brazilian condition was observed that blends of conventional urea and polymer-sulfur coated urea (PSCU), that is a CRU, in a single application incorporated at maize sowing can be an alternative to the conventional fertilization which split urea application (GARCIA et al., 2018; VILLALBA, 2014). These authors also observed that these blends can increase maize N uptake, soil N mineral content during the agriculture crop cycle compared to the control treatment (without N-urea application) and yield in sandy loam soil. It is expected that conventional urea (soluble N source) in the blend provides N at the beginning and PSCU (insoluble source) during the crop cycle. There is also an expectation that these blends would be a better choice in broadcast application at sowing due to the lower application cost compared to the incorporated application. However, the majority of studies in Brazilian conditions evaluated theses blends incorporated at maize sowing (VILLALBA, 2018; GARCIA et al., 2018; VILLALBA, 2014). In the common bean, there are studies evaluating top-dressing application after V4 (third trifoliate leaf unfolded) with only CRU (BERNARDES et al., 2015; SANT'ANA et al., 2011; SOARES, 2011) and there were not studies evaluating these blends with a single application at common bean sowing in Brazilian conditions. Normally, the main evaluations in maize and common bean using only CRU or blended with conventional urea are the total N uptake by plants, soil mineral N content, ammonia volatilization, and NUE that involves indirect calculus (LI et al., 2020a; 2020b; GARCIA et al., 2018; GUO et al., 2017; ZHENG et al., 2016; BERNARDES et al., 2015; VILLALBA, 2014; SANT'ANA et al., 2011; SOARES, 2011). The total N uptake by plants did not differentiate the N from the soil and from the fertilizer, and in the calculus of NUE the total N uptake by plants is used (DOBERMANN, 2007). The use of ¹⁵N tracer in the PSCU and/or conventional urea in the blend can answer the doubts about the N-fertilizer recovery by plants especially in the current cropping systems that involves straw from the previous crop on the soil surface and can help to improve the recommendation of these blends in maize and common bean.

There are different ratios of blends of CRU and conventional urea (U) and the choice can vary according to the climate conditions, soil type, and economic benefits. In sandy loam soil in Brazil the 70% CRU + 30% U was recommended in maize (VILLALBA, 2014), this

ratio was also recommended in North China (GUO et al., 2017), and is considered the most used ratio in cereals (ZHENG et al. 2017). The 30% CRU + 70% U was recommended in Northeast China (LI et al., 2020a). This ratio can be a promising choice in Brazilian conditions because of the lower acquisition costs compared to the 70% CRU + 30% U. And, depending on the straw on the soil surface and the carbon (C) and N ratio can occur the immobilization of N in a higher or lower degree (KONG, 2014), which would justify the use of the higher quantity of U in the blend of CRU + U in order to maintain a C / N ratio suitable for the microbial activity. Moreover, different ratios of CRU + U can also influence the other macronutrient uptake (P, K, Ca, Mg and S) (GARCIA et al., 2018) and it was not evaluated to 30% CRU + 70% U (LI et al., 2020a; 2020b; GARCIA et al., 2018).

The objective of this study was to evaluate the effects of different N fertilization management practices (incorporated, broadcast, and split application), in maize and common bean, with contrasting blends of PSCU and U (70% PSCU + 30% U and 30% PSCU + 70%U) on the ¹⁵N-fertilizer recovery (PSCU and/or U) by plants, macronutrient uptake, and grain yield. In addition, the volatilization of ammonia, and soil N mineral content. Two chapters were developed to confirm or refute the following hypotheses:

- The N fertilization management (incorporated, broadcast, and split application) with straw in the system is more efficient to supply N and the other macronutrients during the maize cycle applying 70% PSCU + 30 %U than 30% PSCU + 70% U. The U source is the likely main N fertilizer supplier at V4, and the PSCU source from V4 to R6 (physiological maturity) applying 70% PSCU + 30% U.
- 2) The N broadcast application using 70% PSCU and 30% U will be a better choice to provide N and the other macronutrients during the common bean growth cycle and to improve grain yield than incorporated or split application using 70% PSCU and 30% U or 30% PSCU and 70%U.

This is the first research evaluating the N-recovery using ¹⁵N-urea coated with sulfur and polymer in maize, and using ¹⁵N-urea treated with NBPT in a mixture of N-fertilizer in common bean. The main findings of this thesis resulted in the following scientific articles:

GARCIA, P. L.; SERMARINI, R. A.; FILHO, C. R. S. A.; BENDASSOLLI, J. A.; BOSCHIERO, B. N.; TRIVELIN, P. C. O. ¹⁵N-Fertizer recovery in maize as an additional strategy for understanding nitrogen fertilization management with blends of controlled-release and conventional urea. **Agronomy**, Basel (Switzerland), v. 10, n. 12, p. 1-21, 2020.

GARCIA, P. L.; SERMARINI, R. A.; TRIVELIN, P. C. O. Nitrogen fertilization management with blends of controlled-release and conventional urea affects common bean growth and yield during mild winters in Brazil. **Agronomy**, Basel (Switzerland), v. 10, n. 12, p. 1-17, 2020.

2 ¹⁵N-FERTILIZER RECOVERY IN MAIZE AS AN ADDITIONAL STRATEGY FOR UNDERSTANDING NITROGEN FERTILIZATION MANAGEMENT WITH BLENDS OF CONTROLLED-RELEASE AND CONVENTIONAL UREA

ABSTRACT

A single application of polymer-sulfur coated urea (PSCU) and conventional urea (U) is expected to ensure nitrogen (N) throughout the maize (Zea mays L.) growth cycle being U the likely main N-fertilizer supplier at the beginning and PSCU during the maize growth cycle. This research aimed to evaluate N fertilization management (split, incorporated, and broadcast application) and fertilizer blends (30% PSCU + 70% U and 70% PSCU + 30% U) on volatilization of ammonia (AV) and soil N mineral content (NM); plant macronutrient uptake and ¹⁵N-fertilizer recovery (NR); and yield (GY). Field experiments were conducted for two growing seasons (2017-2018 and 2019-2020) in Rhodic Eutrustox soil. U was treated with NBPT (N-(n-butyl) thiophosphoric triamide). N rate was 180 kg ha⁻¹. AV reached 12% of the applied N (broadcast-applied 70% PSCU + 30% U, 2017–2018). The 30% PSCU + 70% U application resulted in higher NM at 40–60 cm depth in vegetative and reproductive stages in both seasons. The 70% PSCU + 30% U application resulted in the highest GY in 2017–2018, and the N treatments did not affect GY in 2019–2020. NR was 3% on average at vegetative leaf stage 4 (V4), and PSCU, the main N-fertilizer supplier applying 70% PSCU + 30% U. After V4, the main N-fertilizer supplier is PSCU for 70% PSCU + 30% U and U for 30% PSCU + 70% U application. These blends (incorporated, broadcast, and split application) can ensure N during the maize growth cycle, with NR of 72.5% at maturity (R6) being 47.9% in the grain.

Keywords: polymer-sulfur coated urea; NBPT-treated urea; mixture; nitrogen balance; Zea mays L.

2.1 Introduction

Conventional urea is the nitrogen source most used in maize (*Zea mays* L.) production in China (KE et al., 2017) and Brazil, the second and third world's largest maize producer (USDA, 2020), respectively. Although its lower acquisition cost and higher N content than other N sources (i.e., ammonium nitrate and ammonium sulfate), U is more prone to ammonia volatilization losses when applied on the soil surface, reducing N use efficiency (NUE) in maize (RINALDI et al., 2019). To increase NUE and yield is necessary to split U application (CANTARELLA, 2007), increasing costs with mechanized operations and the risks to lose the second U application (GRAMING et al., 2017).

India and China have a good acceptance for new technologies to improve NUE of U (APOSTOLOPOULOU, 2016), and Brazil and Paraguay have a growing demand for that (GUELFI, 2017). Among the available technologies, there are the coating of U with sulfur and polymers or just polymers, considered controlled-release U (CRU), and the use of urease inhibitors in the U treatment (TRENKEL, 2010). In the first case, the polymers and their micropores provide the U dissolution and diffusion with the soil humidity controlling the N release. It improves the synchronism of N release and maize needs (SHAPIRO et al., 2016), increasing NUE (GUO et al., 2016) by reducing ammonia losses (CANCELLIER et al., 2016) and N leaching (WILSON et al., 2009), but it can provide low N release at the beginning of maize growth cycle and consequently yield reduction (GRANT et al., 2012). In the second case, the urease inhibitor most effective in the U treatment is the NBPT (N-(n-butyl) thiophosphoric triamide) compared to hydroquinone, copper, boric acid, and catechol (RINALDI et al., 2019; CANTARELLA, 2007). The NBPT-treated U reduces N conversion rate from amidic (N-NH₂) to ammoniacal form (N-NH₄⁺) for a period of 3 to 7 days in applications on the soil surface in Brazilian conditions (GUELFI, 2017). After that, it can be incorporated by rain and better used by plants, but it is a soluble N source, and the split N application is also necessary to avoid salt effect in plants (GARCIA et al., 2019a) and N leaching (HONG et al., 2007).

Blends of polymer-sulfur coated urea (PSCU), and U applied incorporated at maize sowing (GARCIA et al., 2018; VILLALBA, 2018) is an alternative to supplying N throughout the maize growth cycle in Brazilian conditions and to avoid low N release at the beginning by single CRU application (GRANT et al., 2012). It probably occurred because U (soluble source) provided N at the beginning and PSCU (controlled-release source) during the maize growth cycle. To confirm that hypothesis, the use of nitrogen-15 (¹⁵N) tracer is necessary for the U and PSCU sources. Although the N incorporated is available at the right time and place for optimal root uptake (NKEBIWE et al., 2016), the N broadcast application on the soil surface can reduce application costs.

In the current crop system management, the straw of the previous crop is left on the soil surface. In that situation, the N-fertilizer dynamic in the soil-plant system probably changes applying different ratios of CRU and U. The optimal ratio can vary based on the soil, climate conditions, and economic benefits, and is normally determined by the ammonia volatilization, N uptake, crop yield, and NUE (LI et al., 2020a; 2020b). 70% CRU + 30% U was recommended in North China (GUO et al., 2017) and in a Typic Haplustox soil in Brazil (GARCIA et al., 2018; 2019b). 30% CRU + 70% U was recommended in Northeast China (LI et al., 2020b). N can also suffer immobilization depending on the straw C/N (carbon/N) ratio (KONG, 2014), and 30% CRU + 70% U would avoid a possible N lack at the beginning of the maize cycle provided by N immobilization.

In this context, we hypothesize that N fertilization management (incorporated, broadcast, and split application) with straw in the system is more efficient to supply nitrogen during the maize cycle applying 70% PSCU + 30% U than 30% PSCU + 70% U in Brazilian conditions. U is the likely main N-fertilizer supplier at V4 (vegetative leaf stage 4), and PSCU from V4 to maturity (R6) applying 70% PSCU + 30% U. This research aimed to evaluate the influence of N fertilization management and blends of U (treated with NBPT) and PSCU on ammonia volatilization, soil N mineral content, macronutrient uptake, ¹⁵N-fertilizer recovery (NR) in plants, and maize yield in Brazilian tropical conditions.

2.2 Materials and Methods

2.2.1 Field Site Description

Two maize field experiments were conducted at Compass Minerals Innovation Center in Iracemápolis, state of São Paulo, Brazil (22°39' S, 47°30' W, 608 m elevation), during 2017– 2018 (season 1) and 2019–2020 (season 2) spring-summer growing seasons. The experimental area has a soil classified as Rhodic Eutrustox (USDA classification; SOIL SURVEY STAFF, 2014) with a clayey texture: 41.9% sand, 11.9% silt, and 46.2% clay (GEE; BAUDER, 1986). Common bean was the previous crop of season 1 and season 2, and 5.6 ± 0.4 Mg ha⁻¹ of straw with a 44:1 C/N ratio was left on the soil for season 1, and 5.7 ± 0.6 Mg ha⁻¹ with a 43:1 C/N ratio was left on the soil for season 2. The straw was quantified using twelve samples of one meter square (three samples per block) before starting the experiments. Each sample was weighed. Subsamples were oven-dried at 65 °C to a constant weight, weighed, and ground with a Wiley mill to pass through a 0.5-mm sieve to analyze C and N contents (BATAGLIA et al., 1983). Plowing, harrowing, and limestone application were performed before the previous crop of season 2. The mean annual temperature and annual precipitations were 21.8 °C and 1200 mm, respectively (3-year average).

The soil chemical characterization was performed in three depths before starting the experiments (Table 1).

Depth	pН	SOM ¹	TSN ²	$\mathrm{NH_{4}^{+}}$	NO ₃ -	S	Р	K	Ca	Mg	Al	CEC ³	AlS ⁴	BS ⁵
cm		g dm ⁻³		mg kg ⁻¹		mg	1m ⁻³		n	nmol _c o	lm ⁻³		%	6
Season 1: 2017-2018														
0-20	5.0	24	1100	3.7	15	125	10	2.5	28	12	1	68	2	63
20-40	4.3	18	800	1.7	4.8	269	< 3	1.3	6	5	11	43	47	28
40-60	4.2	15	600	3.0	4.3	318	< 3	1.0	2	3	11	34	65	18
Season 2: 2019-2020														
0-20	5.5	25	1100	1.5	22	14	30	5.4	39	26	0	95	0	74
20-40	5.1	23	900	1.4	12	38	14	4.7	25	16	3	90	5	62
40-60	5.0	23	700	0.7	9	46	12	4.9	27	16	2	82	4	58

Table 1 - Soil chemical attributes on which maize is grown in Brazil

¹SOM, soil organic matter; ²TSN, total soil nitrogen; ³CEC, cation exchange capacity at pH 7.0; ⁴AlS, aluminum saturation; ⁵BS, base saturation.

Fifteen soil samples per depth were mixed for analysis. The determination of the soil pH was performed using 0.01 mol L⁻¹ CaCl₂ (1:2.5 soil/solution; RAIJ et al., 2001), soil organic matter using the Walkley-Black procedure (NELSON; SOMMERS, 1996), total N content using mass spectrometry (BARRIE; PROSSER, 1996), NH₄⁺-N and NO₃⁻-N contents using 2 mol L⁻¹ KCl (1:5 soil/solution ratio; RAIJ et al., 2001). The extraction of nutrient available was performed by ion-exchange (phosphorus (P), potassium (K), calcium (Ca), and magnesium (Mg)) and using a solution of Ca(H₂PO₄)₂ for SO₄²⁻-S. Al was extracted by KCl solution. The quantification was performed by colorimetric (P), flame photometric (K), and atomic absorption (Ca and Mg) spectroscopy. Al was determined by titration, while SO₄²⁻-S by turbidimetry (RAIJ et al., 2001). Summing exchangeable cations (Ca, Mg and K) and potential acidity (H + Al) determined the cation exchange capacity (CEC) at pH 7.0. Dividing the total exchangeable cations by CEC and multiplying by 100 determined the lase saturation.

2.2.2 Experimental Setup and Treatment Description

The experiments were in a factorial $(3 \times 2) + 1$ randomized block design with four replications. The treatments consisted of three N fertilization management practices (split application: 1/3 of N applied incorporated at sowing and 2/3 as a side-dressing at V4; broadcast application: a single topdressing N application at sowing; incorporated application:

a single N application incorporated at sowing). Two blends: 70% PSCU + 30% U and 30% PSCU + 70% U. A control treatment (without N-urea) was also included. The N rate used was 180 kg ha⁻¹, which would be expected to produce maize grain yields higher than 10 Mg ha⁻¹ in São Paulo state, Brazil (RAIJ; CANTARELLA, 1997). All treatments were applied by hand (Appendix A). The fertilizer incorporation was performed at 15 cm depth and 10 cm to the side of the seed row to avoid salt effect in the maize plant (GARCIA et al., 2019a). The U was treated with 530 mg NBPT kg⁻¹ and has 45% N. The PSCU (Patent n. EP 0574541) was manufactured by an industrial process in which a large quantity of U (prill) is sprayed by molten elemental sulfur (S⁰) and then by polymers (biodegradable and insoluble in water). In the final process, the PSCU has 39% N and 12% S, and the manufacturer indicates that ~80% of the N is released within 60 days after application (MARIANO et al., 2019).

To evaluate the ¹⁵N-fertilizer recovery by plants, the ¹⁵N-enriched U (CO(¹⁵NH₂)₂) with 1.6 and 1.15 atom % ¹⁵N was manufactured in a small quantity at Stable Isotopes Laboratory from the Center for Nuclear Energy in Agriculture (CENA/USP). Firstly, the ¹⁵N-U (powder) went through a granulation process (FRITSCHE, 2019). Secondly, the particle size distribution of granules were classified using ABNT (Brazilian Association of Technical Standards) sieve n. 6 and 10 (2 and 3.35 mm), and the hardness of granules was 2 kgf. Finally, the ¹⁵N-U with 1.6 atom % ¹⁵N was treated with NBPT (530 mg NBPT kg⁻¹), and the ¹⁵N-U with 1.15 atom % ¹⁵N was coated (by the industrial manufacturer) with elemental sulfur and polymers using a similar industrial process, it has 38.6% N and 11.8% S. The microtomography (Micro-CT; LANDIS; KEANE, 2010) shows the PSCU granules after both manufacture processes (Figure 1). The N cumulative release of the ¹⁵N-PSCU (Figure 2) was tested in the water at 25 °C (1:5 fertilizer/deionized water ratio), the supernatant sampled and replaced at 1, 5, 10, 20, 30, 40, 50, and 60 days, and the N released were measured in a mass spectrometer.



Figure 1 - Microtomography images of granules of polymer-sulfur coated urea (PSCU- 15 N and PSCU) applied at sowing (A, C) and its residual granules at R6 maize growth stage (B, D). The PSCU- 15 N (A) was manufactured with a similar process to the industrial PSCU (C). The empty granules (B, D) represent the N-fertilizer applied incorporated, broadcast, and split



Figure 2 - Cumulative nitrogen release of the polymer-sulfur coated urea with ¹⁵N (PSCU-¹⁵N) in the water at 25 °C. Vertical bars indicate the standard error of the mean (n = 3)

The maize experimental plot had 45 m² with 10 maize rows (10 m in length and 0.45-m spacing) and a density of 79,500 plants ha⁻¹. In season 2, two microplots were setup within plots to apply ¹⁵N-fertilizer (¹⁵N-PSCU + U and PSCU + ¹⁵N-U). PSCU without ¹⁵N was also manufactured by the adapted method to mix with ¹⁵N-U to apply in microplots. Each microplot had 2.7 m²: 2 m long and 1.35 m wide that includes three maize rows (Figure 3).



Figure 3 - The plot and microplot representation. The microplots are inside the plot (Appendix A). The ¹⁵N-fertilizers were applied, incorporated, broadcast, and split in the microplots. A sampling of plants were performed in adjacent rows at V4, V12, R2, and R4 maize growth stage. At R6, plants were sampled in the central row

The maize hybrid (DKB 390) was sown on 1 December 2017 in season 1 and on 26 November 2019 in season 2. In season 1 and season 2, 120 kg P_2O_5 ha⁻¹ as triple superphosphate was applied at sowing (beneath of seed row). Sixty and one-hundred and twenty kg K₂O ha⁻¹ as potassium chloride (KCl) was applied broadcast on the soil surface at V4 (season 1) and before sowing (season 2), respectively. S⁰ was applied at sowing in the control and in the treatments with 30% PSCU + 70% U to equalize the S⁰ that had in the 70% PSCU + 30% U. 3 kg B ha⁻¹ and 2 kg Zn ha⁻¹ were applied at sowing mixed with N treatments and in the control with S⁰ in both seasons. The control of weeds, insects, and diseases were performed in season 1 and season 2 when needed. The maize was harvested on 9 April 2018 in season 1 and 15 April 2020 in season 2. Two maize rows of 5 m long were selected to measure yield in season 1 and season 2 (Figure 3).

2.2.3 Quantification of Ammonia Volatilization

In season 1, ammonia (NH₃-N) volatilization was quantified during 34 days after N broadcast application (180 kg N ha⁻¹) and split application (side dressing at V4: 120 kg N ha⁻¹) on the soil surface. The ammonia capture was performed using open collectors (14 cm \times 14 cm \times 7cm) with a foam disc (15 cm in diameter, 6 cm in height and density of 0.02 g cm⁻³) soaked in 25 mL of phosphoric acid (1.5 mol L^{-1} and 5% of glycerol) allocated on the open side of the collectors. It was positioned 1 cm above the soil surface in the N-fertilizer application region. The foams were sampled and replaced at 2, 3, 4, 5, 6, 7, 9, 12, 16, 21, 27, and 34 days after N application. To extract NH₄⁺-N retained in the foams as ammonium phosphate, each foam was put in a beaker contained 300 mL of deionized water and squeezed. After that, beakers were weighted, and an aliquot of each solution was analyzed by flow injection analysis (FIA) to determine the NH₄⁺-N (REIS et al., 1997). The NH₃-N volatilization was determined by dividing the NH₄⁺-N (mg per collector) by the N-fertilizer applied per collector (broadcast: 350 mg; split (side dressing at V4): 792 mg) and multiplying the result by the N rate (kg ha⁻¹). The capture efficiency of the collector (26%) was also considered (GONZAGA; TRIVELIN, 2018). The NH₃-N daily losses (kg ha⁻¹ day⁻¹) were calculated by dividing the losses by the sample time (day), and the cumulative losses (kg ha⁻¹) were determined by summing the losses from each sample. The ammonia volatilization was not evaluated in season 2 because of the limited space in the plot, due to microplots (Figure 3), and the evaluations in season 2 were focused on soil and plant in the plot and microplots.

2.2.4 Analyses of Plant and Soil Samples

In season 1 and season 2, soil and plant sampling were performed during the maize cycle at the V4, V12 (vegetative leaf stage 12), R2 (blister stage), R4 (dough stage), and R6 (physiological maturity) stages (RITCHE et al., 1997). Three soil samples per plot were taken at three depths (0–20, 20–40, and 40–60 cm) based on the N fertilizer row. The samples of each depth were mixed for analysis. NO₃⁻-N and NH₄⁺-N content (Annex A) were extracted by 2 mol L⁻¹ KCl and determined by FIA (1:5 soil/solution ratio; RAIJ et al., 2001). The soil results were expressed as mineral N content (NO₃⁻-N + NH₄⁺-N). Four plants per plot were cut on the soil surface, separated (leaves, stalk, cobs, and grain), oven-dried to a constant weight (at 65 °C), weighed, and ground in a Wiley mill (0.5-mm sieve). The nutrient (N, P, K, Ca, Mg and S) concentration (g kg⁻¹) of each plant component was determined by titration (N, micro-Kjedahl), colorimetric method (P), turbidimetry (S), and atomic absorption spectroscopy (K, Ca, and MG) after sulfuric or nitroperchloric digestion (BATAGLIA et al., 1983). The dry weight of each plant component was multiplied by its nutrient (N, P, K, Ca, Mg, or S) concentration to determine the nutrient (N, P, K, Ca, Mg, or S) uptake (kg ha⁻¹). The nutrient uptake of each plant component was summed to determine the total nutrient uptake. Plant dry matter was expressed as Mg ha⁻¹. The maximum rates of dry matter and nutrient accumulation were determined by subtracting the accumulation (dry matter and nutrient) at the inflection point of a nonlinear sigmoid regression (Equation 1) by the previous day's accumulation (GARCIA et al., 2018; LAVIOLA et al., 2009). Two rows of 5 m were harvested by hand to determine the grain yield (13% moisture content) expressed as Mg ha⁻¹.

$$Y = Y \max / (1 + (X / X_0)^B)$$
(1)

where Y is the nutrient or dry matter accumulation (kg ha⁻¹); Ymax is the maximum accumulation of nutrient or dry matter (kg ha⁻¹); X is the period (day) between maize emergence (VE) and physiological maturity; X_0 is the moment (days after VE (DAE)) which is occurring the maximum rate of dry matter or nutrient uptake.

2.2.5 ¹⁵N-Fertilizer Recovery Analyses

In season 2, the ¹⁵N-PSCU + U was applied by hand in one microplot and PSCU + ¹⁵N-U in the other one in the same manner as the N treatments in the plot. At V4, V12, R2, and R4 stages, the aboveground of two plants were sampled in the internal and adjacent external row of the microplots (Figure 3) and were analyzed separately. The results of ¹⁵N-fertilizer recovery in these plants were summed in each abovementioned growth stage: plants in the adjacent external row (N-fertilizer without ¹⁵N) can recovery the ¹⁵N-fertilizer applied to the plants in the adjacent internal row of microplots (TRIVELIN et al., 1994). At R6, the aboveground of two plants were sampled in the central row of the microplot (Figure 3). Roots of a plant and soil were sampled in the center of the microplot (40 cm length × 40 cm width × 20 cm depth) at R6 to include in the N balance. Roots and the aboveground plant components separated (leaf, stalk, cob and grain) were oven-dried (at 65 °C), weighed, and ground (0.5-mm sieve). The total N concentration and ¹⁵N abundance in the soil and each plant component were determined in an automatic N analyzer interfaced to an isotope ratio mass spectrometer (PDZ Europa ANCA-GLS, 20-20, Sercon Ltd., Crewe, UK).

The control treatment was also analyzed. The ¹⁵N recovery was determined according to the following equations (TRIVELIN et al., 1994; HAUCK; BREMMER, 1976):

Ndff (%) =
$$\left(\frac{a-c}{b-c}\right) \times 100$$
 (2)

$$Ndff(kg ha^{-1}) = \left[\frac{Ndff(\%)}{100}\right] \times Total N$$
(3)

¹⁵N recovery (%) =
$$\left[\frac{Ndff(kgha^{-1})}{Nrate}\right] \times 100$$
 (4)

where Ndff (% and kg ha⁻¹) is the N derived from the fertilizer in the plant components or in the soil; a is the ¹⁵N abundance (atom % ¹⁵N excess) in the plant components or in the soil; b is the ¹⁵N abundance (atom % ¹⁵N excess) in the fertilizer; c is the natural ¹⁵N abundance (atom % ¹⁵N) in the control treatment. Total N is the plant N content (kg N ha⁻¹). ¹⁵N recovery is the N-fertilizer recovered by the maize plant (%). N rate is the N-fertilizer rate in kg N ha⁻¹ that was 180 kg N ha⁻¹.

2.2.6 Statistical Analyses

A combined analysis of variance (ANOVA) was performed for the variables measured in season 1 and season 2. N fertilization management practices, fertilizer blend, and season, were considered fixed effects. A new factor was also included to compare the control treatment. The ANOVA ($p \le 0.05$) was performed using the PROC MIXED procedure of SAS (version 9.0, SAS Institute Inc., Cary, NC, USA), and the means were tested using Fisher's least-test difference (LSD) at the 0.05 significance level. Mixed models were performed for variables from the ¹⁵N-fertilizer analyses in season 2. N fertilization management, fertilizer blend, and ¹⁵N-fertilizer were considered fixed effects and were tested by the Wald-F test. Multiple comparisons were performed by the LSD test. The software R (R DEVELOPMENT CORE TEAM, 2015) and its asreml and asremlPlus package were used. The level of significance was 0.05. The seasonal dry matter (biomass) and nutrient partitioning (leaves, stalks, cobs, and grains) during the maize cycle were fitted to a Gaussian equation (Equation 5) (GARCIA et al., 2018). The total nutrient uptake and ¹⁵N recovery (leaves, stalks, and cobs) at R2 were subtracted by that at R6 to estimate the nutrient remobilization based on the nutrient accumulation models.

$$Y = A x \exp(-0.5 x ((DAE - B)/C^{2}))$$
(5)

where Y is the aerial part biomass (Mg ha⁻¹) or nutrient uptake (kg ha⁻¹), DAE is the day after maize emergence, and A, B and C are constants.

2.3 Results

2.3.1 Weather Conditions

The average daily air temperature was 25 °C during the maize growing seasons (Figure 4A, B). In season 1, the total precipitation was 643 mm, of which 421 mm occurred from maize sowing to V12, 204 mm occurred from V12 to R5 (dent stage), and 18 mm occurred from R5 to R6 (Figure 4A). In addition, three irrigations of 3 mm were performed three days after sowing and 8 mm at V4 (Figure 4A). In season 2, the total precipitation was 669 mm, of which 394 mm occurred from maize sowing to V12, 199 mm occurred from V12 to R5, and 76 mm occurred from R5 to R6 (Figure 4B). In addition, two irrigations of 8 mm were performed between V4 and V6 (vegetative leaf stage 6) (Figure 4B).



Figure 4 - Daily minimum and maximum air temperature, daily rainfall, and irrigation for the maize growing seasons (A: Season 1 (2017–2018); B: Season 2 (2019–2020). GDDc: Growing degree days in Celsius calculated according to (KARLEN et al., 1988)

2.3.2 Ammonia Volatilization

In season 1, the 70% PSCU + 30% U broadcast application resulted in the maximum daily NH₃ -N loss (2% of the applied N) on the sixth day after N application and a cumulative NH₃ -N loss of 12% of the applied N on the 34th day after N application. It was higher than the 30% PSCU + 70% U broadcast application ($p \le 0.05$) that resulted in the maximum daily and cumulative NH₃ -N loss of 1.4% and 9% of the applied N, respectively (Figure 5A,B). The split N application at V4 resulted in the maximum daily NH₃ -N loss (0.5% of the applied N) on the second day after the N application and a cumulative NH₃ -N loss of 2.6% of the applied N on the 34th day after 70% PSCU + 30% U application. It was higher than the 30% PSCU + 70% U split application ($p \le 0.05$) that resulted in the cumulative NH₃ -N loss of 0.8% of the applied N on the 34th day after 70% PSCU + 30% U application. It was higher than the 30% PSCU + 70% U split application ($p \le 0.05$) that resulted in the cumulative NH₃ -N loss of 0.8% of the applied N of the applied N (Figure 5C, D).



Figure 5 - Daily and cumulative losses of NH₃ -N in broadcast N application at sowing (180 kg N ha⁻¹) on the soil surface (A,B) and split N application side dressing at V4 (120 kg N ha⁻¹) maize growth stage (C,D). Vertical bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$).

2.3.3 Mineral N Content in the Soil



In season 1 and season 2, the N fertilization management practice and fertilizer blend affected the mineral N content during the maize growth cycle (Figure 6).

Figure 6 - Effect of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on soil N mineral content during the maize growing seasons (S1: season 1 (2017–2018); S2: season 2 (2019–2020)). Horizontal bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) at each depth

At V4, the incorporated N application resulted in higher mineral N content than the other N fertilization management practices at depths of 0-20 and 20-40 cm, and the experiment in season 1 resulted in higher mineral N content than in season 2 at 40–60 cm depth. At V12, the incorporated N application and split N application resulted in higher mineral N content than the broadcast N application at 0-20 cm depth. At V12, the experiment in season 2 resulted in higher mineral N content than in season 1 at 20–40 cm depth, and the 30% PSCU + 70% U application resulted in higher mineral N content than the 70% PSCU + 30% U application at

40–60 cm depth. At R2, the incorporated N application (in season 1) and the split N application (in season 2) resulted in higher mineral N content than the other N fertilization management treatments at 0–20 cm depth. At R2, the 30% PSCU + 70% U application resulted in higher mineral N content than the 70% PSCU + 30% U application at depths of 20–40 cm (in season 2) and 40–60 cm (both seasons). At R4, N treatments did not affect mineral N content (p > 0.05). At R6, the incorporated N application resulted in higher mineral N content than the broadcast application at 0–20 cm depth, and the N treatments did not affect mineral N content at 20–40 cm (depth).

2.3.4 Biomass (Dry Matter) Accumulation in Plants and Maize Yield

The N fertilization management practice affected the total dry matter accumulation in vegetative and reproductive maize growth stages, and the fertilizer blend affected the maize yield (Figure 7).



Figure 7 - Effect of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on maize yield and dry matter accumulation (aerial part) during the maize growing seasons (Season 1 (S1): 2017–2018; Season 2 (S2): 2019–2020). Vertical bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) in each maize growth stage

At V4, the incorporated N application resulted in higher dry matter accumulation (0.17 Mg ha⁻¹) than the other N management treatments (0.16 Mg ha⁻¹) in both seasons, and the experiment in season 2 resulted in higher dry matter accumulation (0.22 Mg ha⁻¹) than in season 1 (0.1 Mg ha⁻¹). At V12, the experiment in season 1 resulted in higher dry matter accumulation (6.8 Mg ha⁻¹) than in season 2 (5.7 Mg ha⁻¹). The N treatments did not affect the dry matter accumulation (p > 0.05) at R2 (12.7 Mg ha⁻¹) and R4 (16 Mg ha⁻¹). At R6, the N fertilization management practices resulted in higher dry matter accumulation (20 Mg ha⁻¹) than the control (16 Mg ha⁻¹) in season 1. At R6, N treatments did not affect dry matter accumulation (26 Mg ha⁻¹) in season 2, and the experiment in season 2 resulted in higher dry matter accumulation (26 Mg ha⁻¹) than in season 1 (19 Mg ha⁻¹). The 70% PSCU + 30% U application resulted in higher maize yield (8.3 Mg ha^{-1}) than 30% PSCU + 70% U application (7.6 Mg ha⁻¹) in season 1. The N treatments did not affect the maize yield in season 2 (12.1 Mg ha⁻¹), and the experiment in season 2 resulted in higher maize yield (12.1 Mg ha⁻¹) than in season 1 (7.5 Mg ha⁻¹). In season 1, the maximum rate of dry matter accumulation in the control (420 kg ha⁻¹ day⁻¹) occurred on the 58th day after maize emergence (VE) and in the N treatments (361 kg ha⁻¹ day⁻¹) on the 56th day after VE (Figure 8D, E).



Figure 8 - Seasonal dry matter and N accumulation and partitioning in the control (A,D) and in the N treatments (B,E) during the maize growing season 1 (2017–2018) and in the treatments (C,F) during the maize growing season 2 (2019–2020). The arrow indicates the maximum daily rate of dry matter and N accumulation. DAE is the day after maize emergence. The dashed line is the N derived from the fertilizer (Ndff) in each plant component during the maize growing season 2 (C)



Figure 9 - The daily rate of dry matter (A) and N accumulation (B) (kg ha⁻¹ day⁻¹) in maize during two growing seasons (season 1: 2017–2018; season 2: 2019–2020). Average of treatments (two fertilizer blends, three N fertilization management practices, and a control treatment) in each growing season
2.3.5 Nitrogen Uptake in Maize Plants

The N fertilization management practices affected the total N uptake in the vegetative and reproductive maize growth stages (Figure 10).



Figure 10 - Effect of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on N uptake (aerial part) during the maize growing seasons (Season 1 (S1): 2017–2018; Season 2 (S2): 2019–2020). The dashed line is the N derived from the fertilizer (Ndff) in the plant in season 2. Vertical bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) in each maize growth stage

At V4, the incorporated N application resulted in higher N uptake (8 kg ha⁻¹) than the other N fertilization management treatments (7 kg ha⁻¹) in both seasons. At V4, the experiment in season 2 resulted in higher N uptake (9 kg ha⁻¹) than in season 1 (4 kg ha⁻¹), and the Ndff in season 2 was 3.8 kg ha^{-1} on average. At V12, the experiment in season 2 resulted in higher N uptake (138 kg ha⁻¹) than in season 1 (118 kg ha⁻¹), and the Ndff in season 2 was 55 kg ha⁻¹ on average. At R2, the experiment in season 2 resulted in higher N uptake (205 kg ha⁻¹) than in season 1 (171 kg ha⁻¹), and the Ndff in season 2 was 95 kg ha⁻¹ on average.

At R4, the experiment in season 2 resulted in higher N uptake (253 kg ha⁻¹) than in season 1 (221 kg ha⁻¹), and the Ndff in season 2 was 102 kg ha⁻¹ on average. At R6, the N broadcast application resulted in higher N uptake (261 kg ha⁻¹) than split N application (215 kg ha⁻¹) in season 1, the N treatments did not affect total N uptake (324 kg ha⁻¹) in season 2, and the Ndff in season 2 was 130 kg ha⁻¹ on average. The experiment in season 2 resulted in higher total N uptake (324 kg ha⁻¹) than in season 1 (221 kg ha⁻¹). The N broadcast application resulted in higher N uptake in the grain (179 kg ha⁻¹) than the other N fertilization management treatments (144 kg ha⁻¹) in season 1. The N treatments did not affect the N uptake in the grain (216 kg ha⁻¹) in season 2, and the Ndff in the grain in season 2 was 86 kg ha⁻¹ (Figures 8C and 9). The experiment in season 2 provided higher N uptake in the grain (216 kg ha^{-1}) than in season 1 (146 kg ha⁻¹). In season 1, the maximum rate of N uptake in the control $(4.8 \text{ kg ha}^{-1} \text{ day}^{-1})$ occurred on the 55th day after VE and in the N treatments $(5.2 \text{ kg ha}^{-1} \text{ day}^{-1})$ occurred on the 48th day after VE (Figure 8A,B). In season 2, the maximum rate of N uptake in the treatments (4.5 kg ha⁻¹ day⁻¹) occurred on the 48th day after VE (Figures 8C and 9B). The N remobilization from R2 (leaf, stalk, and cob) to R6 (grain) was 57 kg N ha⁻¹ in the control (Figure 8A) and 73 kg N ha⁻¹ in the N treatments (Figure 8B) in season 1, and 77 kg N ha⁻¹ (Figure 8C) in the treatments in season 2. The remobilization in terms of Ndff was $42 \text{ kg N} \text{ ha}^{-1}$ in the N treatments in season 2 (Figure 8C).

2.3.6 ¹⁵N-Fertilizer Recovery in Maize Plants and Nitrogen Balance

In season 2, the N fertilization management practice and fertilizer blend affected the ¹⁵N recovery (NR) from fertilizer N source by plants during the maize growth cycle (Figure 11).



Figure 11. Effect of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on ¹⁵N-fertilizer recovery (aerial part) during the maize growing season (2019–2020). Vertical bars indicate the standard error of the mean. Means followed by different lowercase letters indicate difference ($p \le 0.05$) between ¹⁵N source (¹⁵N-PSCU and ¹⁵N-U) and among treatments, while different capital letters indicate difference ($p \le 0.05$) of total ¹⁵N-fertilizer recovery among treatments in each maize growth stage

At V4, the split N application resulted in higher total NR (3.6%) than the broadcast (3.1%) and incorporated (2.1%) application, and the 70% PSCU + 30% U application resulted in higher total NR (3.4%) than 30% PSCU + 70% U application (2.5%). The PSCU resulted in higher NR (2.6%) than the U (0.8%) under 70% PSCU + 30% U application, and it was also observed under split N application. At V12, the N broadcast application resulted in higher total NR (37%) than the other N fertilization management treatments (27%), and the 30% PSCU + 70% U application resulted in higher total NR (33%) than the 70% PSCU + 30% U application (28%). The PSCU resulted in higher NR (20%) than U (8%) under 70% PSCU + 30% U application, and the U resulted in higher NR (21%) than the PSCU (12%) under 30% PSCU + 70% U application. At R2, the incorporated N application and split N application resulted in higher total NR (60%) than the N broadcast application (39%), and the fertilizer blend did not affect the total NR (54%). The PSCU resulted in higher NR (38%) than the U (16%) under 70% PSCU + 30% U application, and the U resulted in higher NR (35%) than the PSCU (19%) under 30% PSCU + 70% U application. At R4, the split N application resulted in higher total NR (67%) than the N broadcast application (49%), and the 30% PSCU + 70% U application resulted in higher total NR (68%) than the 70% PSCU + 30% U application (50%). The PSCU resulted in higher NR (34%) than the U (16%) under 70% PSCU + 30% U application, and the U resulted in higher NR (49%) than the PSCU (19%) under 30% PSCU + 70% U application. At R6, the N fertilization management practice and fertilizer blend did not affect the total NR (72%). The PSCU resulted in higher NR (51%) than the U (23%) under 70% PSCU + 30% U application, and the U resulted in higher NR (50%) than the PSCU (22%) under 30% PSCU + 70% U application. From the total NR at R6 (plant aboveground), it was in grain (47.91%), leaf (15.4%), stalk (6.84%), and cob (2.34%). It was also found 1.37% of NR in root and 0.02% in the soil at 0–20 cm depth (Figure 12).



Figure 12 - Balance of ¹⁵N-fertilizer recovery (%) in the soil-plant system applying blends of polymersulfur coated urea (PSCU) and conventional urea (U), 70% PSCU + 30% U and 30% PSCU + 70% U, incorporated, broadcast, and split in maize in Brazilian tropical conditions. The graph represents all N treatments, and the rate of N was 180 kg ha⁻¹

The PSCU resulted in higher NR in the grain (34%) than the U (15%) under 70% PSCU + 30% U application, and the U resulted in higher NR in the grain (34%) than the PSCU (14%) under 30% PSCU + 70% U application. The incorporated N application and split N application resulted in higher N remobilization from R2 (leaf, stalk, and cob) to R6 (grain) in terms of NR (30%) than N broadcast application (13%) (Figure 11).

2.3.7 Phosphorus, Potassium and Sulfur Uptake in Maize Plants

The N fertilization management practices affected the total P and K uptake, and the blends affected the S uptake especially at R6 growth stage (Figure 13).



Figure 13 - Effect of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on P, K and S uptake (aerial part) during the maize growing seasons (Season 1 (S1): 2017–2018; Season 2 (S2): 2019–2020). Vertical bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) in each maize growth stage

At V4, the experiment in season 2 resulted in higher P (0.7 kg ha⁻¹), K (6.6 kg ha⁻¹) and S (0.6 kg ha⁻¹) uptake compared to P (0.3 kg ha⁻¹), K (2.8 kg ha⁻¹) and S (0.3 kg ha⁻¹) uptake in season 1. At V12, the N treatments did not affect K (147 kg ha⁻¹) and S (11 kg ha⁻¹) uptake, and the experiment in season 2 resulted in higher P uptake (13 kg ha⁻¹) than in season 1 (11 kg ha⁻¹). At R2, the N treatments did not affect K uptake (194 kg ha⁻¹); the experiment in season 2 resulted in higher P uptake (19 kg ha⁻¹) than season 1 (16 kg ha⁻¹); and the experiment in season 1 resulted in higher S uptake (18 kg ha⁻¹) than season 2 (12 kg ha⁻¹). At R4, the N

treatments did not affect P (26 kg ha⁻¹), K (198 kg ha⁻¹), and S (25 kg ha⁻¹) uptake. At R6, the broadcast N application resulted in higher P (total: 31 kg ha⁻¹; grain: 26 kg ha⁻¹) and K (total: 235 kg ha⁻¹; grain: 72 kg ha⁻¹) uptake compared to P (total: 26 kg ha⁻¹; grain: 20 kg ha⁻¹) and K (total: 156 kg ha⁻¹; grain: 50 kg ha⁻¹) uptake in the control in season 1. The split N application resulted in higher P uptake (total: 60 kg ha⁻¹; grain: 52 kg ha⁻¹) compared to the P uptake (total: 52 kg ha⁻¹; grain: 44 kg ha⁻¹) in the broadcast N application and control in season 2. The N treatments did not affect K uptake in season 2 (total: 211 kg ha⁻¹; grain: 67 kg ha⁻¹). The 70% PSCU + 30 % U treatment resulted in higher S uptake (total: 17 kg ha⁻¹) in season 1, and the 30% PSCU + 70% U treatment resulted in higher S uptake (total: 30 kg ha⁻¹) than 70% PSCU + 30% U (total: 23 kg ha⁻¹) in season 2. In addition, the N treatments did not affect the S uptake in the grain in the both seasons (14 kg ha⁻¹).

The maximum rate of P and K uptake in the treatments in season 2 (Figures 14 L, M; 15 A, B) were higher and occurred earlier than that in the control (Figure 14 A, B) and in the N treatments (Figure 14 F, G) in season 1. In addition, the maximum rate of S uptake in the treatments in season 2 (Figures 14 O; 15 C) was lower and occurred later than that in the control (Figure 14 C) and in the N treatments (Figure 14 H) in season 1. The remobilization from R2 (leaf, stalk, and cob) to R6 (grain) was 21 kg P ha⁻¹, 130 kg K ha⁻¹, and 4 kg S ha⁻¹ in the N treatments in season 2 (Figure 14 L, M, O). In the season 1, the remobilization was 15 kg P ha⁻¹, 180 kg K ha⁻¹, and 10 kg S ha⁻¹ in the control (Figure 14 F, G, H). It can be noted that the K uptake in the models (Figure 14 B, G, M) slightly decreased after R4 growth stage. It cannot be considered with only the remobilization once K can be lost from leaves by leaching (KARLEN et al., 1988), and it can also overestimate the values of the maximum rate of K uptake (Figure 15 B).



Figure 14 - Seasonal P, K, S, Ca, and Mg accumulation and partitioning in the control (A, B, C, D, and E) and in the N treatments (F, G, H, I, and J) during the maize growing season 1 (2017–2018) and in the treatments (L, M, O, Q, and R) during the maize growing season 2 (2019–2020). The arrow indicates the maximum daily rate of P, K, S, Ca, and Mg accumulation. DAE is the day after maize emergence



Figure 15 - The daily rate of P (A), K (B), S (C), Ca (D), and Mg (E) accumulation (kg ha⁻¹ day⁻¹) in maize during two growing seasons (season 1: 2017–2018; season 2: 2019–2020). Average of treatments (two fertilizer blends, three N fertilization management practices, and a control treatment) in each growing season

2.3.8 Calcium and Magnesium Uptake in Maize Plants

The N fertilization management practices affected the Ca and Mg uptake especially at the earlier growth stage (Figure 16). At V4, the broadcast N application resulted in higher Ca (0.8 kg ha⁻¹) and Mg (0.6 kg ha⁻¹) compared to the control (0.6 kg Ca ha⁻¹; 0.4 kg Mg ha⁻¹) and split N application (0.7 kg Ca ha⁻¹; 0.5 kg Mg ha⁻¹) in season 2. In addition, the N treatments did not affect Ca (0.4 kg ha⁻¹) and Mg (0.2 kg ha⁻¹) uptake in season 1. From V12 to R6 the experiment in season 1 resulted higher Ca uptake than that in season 2, reaching a total of 64 kg Ca ha⁻¹ (23 kg Ca ha⁻¹ in the grain) at R6 in season 1, and a total of 42 kg Ca ha⁻¹ (6 kg Ca ha⁻¹ in the grain) in season 2. Moreover, from V12 to R6 the experiment in season 1 resulted in lower Mg uptake than that in season 2, reaching a total of 30 kg Mg ha⁻¹ (14 kg Mg ha⁻¹ in the grain) at R6 in season 1, and a total of 30 kg Mg ha⁻¹ in the grain) in season 2.

The maximum rate of Ca uptake in the N treatments in season 2 (Figures 14 Q; 15 D) was lower and occurred earlier than that in the control (Figure 14 D) and in the N treatments in season 1 (Figure 14 I). In addition, the maximum rate of Mg uptake in the N treatments in season 2 (Figure 14 R, 15 E) was higher and occurred at the same moment than that in the control (Figure 14 E) and in the N treatments in season 1 (Figure 14 E).



Figure 16 – Effect of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on Ca and Mg uptake (aerial part) during the maize growing seasons (Season 1 (S1): 2017–2018; Season 2 (S2): 2019–2020). Vertical bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) in each maize growth stage

The remobilization from R2 (leaf, stalk and cob) to R6 (grain) was 14 kg Mg ha⁻¹ in the N treatments in season 2 (Figure 14 R), and was 2 kg Mg ha⁻¹ in the control (Figure 14 E) and 4 kg Mg ha⁻¹ in the N treatments (Figure 14 J) in season 1. The Ca uptake in the models slightly decreased after R5 growth stage (Figure 14 D, I, Q), it can be associated with the falling of senescent leaves (KARLEN, 1988), and it can also overestimate the values of nutrient remobilization.

2.4 Discussion

In season 1, the 70% PSCU + 30% U application produced 1552 kg grain ha^{-1} more than the control treatment that had no difference compared to the 30% PSCU + 70% U application. In terms of agronomic efficiency, it is the same to say that each 1 kg N ha⁻¹ applied with 70% PSCU + 30% U produced 8.6 kg grain ha^{-1} more than the control. However, the N rate (180 kg N ha⁻¹) adopted in our experiments was aiming yields higher than 10 Mg ha⁻¹ (RAIJ et al., 1997) and the highest yield obtained in season 1 was 8.3 Mg ha⁻¹. Another study in Brazilian condition evaluating blends of PSCU + U (180 kg N ha⁻¹) applied incorporated at maize sowing in a sandy loam soil found the agronomic efficiency of 16 kg grain ha^{-1} and a yield of 10.5 Mg ha⁻¹ (GARCIA et al., 2018). In Chinese conditions, blends of CRU + U applied as basal fertilizer in maize provided an agronomic efficiency of 21 kg grain ha⁻¹ and a yield of 9.7 Mg ha⁻¹ in sandy soil (185 kg N ha⁻¹), and 15.7 kg grain ha⁻¹ and a yield of 12.2 Mg ha⁻¹ in a clayey soil (171 kg N ha⁻¹) (LI et al., 2020a). The maize yield can be affected by the genetic of hybrids, pests and diseases, weather conditions, and nutritional management (IPNI, 2003). The loss of NH₃ -N by volatilization reached 22 kg N ha⁻¹ under the broadcast-applied 70% PSCU + 30% U treatment that represented 6 kg N ha⁻¹ more than the observed under the broadcast-applied 30% PSCU + 70% U treatment in season 1. Subtracting that loss by the N rate (180 kg N ha⁻¹), the result is close to the N rate of 150 kg N ha⁻¹ that is recommended expecting maize grain yields between 8 and 10 Mg ha⁻¹ (RAIJ et al., 1997) in São Paulo state, Brazil. The irrigations performed days after the N broadcast application favored the volatilization of ammonia. After the 50 mm precipitation in one day, the U probably incorporated and reduced the ammonia volatilization. The PSCU (the insoluble source) probably stayed on the soil surface, providing more ammonia volatilization in the blend with more PSCU. The U, treated with NBPT, probably inhibited the ammonia volatilization during the irrigated days. It also occurred on a lesser scale in the split N application (side dressing at V4) followed by the precipitations after N application. In a study evaluating blends of PSCU + U in coffee in Brazilian conditions were observed 20 kg ha⁻¹ of NH₃ -N volatilization applying 220 kg N ha⁻¹ using another type of ammonia collector at 42 days after fertilizer application (CHAGAS et al., 2016). In that study, was also found loss of 40 kg ha⁻¹ of NH₃ -N applying just U (150 kg N ha⁻¹). In our study, the precipitation also provided the soil N mineral percolation to 40-60 cm soil layer at the V12 and R2 growth stages in both seasons. It disfavors 30% PSCU + 70% U application that resulted in higher soil N mineral content at 40-60 cm depth than 70% PSCU + 30% U application, probably by the N from the U that is a soluble N

source, and 30% PSCU + 70% U application consequently provided lower yield in season 1. In Chinese conditions, blends of CRU + U resulted in a similar soil N mineral content at 40-60 cm depth in a clayey soil, but at R6 maize growth stage (LI et al., 2020a). In that study, the authors used a CRU with a different pattern of N release (80% of N was released within 120 days after application) (LI et al., 2020a) compared to our study. Other effects of blends of CRU and U and just CRU (with polymer without S⁰) on ammonia volatilization, maize yield, and soil N mineral in Chinese conditions can be found in (LI et al., 2020b; KE et al., 2017), and in Brazilian conditions in (MIYAZAWA et al., 2020).

The experiment in season 2, in general, resulted in higher dry matter, N uptake, P uptake, Mg uptake, and maize yield than in season 1. It can be explained by the difference in the daily rate of dry matter and nutrient accumulation during the maize growth cycle (Figures 9; 15) that was probably affected by the agricultural history of the experiments. The better condition observed in season 2 for the maize growth, that was non-responsive to N-fertilizer application, can be associated with the recent application of limestone and the soil preparation (plowing and harrowing), that provided an optimal base saturation to maize production, and accelerated the mineralization of soil organic matter (LOSS et al., 2014). In addition, the precipitation in season 2 was better distributed than in season 1, especially between R5 and R6, and hydric stress at R5 can anticipate the physiological maturity and reduce the weight of grains (MAGALHÃES; DURÃES, 2006). The roots growth of maize plants in season 1 probably was limited at 0-20 cm depth based on the aluminum saturation (>20%) at 20-40 and 40-60 cm depth, and in favorable conditions, 48% of maize roots can develop below of 30 cm depth depending on soil texture (FELDMAN, 1994). It probably limited the water and nutrient uptake at 0-20 cm depth, favoring the N broadcast application and the N-fertilizer with more PSCU that stayed on the soil surface, and limiting maize yield in season 1. The aluminum saturation higher than 20% is typically found in no-tillage systems, and it is normally recommended to apply gypsum to reduce the aluminum saturation, in-depth (VICENSI et al., 2020). The nonresponse to N fertilization using blends of CRU and U and just U was also observed in maize in Rhodic Haplustox sites (SCHONINGER et al., 2018; GARCIA et al., 2018), which also has a clayey texture, and in other crops (BOSCHIERO et al., 2020; GARCIA et al., 2020a) in Brazilian conditions.

The incorporated N-fertilizer application tended to result in higher soil N mineral content at 0-20 cm depth during the maize growth cycle than the other N fertilization management treatments once this application is concentrated in the fertilizer row and the soil samples were performed in this region. However, the incorporated N application resulted in higher N uptake and dry matter accumulation only at V4 (both seasons) and the incorporated N application was not sufficient to affect the maize yield potential (SANGOI, 2001). Similar results of dry matter accumulation and N uptake in maize at the V4 growth stage applying blends of PSCU + U just incorporated at sowing were observed by (GARCIA et al., 2018) in a sandy loam soil and a clayey soil. Contrary to our hypothesis, the CRU was the main N-fertilizer supplier at the V4 growth stage based on the NR in the 70% PSCU + 30% U application, and the CRU resulted in the same NR compared to the U in the 30% PSCU + 70% U application at V4. It shows that the PSCU of our study applied in Brazilian conditions can provide N to maize plants in the early stages, different from what happened in North America using another CRU based on the low soil N availability (GRANT et al., 2012). It probably can be explained by the N released from our PSCU that reached ~20% of the applied N in the first day tested in water. Although the 30% PSCU + 70% U application resulted in lower total NR at V4 and higher soil N mineral content at 40–60 cm (V12 and R2 stages) than the 70% PSCU + 30% U application, 30% PSCU + 70% U resulted in the same and in some cases higher total NR than 70% PSCU + 30% U after V4 maize growth stage. It probably is associated with the development of roots below of 0-20 cm soil layer in season 2 (low in-depth aluminum saturation), resulting in the NR of the U (30% PSCU + 70% U application) similar to the NR of the CRU (70% PSCU + 30% U application). The split N application resulted in higher NR at V4 than the other N fertilization management treatments, but the split N application treatments just received 30% of N-fertilizer at sowing; lower N rates tend to provide higher NR (ROBERTS et al., 2016; WALSH et al., 2012). The N broadcast application resulted in higher total NR at V12 and lower total NR at R2 and R4 growth stages than other N management treatments, but the total N uptake had no difference among treatments. In this situation, the priming effect (CHEN et al., 2019) could be higher after V12 and until the R4 growth stage in the N broadcast application, although the control treatment resulted in similar N uptake. The broadcast application also resulted in lower remobilization of NR to the grain from R2 to R6 compared to the other N fertilization management treatments; it indicated that maize plants tended to absorb more N from the fertilizer after R2 in the broadcast application. At R6, the N treatments resulted in the same NR in season 2, and the unrecovered N-fertilizer (26.12%) can be associated with the ammonia volatilization and the N percolation. This is the first study that evaluated the ¹⁵N-fertilizer recovery in maize using blends of PSCU and U with ¹⁵N in both sources of the blends. Other studies just evaluated the ¹⁵N-fertilizer recovery from the U source of a blend of PSCU + U, and just applied 70% PSCU + 30% U broadcast (MOSCHINI, 2019) or incorporated (VILLALBA, 2018) in the same type of soil of our experiment. They found an average of 12% of NR from the U source by maize using N rate around 180 kg N ha⁻¹. The main challenge in a study like that is to manufacture the PSCU-¹⁵N with the same characteristics as the industrial product, providing high costs to the research. It can restrict the studies on this topic. In our study, it was just possible to work in one-year experiment using ¹⁵N-fertilizers. If we used ¹⁵N in season 1, the results of NR could change because of the aluminum saturation at 20-40 and 40-60 cm depths (>20%) that can restrict the N uptake at 0-20 cm depth, and plants could likely recovery more N from the 70% PSCU + 30% U compared to the 30% PSCU + 70% U application. The average of NR in cereals worldwide is 33% (LADHA, et al., 2005). In specific conditions in Brazil, the NR of maize applying different N sources ranged from 19% (GAVA et al., 2010) to 89% (LARA CABEZAS; COUTO, 2007). In China, the NR ranged from 26% to 35% (WANG et al., 2016), in America can be found 52%, and in Europe, 62% on average (LADHA et al., 2005) in most cereals. These variations of NR are normally attributed to the type of the soil, weather conditions (WALLACE et al., 2020; TORBERT et al., 1992; HAUCK, 1973), the N-fertilizer source (LANGE et al., 2010; LARA CABEZAS; COUTO, 2007), the N fertilization management practice, the N rate, and the soil management practice (GAVA et al., 2010; LANGE et al., 2010, GAVA et al., 2006). It would be of interest in future studies to evaluate the dynamics of blends of CRU + U and other enhanced efficiency fertilizers using ¹⁵N in sandy loam soil. This soil is normally more responsive for N fertilizer application in maize than clayey soil (GARCIA et al., 2020; LI et al., 2020a), and it would improve the recommendation for these fertilizers in Brazilian conditions. Our study can help future studies that will use ¹⁵N in blends of CRU and U and help to improve CRU fertilizers aiming to increase the N-fertilizer recovery.

2.5 Conclusions

In Rhodic Eutrustox soil, blends of PSCU and U treated with NBPT (70% PSCU + 30% U and 30% PSCU + 70% U), at a rate of 180 kg N ha⁻¹, applied incorporated at sowing, broadcast on the soil surface at sowing, and split (30% incorporated at sowing and 70% side-dressing at V4) can ensure N throughout the maize growth cycle in Brazilian condition.

In season 2, the PSCU was the main N-fertilizer supplier at the V4 growth stage, applying 70% PSCU + 30% U, and both blends can ensure 73.8% of N-fertilizer recovery (maize aerial part + root) of which 47.9% in the grain. The unrecovered N can be attributed to the ammonia volatilization losses that reached 11% on average in season 1 and the N percolation that was prominent at 40–60 cm depth in important maize growth stages in applications with 30% PSCU + 70% U in both seasons. The agricultural history of the experiments can affect the macronutrient uptake. Recent application of limestone, plowing, and harrowing can provide a nonresponse to N fertilization in a Rhodic Eutrustox soil with common bean straw left on the soil surface, and to replace the N extracted by harvest, farmers can choose the most cost-effective option.

3 NITROGEN FERTILIZATION MANAGEMENT WITH BLENDS OF CONTROLLED-RELEASE AND CONVENTIONAL UREA AFFECTS COMMON BEAN GROWTH AND YIELD DURING MILD WINTERS IN BRAZIL

ABSTRACT

The common bean (Phaseolus vulgaris L.) requires nitrogen (N) during its vegetative and reproductive stages. A single application of a blend of polymer-sulfur coated urea (PSCU) and conventional urea (U) treated with NBPT (N-(n-butyl) thiophosphoric triamide) can meet that demand. Broadcast application could improve yield than other N management practices. This research evaluated two blends (70% PSCU + 30% U and 30% PSCU + 70% U) and three N fertilization managements (incorporated, broadcast, and split application) on soil ammonia volatilization (AV) and N mineral content (NM); plant macronutrient uptake and ¹⁵N recovery from U (NUR); and yield (GY). Irrigated field experiments were conducted in 2018 and 2019 in Rhodic Eutrustox soil. The N application rate was 90 kg ha⁻¹. AV reached 12% (30% PSCU + 70% U, broadcast application) and 14% of the applied N (split application at the third trifoliate leaf unfolded stage (V4)). The incorporated application resulted in higher NM in the vegetative and reproductive stages than the other management practices. Broadcast application resulted in higher N uptake than split application at physiological maturity. Split application resulted in higher NUR (grain) and GY than broadcast application. There was a positive correlation between NUR (grain) and GY in all N fertilization management treatments. The NUR values reached 48% (30% PSCU + 70% U) and 18% (70% PSCU + 30% U). Split N application using these blends can improve NUR in grain and GY compared to broadcast application in Rhodic Eutrustox soil. This information can help farmers improve the fertilization management practices used with these blends, and thereby avoid economic losses and environmental pollution.

Keywords: polymer-sulfur coated urea; NBPT-treated urea; ammonia volatilization; soil N mineral; ¹⁵N-urea recovery; *Phaseolus vulgaris* L.

3.1 Introduction

The common bean is a legume cultivated in three growing seasons per year in Brazil, the world's largest consumer and producer of the common bean (3.3 million tons) (CONAB, 2020). The first growing season of common bean in Brazil occurs between July and October, the second occurs between January and March, and the third occurs between April and July. The average common bean yield in Brazil is 1100 kg ha⁻¹ and reaches 1800, 850, and 2511 kg ha⁻¹ in the first, second and third growing seasons in São Paulo state, respectively (CONAB, 2020). The difference in yield between the first and second growing seasons is related especially to climatic conditions (high temperatures and hydric stress) and is associated with low soil fertility, soil acidity, pests and diseases, and low technology use by farmers (FAGERIA et al., 2015). The third growing season, which occurs during the mild winters, emerged in Brazil simultaneously with center-pivot irrigation systems (ZIMMERMANN, 1988). These systems prompted farmers to invest in irrigation to cultivate crops during this period, in which almost no rain falls. The third season produces higher yields than the first and second season due to irrigation, as well as fertilization and pest and disease control.

The common bean is a legume, but the symbiotic bacterial association (Rhizobium leguminosarum bv. phaseoli) with its roots provides low-efficiency biological nitrogen (N) fixation (REINPRECHT et al., 2020), and N fertilizer application is recommended to meet its N demand in most cases. N is the most limiting nutrient for common bean yield (FAGERIA et al., 2015), and conventional urea (U) is the main N source because of its high N concentration and low price compared to other N sources (CARNEIRO et al., 2015). However, N fertilizer recovery from U application in common bean can be lower than 50% (FAGERIA et al., 2015), resulting in ammonia volatilization losses (RINALDI et al., 2019) when U is applied on the soil surface, nitrate leaching (HONG et al., 2007) and denitrification (DUSENBURY et al., 2008). N immobilization (KONG et al., 2014) must also be considered, especially under current crop system management practices in which the straw of the previous crop is left on the soil surface. Thus, N fertilization management using U in common bean is a challenge for the current cropping systems, and split U application is necessary aiming to supply N in early growth (MEIRA et al., 2005), before flowering (HENSON; BLISS, 1991), and during flowering and grain filling (ARAÚJO; TEIXEIRA, 2008).

Controlled-release U (CRU) provides an alternative to split U application in the common bean due to the gradual N release provided by the micropores of its polymers (SNYDER, 2017). CRU is more expensive than U, and although CRU provides a reduction in ammonia volatilization losses (TIAN et al., 2017), nitrate leaching (TIAN et al., 2018), and denitrification (HALVORSON; DEL GROSSO, 2012), it can result in low N availability after application (GRANT et al., 2012), compromising common bean growth (ARAÚJO; TEIXEIRA, 2008).

Blending CRU and U could be an alternative for supplying N during early growth and throughout the common bean growth cycle. U (the soluble source) would be readily available at the beginning of the common bean growth cycle, and if it were treated with NBPT (N-(n-butyl) thiophosphoric triamide), ammonia volatilization losses in the first days of U application on the soil surface would be reduced (RINALDI et al., 2019; GUELFI, 2017), thereby improving N recovery from U. CRU (the insoluble source) would supply N after initial growth at high-demand growth stages until the end of the common bean growth cycle, without compromising yield. Moreover, blending CRU and U can decrease the application cost compared with that of using only CRU.

A single application incorporated at sowing using blends of polymer-sulfur coated urea (PSCU), which is a type of CRU (SNYDER, 2017), and U have proven to be efficient for supplying N in other cropping systems in Brazil (GARCIA et al., 2020b; GARCIA et al., 2018; VILLALBA, 2018). There are no studies of a single broadcast application during common bean sowing using these blends. Broadcast application would reduce the costs associated with fertilizer incorporation. The most common proportion of CRU and U in annual crops in North China is 70% CRU + 30% U (GUO et al., 2017), and this proportion was also recommended for soils from Brazil (GARCIA et al., 2018; 2019a; VILLALBA, 2018). 30% CRU + 70% U was also recommended in Northeast China (LI et al., 2020a; LI et al., 2020b) and it represents a potential way to avoid low N levels due to N immobilization, especially at the beginning of the common bean growth cycle, in the current crop system in Brazil, in which the straw from the previous crop is left on the soil surface.

In this context, our hypothesis is that N broadcast application using 70% PSCU and 30% U will be a better choice to provide N during the common bean growth cycle and to improve grain yield than incorporated or split application using 70% PSCU and 30% U or 30% PSCU and 70% U. The N recovery from the U sources treated with NBPT would be higher than 50%

in both blends. This research evaluated the effect of N fertilization practices and blends of PSCU and U (treated with NBPT) on ammonia volatilization, soil mineral N content, macronutrient uptake, ¹⁵N-fertilizer recovery from U (NUR) in plants and common bean yield in irrigated field experiments with straw on the soil surface during the mild winters in Brazil.

3.2 Materials and Methods

3.2.1 Field Site Description

Two irrigated field experiments were performed at Compass Minerals Innovation Center in Iracemápolis, São Paulo state, Brazil (22°39′ S, 47°30′ W, 608 m elevation) during 2018 and 2019 mild winter growing seasons. The soil of the experimental area was classified as a Rhodic Eutrustox soil (SOIL SURVEY STAFF, 2014) with a clayey texture: 41.9% sand, 11.9% silt, and 46.2% clay (GEE; BAUDER, 1986). Millet was the previous crop in 2018, and 4.2 ± 0.3 Mg ha⁻¹ of straw with a 22:1 C/N ratio was left on the soil. Oat was the previous crop in 2019, and 3.5 ± 0.2 Mg ha⁻¹ of straw with a 31:1 C/N ratio was left on the soil. Three samples of one square meter of straw per block were weighed and subsamples were oven-dried (65 °C) and ground with a Wiley mill (0.5-mm sieve) to quantify the straw and analyze its N and C contents (BATAGLIA et al., 1983). Limestone application, plowing, and harrowing were performed in 2019 before oat sowing. The three-year average temperature and precipitation were 21.8 °C and 1200 mm, respectively.

Fifteen soil samples at intervals 20 cm in the soil (0- to 60 cm depth) were performed for chemical characterization in both years prior to the beginning of the experiment (Table 1).

Depth	pН	SOM ¹	TSN ²	$\mathbf{NH4}^{+}$	NO ₃ -	S	Р	K	Ca	Mg	Al	CEC ³	AlS ⁴	BS ⁵	
cm		g dm ⁻³	mg kg ⁻¹			mg dm ⁻³		mmol _c dm ⁻³					%		
	2018														
0-20	5.0	27	1130	1.1	2.4	30	64	2.4	29	7	2	58.4	5	66	
20-40	4.2	22	883	3.4	3.1	183	13	1.3	12	3	14	36.3	46	45	
40-60	4.2	20	820	1.3	2.9	207	13	1.3	12	3	12	38.3	42	43	
	2019														
0-20	5.1	27	1300	7	9	26	46	5.3	29	12	0	84	0	55	
20-40	4.5	16	1000	9.6	5.2	67	10	3.5	19	8	0	83	0	37	
40-60	4.4	14	800	3.2	4.7	135	5	2.7	12	6	2	79	9	26	

Table 1 - Soil chemical attributes on which common bean is grown in Brazil

¹SOM: soil organic matter; ²TSN: total soil nitrogen; ³CEC: cation exchange capacity at pH 7.0; ⁴AIS: aluminum saturation; ⁵BS: base saturation.

The samples from each soil layer were mixed for analysis. To determine the soil pH 0.01 mol L⁻¹ CaCl₂ (RAIJ et al., 2001) (1:2.5 soil/solution) was used. The Walkley–Black procedure (NELSON; SOMMERS, 1996) was used to determine the soil organic matter. Mass spectrometry (BARRIE; PROSSER, 1996) was used to determine the total N content. A solution of 2 mol L⁻¹ KCl (RAIJ et al., 2001) (1:5 soil/solution ratio) was used to determine the NH₄⁺-N and NO₃⁻-N contents. The available nutrients (phosphorus (P), potassium (K), calcium (Ca), and magnesium (Mg)) were extracted by ion exchange and quantified by colorimetric (P), flame photometric (K), and atomic absorption spectroscopy (Ca and Mg) analyses. Al and SO₄⁻²-S were determined by titration and turbidimetry, respectively (RAIJ et al., 2001). To determine the cation exchange capacity (CEC), the exchangeable cations (Ca, Mg and K) were summed by potential acidity (H + Al). The base saturation (%) was determine the Al saturation (%), the Al was multiplied by 100 and divided by (Ca + Mg + K + Al).

3.2.2 Experimental Setup and Treatment Description

A randomized block design were performed in the experiments in a factorial $(3 \times 2) + 1$ with four replications. The treatments were two blends (70% PSCU + 30% U and 30% PSCU + 70% U) and three N fertilization management practices (broadcast application: a single topdressing N application at sowing; split application: 1/3 of N incorporated at sowing and 2/3 as a side-dressing at V4 (third trifoliate leaf unfolded); incorporated application: a single N application incorporated at sowing). A control treatment (without U) was included. The N rate used was 90 kg ha⁻¹, which would be expected to produce common bean yields between 2.5 and 3.5 Mg ha⁻¹ in São Paulo, Brazil (RAIJ et al., 1997). The incorporation of N fertilizer was 15 cm depth and 10 cm to the side of seed row, and all N treatments were applied by hand. The U (45% N) fertilizer was treated with NBPT (530 mg NBPT kg⁻¹). The manufacturer of PSCU (patent n. EP 0574541), which is 39% N and 12% S, suggests that ~80% of the N is released within 60 days after application (MARIANO et al., 2019) considering that it contains 0.78% of polymers that are biodegradable and insoluble in water. The ¹⁵N-enriched U with 1.6 atom % ¹⁵N was manufactured at Stable Isotopes Laboratory at CENA/USP (FRITSCHE, 2019) and treated with NBPT as mentioned above to evaluate the ¹⁵N recovery of U by plants in 2019.

The experimental plot had 10 common bean rows of 10 m in length with 0.45-m spacing and a density of 244,000 plants ha⁻¹. One microplot (2 m²: 1.5 m long and 1.35 m wide) that included 3 sections of 1.5 m of common bean row was set up within the plots to apply the ¹⁵N-fertilizer (PSCU + ¹⁵N-U).

The common bean cultivar (Pérola: carioca-grain group) was sown on 7 June 2018. Pérola is the most common bean cultivated in the mild winter season in Brazil (CONAB, 2020), and it has a semi-upright architecture and indeterminate growth habit (between type II and III; LISBOA et al., 2018). BRS Estilo, which is also a cultivar of the carioca-grain group, was sown on 10 June 2019. BRS Estilo has an upright architecture and indeterminate growth habit (type II; MELLO et al., 2010), and it was chosen to facilitate plant sampling in the microplots in 2019. 80 kg P_2O_5 ha⁻¹ as triple superphosphate was applied under the seed row at sowing in 2018 and 2019. 40 kg K_2O ha⁻¹ as KCl was broadcast-applied at sowing. S⁰ was applied at sowing in the treatments of 30% PSCU + 70% U and in the control to equalize the S⁰ contained in the 70% PSCU + 30% U treatments. One-kilogram B ha⁻¹ and 1 kg Zn ha⁻¹ were applied at sowing with the N treatments and with S⁰ in the control in 2018 and 2019. Insects, diseases and weeds were controlled in both years when needed. Two common bean rows of 5 m long were harvested by hand on 2 October 2018 and on 3 October 2019 to measure yield (13% moisture content).

3.2.3 Quantification of Ammonia Volatilization

Ammonia (NH₃-N) volatilization was evaluated at 2, 3, 4, 5, 6, 7, 9, 12, 16, 21, 27, and 34 days after N broadcast application (90 kg N ha⁻¹) and split application (side dressing at V4: 60 kg N ha⁻¹) on the soil surface in 2018. To capture ammonia volatilization, open collectors (14 cm \times 14 cm \times 7 cm) with a foam disc (15 cm in diameter, 6 cm in height and density of 0.02 g cm⁻³) soaked in 25 mL of phosphoric acid (1.5 mol L⁻¹ and 5% glycerol) attached to the open side of the collectors were used. The collectors were placed 1 cm above the N-fertilizer application region on the soil surface. The NH₄⁺-N retained as ammonium phosphate in the sampled foam discs was extracted by squeezing each disc into 300 mL of deionized water in a beaker. After weighing the beaker, an aliquot of each sample solution was analyzed by flow injection analysis (FIA), to determine the NH₄⁺-N (REIS et al., 1997). The NH₄⁺-N (mg per collector) was divided by the N-fertilizer applied per collector (broadcast: 176 mg; split (side dressing at V4): 400 mg) to determine the NH₃-N volatilization.

The efficiency of the collector (26%) capturing ammonia was considered (GONZAGA; TRIVELIN, 2018). The daily losses of NH₃-N (kg ha⁻¹ day⁻¹) were determined by dividing the losses by the sample time (day), and the cumulative losses by summing the losses from each sample (kg ha⁻¹).

3.2.4 Analyses of Plant and Soil Samples

Plant and soil sampling were performed at the V4, R6 (flowering), R7 (pod formation), R8 (grain filling) and R9 (physiological maturity) stages (EMBRAPA, 2018). Three samples of soil per depth (0–20 cm, 20–40 cm, and 40–60 cm) in each plot, sampled in the N fertilized row, were mixed for analysis. The mineral N content (NO₃⁻-N and NH₄⁺-N (Annex B)) was extracted by 2 mol L⁻¹ KCl and determined by FIA (1:5 soil/solution ratio; RAIJ et al., 2001) and expressed in the results as NO₃⁻-N + NH₄⁺-N. The aerial-part samples of eight plants per plot were divided (leaf, stem, pod, and grain), oven-dried at 65 °C to a constant weight, weighed and ground in a Wiley mill using a 0.5-mm sieve. The micro-Kjeldahl method (BATAGLIA et al., 1983) was performed to determine the N concentration in each plant component; the atomic absorption spectroscopy to determine the K, Ca and Mg content; and the colorimetric method and turbidimetry to determine P and S content, respectively (BATAGLIA et al., 1983). The nutrient concentration was multiplied by the dry weight to determine the nutrient uptake (kg ha⁻¹). The maximum rates of dry matter and nutrient accumulation were determined according to Laviola et al. (2009) and Garcia et al. (2020).

3.2.5 ¹⁵N-Fertilizer Recovery Analyses

The PSCU + ¹⁵N-U was applied by hand (incorporated, broadcast and split application) in the microplots in 2019. The ¹⁵N-U recovery (NUR) in two plants (aerial parts) sampled in the internal adjacent rows of the microplots was added to the NUR of two plants (aerial parts) in the external adjacent rows to determine the NUR at V4, R6, R7 and R8 (TRIVELIN et al., 1994). At R9, two plants (aerial parts) were sampled at the center of the microplots to determine the NUR. The plant components (leaf + stem + pod, and grain) were oven-dried at 65 °C to a constant weight, weighed, and ground (0.5-mm sieve). The ¹⁵N abundance and total N concentration of the plant components were determined in a mass spectrometer

(PDZ Europa ANCA-GLS, 20-20, Sercon Ltd., Crewe, UK). The following equations were used to determine the ¹⁵N recovery (TRIVELIN et al., 1994):

Ndff (%) =
$$\left(\frac{a-c}{b-c}\right) \times 100$$
 (1)

$$Ndff(kg ha^{-1}) = \left[\frac{Ndff(\%)}{100}\right] \times Total N$$
(2)

¹⁵N recovery (%) =
$$\left[\frac{Ndff(kg ha^{-1})}{N rate}\right] \times 100$$
 (3)

where Ndff (% and kg ha⁻¹) is the N in the plant components derived from the fertilizer; a is the ¹⁵N abundance (atom % ¹⁵N excess) in the plant components; b is the ¹⁵N abundance in the fertilizer (atom % ¹⁵N excess); c is the natural ¹⁵N abundance in the control treatment (atom % ¹⁵N). Total N is the plant N content (kg N ha⁻¹). ¹⁵N recovery is the N of U treated with NBPT recovered by common bean plant (%). N rate (kg N ha⁻¹) is the N-fertilizer rate used in common bean (90 kg N ha⁻¹).

3.2.6 Statistical Analyses

The PROC MIXED procedure in SAS (version 9.0, SAS Institute Inc., Cary, NC, USA) was used to perform a combined analysis of variance (ANOVA) for the variables evaluated in 2018 and 2019. Fertilizer blend, N fertilization practice and year were considered fixed effects. The control treatment, included as a new factor, was also compared. The means were compared using Fisher's least-test difference (LSD) at the 0.05 significance level. Fertilizer blend and N fertilization management were considered fixed effects for the variable ¹⁵N-fertilizer recovery, the software R (R DEVELOPMENT CORE TEAM, 2015), and its ExpDes package were used, and the means were tested by the LSD test ($p \le 0.05$). The seasonal biomass (dry matter) and N partitioning (leaf, stem, pod and grain) during common bean growth were fitted to a Gaussian equation (GARCIA et al., 2020b). The nutrient remobilization was estimated by subtracting the total nutrient uptake and ¹⁵N recovery (leaf, stem and pod) at R7 from those at R9 based on the N accumulation models.

3.3 Results

3.3.1 Weather Conditions

The average daily air temperature was 20 °C during the mild winter common bean growing seasons (Figure 1A, B). The total irrigation was 171 mm, of which 130 mm occurred from sowing to R6, and 41 mm occurred from R6 to R9 in 2018 (Figure 1A). In addition, 3 mm of precipitation occurred after sowing, 109 mm occurred between R6 and R8, and 49 mm occurred between R8 and R9 (Figure 1A). The total irrigation was 194 mm of which 144 mm occurred from sowing to R6, and 50 mm occurred from R6 to R8 in 2019 (Figure 1B). In addition, 30 mm of precipitation occurred between R7 and R8 (Figure 1B).



Figure 1 - Daily minimum and maximum air temperature, daily irrigation and rainfall during mild winter growing seasons of common bean ((A): 2018; (B): 2019)

3.3.2 Ammonia Volatilization

The 30% PSCU+70% U broadcast application resulted in the maximum daily loss of NH₃-N (1.6% of the applied N) on the fourth day after N application and a cumulative loss of 12% of the applied N on the 34th day after N application in 2018. The 70% PSCU + 30% U broadcast application provided lower daily (0.9% of the applied N) and cumulative (9% of the applied N) losses than the 30% PSCU + 70% U treatment (Figure 2A, B). The maximum daily NH₃-N loss (2.5% of the applied N) occurred on the ninth day after split N application, and cumulative losses of 14% of the applied N were determined on the 34th day (at V4) for both blends (Figure 2 C, D).



Figure 2 - Daily and cumulative losses of NH₃-N in the N broadcast application at sowing (90 kg N ha⁻¹) on soil surface (A,B) and split N application side dressing at V4 (60 kg N ha⁻¹) common bean growth stage (C,D). Vertical bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$)

3.3.3 Mineral N Content in the Soil

The N fertilization management practice affected the mineral N content during the common bean growth cycle in 2018 and 2019 (Figure 3). At V4, the incorporated N application resulted in higher mineral N content than the other N fertilization management practices at depths of 0–20, 20–40, and 40–60 cm. At R6, incorporated and split N applications resulted in higher mineral N content than broadcast application at 0–20 and 40–60 cm depth, and incorporated application resulted in higher mineral N content than broadcast application resulted in a higher mineral N content than the other N fertilization management practices at 20–40 cm depth. At R7, incorporation resulted in a higher mineral N content than the other N management practices and broadcast application at 0–20 cm depth and 40–60 cm depth, respectively. Incorporated and split N applications resulted in higher mineral N content than broadcast application at 20–40 cm depth at R7, R8, and R9.



Figure 3 – Influence of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast and split on soil N mineral content during mild winter growing seasons of common bean (2018 and 2019). Horizontal bars indict the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) at each depth

3.3.4 Biomass (Dry Matter) Accumulation in Plants and Common Bean Yield

The N fertilization management practice influenced the dry matter accumulation (aerial part) in the vegetative and reproductive growth stages of common bean and its yield in 2018 and 2019 (Figure 4). At V4, broadcast application resulted in higher dry matter accumulation $(0.56 \text{ Mg ha}^{-1})$ than the other N treatments in 2018 (0.5 Mg ha^{-1}) and 2019 $(0.45 \text{ Mg ha}^{-1})$; that value in the broadcast application was similar to that in the control treatment in 2018. Incorporated application $(0.48 \text{ Mg ha}^{-1})$ resulted in higher dry matter accumulation than split N application $(0.43 \text{ Mg ha}^{-1})$ and the control $(0.37 \text{ Mg ha}^{-1})$ in 2019.



Figure 4 - Influence of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on common bean yield and dry matter accumulation (aerial part) during mild winter growing seasons of common bean (2018 and 2019). Vertical bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) in each common bean growth stage

At R6, broadcast application resulted in higher dry matter accumulation (2.53 Mg ha⁻¹) than the other N treatments (2.3 Mg ha⁻¹) and the control (2 Mg ha⁻¹) in 2018 and 2019, and the experiment in 2019 generated more dry matter (4.3 Mg ha⁻¹) than in 2018 (3.5 Mg ha⁻¹) at R7. At R8, the broadcast and incorporated application resulted in higher dry matter (6.5 Mg ha⁻¹) than split N application (5.9 Mg ha⁻¹) and the control (6 Mg ha⁻¹) in 2018 and 2019, and the experiment in 2019 generated more dry matter (7.2 Mg ha⁻¹) than in 2018 (5.3 Mg ha⁻¹). At R9, the experiment in 2019 generated more dry matter (8.1 Mg ha⁻¹) than in 2018 (5.6 Mg ha⁻¹). The yield under split N application (3.63 Mg ha⁻¹), which was similar to that in the control, was higher than that under broadcast application (3.34 Mg ha⁻¹) in 2018 and 2019, and the experiment in 2019 generated a higher yield (3.65 Mg ha⁻¹) than in 2018

(3.34 Mg ha⁻¹). The maximum rate of dry matter accumulation (110 kg ha⁻¹ day⁻¹) occurred on the 51st day after common bean emergence (V1) in 2018 (Figures 5C and 6A) and on the 60th day (178 kg ha⁻¹ day⁻¹) after V1 in 2019 (Figures 5D and 6A).



Figure 5 - Seasonal N uptake and dry matter accumulation and partitioning during mild winter growing seasons of the common bean (2018 (A,C) and 2019 (B,D)) using blends of polymer-sulfur coated urea (PSCU) and urea (U) treated with NBPT (broadcast application: (E); incorporated application: (F); split application: (G); average of two years). The arrow indicates the maximum daily rate of dry matter and N accumulation. DAE is day after common bean emergence. The dashed line is the N derived from the fertilizer (Ndff), U treated with NBPT in the blend, in plant components during the 2019 growing season of the common bean (E–G)



Figure 6 - Daily rate of dry matter accumulation (A) and N uptake (B) (kg ha⁻¹ day⁻¹) in common bean during mild winter growing season (2018 and 2019), average of all treatments. Daily rate of N uptake in broadcast, incorporated and split N application (D), average of two years. (C): Chlorosis in primary leaves of common bean (V2) after broadcast application and incorporated application with 30% PSCU + 70% U (PSCU: polymer-sulfur coated urea; U: conventional urea treated with NBPT)

3.3.5 Nitrogen Uptake in Common Bean Plants

The N fertilization management practices influenced the total N uptake in the vegetative and reproductive stages of common bean (Figure 7). At V4, the N application broadcast resulted in higher N uptake (25 kg ha⁻¹) than the other N management practices in 2018 (21 kg ha⁻¹) and 2019 (19 kg ha⁻¹), and the incorporated application resulted in higher N uptake (21 kg ha⁻¹) than split N application (18 kg ha⁻¹) and the control (14 kg ha⁻¹) in 2019. The Ndff was 8 kg ha⁻¹ on average in 2019 (V4). At R6, higher N uptake was observed in 2019 (82 kg ha⁻¹) than in 2018 (73 kg ha⁻¹). At R7, higher N uptake was observed in 2019 (122 kg ha⁻¹) than in 2018 (98 kg ha⁻¹). At R8, all treatments did not affect N uptake (126 kg ha⁻¹) in 2018, and the split N application resulted in lower N uptake (100 kg ha⁻¹) than the other treatments (147 kg ha⁻¹) in 2019. At R9, broadcast application resulted in higher total N uptake (163 kg ha⁻¹) and N uptake in grain (142 kg ha⁻¹) than split N application (total: 146 kg ha⁻¹; grain: 127 kg ha⁻¹). The Ndff was 30 kg ha⁻¹ on average from R6 to R9 in 2019 and was 23 kg ha⁻¹ in grain at R9 in 2019. Split N, incorporated and broadcast application resulted in Ndff values of 34 kg ha⁻¹ (28 kg ha⁻¹ in the grain), 30 kg ha⁻¹ (25 kg ha⁻¹ in grain), and 23 kg ha⁻¹ (18 kg ha⁻¹ in grain), respectively (Figures 5E–G and 7). The maximum rate of N uptake in 2018 (2.2 kg ha⁻¹ day⁻¹) occurred on the 38th day after V1, and in 2019 (2.4 kg ha⁻¹ day⁻¹) occurred on the 45th day after V1 (Figures 5A, B and 6B). The remobilization from R7 (leaf, stem and pod) to R9 (grain) was 82 kg N ha⁻¹ in 2018 and 97 kg N ha⁻¹ in 2019 (Figure 5A, B). The remobilization in terms of Ndff was, on average, 27 kg N ha⁻¹. It is possible for remobilization values to be overestimated once the plant leaves fall at R9.



Figure 7 - Influence of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on N uptake (aerial part) during the mild winter growing season of the common bean (2018 and 2019). The dashed line is the N derived from the fertilizer (Ndff), U treated with NBPT in the blend, in the plant in 2019 growing season of common bean. Vertical bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) in each common bean growth stage

3.3.6 ¹⁵N-Fertilizer Recovery in Common Bean Plants

The N fertilization management practice and fertilizer blend affected the ¹⁵N recovery from U treated with NBPT (NUR) by plants during the common bean growth cycle in 2019 (Figure 8). At V4, broadcast application provided a higher NUR (12%) than the other N

management practices (7%), and 30% PSCU + 70% U provided a higher NUR (11%) than 70% PSCU + 30% U (6%). From R6 to R8, 30% PSCU + 70% U provided a higher NUR (43% on average) than 70% PSCU + 30% U (18% on average). At R9, 30% PSCU + 70% U provided a higher NUR (48%) and NUR in grain (39%) than 70% PSCU + 30% U (NUR: 18%; NUR in grain: 14%), and split N application and incorporation provided higher NUR in grain (30%) than broadcast application (21%). The NUR in the grain was positively correlated with common bean yield (Figure 9).



Figure 8 - Influence of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast and split on ¹⁵N-fertilizer recovery (aerial part) of U treated with NBPT during mild winter growing season of common bean (2018 and 2019). Vertical bars indicate the standard error of the mean. Means followed by different letters indicate difference ($p \le 0.05$) among treatments. The rate of N was 90 kg ha⁻¹ and the results are in % of total N applied



Figure 9 - Correlation between common bean yield and ¹⁵N-fertilizer recovery in the grain (%) of conventional urea (U) treated with NBPT, in application broadcast, incorporated and split using blends of polymer-sulfur coated urea (PSCU) and U. (A): average two blends (70% PSCU + 30% U and 30% PSCU + 70% U). (B): application of 30% PSCU + 70% U

3.3.7 Phosphorus, Potassium, Sulfur, Calcium and Magnesium Uptake in Common Bean Plants

The N fertilization management practices affected the P, K and S at earlier common bean growth stage (Figure 10, 11).



Figure 10 - Influence of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on P, K and S uptake (aerial part) during mild winter growing season of common bean (2018 and 2019). Vertical bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) in each common bean growth stage



Figure 11 - Influence of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on Ca and S uptake (aerial part) during mild winter growing season of common bean (2018 and 2019). Vertical bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) in each common bean growth stage

At V4, the broadcast N application resulted in higher P (2 kg ha⁻¹), K (16 kg ha⁻¹), S (2 kg ha⁻¹) Ca (10 kg ha⁻¹) and Mg (2.2 kg ha⁻¹) uptake compared to the other treatments (average: 1.4 kg P ha⁻¹; 12 kg K ha⁻¹; 1.4 kg S ha⁻¹; 8 kg Ca ha⁻¹; and 1.7 kg Mg ha⁻¹) in 2019. In 2018, At V4, the broadcast N application resulted in higher P (1.4 kg ha⁻¹) and Mg (1.8 kg ha⁻¹) uptake compared to the incorporated N application (1.2 kg P ha⁻¹; 1.6 kg Mg ha⁻¹). In addition, the N treatments resulted in higher K uptake (15 kg ha⁻¹) than control (12 kg ha⁻¹); and the treatments did not affect S (1.4 kg ha⁻¹) and Ca (6 kg ha⁻¹) uptake. From R8 to R9 the experiment in season 2 resulted in higher P, K, S, Ca and Mg uptake than that in season 1. Reaching at R9, in season 2, a total of 16 kg P ha⁻¹ (14 kg P ha⁻¹ in the grain), 124 kg K ha⁻¹ (63 kg K ha⁻¹ in the grain), and 14 kg S ha⁻¹ (10 kg S ha⁻¹ in the grain). In addition, a total of 66 kg Ca ha⁻¹ in the grain), and 23 kg Mg ha⁻¹ (9 kg Mg ha⁻¹ in the grain). And in season 1, a total of 14 kg P ha⁻¹ (12 kg P ha⁻¹ in the grain), 76 kg K ha⁻¹

(40 kg K ha⁻¹ in the grain), and 10 kg S ha⁻¹ (6 kg S ha⁻¹ in the grain). Moreover, a total of 20 kg Ca ha⁻¹ (12 kg Ca ha⁻¹ in the grain), and 15 kg Mg ha⁻¹ (7 kg Mg ha⁻¹ in the grain). The maximum rate of nutrient uptake occurred between V4 and R7 (Figure 12).



Figure 12 - Seasonal P, K, S, Ca, and Mg accumulation and partitioning in the treatments during the mild winter growing season of common bean in 2018 (A, B, C, D, and E) and in 2019 (F, G, H, I, and J). The arrow indicates the maximum daily rate of P, K, S, Ca, and Mg accumulation. DAE is the day after common bean emergence. The daily rate of P (L), K (M), S (O), Ca (Q), and Mg (R) accumulation (kg ha⁻¹ day⁻¹) in common bean during mild winter growing seasons (2018 and 2019). Average of treatments (two fertilizer blends, three N fertilization management practices, and a control treatment) in each growing season

The maximum rate of P, S and Mg uptake in 2019 (Figure 12 F, H, J) occurred later than that in 2018 (Figure 12 A, C, E, L, O, R). In addition, the maximum rate of K uptake in 2019 (Figure 12 B) occurred earlier than that in 2018 (Figure 12 G, M), and the maximum rate of Ca in 2019 (Figure 12 I) occurred at the same time than that in 2018 (Figure 12 D, Q). The maximum rate of P, Ca and Mg uptake in 2019 was higher than that in 2019. Moreover, the maximum rate of K uptake in 2019 was lower than that in 2018, and the maximum rate of S in 2019 was similar than that in 2018. The remobilization from R7 (leaf, stem and pod) to R9 (grain) was 18 kg P ha⁻¹, 166 kg K ha⁻¹, 16 kg S ha⁻¹, and 11 kg Mg ha⁻¹ in the treatments in 2019 (Figure 12 F,G,H,J). In 2018, the remobilization was 23 kg P ha⁻¹, 174 kg K ha⁻¹, 17 kg S ha⁻¹, and 18 kg Mg ha⁻¹ in the treatments (Figure 12 A,B,C,E). The nutrient uptake in the models (Figure 12) slightly decreased after R8 because of the falling of senescent leaves, which can overestimate the values of nutrient remobilization, and the maximum rate of nutrient uptake as can be observed in the Figure 12 (M).

3.4 Discussion

The common bean growth and yield were affected by precipitation in the different years of the experiments in another study (CHEKANAI et al., 2018). In our irrigated experiments, the daily rate of dry matter accumulation and nutrient uptake during the common bean growth cycle were probably affected by the agricultural history in 2018 and 2019, which would explain the higher dry matter accumulation, nutrient uptake and yield in 2019 than in 2018. The common bean has a low tolerance to low soil fertility (CHEKANAI et al., 2018; SINGH et al., 2003). The better conditions for common bean growth in 2019 than in 2018 are partially explained by the mechanized operations (plowing and harrowing) and limestone application performed before the sowing of the previous crop in 2019. These factors probably contributed to accelerating the decomposition and mineralization of soil organic matter (LOSS et al., 2014) and increasing the plant nutrient uptake, dry matter accumulation and yield in 2019. Limestone also improved common bean yield during three growing seasons in the Cerrado region in Brazil (CARVALHO; NASCENTE, 2018). Ammonia volatilization reached 11 kg ha⁻¹ under the broadcast-applied 30% PSCU + 70% U treatment, which was 3 kg ha⁻¹ more than that under 70% PSCU + 30% U, and ammonia volatilization increased after the first precipitation event in the growing season (4 mm, in 2018). After the fourth irrigation event, ammonia volatilization decreased, probably due to the incorporation of U. Ammonia volatilization also occurred in the
split N application at V4 followed by irrigation and precipitation. The blend with more PSCU tended to result in lower ammonia volatilization because of the controlled N release provided by the insoluble polymers. The intervals of irrigation also contributed to minimizing the daily ammonia volatilization in the PSCU. In situations with more daily precipitation, the daily volatilization of CRU tends to be higher than that observed in our irrigated experiment (KE et al., 2017). The ammonia volatilization probably had the same pattern in 2019, based on the similar irrigation and temperature in the first days after N-fertilizer application compared to those in 2018. The poorer growth performance of common bean in 2018 can also be attributed to the aluminum saturation of the soil, which was higher than 20% below 20 cm; common bean roots in favorable conditions can reach 30 cm depth depending on the soil texture (PIRES et al., 1991). The aluminum saturation probably restricted common bean root growth at 0-20 cm depth in 2018, limiting water and nutrient absorption and favoring nutrient percolation. Roots were likely more developed at deeper depths in 2019 than in 2018; this would improve plant water and nutrient absorption, especially during periods without rain or irrigation such as during R8 in 2019. Conditions with high A1 saturation, as observed in 2018, are normally found in no-tillage systems where limestone is applied to the soil surface without incorporation and reacts with the soil in the application region (SORATTO; CRUSCIOL, 2008). To mitigate toxic levels of aluminum in deeper soils, gypsum (VICENSI et al., 2020) can be applied in no-tillage systems.

The N fertilization management treatments also affected the daily rate of N uptake in 2018 and 2019 and influenced the differences in N uptake during the common bean growth cycle. Moreover, chlorosis (Figure 6 C) in the primary leaves (V2) occurred in 2018 and 2019 after broadcast and incorporated application using 30% PSCU + 70% U. This can be attributed to the salt toxicity (ZHU, 2007) caused by the excess N, especially that from U (soluble source), which was mostly concentrated in the incorporated fertilizer application treatments. This result can be explained by the higher soil N mineral levels under incorporated application than under the other N management practices in the N application region, especially at the V4 growth stage. A similar level of soil N mineral was also observed at the V4 maize growth stage at 0–20 cm depth in the fertilizer application region, in studies incorporating 70% PSCU + 30% U and 100% PSCU at sowing (VILLALBA, 2014). Other studies associated the soil salinity, provided by greater amounts of fertilizers in the seed row, with damage in common bean roots (LACERDA et al., 2015). Chlorosis in primary leaves of the common bean was also observed when applying greater amounts of potassium fertilizer close

to the seeds (LACERDA et al., 2015; KLUTHSCOUSKI; STONE, 2003). K₂O (as KCl) was broadcast applied at sowing in our experiments, and it probably enhanced the effects of salt on plants that underwent broadcast application with 30% PSCU + 70% U. To provide better conditions for N broadcast application using these blends, it is necessary to determine a better rate of N and K₂O for broadcast application at sowing in Rhodic Eutrustox soil. Lacerda et al. (2015) observed that 22.5 kg N ha⁻¹ and 67.5 kg K₂O ha⁻¹ applied broadcast at common bean sowing is an alternative to the application incorporated at sowing. The authors also applied 60 kg N ha⁻¹ (as urea) topdressing at V4 common bean growth stage in their treatments. Other studies evaluated the salt effect in maize plants using 70% PSCU + 30% U (GARCIA et al., 2019a; 2019b), and a deleterious effect, caused by the excess N, was observed in early growth. Based on that result the optimal N rate and application mode with 70% PSCU + 30% U in maize were recommended. Similar studies would be helpful in determining the ideal application mode and N rate using the blends in our study for common bean. The optimal method would avoid the possible effects of salt on the plants. Moreover, excess N can accelerate the vegetative growth of common bean, which negatively affects the reproductive stages by retarding flowering and disrupting the nutrient balance (ZHU, 2017; RABELO et al., 2017; ALMEIDA et al., 2016). The split N application in our research provided better conditions for the early growth of common bean than other N management practices, based on the slightly higher daily rate of N uptake observed from V2 to V4 (Figure 6 D). In this period, plant stress can indirectly affect common bean yield (EMBRAPA, 2018). From V4 until R8, broadcast application resulted in a higher daily rate of N uptake than the other N fertilization management practices. Broadcast application resulted in higher N uptake and NUR at V4 than the other N management practices, and higher total N uptake at R9 than split N application, especially in the grain. These results suggest that broadcast application probably provided a greater priming effect (QIAO et al., 2016) than the other N management practices between V4 and R8, as the plants had lower NUR in the grain at R9 in the broadcast application than in the split N application. In addition, the plants tended to reach their maximum NUR at the R6 growth stage with both blends. This tendency probably occurred because U is a soluble N source; after R6, the N from this source was probably located below the roots, and the plants probably absorbed more N from the soil and from PSCU which provided N higher on the soil surface. It would be of interest to confirm this hypothesis in another study with ¹⁵N in the PSCU source.

We also found an imbalance between the N uptake and the NUR in the grain in the broadcast application but no in the other management treatments, and the NUR in the grain was positively correlated with common bean yield (Figure 9). Our hypothesis explaining that correlation is that the salt effect observed in common bean plants, especially under the broadcast application of 30% PSCU + 70% U, probably affected the NUR, and plants in that situation tended to absorb less N from the fertilizer than those in split N application treatments. The stress probably disrupted the balance between the N uptake from the U source and the total N uptake, and indirectly interfered with the common bean yield; the yield was lower in the broadcast application treatment than in split N application treatment and the control treatment (without N-fertilizer application) in 2018 and 2019. Similar results have not been reported in other studies in common bean using these blends, and studies evaluating the influence of salt effects on common bean plants and their association with common bean yield would clarify these results. Other studies observed that the split N application in the proportion 1:1 (basal N application:topdressed N application) resulted in a higher N uptake, NUR and yield than the proportion 2:1 (WANG et al., 2016; YI et al., 2008). Contrary to our results, Oliveira et al. (2019) did not observe a difference in yield for N fertilization management practices with a controlled-release fertilizer in common bean. Moreover, in our study, the control treatment provided similar conditions for better common bean growth and yield. Nonresponse to N-fertilizer application were also observed in maize production at Rhodic Haplustox and Rhodic Eutrustox sites using blends of PSCU + U and only U (GARCIA et al., 2018; 2020b; SCHONINGER et al., 2018) and in sugarcane in a Typic Hapludox soil (BOSCHIERO et al., 2020).

The challenges of U application that have been observed in other studies (ammonia volatilization (RINALDI et al., 2019), salt effect (GARCIA et al., 2019b), and N percolation (HONG et al., 2007)) were observed in our research despite the N-fertilizer recovery of U treated with NBPT being higher than 50% for both blends. Neptune and Muraoka (1978) observed that NUR in common bean varied from 11% to 35% in N fertilization management practices with U, and the N application at sowing resulted in lower N uptake than the N application before or at flowering stage. Based on our results, split N application using the less expensive blend can be recommended. Fertilizer incorporation can also be recommended with adjustments associated with the distance from the N-fertilizer application region to the seed row to avoid salt toxicity. Similar recommendations were also made in a study evaluating N fertilization management using only U and based only on common bean

yield (KIEHL et al., 1993). At nonresponsive sites, it is advantageous to apply N to replace the N extracted by plants, thereby avoiding economic losses and pollution. This suggests the benefits of a reduction in the N application rate, which would reduce costs and, in this study, minimize the salt toxicity observed in the broadcast and incorporated applications for common bean in Rhodic Eutrustox soil.

3.5 Conclusions

Broadcast application using blends of PSCU and U (70% PSCU + 30% U and 30% PSCU + 70% U) did not improve grain yield compared to N incorporated application at sowing and split N application in irrigated experiments in Rhodic Eutrustox soil during the mild winter in Brazil. Broadcast application also resulted in lower grain yield than the control treatment (without N-fertilizer application) and the split N application treatment. Broadcast application resulted in lower NUR in the grain at harvest than split N application, which indicates that broadcast application was less efficient at supplying N from the U source in the blends. The U source in the blend provided 18% of the NUR in common bean plants with the 70% PSCU + 30% U blend and 48% with the 30% PSCU + 70% U blend. These NURs are higher than 50% considering the proportion of N in the U in each blend. The nonrecovery of N from U by plants can be attributed to ammonia volatilization, which reached 12% of the total N applied, on average, under split N application and broadcast application, and the likely percolation of N below the common bean root zone.

4 FINAL REMARKS

Despite this unprecedented investigation provided only one-season information about the ¹⁵N-fertilizer recovery in maize using blends of polymer-sulfur coated urea and NBPTtreated urea, it helped to understand the behavior of ¹⁵N-fertilizer recovery during the maize growth cycle in a better soil fertility situation compared to the previous season. In addition, it helped to provide a recommendation to use the most cost-effective option in this situation, i.e. the N broadcast application using 30% PSCU + 70% U, once the ¹⁵N-fertilizer recovery at physiological maturity (73.8%) was similar among N treatments with a good rainfall distribution during maize cycle. It would not be possible to make a precise recommendation evaluating only the N uptake, which does not differentiate the N from the soil and the fertilizer, as observed in other studies in non-responsive sites (MOSCHINI, 2019; GARCIA et al., 2018; VILLALBA, 2014). The soil was the main N supplier providing 191 kg ha⁻¹ of the total N uptake in maize at R6 (324 kg N ha⁻¹) and the N fertilizer contributed with 133 kg N ha⁻¹.

It is important to note that this study only evaluates one N rate (180 kg N ha⁻¹) that is recommended to high maize yield (RAIJ et al., 1997). To minimize the N losses observed in the N broadcast application (12% of the applied N) and to reduce costs it would be interesting to reduce the N rate with 30% PSCU + 70% U in the soil of the experiment aiming to replace the N extracted by grain harvest once the N treatments did not affect maize yield. Villalba (2018) observed that can be possible to reduce N rate using 70% PSCU + 30% U incorporated at maize sowing in the soil of our experiment and more studies for N broadcast application with 30% PSCU + 70% U would be necessary. It can help to attend the 4 R's concept (SNYDER, 2017) once the right place, right source and right time were possible to recommend in this study.

In irrigated common bean, the effect of N fertilization management practices with blends of controlled-release urea and NBPT-treated urea drawn attention to the N broadcast application in Brazilian clayey soil. If on one hand, it resulted in the lowest common bean yield, on the other it resulted in the lowest ¹⁵N recovery of NBPT-treated urea in the common bean grain. Despite this research did not provide information about the ¹⁵N recovery of PSCU in common bean, it is evident that the N broadcast application was less efficient to provide N during the common bean growth cycle compared to the other N management practices. As we mentioned above to maize about N rate is valid to the common bean, and in this case a rate study would be of interest in incorporated and split N application using the most cost-effective blend aiming to attend the 4 R's concept (SNYDER, 2017) in the experiment soil.

To finalize, this study focused on evaluating the most used ratios of blends of controlledrelease and conventional urea in maize and common bean using isotopic technic (¹⁵N) to improve the recommendation of N fertilization management practices. This technic, which helped us in clayey soil, can be used to clarify the recommendation of these blends in sandy loam soils, and with other types of enhanced efficiency fertilizers, and compare it with conventional urea that normally has low N recovery by plants (GAVA et al., 2006).

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APPENDIX

Appendix A: Experiment with two microplots at maize sowing (2019-2020 growing season)
(a); incorporated N application (b), broadcast N application in the microplots at maize sowing (c); split N application in the microplots at V4 maize growth stage; open collector used to capture the ammonia volatilization (e); common bean experiment in 2018 (f); common bean microplot in 2019 (g)



ANNEXES

Annex A: Effect of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast, and split on soil N mineral content (nitrate and ammonium) during the maize growing seasons (S1: season 1 (2017–2018); S2: season 2 (2019–2020)). Horizontal bars indicate the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) at each depth.



Annex B: Influence of blends of polymer-sulfur coated urea (PSCU) and conventional urea (U) (70% PSCU + 30% U and 30% PSCU + 70% U) applied incorporated, broadcast and split on soil N mineral content (nitrate and ammonium) during mild winter growing seasons of common bean (2018 and 2019). Horizontal bars indict the standard error of the mean. Means followed by different letters are significantly different ($p \le 0.05$) at each depth



treatment	Ν	Р	K	Ca	Mg	S	Yield					
season 1: total nutrient uptake (kg ha ⁻¹)												
control	178	20	156	52	23	17	6744					
70PSCU + 30U	237	30	216	66	32	26	8298					
30PSCU + 70U	233	27	203	66	30	23	7592					
broadcast	260	31	235	67	33	26	7690					
split	215	26	181	64	29	23	7980					
incorporated	230	28	213	66	30	25	8165					
average	227	27	202	64	29	24	7774					
season 1: grain nutrient uptake												
control	118	15	50	18	10	9						
70PSCU+30U	155	24	65	24	15	14						
30PSCU + 70U	155	22	61	24	14	13						
broadcast	178	26	72	25	16	14						
split	144	21	56	24	13	13						
incorporated	142	22	62	22	13	13						
average	150	22	61	23	14	13						
season 2: total nutrient uptake												
control	307	51	203	39	40	25	12588					
70PSCU+30U	319	56	212	41	44	23	11621					
30PSCU + 70U	329	55	212	44	42	31	12203					
broadcast	302	52	194	42	42	31	12147					
split	344	60	222	45	47	26	11438					
incorporated	327	54	221	41	40	24	12151					
average	322	55	211	42	43	27	12009					
season 2: grain nutrient uptake												
control	215	43	65	4	20	13						
70PSCU + 30U	213	48	68	4	21	13						
30PSCU + 70U	218	47	67	7	21	17						
broadcast	204	44	65	7	20	19						
split	226	52	73	5	23	14						
incorporated	216	46	65	4	21	13						
average	216	47	67	5	21	15						

Annex C: Average value (kg ha⁻¹) of N, P, K, Ca, Mg and S uptake at R6 (total and in the grain) and grain yield (kg ha⁻¹) in the maize growing seasons (season 1 (2017–2018); season 2 (2019–2020)). PSCU: polymer-sulfur coated urea; U: conventional urea

Annex D: Average value (kg ha⁻¹) of N, P, K, Ca, Mg and S uptake at R9 (total and in the grain) and grain yield (kg ha⁻¹) in the mild winter growing seasons of common bean (2018 and 2019). PSCU: polymer-sulfur coated urea; U: conventional urea

treatment	N	Р	K	Ca	Mg	S	Yield				
2018: total nutrient uptake (kg ha ⁻¹)											
control	127	14	78	28	13	10	3581				
70PSCU+30U	142	15	80	32	16	10	3357				
30PSCU + 70U	123	13	73	28	14	9	3249				
broadcast	147	15	81	32	17	10	3173				
split	120	13	77	28	13	10	3485				
incorporated	130	13	72	29	15	10	3250				
average	132	14	77	30	15	10	3342				
-	20)18: grain	nutrient	uptake							
control	111	12	44	11	6	6					
70PSCU+30U	125	13	42	13	7	7					
30PSCU + 70U	108	12	38	12	7	6					
broadcast	131	13	41	14	8	6					
split	104	12	40	11	6	6					
incorporated	114	12	38	12	7	7					
average	116	12	40	12	7	6					
2019: total nutrient uptake											
control	179	16	129	64	22	15	3618				
70PSCU+30U	178	16	125	67	23	14	3664				
30PSCU + 70U	177	17	121	66	22	14	3650				
broadcast	178	15	118	64	21	15	3526				
split	172	16	122	65	23	13	3771				
incorporated	182	17	129	71	24	14	3674				
average	178	16	124	66	23	14	3657				
2019: grain nutrient uptake											
control	155	14	69	17	10	11					
70PSCU+30U	153	14	64	15	9	9					
30PSCU + 70U	152	15	61	14	9	10					
broadcast	153	13	57	13	8	10					
split	149	14	65	16	10	9					
incorporated	156	15	66	15	10	9					
average	153	14	64	15	9	10					